QUALITY ASSURANCE PROJECT PLAN
Riverside Avenue Asbestos
Newark, New Jersey

Prepared for:
United States Environmental Protection Agency/Environmental Response Team
Edison, New Jersey

By:
Lockheed Martin/Scientific, Engineering, Response and Analytical Service (SERAS)
Work Assignment Number: SERAS-001

Based on the Intergovernmental Data Quality Task Force Uniform
Federal Policy for Quality Assurance Project Plans
(Final Version 1.1, June 2006)

June 30, 2017

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QAPP Worksheet #1
Title and Approval Page

Site Name/Project Name: Riverside Avenue Asbestos
Site Location: Newark, New Jersey (NJ)

Document Title: Quality Assurance Project Plan (QAPP) for Riverside Avenue Asbestos Emergency Response

Lead Organization: Environmental Protection Agency/Environmental Response Team (EPA/ERT)

Preparer's Name and Organizational Affiliation: Amy DuBois, Lockheed Martin/Scientific, Engineering, Response and Analytical Services (SERAS)

Preparer's Address, Telephone Number, and E-mail Address: 2890 Woodbridge Ave, Edison, New Jersey 08226 (732) 494-4007, amy.e.dubois@leidos.com

Preparation Date (Month/Day/Year): June 30, 2017

Investigative Organization's Project Manager/ Date: _____
Signature
Printed Name/Organization: Sella Burchette/ERT Work Assignment Manager

Investigative Organization's Response Project Manager/Date: _____
Signature
Printed Name/Organization: Michael Hoppe/ERT Response Work Assignment Manager

Investigative Organization's Project QA Officer/Date: _____
Signature
Printed Name/Organization: Stephen Blaze/ERT Quality Coordinator

Lead Organization's Project Manager/Date: _____
Signature
Printed Name/Organization: Philip Solinski/SERAS Task Leader

Approval Signatures/Date: _____
Signature
Printed Name/Title: Deborah Killeen/SERAS QA/QC Officer

Approval Authority: SERAS

Other Approval Signatures/Date: _____
Signature
Printed Name/Title: Kevin Taylor/SERAS Program Manager

Document Numbering System: SERAS-001-DQAPP-063017_89001

QAPP Worksheet #2
QAPP Identifying Information

Site Name/Project Name: Riverside Avenue Asbestos
Site Location: Newark, NJ
Site Number/Code:
Operable Unit:
Contractor Name: Lockheed Martin
Contractor Number: EP-W-09-031

Contract Title: SERAS
Work Assignment Number: SERAS-001

1. Identify regulatory program: Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA)
2. Identify approval entity: EPA/ERT
3. The QAPP is (select one): Generic X Project Specific
4. List dates of scoping sessions that were held: NA
5. List dates and titles of QAPP documents written for previous site work, if applicable:

Title	Approval Date

6. List organizational partners (stakeholders) and connection with lead organization:
   EPA Region 2
7. List data users:
   EPA Region 2
8. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusions below:

Worksheet 13 – No existing data are available for this project
Worksheet 22 – No field equipment requiring calibration will be used on site.
Worksheet 37 – EPA Region 2 will be responsible for assessing the usability of the data.

QAPP Worksheet #2
QAPP Identifying Information
(Continued)

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QAPP Worksheet #2
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☐ Worksheet Not Applicable (State Reason)

QAPP Worksheet #3
Distribution List

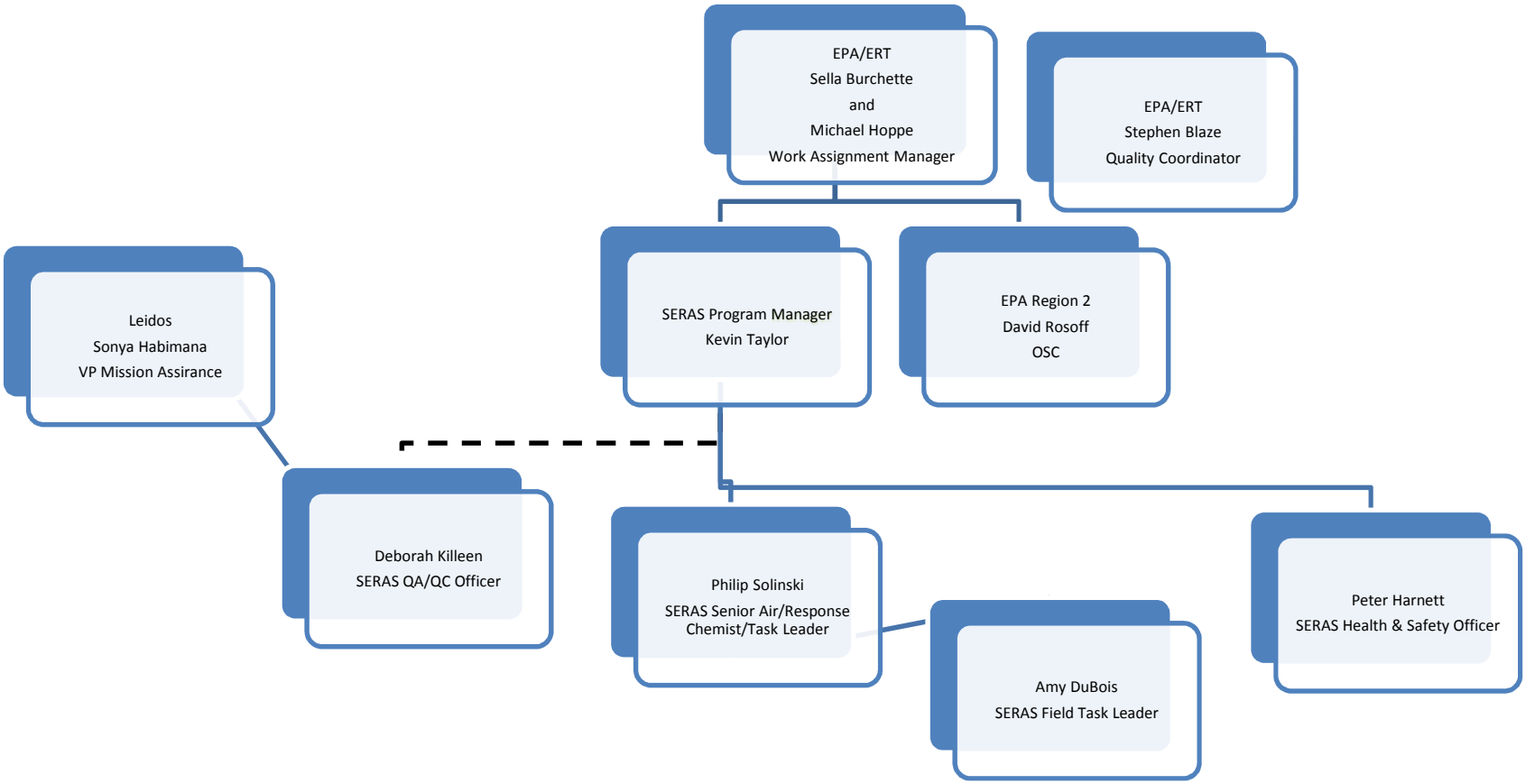
QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Number
Sella Burchette	Work Assignment Manager (WAM)	ERT	(732) 321-6726	(732) 321-6724	burchette.sella@epamail.epa.gov	SERAS-001-DQAPP-063017_89001
Michael Hoppe	On-Site Response WAM	ERT	(732) 906-6908	(732) 321-6724	Hoppe.michael@epa.gov	SERAS-001-DQAPP-063017_89001
Stephen Blaze	Quality Coordinator	ERT	(732) 906-6921	(732) 321-6724	blaze.stephen@epamail.epa.gov	SERAS-001-DQAPP-063017_89001
David Rosoff	On-Scene Coordinator (OSC)	EPA Region 2	(732) 906-6879	NA	rosoff.david@Epa.gov	SERAS-001-DQAPP-063017_89001
Amy DuBois	Sr. Environmental Scientist/Field Task Leader (TL)	SERAS	(732) 494-4007	(732) 494-4021	amy.e.dubois@leidos.com	SERAS-001-DQAPP-063017_89001
Philip Solinski	Air Response Chemist/Emergency Response (ER) TL	SERAS	(732) 321-4283	(732) 494-4021	Philip.j.solinski@leidos.com	SERAS-001-DQAPP-063017_89001
Deborah Killeen	Quality Assurance/Quality Control (QA/QC) Officer	SERAS	(732) 321-4245	(732) 494-4021	deborah.a.killeen@leidos.com	SERAS-001-DQAPP-063017_89001
Kevin Taylor	Program Manager	SERAS	(732) 321-4202	(732) 494-4021	kevin.c.taylor@leidos.com	SERAS-001-DQAPP-063017_89001

QAPP Worksheet #4
Project Personnel Sign-Off Sheet

Organization: SERAS/ERT/EPA

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Amy DuBois	SERAS Environmental Scientist/Response TL	(732) 494-4007		
Michael Hoppe	On-site Response ERT WAM	(732) 906-6908		
David Rosoff	EPA Region 2 OSC	(732) 906-6879		

QAPP Worksheet #5
Project Organizational Chart



QAPP Worksheet #6
Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, etc.)
Approval of initial QAPP and any amendments	ERT WAM ERT Quality Coordinator SERAS Program Manager SERAS QA/QC Officer SERAS TL	Michael Hoppe Stephen Blaze Kevin Taylor Deborah Killeen Philip Solinski	(732) 906-6908 (732) 906-6921 (732) 321-4202 (732) 321-4245 (732) 321-4283	SERAS internal peer review, followed by ERT approval, implementation of changes effective only with approved QAPP or QAPP Change Form.
Nonconformance and Corrective Action	SERAS Response TL ERT WAM SERAS QA/QC Officer	Amy DuBois Michael Hoppe Deborah Killeen	(732) 494-4007 (732) 906-6908 (732) 321-4245	Use of the Work Assignment Field Change Form for field issues.
Posting of Deliverables to the ERT-Information Management System (IMS) website	SERAS Response TL SERAS QA/QC Officer SERAS Administrative Support SERAS Air Response Chemist/TL	Amy DuBois Deborah Killeen Eileen Ciambotti Philip Solinski	(732) 494-4007 (732) 321-4245 (732) 321-4255 (732) 321-4283	As per work assignment, posting of deliverables to ERT-IMS website constitutes delivery to the WAM.
Work Assignment	SERAS Program Manager	Kevin Taylor	(732) 321-4202	Describes scope of work to SERAS personnel from the ERT WAM.
Projected Work Assignment/Analytical Services Resource Requirements (PWA/ASRR)	SERAS Response TL	Amy DuBois	(732) 494-4007	Filled out by the TL upon receipt of the work assignment and following the project scoping meeting, and distributed to field, analytical, and support personnel.
Health and Safety On-Site Meeting	SERAS Response TL and/or Site Health and Safety Officer	Amy DuBois	(732) 494-4007	Describe potential site hazards, required personal protective equipment, and access to local emergency services.

QAPP Worksheet #7
Personnel Responsibilities and Qualification Table

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Amy DuBois	Response TL/Environmental Scientist	SERAS	Project Supervision/Site Health and Safety Officer/Field Operations	Minimum B.S. degree plus 8 years of related experience/LM Employee Files
Philip Solinski	Air Response Chemist/ER TL	SERAS	Oversight	Minimum BS degree plus 14 years related experience/LM Employee Files
Deborah Killeen	QA/QC Officer	SERAS	QA Oversight/Deliverable Review	Minimum B.S. degree plus 14 years of related experience/LM Employee Files
David Rosoff	OSC	EPA Region 2	Project Oversight	EPA job-specific qualifications/EPA Files
Michael Hoppe	WAM	EPA/ERT	Technical Direction-Project	EPA job-related qualifications/EPA Files
Stephen Blaze	Quality Coordinator	EPA/ERT	QA Oversight	EPA job-related qualifications/EPA Files

☐ Worksheet Not Applicable (State Reason)

QAPP Worksheet #8
Special Personnel Training Requirements Table

Project Function	Specialized Training – Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates
Project Oversight	Task Leader	REAC	2002	Amy DuBois	Response TL/SERAS	Quality Files
Project Oversight/Field Operations	Occupational Safety and Health Administration (OSHA) 8-hour Refresher	SERAS	August 2016	Amy DuBois	Response TL/SERAS	Health & Safety Files
QA Oversight	Uniform Federal Policy for Quality Assurance Project Plans	Advanced Systems	January 2006	Deborah Killeen	QA/QC Officer/SERAS	Quality Files
QA Oversight	Lead Auditor Training	IT Corp	Sept 1991	Deborah Killeen	QA/QC Officer/SERAS	Quality Files

☐ Worksheet Not Applicable (State Reason)

QAPP Worksheet #9
Project Scoping Session Participants Sheet

Project Name: Riverside Avenue Asbestos ER **Projected Date(s) of Sampling:** 7/5/17 **Project Manager:** Amy DuBois	**Site Name:** Riverside Avenue **Site Location:** Newark, NJ

Date of Session: 6/27/17
Scoping Session Purpose: Discussion of laboratory subcontracting requirements

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Deborah Killeen	QA/QC Officer	SERAS	(732) 321-4245	deborah.a.killeen@leidos.com	Quality Assurance
Amy DuBois	Environmental Scientist	SERAS	732-494-4007	amy.e.dubois@leidos.com	Response Task Lead (TL)
Philip Solinski	Air/Response Chemist	SERAS	732-321-4283	Philip.j.solinski@leidos.com	TL/Project Oversight
Stephen Simonetti	Environmental Scientist	SERAS	732-321-4293	Stephen.j.simonetti@leidos.com	Support
Misty Barkley	Property Coordinator	SERAS	732-321-4205	Misty.barkley@leidos.com	Analytical Subcontract
Mike Hoppe (phone)	WAM	ERT	732-906-6908	Hoppe.michael@epa.gov	Response WAM

Comments/Decisions: Non-site-related debris has been dumped at the Riverside Avenue site in Newark, NJ. Some of this debris is believed to contain asbestos. Sampling and analysis have been requested to ascertain whether the bulk material does contain asbestos. Approximately 12 samples plus 2 duplicates will be collected and sent to the laboratory for polarized light microscopy (PLM) EPA Non-Friable Organically Bound (NOB)-EPA/600/R-93/116 (target reporting limit 0.5%) with gravimetric reduction reporting by % mass – one week turn around for tabulated results.

Action Items: Find a laboratory that can handle the analysis on short notice.

Consensus Decisions: Sampling will be conducted in Level C. Batta Laboratories will perform the analysis. Reporting limits will be 0.5%. Percent mass will be reported.

QAPP Worksheet #10
Problem Definition

The problem to be addressed by the project: During a site visit on June 14, 2017 with the Remedial Program to support EPA cost recovery efforts, the OSC witnessed large scale construction and debris (C&D) dumping taking place at the site. Amongst the C&D debris were torn bags of construction debris labeled "Asbestos". During a follow up site visit on June 22, 2017, EPA OSCs determined that the material in and around these bags as well as other material in the piles of C&D waste were potentially asbestos containing material (PACM). The Site has a long history of industrial use and EPA has been involved in the Site since 2009. EPA performed a removal assessment in 2009-2010 which lead to a removal action to address several areas of contamination. The Site was put on the National Priorities List (NPL) in 2013 and is currently in the Remedial Investigation Stage. The illegal dumping on the part of the site owned by the City of Newark is relatively recent. ERT has requested assistance in collecting bulk samples from the removal site (Block 614, Lots 63 and 64) to collect up to 12 samples from the construction and debris piles to be analyzed for asbestos to determine if there is regulated friable asbestos material in the debris dumped on site. Sampling will occur on July 5, 2017.
The environmental questions being asked: Does the material dumped on site contain regulated friable asbestos material?
Observations from any site reconnaissance reports: Construction and debris piles were found to be dumped at the removal site.
A synopsis of existing data or information from site reports: Not applicable
The possible classes of contaminants and the affected matrices: Asbestos in bulk materials.
The rationale for inclusion of chemical and nonchemical analyses: Torn bags of construction debris labeled "Asbestos".
Information concerning various environmental indicators: Not applicable.
Project decision conditions ("If..., then..." statements): If regulated friable material is detected in the samples, EPA Region 2 personnel will determine the need for any enforcement actions.

☐ Worksheet Not Applicable (State Reason)

QAPP Worksheet #11
Project Quality Objectives /Systematic Planning Process Statements

Who will use the data? EPA Region 2 and ERT
What will the data be used for? Data will be used to assess the level of asbestos contamination in bulk materials from debris piles on site.
What type of data is needed? (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques) Asbestos mineralogy and percent mass from bulk materials – off-site laboratory –PLM and/or transmission electron microscopy (TEM) Bulk construction material will be deposited in jars by hand, samplers will wear a discrete pair of gloves for each sample. If needed, sampling procedures in SERAS SOP #2011, *Chip, Wipe and Sweep Sampling* or SOP #2012, *Soil Sampling* will be utilized.
How "good" do the data need to be in order to support the environmental decision? Data must meet definitive data requirements. The quantitation levels are specified in Worksheet #15. Worksheets #12 and #28 show the measurement performance criteria that are needed for the analytical quality indicators. Worksheet #20 outlines the field quality control (QC) samples required. All analytical data will also be verified and validated by SERAS personnel.
How much data are needed? (number of samples for each analytical group, matrix, and concentration) Up to 12 samples and two duplicates will be collected from bulk materials from site debris piles.
Where, when, and how should the data be collected/generated? SERAS personnel, based on discussions with EPA/ERT and Region 2 personnel, will collect up to 12 samples on Wednesday 7/5/17. Samples will be collected into sampling jars provided by the laboratory, labeled, custody sealed and shipped to Batta Laboratories for analysis.
Who will collect and generate the data? SERAS personnel will collect the samples and Batta Laboratories will perform the analysis.
How will the data be reported? A trip report prepared in accordance with SERAS SOP #4017, *Preparation of Trip Reports* will be the final deliverable to the EPA/ERT WAM. Data will be disseminated to EPA Region 2 by the ERT WAM.
How will the data be archived? Hard copies will be stored in SERAS Central Files and e-copies will be stored on SERAS Local Area Network (LAN). Data will be imported into a Scribe database and posted to the ERT- Information Management System (IMS) website. Data will be archived by SERAS in accordance with Administrative Procedure (AP) #34, *Archiving Data Electronic Files.*

QAPP Worksheet 12
Measurement Performance Criteria Table

Matrix	Bulk material
Analytical Group	Asbestos
Concentration Level	low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
Bulk construction material will be deposited in jars by hand, samplers will wear a discrete pair of gloves for each sample. If needed, sampling procedures in SERAS SOP #2011, *Chip, Wipe and Sweep Sampling* or SOP #2012, *Soil Sampling* will be utilized.	Batta Laboratories SOP **M25:** *Preparation and Analysis of Non-Friable Building Materials for PLM and TEM Analysis using EPA, Chatfield and NY-ELAP Protocol (additional instrument information can be found in Batta QM2 and QM3)*	Precision	Relative Percent Difference (RPD) within lab limits	Lab Duplicates	A
		Precision	RPD ±50%	Field Duplicates	S & A
		Accuracy/Bias (Contamination)	No fibers present	Method Blank	A
		Intra-Lab Precision	Within control chart limits	Standard Reference Material (SRM) or NOB-containing sample	A
		Intra-Lab Accuracy	Within control chart limits	Standard Reference Material (SRM) or NOB-containing sample	A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2)
[2]Reference number from QAPP Worksheet #23 (see Section 3.2)

☑ Worksheet Not Applicable (State Reason) No existing data are available for this response.

QAPP Worksheet #13
Existing Data Criteria and Limitations Table

Existing Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Org., Data Types, Data Generation/ Collection Dates)	How Data Will Be Used	Limitations on Data Use

QAPP Worksheet #14
Summary of Project Tasks

Sampling Tasks: The purpose of this sampling effort is to collect samples of bulk materials from construction and debris piles dumped at the site. Samples will be stored in glass sample jars provided by the laboratory. Discrete and/or disposable sampling materials (gloves, spoons/scoops, etc.) will be used, as needed, to collect each sample. If large bulk material is present, any tools used to mechanically break up the sample will be decontaminated between samples.
Analysis Tasks: Samples will be prepped for asbestos analysis following Batta Laboratories SOP **M25:** Preparation and Analysis of Non-Friable Building Materials for PLM and TEM Analysis using EPA, Chatfield and NY-ELAP Protocol. This method combines the tracked gravimetric-reduction preparation of 198.4 with the PLM point-counting of 198.1. Reports for each negative result will include "Inconclusive" and a specific disclaimer that quantitative TEM is the only method that can verify that a NOB is not an asbestos-containing material (ACM).
Quality Control Tasks: Field duplicate samples will be collected at a rate of 10%. Field QC samples are described in Worksheet #20. Analytical QC samples can be found on Worksheets 12 and 28. Additional QA/QC used by the laboratory for PLM and TEM analyses can be found in the attached PLM and TEM Quality Assurance Manuals.
Existing Data: Not applicable
Data Management Tasks: All sampling locations will be identified by a field assigned number. Field sampling data will be recorded on field data sheets or in field books and transferred into a Scribe database. All samples will be delivered under chain of custody (COC) to the specified laboratory. Laboratory procedures will be reviewed and the data verified for the appropriate quality assurance objectives. All deliverables will be generated in accordance to the appropriate SERAS SOP and posted to the ERT- IMS website upon completion. Posting to the ERT-IMS site will be considered as completion of the deliverable.
Documentation and Records: All documentation will be recorded in accordance with SERAS SOP #4001, *Logbook Documentation* and SOP #2002, *Sample Documentation*. A verification/validation report will be prepared by the SERAS QA/QC Group. The Trip Report will provide a description of the project; field and laboratory methodologies and results, and will be prepared in accordance with SERAS SOP #4017, *Preparation of Trip Reports*. Documents and records that may be generated during this project include: WP, QAPP, HASP, Field and Laboratory Logbooks, Site Map, Sample Labels, COC Records, Custody Seals, Air Sampling Worksheets, PWA/ASRR, Data Review Records, Data Reduction Records, Data Assessment Forms, Data Validation Records, Instrument Printouts, Verification/Validation Report, Scribe Database, Trip Report, and Field Change Form.
Assessment/Audit Tasks: No performance audit of field operations is anticipated for this project. The tasks associated with this QAPP are assessed using peer reviews and management system reviews. Peer review enables the chemist to identify and correct reporting errors before reports are submitted. Management system reviews establish compliance with prevailing management structure, policies and procedures, and ensures that the required data are obtained.
Data Review Tasks: All project deliverables will receive an internal peer review prior to release, per guidelines established in the SERAS AP #22, *Peer Review of SERAS Deliverables*.

☐ Worksheet Not Applicable (State Reason)

QAPP Worksheet 15
Reference Limits and Evaluation Table

Matrix: Bulk

Analytical Group: Asbestos

Concentration Level: Low

Analyte	CAS Number	Project Action Limit	Project Quantitation Limit (% mass)	Analytical Method		Achievable Laboratory Limits	
				Analytical Sensitivity	Method QLs	Analytical Sensitivity	QLs
Asbestos	1332-21-4 (amphibole asbestos)	Presence/Absence	0.5%	NA	PLM – 0.2% TEM – 0.05%	NA	0.5%

% = percent

QAPP Worksheet #16
Project Schedule Timeline Table

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Field Activities	SERAS	07/05/17	07/05/17	NA	NA
Sample Analysis	Batta Lab	07/06/17	07/13/17	Final Data Package	5 BD after receipt of samples
Data Validation/ Verification/Validation Report Preparation	SERAS	07/14/17	07/28/17	Verification/Validation Report	10 BD after receipt of data package
Trip Report	SERAS	07/28/17	08/18/17	Final Trip Report	15 BD after receipt of Verification/Validation Report

QAPP Worksheet #17
Monitoring Design and Rationale

Describe and provide a rationale for choosing the monitoring approach (e.g., grid system, biased statistical approach): Biased sampling based on visual observations will be conducted to collect samples from bulk materials in construction and debris piles deposited on site in consultation with WAM and OSC.
Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the monitoring locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations). Up to 12 bulk samples will be collected from debris piles during a single sampling event July 5, 2017. Samples will be analyzed for asbestos in non-friable organic materials and percent mass will be calculated.

QAPP Worksheet #18
Sampling Locations and Methods/SOP Requirements Table

Sampling Location/ID Number*	Matrix	Depth ( )	Analytical Group	Concentration Level	Number of Samples (identify field duplicates	Sampling SOP Reference[1]	Rationale for Monitoring Location
TBD	Bulk Material	Surface	Asbestos	Low	12+2 duplicate	N/A	Judgmental - Debris and construction piles

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #21)

QAPP Worksheet #19
Analytical SOP Requirements Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference [1]	Sample Volume	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Bulk Material	Asbestos	Low	Batta M25 Batta QM2 Batta QM3	NS[2]	1 glass jar	none	none

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).
[2] Not specified – the sample should be representative of the bulk material in question

QAPP Worksheet #20
Field Quality Control Sample Summary Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation SOP Reference[1]	No. of Sampling Locations	No. of Field Duplicate Pairs	Inorganic No. of MS	No. of Trip Blanks	No. of Equip. Blanks	No. of PT Samples	Total No. of Samples to Lab
Bulk Material	Asbestos	Low	Batta SOP M25 Batta QM2 Batta QM3	12	2	NA	NA	NA	NA	14

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

QAPP Worksheet #21
Project Sampling SOP References Table

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Check if yes)	Comments
EPA 560/5-85-030a	Asbestos in Buildings: Simplified Sampling Scheme for Friable Surface Materials	EPA	Glass vial, metal or plastic container	Y	Used for reference only. Method is for intact buildings, not construction debris.
2011[1]	Chip, Wipe and Sweep Sampling	SERAS	Chisel, sample containers	Y	Reference only. No template needed, samples are of bulk material and are not concentrations per area
2012[1]	Soil Sampling	SERAS	Trowels, scoops, etc.	Y	Reference only.

NA = Not applicable
[1]It is anticipated that samples will be collected by hand using a discrete pair of disposable nitrile gloves for each sample. SOP 2011 and 2012 are provided in case additional tools are needed for sample collection.

☑ Worksheet Not Applicable (State Reason) No field equipment requiring calibration will be used for this project.

QAPP Worksheet #22
Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]

NA = Not applicable

SOPs can be found at https://www.epaosc.org/site/site_profile.aspx?site_id=2107

QAPP Worksheet #23
Analytical SOP References Table

Reference Number*	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
Batta M25	Preparation and Analysis of Non-Friable Building Materials for PLM and TEM Analysis using EPA, Chatfield and NY-ELAP Protocol	Definitive	Asbestos	PLM TEM	Batta Laboratories	No
Batta QM2	Analytical and QA/QC Program for Bulk Asbestos	Definitive	Asbestos	PLM	Batta Laboratories	No
Batta QM3	Quality Control/Quality Assurance Manual for TEM Asbestos Analysis	Definitive	Asbestos	TEM	Batta Laboratories	No

*SOPs and Quality Manuals are attached to this QAPP

QAPP Worksheet #24
Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
PLM	Refer to Batta QM2	Refer to Batta QM2	Refer to Batta QM2	Refer to Batta QM2	Lab Analyst	QM2
TEM	Refer to Batta QM3	Refer to Batta QM3	Refer to Batta QM3	Refer to Batta QM3	Lab Analyst	QM3

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23)
QM2 and QM3 are attached at the end of this QAPP

QAPP Worksheet #25
Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
PLM TEM	As per Batta Laboratories' Quality Assurance Manual	As per Batta Laboratories' Quality Assurance Manual	As per Batta Laboratories' Quality Assurance Manual	As per Batta Laboratories' Quality Assurance Manual	As per Batta Laboratories' Quality Assurance Manual	As per Batta Laboratories' Quality Assurance Manual	Analyst	Batta QM2 QM3

[1]Specify the appropriate reference letter or number from Analytical SOP References table (Worksheet #23)

QAPP Worksheet #26
Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Mike Hoppe, ERT WAM, Amy Dubois, SERAS Response TL
Sample Packaging (Personnel/Organization): SERAS personnel
Coordination of Shipment (Personnel/Organization): SERAS personnel
Type of Shipment/Carrier: FedEX
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Custodian/Batta Laboratories
Sample Custody and Storage (Personnel/Organization): Sample Custodian/Batta Laboratories
Sample Preparation (Personnel/Organization): Analyst/Batta Laboratories
Sample Determinative Analysis (Personnel/Organization): Analyst/Batta Laboratories
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): Samples to be shipped within 1 day to laboratory
Sample Extract/Digestate Storage (No. of days from extraction/digestion): Not applicable
Biological Sample Storage (No. of days from sample collection): Not applicable
SAMPLE DISPOSAL
Personnel/Organization: Hazardous Waste Coordinator/Batta
Number of Days from Analysis: Based on laboratory's standard protocol.

QAPP Worksheet #27
Sample Custody Requirements

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory): Samples will be collected by SERAS personnel and packed and shipped in accordance with SOP 2004, *Sample Packing and Shipment,* to Batta Laboratories under proper chain of custody via Fedex.
Laboratory Sample Custody Procedures (receipt of samples, archiving and disposal): A sample custodian at the laboratory will accept custody of the samples, check them for discrepancies, integrity, etc. and log them into the laboratory's sample receipt log. Transfer of samples within the laboratory will be documented using internal chain of custody.
Sample Identification Procedures: The outside laboratories will assign a unique laboratory identifier to each sample during sample login.
Chain-of-custody (COC) Procedures: SERAS personnel will pack and ship the samples in accordance with SERAS SOP #4005, *Chain of Custody Procedures*.

QAPP Worksheet #28-1
QC Samples Table

Matrix	Bulk
Analytical Group	Asbestos
Concentration Level	Low
Sampling SOP	Discreet and/or disposable sampling materials
Analytical Method/ SOP Reference	EPA 600/R-93/116/Batta M25, QM2, QM3
Sampler's Name	DuBois
Field Sampling Organization	SERAS
Analytical Organization	Batta
No. of Sample Locations	12

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Lab Duplicates	10%	Refer to Batta SOP	Refer to Batta SOP	Lab Analyst	Precision	Relative Percent Difference (RPD) within lab limits
Field Duplicates	10%	NA	Document in final deliverable	SERAS Task Leader	Precision	RPD ±50%
Method Blank	5%	Refer to Batta SOP	Refer to Batta SOP	Lab Analyst	Accuracy/Bias (Contamination)	No fibers present
Standard Reference Material (SRM) or NOB-containing sample	5%	Refer to Batta SOP	Refer to Batta SOP	Lab Analyst	Intra-Lab Precision	Within control chart limits

QAPP Worksheet #28-1
QC Samples Table

Matrix	Bulk
Analytical Group	Asbestos
Concentration Level	Low
Sampling SOP	Discreet and/or disposable sampling materials
Analytical Method/ SOP Reference	EPA 600/R-93/116/Batta M25, QM2, QM3
Sampler's Name	DuBois
Field Sampling Organization	SERAS
Analytical Organization	Batta
No. of Sample Locations	12

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Standard Reference Material (SRM) or NOB-containing sample	5%	Refer to Batta SOP	Refer to Batta SOP	Lab Analyst	Intra-Lab Accuracy	Within control chart limits

QAPP Worksheet #29
Project Documents and Records Table

Sample Collection Documents and Records	On-site Analysis Documents and Records	Off-site Analysis Documents and Records	Data Assessment Documents and Records	Other
Chain of Custody Records Sample Labels Custody Seals Field Change Form (if necessary)	NA	Asbestos Worksheets Refractive Index Liquid Calibrations Alignment Checks Preventive Maintenance logs Internal COC Records Asbestos Results	Data Assessment Forms Data Validation Check Records	Verfication/Validation Report GIS Maps Trip Report WP QAPP HASP

NA = Not Applicable

QAPP Worksheet #30
Analytical Services Table

Matrix	Analytical Group	Concentration Level	Sample Location/ID Numbers	Analytical SOP	Data Package Turnaround Time	Laboratory/Organization (Name and Address, Contact Person and Telephone Number)	Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number
Bulk	Asbestos	Low	TBD	Batta M25 Batta QM2 Batta QM3	5 business days for preliminary results 10 business days for final data package	Batta Laboratories Robert Shumate, Jr. 6 Garfield Way Newark, DE 19713 Cell: (302) 943-5569 Tel: (302) 737-3376, ext. 125	NA

NA = Not applicable

QAPP Worksheet #31
Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation)	Person(s) Responsible for Responding to Assessment Findings (Title and Organizational Affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (Title and Organizational Affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organizational Affiliation)
Laboratory Accreditation Audit	As per NYSDOH program	External	NYSDOH	Regulating Agency	QA/QC Manager, Batta Laboratories	Lab Manager, Batta Laboratories	NYSDOH
Laboratory Audit	As per QM	Internal	Contract laboratory	QA/QC Manager, Batta Laboratories	Lab Operations Personnel, Batta Laboratories	Lab Operations Personnel, Batta Laboratory	QA/QC Officer, Batta Laboratories
Performance Evaluation Sample	3-4 times/year	Internal	NVLAP	NVLAP	QA/QC Manager or designee, Batta Laboratories	QA/QC Manager or designee, Batta Laboratories	NVLAP

QAPP Worksheet #32
Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title, Organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (Name, Title, Org.)	Timeframe for Response
Field Observations/ Deviations from Work Plan	Logbook	Amy DuBois/Response TL/SERAS	Immediately	Field Change Form	Amy DuBois/TL/SERAS	Within 24 hours of change
Peer Review	In the deliverable	Amy DuBois/Response TL/SERAS	Prior to deliverable due date	Comments directly in the deliverable	Amy DuBois/TL/SERAS	Prior to deliverable due date
External Lab Performance Audits	Audit Report	QA/QC Manager, Batta Laboratories	Within time allotted by regulatory agency	Corrective Action Plan	Regulatory Agency	Within time allotted by the regulatory agency
Internal Lab Performance Audits	Audit Report	Laboratory Manager, Batta Laboratories	Within time defined in laboratory QA Manual	Corrective Action Plan	QA/QC Manager, Batta Laboratories	Within time defined in laboratory QA Manual

QAPP Worksheet #33
QA Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (Title and Organizational Affiliation)	Report Recipient(s) (Title and Organizational Affiliation)
Technical Report	Monthly	20th of the month following performance period	TL/SERAS	ERT Project Officer and WAM
QA Report	Quarterly	February, May, August, November	QA/QC Officer/SERAS	ERT Project Officer and Quality Coordinator

QAPP Worksheet #34
Verification (Step I) Process Table

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
Trip Report	Reviewed for accuracy	Internal	Peer Review Team
Completeness Check	Review of Planning Documents, Analytical Data package, Sampling Documents and External Reports, as applicable, using the UFP-QAPP Checklist	Internal	SERAS TL QA/QC Chemist

QAPP Worksheet #35
Validation (Steps IIa and IIb) Process Table

Step IIa/IIb	Validation Input	Description	Responsible for Validation (Name, Organization)
IIa	SOPs	Ensure that the sampling methods/procedures outlined in the QAPP were followed and any deviations noted.	SERAS TL, ERT WAM
IIa	COC records	Examine COC records and match with requested analyses.	SERAS QA/QC Chemist
IIa	Lab Data Package	Examine packages against COC forms (holding times, sample handling, methods, sample identifications, qualifiers).	SERAS QA/QC Chemist, TL
IIb	Lab Data Package	Quantify data based on QC deficiencies (precision/accuracy, %RSD, %D, etc.).	SERAS QA/QC Chemist, QA/QC Officer

QAPP Worksheet #36
Validation (Steps IIa and IIb) Summary Table

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa	Bulk Materials	Asbestos	Low	Data Assessment Form for Asbestos in Bulk	QA/QC Chemist, SERAS

☑ Worksheet Not Applicable (State Reason)

EPA Region 2 will be responsible for assessing the usability of the data.

QAPP Worksheet #37
Usability Assessment

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:
Describe the evaluative procedures used to assess overall measurement error associated with the project:
Identify the personnel responsible for performing the usability assessment:
Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:

ATTACHMENT A
Site Location
UFP-QAPP for Riverside Avenue Asbestos ER
June 2017


BLOCK 614
LOT 64
Building # 12
5 Story Brick Building
LOT 63
Building # 7
3 Story Brick Building
Passaic River
50
0
50
100
150
Feet

ATTACHMENT B
Batta Laboratories SOP #M25
UFP-QAPP for Riverside Avenue Asbestos ER
June 2017

STANDARD OPERATING PROCEDURE

M25: Preparation and Analysis of Non-Friable Building Materials for PLM and TEM Analysis using EPA, Chatfield and NY-ELAP Protocol

Prepared by:_____ Date: 4/21/17
Lab Manager – Robert Shumate

Approved by:_____ Date: 4/21/17
QA/QC Manager – Naresh C. Batta

Annual Review Log

	Initials/Date	Initials/Date	Initials/Date	Initials/Date	Initials/Date	Initials/Date	Initials/Date	Initials/Date	Initials/Date	Initials/Date
2017										
2018										
2019										
2020										

1. **TABLE OF CONTENTS**

batta LABORATORIES

2.0 SOP APPROVAL, REVIEW AND UPDATES

2.1 SOP APPROVAL. The approval signature of the below individual can be found on the hard copy version of this document. The approval signature indicates that this Standard Operating Procedure (SOP) is complete and meets the requirements specified in the method reference(s) within it.

Initial Approval By: QA/QC Manager – Naresh C. Batta (see page 3)
Date of Initial Approval: (see page 3)

2.2 SOP REVIEW. This document is reviewed annually by all personnel subject to perform the tasks within it. At the time of review, personnel shall inform lab management when inconsistencies between SOP and actual practices are noted.

2.3 SOP UPDATES. This document is subject to being updated at any time deemed necessary as practices and regulatory requirements mandate. The Document Control Master File is the repository location of all Batta document updates. Updates to this document which are entered within the Document Control Master File are automatically updated within the **Section 3 - Table of Revisions** within this document. Once approved, items listed within the Table of Revisions are considered official changes to the SOP, even if not permanently addressed within the pertinent section of the document. As such, technicians are urged to review this table BEFORE referring to any steps within the SOP. The updates listed within this table will be annotated permanently in its appropriate section at the time of the annual SOP review.

batta LABORATORIES

3.0 Table of Revisions (updated automatically from Document Control Master File):

M25 Preparation of Non-Friable Building Materials for PLM and TEM Analysis

Revision ID	Date of Revision	Description of Update (include applicable Section ID and pages, use multiple rows if needed)	Approval Status	Updated By
1	4/21/17	Insertion of SOP into Document Control registry.	Approved	RGS

4.0 Scope of Application: This method describes the sample matrix reduction and analysis steps for non-friable, organically-bound materials. This method can be employed with any building material, however it is specifically designed for the material types listed in Table 1 of ELAP Item 198.6. For PLM Asbestos is reliably detectable down to a level of 0.2%. For TEM, asbestos concentrations down to 0.05% are quantifiable.

5.0 References:

A. ELAP Item 198.4 and ELAP Item 198.6 for samples using by NYDOH protocol, and

B. EPA/600/R-93/l 16 for Chatfield and samples not under NYDOH purview, and

C. 40 CFR Part 763, Appendix A to Subpart E

D. NESHAP 1990. National Emission Standards for Hazardous Air Pollutants; Asbestos NESHAP Revision, Final Rule. Federal Register, 55(224):48405-48433. Tuesday, November 20, 1990.

6.0 Summary: This SOP is written in compliance with ELAP Item 198.4 and ELAP Item 198.6 for BATTA laboratory sample preparation operations only. The sampling method is not covered. The analytical portion is not discussed in its entirety. Please refer to the BATTA PLM Analytical SOP or the TEM Analytical SOP for more complete analytical guidelines and procedures. Clients or customers are advised at the time of service that proper sampling, packaging and transportation are their responsibility. A disclaimer referring to this in included on the certificate of analysis. However, clients or customers may obtain information regarding sampling, preservation, and transportation from the laboratory if requested. Any omissions which exist in this SOP in relation to ELAP Item 198.4 or 198.6, the steps outlined in the parent method prevail. In the case that this SOP presents an improper deviation from the parent method, technicians shall notify laboratory supervision for immediate procedural review.

7.0 Glassware, Materials, and Apparatus: Refer to Section 3.1 to 3.20 for ELAP Item 198.6 for a complete listing of materials needed. Notable departures from these items are:

a. Centrifuge. While BATTA has a centrifuge, it is not employed in this procedure.

b. Polycarbonate filters. This procedure uses MCE filters.

c. Heat lamp. For drying filters, the SOP uses a drying oven.

d. Melting point solids. To validate the operating temperatures of the muffle furnace, BATTA calibrates the muffle furnace temperature in the 250-600°C range using a digital thermometer calibrated to ISO 17025 standards and uses linear regression to indicate the appropriate adjustment to reach 480°C.

8.0 Health and Safety: Technicians and analysts shall assume all samples received are asbestos-containing and follow safety procedures as described in Batta QAM for sample handling, analysis, storage and disposal. Samples shall be handled inside a HEPA-filtered, negative pressure hood whenever possible. Adequate protection such as gloves, safety glasses and lab coats are mandatory. Samples are securely held for a period of at least six months prior to contract disposal.

9.0 Acceptance Criteria and Frequency of QC Samples

By standard practice, BATTA Labs includes its PLM-NOB and TEM-NOB QC analysis on its certificates of analysis. By doing so, there is added confidence that QC is completed in real time, and that any discrepancies will be brought to light prior to results being issued to the client.

Acceptance Criteria – The range of acceptability for NOBs containing regulated asbestos is dictated by the intra-lab precision and accuracy statistics developed.

Frequency of QC Samples – Preparation and analysis of duplicates is done at a rate of at least 10% of client samples analyzed. Preparation and analysis of blanks (using Non-ACM joint compound) is completed at a rate of at least 5% of client samples analyzed. With a minimum of one lab blank in each preparation batch. The current frequency of QC sample preparation (duplicates and blanks) is viewable in real time on the NOB Prep Log.

10.0 Definitions:

A. Asbestos: "Asbestos" refers to the asbestiform varieties of: chrysotile (serpentine); crocidolite (riebeckite); amosite (cummingtonite-grunerite); anthophyllite; tremolite; and actinolite (AHERA, 1987).

B. Non-friable Organically-bound (NOB): This term (NOB) refers to a wide variety non-friable building materials embedded in flexible-to-rigid asphalt or vinyl matrices. This includes VAT, mastic, asphalt shingles, roofing materials, caulking and glazing, etc.

C. Friable: "Friable" materials are those materials that, when dry, may be crumbled, pulverized, or reduced to powder by hand pressure, and includes previously non-friable material after such previously non-friable material becomes damaged to the extent that when dry it may be crumbled, pulverized, or reduced to powder by hand pressure (AHERA, 1987).

D. Asbestos-Containing Materials (ACMs): "Asbestos-containing materials" (ACM) means any material or product that contains more than 1 percent asbestos (AHERA, 1987; NESHAP, 1990).

11.0 Method Performance: Statistics to characterize the performance of this procedure are being compiled.

12.0 Interferences: None

13.0 NOB Sample Preparation and Analysis:

A. Sample Receiving: This method does not cover sample accessioning procedures. Please follow the general procedures as described in the BATTA QAM for sample receiving, rejection, login, client contacts and documentation.

B. Sample Preparation and Use of the NOB Prep Log

1 Open the NOB Prep Log and use this file to enter all weights and related sample information. This file houses NOB preparation data (PLM and TEM) for all samples prepped by ELAP 198.4, ELAP 198.6, EPA 600/R-93/116 and Chatfield. Data entered onto this spreadsheet will be accessible to PLM and TEM analysts from their analytical files. Onto the NOB Prep Log, fill in the applicable info (i.e. client info, project name, date of prep, weights, etc.) as data becomes available.

2.0 **Uploading Samples from the PLM Login Database:** If the samples were logged into the PLM log database, choose 'Ctrl-Shift-P' to upload PLM login data. Data from the PLM login database will automatically load onto the worksheet entitled "PLM Login Upload to Paste".

2.1 Once PLM login data is uploaded, go to "PLM Login Upload to Paste" and block all the uploaded data. Using your mouse, right click the blocked data and choose "Copy" from the menu. Go back to the worksheet entitled "NOB Prep Log" and place your cursor in column U at the next available row and, using your mouse, right click and choose "Paste special…". From the menu, then choose "Values".

2.2 Once the data is in place, you can begin entering data into NOB Prep Log columns A through S. **Uploading Samples from the TEM Login Database:** If samples have been upgraded from PLM NOB to TEM NOB, there is no need to upload data from the TEM Login database. The TEM lab sample number is simply added into the appropriate location (in Column D) next to its corresponding PLM lab sample number.

2.3 For samples logged exclusively into the TEM database, choose 'Ctrl-T'. Next, follow the steps above for PLM upload, only go to the worksheet entitled "Database" to retrieve the pertinent data.

2.4 For documenting weights, use the **handwritten worksheet** found on the worksheet tab of the same name. Data written on this sheet will be manually transcribed into cells in columns A through L. Other information required to be entered into columns Q through S should be located on the client chain of custody. If this information is not available, leave the cells blank.

2.5 **Quality Assurance Samples:** Regulatory agencies require that we prepare duplicate samples and lab blanks at a rate of 10% and 5%, respectively. For sample duplicates, it is important that the duplicate sample accurately represent the original sample prep. When preparing the duplicate sample, make every effort to duplicate the specific layers taken in the original sample prep. For lab blanks, we use commercially obtained asbestos-free (not traceable) joint compound. At least one lab blank (or 5%, whichever is greater) will be prepared with each prep batch. Amounts of blank material taken should mimic the weights of the field samples within that batch. For samples elevated from PLM to TEM, it is acceptable to use the same prepped material for TEM analysis – there is no need to re-prep the samples from "square one". However, the identification number given the corresponding TEM duplicate will reflect the TEM sample number, not the PLM sample number. For lab blanks, always choose the next

available lab blank (LBxxx) number – do not reuse LB numbers. The next available lab blank number is viewable in cell B3 of the NOB Prep Log. If the incorrect Lab Blank number has been assigned, you will see the message "LB number is not correct" in cell E2 of the NOB Prep Log.

2.6 For each sample, in addition to the crucible ID number, enter the type of sample (choose from: field, reanalysis or blank), NOB Prep Tech initials and date prepped, and enter each sample weights into the NOB Prep Log. Document weights involving the crucible without a lid, and document all filter weights with their corresponding petri dishes. The following weights (recorded in grams) will be documented:

- 2.6.1 Crucible (Column H)
- 2.6.2 Crucible plus Sample, pre-ash (Column I)
- 2.6.3 Crucible plus Sample, post-ash (Column J)
- 2.6.4 Filter only (Column K)
- 2.6.5 MCE Filter plus Sample, post-acid (Column L)

2.7 Take a representative fraction amount (~0.1 – 0.5 gram) of material from the original sample and place into the corresponding sample crucibles designated for each sample to be analyzed. Materials taken from their original samples must be free of foreign material content to avoid possible cross-contamination.
Weigh each crucible, and record their weights on the Prep Log as Cruc+Smpl Wt (pre-ash).

2.8 Place lids on all crucibles prepped above and place them inside the muffle furnace oven. When setting the oven for an oven temperature to 480°C, refer to the current adjusted value on the calibration chart posted on the oven door. Leave samples in the oven for a minimum of 4 hours (do not exceed 12 hours).

2.9 While waiting for the ashing during Step 2.8, place a 47 mm diameter MCE filter with pore size of 0.45µm in each empty petri dish and label accordingly according to sample IDs. Then weigh each assembly (with the filter) and record in the Filter Only column in the Prep Log.

2.10 After at least four hours has passed, turn off oven and open door slightly to cool oven to room temperature. Crucibles can be removed directly at the end of the 4 to 12-hour period, however ensure proper heat protection equipment (tongs, gloves, etc.) are used.

Weigh each sample crucible and record as post-ash weights in the Cruc+Smpl column of the Prep Log.

2.11 Acid digestion: after weighing of the crucibles in Step 2.10, add a few drops of concentrated HCL, then 1-3 ml of 10% HCl (~1N) in each crucible for acid digestion. Grind the residue during acid reaction to a fine suspension. Proceed to filtration step.

2.12 Filtration: Take the filter prepped in Step 2.11 and place it evenly and centered on a wetted filtration surface. Slightly apply vacuum so that the filter can be bubble free underneath. Secure the glass funnel on top of the filter, and then pour the suspension and rinse the crucible with 0.1µm DI water for each sample to be filtered into the filtration device while apply the full vacuum. Rinse off the residue from the crucible well with the same diluent.

Note: make sure not to mix samples with different IDs during filter placement and filtration.

2.13 After filtration, place the filter into its original petri dish assembly with correct labels and place all dishes (unsealed to release moisture) with filters inside a pre-heated drying oven set at 50-100°C for 15-30 minutes. Have the petri dish assemblies cool to room temperature after drying. Weigh each container and record their weights to the Smpl+Filter column on the NOB Prep Log. If sample is going to PLM analysis next, this prep procedure is complete. If filters are to be prepped for TEM analysis, proceed to step 2.14.

2.14 Take a certain amount of residue or a portion of the filter prepped above with the deposit intact and place it into a 25ml vial. Use a clean non-colored vial. Fill the vial with ~5ml of 50/50 aqueous ethanol solution and place it in the sonicator for about 1-5 minutes.

Note:

1. Break the tip of pipette before drawing the solutions;

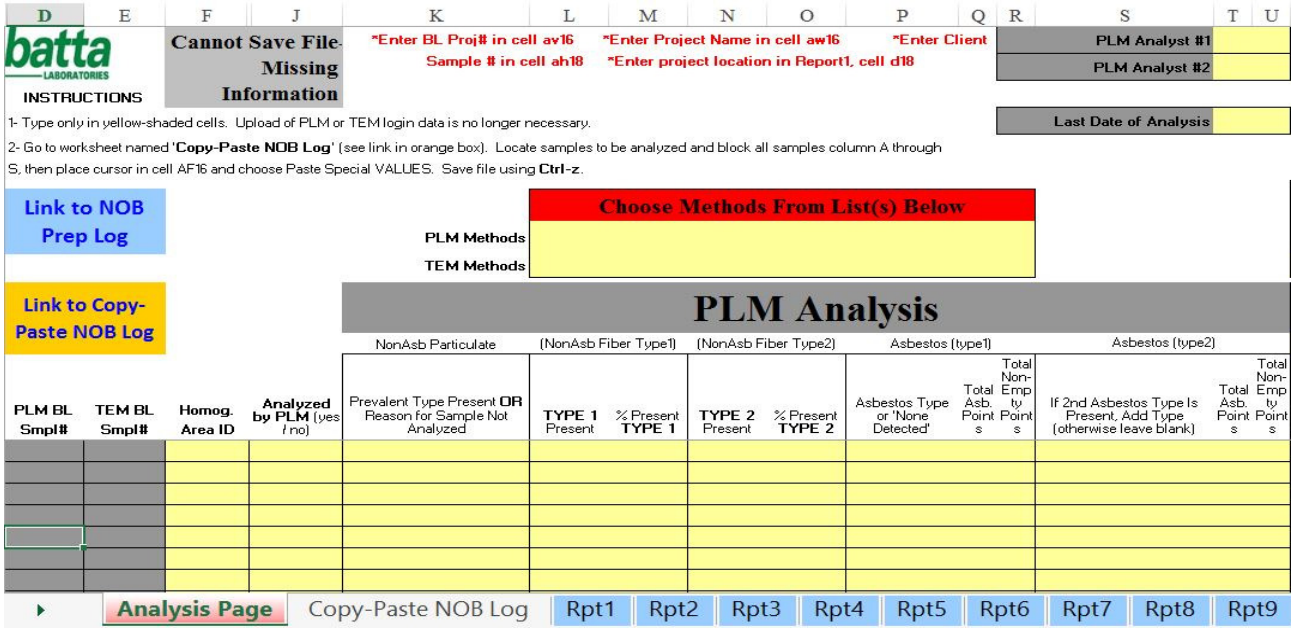
2. Apply each drop after the prior drop is dry or almost dry; and 3. Label clearly each pair of grids prepped to corresponding sample IDs.

2.15 From each vial prepped above, use the glass pipette dropper to drop 3-6

drops solutions over each of the two TEM blank grids (with carbon side up) prepared beforehand. It is better to lay the TEM blank grids on C-fold tissue paper for better moisture suction. Exact number of drops depends on the turbidity of solution.

C. Procedure – PLM Analysis Using the PLM-TEM Combo Analytical Spreadsheet

3.1 This section of the SOP focuses on procedures specific to PLM NOB analysis using the PLM-TEM NOB Excel spreadsheet for analysis and report generation. For PLM analytical procedures, refer to the main BATTA PLM SOP. The PLM-TEM NOB template used for analysis is here. There are other templates used at BATTA, but the benefit of this template is that it can be used to analyze and report PLM and TEM NOB (PLM only, TEM only or both). An example of the layout of the PLM section of the data entry page is below:



3.2 **Getting Started:** Begin with the 'Copy-Paste NOB Log' worksheet. With your mouse, block the rows for cells to include all the samples to be analyzed (include duplicates and lab blanks) from column A through column S, then right click and choose Copy from the menu. Go to the 'Analysis Page' and single click on cell AF16. Right click, then choose Paste Special, then choose Values. All pertinent login and sample prep information for the samples to be analyzed will be placed into the appropriate cells.

3.3 **The Analysis Portion:** For the analysis portion, the PLM analytical benchsheet is completed by hand in the same manner as other PLM analyses. Wherever appropriate, the PLM-TEM NOB analytical worksheet will offer pull-down menus to direct/guide your entry. Where needed, you will have the opportunity to manually type information into the cell if it is not available as an option in the

pull-down menu. The following info is requested:

Location	Description	Mandatory/Optional
Column F	Homogenous Area ID	Optional
Column J	Analyzed by PLM (y/n)	Mandatory
Column K	NonAsb Particulate Type/Reason not Analyzed	Mandatory
Column L	NonAsb Fiber Type 1, if present	Mandatory
Column M	NonAsb Fiber Percent 1, if present	Mandatory
Column N	NonAsb Fiber Type 2, if present	Mandatory
Column O	NonAsb Fiber Percent 2, if present	Mandatory
Column P	Asb Fiber Type 1, if present	Mandatory
Column Q	Asb 1, Total Asb Pts, if present	Mandatory
Column R	Asb 1, Total NonEmpty Pts, if present	Mandatory
Column S	Asb Fiber Type 2, if present	Mandatory
Column T	Asb 2, Total Asb Pts, if present	Mandatory
Column U	Asb 2, Total NonEmpty Pts, if present	Mandatory
Cell T1	PLM Analyst #1	Mandatory
Cell T2	PLM Analyst #1, if applicable	Mandatory
Cell L8	PLM Method Employed	Mandatory
G19*	Project Location	Mandatory

*This cell is located on the 'Analysis Page' worksheet. Prior to being able to save the file, you must have this cell populated, even if only with "n/a".

3.4 **Saving the File**: The analytical file is able to be saved to a very unique, automatically-generated filename that is constructed from specific information added to the spreadsheet by the analyst. Information required to properly save the file to its unique name is:

3.4.1 -BL Project Number

3.4.2 -The Initial BL Sample Number assigned to the batch

3.4.3 -Client Project Name (entered onto the 'Analysis Page' worksheet, cell G19)

3.4.4 -The Initial Client Sample Number assigned to the batch

3.4.5 -Client Name

Once all the above information is placed properly, cell F1 should state "Okay to save file! Use 'Ctrl-z". Until that time, the cell will state "Cannot Save File Missing Information".

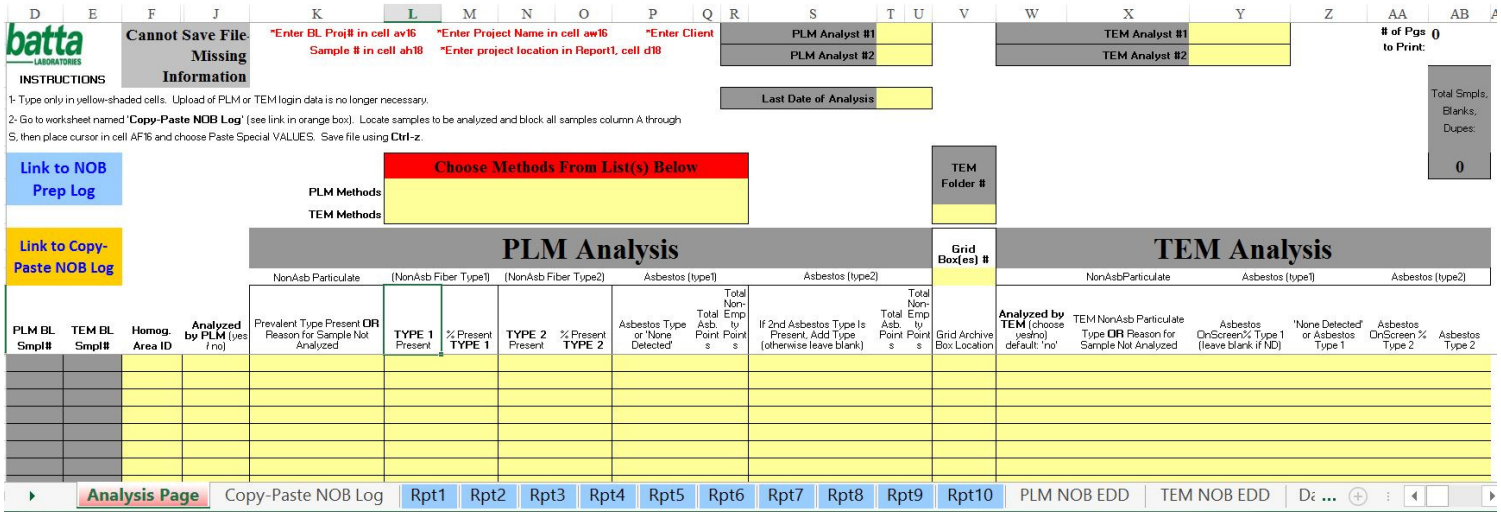
3.5 **Reporting the Results:** Once the analysis is completed and the file is saved to its unique filename, print out the 'Analysis Page'. View the report pages to ensure the data accurately represents the analysis activities that occurred. If the 'Date Sampled' cell (AK17) on worksheet Rpt1 is colored red, that means that the Login database uploaded a date that is not Y2K compatible. This cell has been left unprotected so that you can easily change the date (e.g. from 4/13/1917 to 4/13/2017). Print out all applicable report pages and submit the entire package to an approved signatory for QA review and signing. Ensure that the submitted package

includes:

3.5.1 Analytical Page(s)
3.5.2 Report page(s)
3.5.3 PLM Analytical Benchsheet(s)
3.5.4 Chain of Custody/Client Field Sampling Sheet(s)
3.5.5 Client communications

D. Procedure – TEM Analysis Using the PLM-TEM Combo Analytical Spreadsheet

3.6 This section of the SOP focuses on procedures specific to TEM NOB analysis using the PLM-TEM NOB Excel spreadsheet for analysis and report generation. For TEM analytical procedures, refer to the main BATTA TEM SOP. Just as with other TEM analyses at BATTA, Chrysotile is identified using on-screen recognition of SAED (taking occasional micrographs are required) and Amphiboles require EDS confirmation. At least one EDS (data file or hard copy) must be documented for each type identified. The PLM-TEM NOB template used for analysis is here. There are other templates used at BATTA, but the benefit of this template is that it can be used to analyze and report PLM and TEM NOB (PLM only, TEM only or both). An example of the layout of the TEM section of the data entry page is below:



3.7 **Getting Started:** Begin with the 'Copy-Paste NOB Log' worksheet. With your mouse, block the rows for cells to include all the samples to be analyzed (include duplicates and lab blanks) from column A through column S, then right click and choose Copy from the menu. Go to the 'Analysis Page' and single click on cell AF16. Right click, then choose Paste Special, then choose Values. All pertinent login and sample prep information for the samples to be analyzed will be placed into the appropriate cells.

3.8 **The Analysis Portion:** Wherever appropriate, the PLM-TEM NOB analytical

worksheet will offer pull-down menus to direct/guide your entry. Where needed, you will have the opportunity to manually type information into the cell if it is not available as an option in the pull-down menu. The following info is requested:

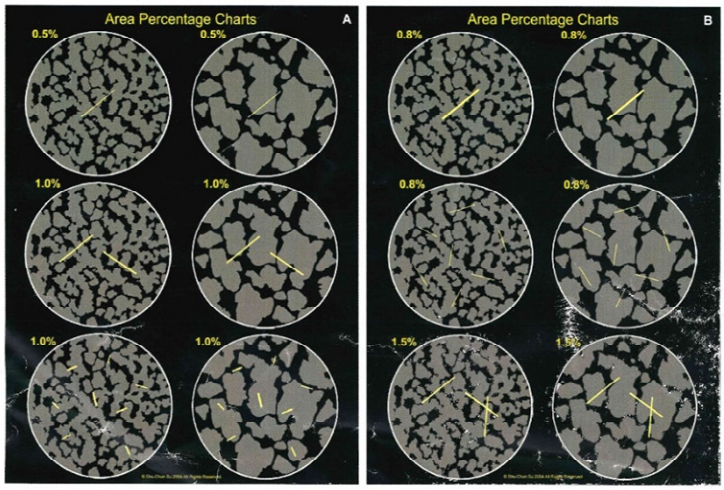
Location	Description	Mandatory/Optional
Column F	Homogenous Area ID	Optional
Column W	Analyzed by TEM (y/n)	Mandatory
Column X	NonAsb Particulate Type/Reason not Analyzed	Mandatory
Column Y	Asb Fiber On-Screen Percent 1, if present	Mandatory
Column Z	ND or Asb Type 1, if present	Mandatory
Column AA	Asb Fiber On-Screen Percent 2, if present	Mandatory
Column AB	Asb Fiber Type 2, if present	Mandatory
Cell Y1	TEM Analyst #1	Mandatory
Cell Y2	TEM Analyst #1, if applicable	Mandatory
Cell L9	TEM Method Employed	Mandatory
G19*	Project Location	Mandatory

*This cell is located on the 'Analysis Page' worksheet. Prior to being able to save the file, you must have this cell populated, even if only with "n/a".

3.1 **Particulate Loading:** Particulate loading should be between 10-50% to be appropriate for analysis. If loading is below 10%, verify whether loading can be increased by examining the vial suspension. If loading is >50% reject prep and submit for a re-drop of the prep

3.2 **Scanning and Estimating On-Screen Percentages:** Scanning of appropriately-loaded grid areas shall take place at a magnification of 14,000x or greater. If during the grid area scan, asbestos is found in area concentrations below 1%, enter 0.5 into column Y (or column AA for the second asbestos type). To aid analysts in estimating on-screen percentages of particulate and regulated asbestos fibers, there are graphic representations of loadings posted on the wall of the TEM Room. The graphic is included below.

Area% Chart With Matrix



3.3 **Saving the File**: The analytical file is able to be saved to a very unique, automatically-generated filename that is constructed from specific information added to the spreadsheet by the analyst. Information required to properly save the file to its unique name is:

3.3.1 -BL Project Number
3.3.2 -The Initial BL Sample Number assigned to the batch
3.3.3 -Client Project Name (entered onto the 'Analysis Page' worksheet, cell G19)
3.3.4 -The Initial Client Sample Number assigned to the batch
3.3.5 -Client Name

Once all the above information is placed properly, cell F1 should state "Okay to save file! Use 'Ctrl-z". Until that time, the cell will state "Cannot Save File Missing Information".

3.4 **Reporting the Results:** Once the analysis is completed and the file is saved to its unique filename, print out the 'Analysis Page'. View the report pages to ensure the data accurately represents the analysis activities that occurred. If the 'Date Sampled' cell (AK17) on worksheet Rpt1 is colored red, that means that the Login database uploaded a date that is not Y2K compatible. This cell has been left unprotected so that you can easily change the date (e.g. from 4/13/1917 to 4/13/2017). Print out all applicable report pages and submit the entire package to an approved signatory for QA review and signing. Ensure that the submitted package includes:

3.4.1 Analytical Page(s)
3.4.2 Report page(s)
3.4.3 PLM Analytical Benchsheet(s)
3.4.4 Chain of Custody/Client Field Sampling Sheet(s)
3.4.5 Client communications

14.0 NOB Report Calculations

A. Ashed Residue: 100-(((A-B)-(C-B))*(100/(A-B))))
B. Insoluble Residue: ((D-E)*(100/(C-B)))) = F
C. Percent Asbestos, by PLM: ((((G/H)*100)*F)/100))))
D. Percent Asbestos, by TEM: ((I*F)/100))

Where,
A = initial mass
B = crucible weight
C = crucible + sample weight (pre-ash)
D = Filter + sample weight
E = Filter (only) weight
F = Insoluble Residue
G = Number of Asbestos Points (PLM)
H = Total Non-Empty Points (PLM)
I = On-screen Asbestos Percentage (TEM)

15.0 Calibration and Standardization

Steps regarding microscope calibration and standardization using reference materials is covered as part of the PLM and TEM analytical procedures.

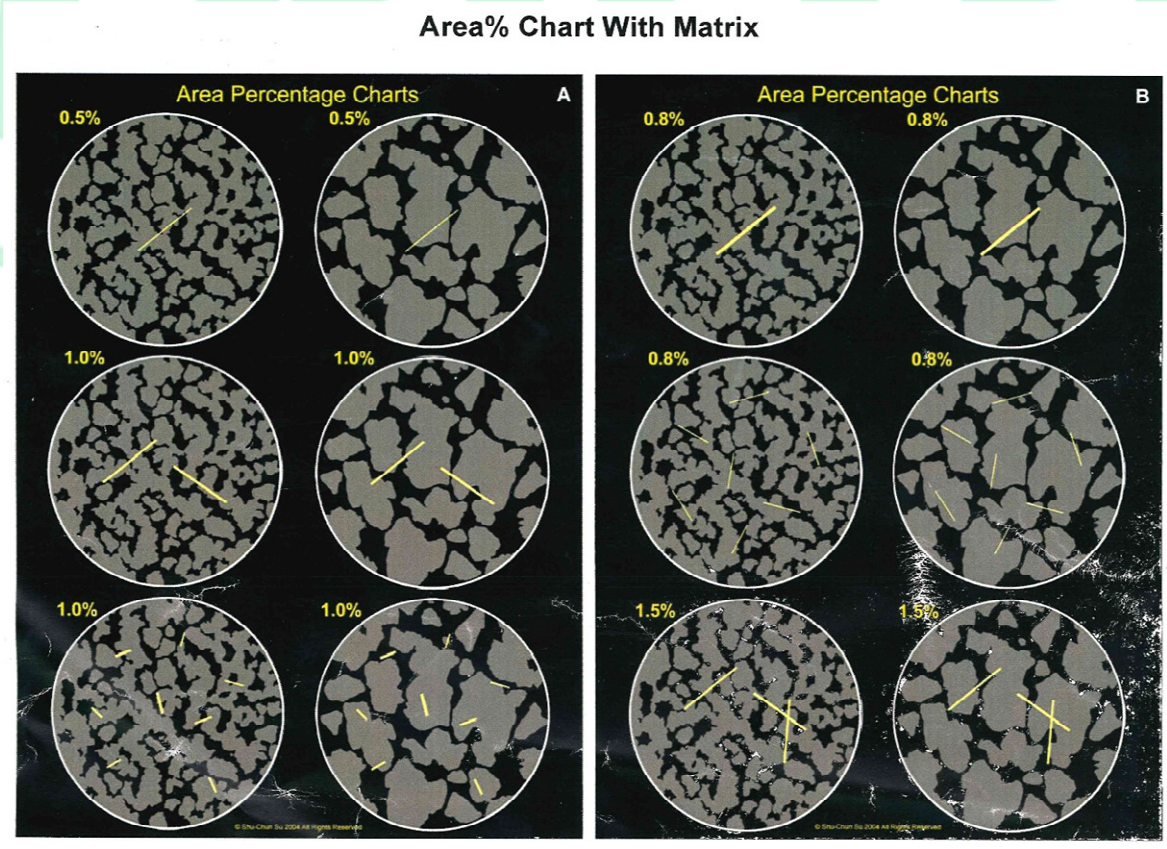
16.0 Corrective Actions for Out of Control Data

Analytical data is evaluated at three main stages: point of data entry, point of data review and point of report review. The data is reviewed by the prep technician and the analyst as they are entering the NOB Prep Log and analytical data into the Excel spreadsheets, respectively. Data is reviewed by the analyst when the preliminary report is printed. The final report is compared against the raw data by an approved signatory at the time the final report is signed.

Whenever out of control data is observed, the data for that entire batch is scrutinized beginning at the prep stage and throughout the analysis. The actions taken will depend upon the issue observed. The end result may be a need to re-accomplish the entire prep batch, only a sample or none at all. If the out of control data is observed later in the process, there may be a need to recall results from the client. If out of control data causes errant results to be sent to a client, a corrective action is initiated.

17.0 Images

A. Particulate Loading Quantification Graphic (posted on TEM Room wall)



B. Graphic of revision to ELAP Item 198.6

New York State Department of Health
Wadsworth Center

Environmental Laboratory Approval Program

Revisions to ELAP Certification Manual for Analysis of Asbestos in Bulk Samples

A new Item (198.6) has been generated for PLM analysis of non-friable organically bound (NOB) materials. This method combines the tracked gravimetric-reduction preparation of 198.4 with the PLM point-counting of 198.1. Note that reports for each negative result (1% asbestos or less) must include "Inconclusive" and a specific disclaimer that quantitative TEM is the only method that can verify that an NOB is not an asbestos-containing material (Section 6.3.2.1).

The application of PLM for NOB analysis has been clarified, as in Section 2 of item 198.1, so that "*...Item (198.1) shall not be used for VAT, resilient floor tiles, mastic, asphalt shingles, roofing materials, paint chips, caulking, glazing and other NOB materials.*"

Item 198.4 was revised to exclude the PLM portion assigned to Item 198.6.

All three methods (Items 198.1, 198.4, 198.6) now require a laboratory thermometer for measurement of room temperature when using RI liquids. This thermometer must be calibrated to NIST-traceable standards.

All three methods (Items 198.1, 198.4, 198.6) have revised refractive-index (RI) measurement requirements for consistency with NELAC. Measurements and calibrations must now be performed within ±0.004 rather than±0.005.

Laboratories seeking accreditation for NOB by PLM must apply for method 198.6. Proficiency testing is required and provided by ELAP.

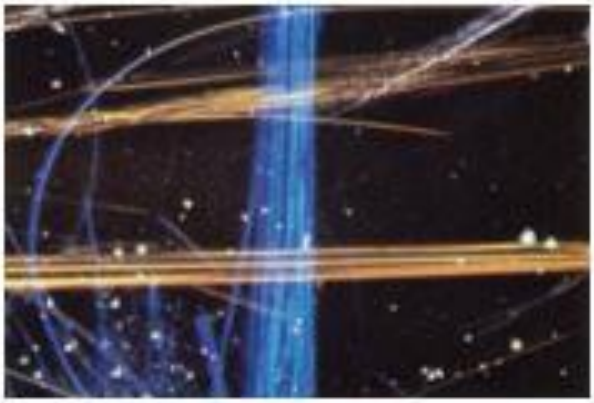
It is no longer acceptable to reference Item 198.1 or EPA 600/M4/82/020 for NOB by PLM. Also, EPA 600/R-93/116 is not an approved method in New York State.

18.0 Required Components Within This Procedure

Description	Section Covering This Component
Identification of the test method	Cover page, page 1
Applicable matrix or matrices	Section 4, page 5
Detection limit	Section 4, page 5
Scope and application	Section 4, page 5
Summary of the test method	Section 6, page 5
Definitions	Section 10, page 6
Interferences	Section 12, page 7
Safety	Section 8, page 6
Equipment and supplies	Section 7, page 5
Reagents and standards	Section 7, page 5
Sample collection, preservation, shipment and storage	Section 8, page 6, Section 6, page 5
Quality Control	Section 9, page 6
Calibration and standardization	Section 15, page 16
Procedure	Section 13, page 7
Data analysis and calculations	Section 14, page 15
Method performance	Section 11, page 7
Pollution prevention	Section 8, page 6
Data assessment and acceptance criteria for quality control measures	Section 9, page 6
Corrective actions for out of control data	Section 16, page 16
Contingencies for handling out of control or unacceptable data	Section 16, page 16
Waste management	Section 8, page 6
References	Section 5, page 5
Tables, diagrams, flow charts and validation data	Section 17, page 16

ATTACHMENT C
Batta Laboratories QM2
UFP-QAPP for Riverside Avenue Asbestos ER
June 2017

ANALYTICAL & QA/QC PROGRAM FOR BULK ASBESTOS



9th Edition, 2017

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STANDARD OPERATING PROCEDURE

QM2 – PLM Analysis and QA/QC Procedures

Prepared by:_____ Date: 3/10/17
Lab Manager – Robert Shumate

Approved by:_____ Date: 3/13/17
QA/QC Manager – Naresh C. Batta

Annual Review Log

	Initials/Date	Initials/Date	Initials/Date	Initials/Date	Initials/Date	Initials/Date	Initials/Date	Initials/Date	Initials/Date	Initials/Date
2017										
2018										
2019										
2020										

1.0 CONTENTS

batta LABORATORIES

2.0 SOP APPROVAL, REVIEW AND UPDATES

2.1 SOP APPROVAL. The approval signature of the below individual can be found on the hard copy version of this document. The approval signature indicates that this Standard Operating Procedure (SOP) is complete and meets the requirements specified in the method reference(s) within it.

Initial Approval By: QA/QC Manager – Naresh C. Batta (see pages 1 & 3)
Date of Initial Approval: (see pages 1 & 3)

2.2 SOP REVIEW. This document is reviewed annually by all personnel subject to perform the tasks within it. At the time of review, personnel shall inform lab management when inconsistencies between SOP and actual practices are noted. Placement of initials and date on the cover page of this document signifies that all items within have been reviewed and are understood. Only the hard copy of the SOP will show review initials and their corresponding dates.

2.3 SOP UPDATES. This document is subject to being updated at any time deemed necessary as practices and regulatory requirements mandate. The Document Control Master File is the repository location of all Batta document updates. Updates to this document which are entered within the Document Control Master File are automatically updated within the **Section 3 - Table of Revisions** within this document. Once approved, items listed within the Table of Revisions are considered official changes to the SOP, even if not yet permanently addressed within the pertinent section of the document. As such, technicians are urged to review this table BEFORE referring to any steps within the SOP. The updates listed within this table will be annotated permanently in its appropriate section at the time of the annual SOP review.

3.0 Table of Revisions (updated automatically from Document Control Master File):

QM2 Manual of Technical and Quality Procedures (PLM)

Revision ID	Date of Revision	Description of Update (include applicable Section ID and pages, use multiple rows if needed)	Approval Status	Updated By	Approved By
1	10/5/16	ient to the document control listing. Form has been used previously, but ha	Approved	RGS	NCB

CHAPTER I

GENERAL STATEMENT

I-I: STATEMENT OF PURPOSE

BATTA LABORATORIES, LLC has developed a comprehensive management system of quality assurance and quality control practices to ensure the consistent accuracy and validity of the data/results that BLI produces. This management system, designed to optimize the quality of the laboratory operations for quality products, consists of quality, administrative and technical systems that govern the proper operations of this laboratory in fulfilling the requirement of ISO/IEC 17025-2005, and in agreement with the NVLAP procedures and general requirements provided in NIST handbook 150, the 2006 edition. The Batta management, through rigorous internal and external reviews and audits, is fully committed to continual improvement of the current QA system for better services.

Batta has been striving to deliver its customers reliable results by developing, improving and maintaining a rigorous quality assurance and quality control system. As such, Batta has constructed a viable organization system for the general laboratory operation, including QA/QC procedures and practices of conducting routine customer analysis. As part of the quality control system, Batta is willing to cooperate with customers or their representatives in clarifying the customer's request and in monitoring the laboratory's performance in relation to the work performed while the confidentiality of the customers are protected.

In Batta, well-established polices, documentation and quality control and quality assurance programs assure that the laboratory's management and personnel are free from any undue internal and external commercial, financial and other pressures and influences that may adversely affect the quality of our work. While data quality and the needs of our clients are vital to Batta being successful as a regional environmental laboratory, our quality management system assures there is no involvement in any activities that will diminish confidence in our laboratory's competence, impartiality, judgment or operational integrity.

Batta's management is committed to professional practices and to the quality of its testing in serving its customers. The laboratory is committed to complying with ISO requirements and NVLAP guidelines, and to continually improve the effectiveness of its management system.

I-II: MODIFICATION HISTORY

This laboratory QA/QC manual for Bulk Sample Analysis using Polarizing Light Microscopy (PLM) is its 8th (8) edition to its previous version made in May 2014. In this edition, the following significant changes were made:

1. Changes to reflect the revised quality assurance values and practices made in an attempt to increase real-time feasibility of analyst precision and accuracy determination.
2. Updating all references to the organizational name from Batta Laboratories, Inc. to Batta Laboratories, LLC.

The QA/QC manual is assessed for revision on an annual basis unless requested otherwise by the lab QA/QC officer in pursuant to the improvement and assurance of the quality of the laboratory operation and quality systems. Modifications can be suggested at any time by all lab personnel. Modifications/revisions may include: changes in specific laboratory procedures reflecting current practices, responses to changes in accreditation programs (NVLAP, EPA, AIHA, etc.), responses to inspections (accreditation agency site visits and comments), methodological changes, and editorial revisions. However, a revision of the laboratory standard operational procedures (SOP) and changes in QA/QC protocols must be approved by the laboratory director and a revision request form shown in Page B-19 of Appendix B must be filed prior to any changes applied. Upon its completion, the manual has to be cosigned by the laboratory director, the laboratory manager, and the laboratory QA/QC officer. Laboratory staff will also have to sign a form acknowledging their having reviewed and/or read through the revision.

The following changes have been made to the 2016 edition:

1. Updated **Section I-II** (above) to reflect the frequency of manual updates.
2. Updated **Section I-III** to reflect the current objectives of the QA-QC Program.
3. Updated Appendix B (Forms) to reflect the current forms used by the lab.
4. Updated **Section IV-VIII** of Chapter **IV** (policies and procedures pertaining to using contract laboratories and vendors for equipment and supplies) to reflect current practices.
5. Updated biography
6. Updated organization chart
7. Typo and grammar-related corrections

I-III: OBJECTIVES OF THE QUALITY ASSURANCE PROGRAM

1. Accurately represent the current quality state of the laboratory.

2. To establish procedures to ensure the data and results generated in the laboratory are within known limits of accuracy and precision.

3. To establish procedures to document and verify that these quality control measures are being carried out in real time.

4. To establish procedures which ensure that, at any future date, results reported to a client or to a government regulatory agency can be traced to:

   a. The date and procedures of sampling

   b. The date the analysis was performed

   c. The analyst who performed the tests

   d. The raw data generated during the performance of the tests

   e. The condition of any instrument, reagent, or equipment used in the analysis, at the time it was run

   f. The status of the quality control system at the time the test was run

5. To establish procedures which minimize the possibility of erroneous data, or the deliberate falsification of data. This will be done by observing the limits derived from control charts and reference standards and periodically performing Data Integrity Audits.

6. Periodic review (at least once per year) of established procedures by QA Coordinator or Lab Manager for potential updates in personnel and/or procedures.

7. To make necessary changes to established procedures as they occur and are implemented.

8. To reveal technical limitations and weaknesses in equipment, procedures and personnel such that improvements can be made swiftly and elevate analytical confidence.

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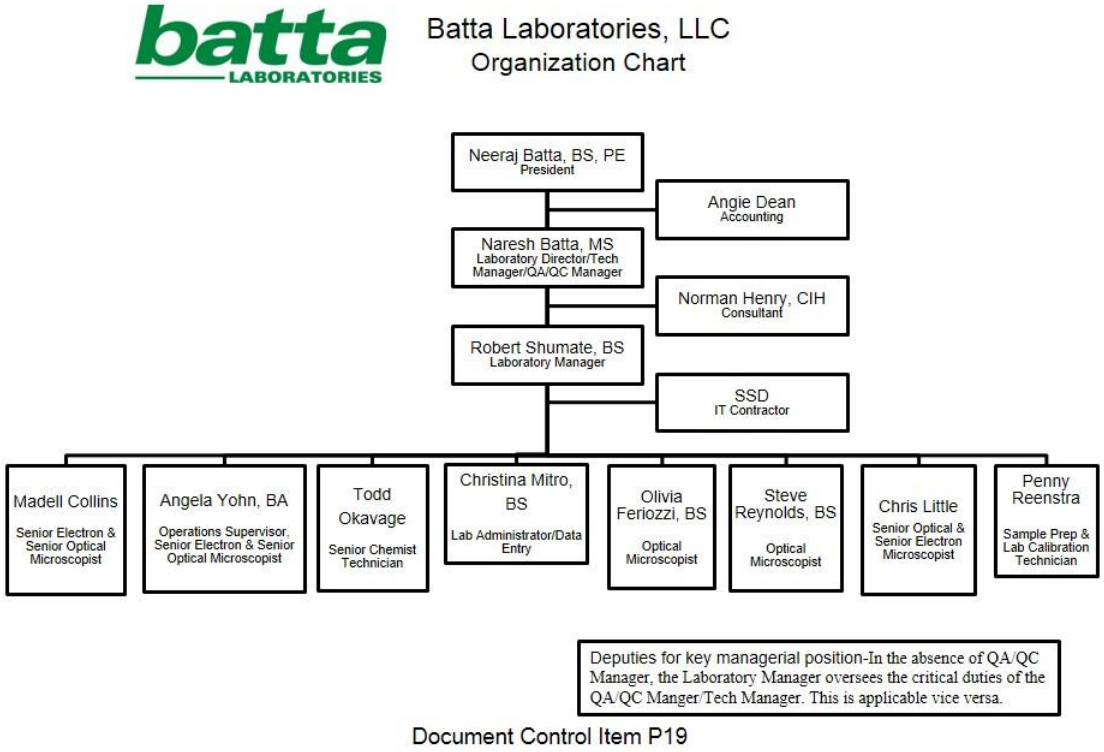
I-IV: ORGANIZATION CHART



Document Control Item P19

I-V: STATEMENT OF AUTHORITY AND DUTIES

The managerial and technical personnel as listed in the above organization chart, irrespective of other responsibilities, have the authority and due resources needed to carry out their duties, including the implementation, maintenance and improvement of the management system (quality assurance and quality control in specific), and to identify the occurrence of departures from the management system or from the procedures for performing tests and/or calibrations, and to initiate actions to prevent or minimize such departures, as defined (but not limited) in the following section.

I-VI: DECLARATION OF TITLES, DUTIES AND QUALIFICATIONS

LABORATORY DIRECTOR

MINIMUM REQUIREMENTS:

Ph.D. in Science with a major in Chemistry, **OR**
MS in Science with a major in Chemistry + Six (6) years experience in a laboratory of which at least three (3) years involved management responsibilities, **OR**
BS in Science with a major in Chemistry + Eight (8) years experience in a laboratory of which at least four (4) years involved management responsibilities

Direct experience with analytical instrumentation (GC, AA, UV/Visible Spectrophotometry, etc.)

Experience with and/or knowledge of TEM and light microscopy

RESPONSIBILITIES:

Manage, in conjunction with the Laboratory Manager, the activities of the laboratory

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LABORATORY MANAGER

RESPONSIBLE TO: Laboratory Director

MINIMUM POSITION REQUIREMENTS:

Ph.D. in Life or Physical Sciences, **OR**
MS in Life of Physical Science + four (4) years experience in an analytical laboratory of which at least two (2) years involved management experience, **OR**
BS in Life or Physical Science + ten (10) years experience in an analytical laboratory of which at least four (4) years involved management experience

Experience with analytical instrumentation (GC, AA, GC/MS)

Experience with and/or knowledge of electron and light microscopy

DUTIES AND RESPONSIBILITIES:

Develop and implement new analytical methods appropriate to the laboratory's and/or customer's needs

Evaluate laboratory materials needs and make recommendations

Manage laboratory staff by making task assignments, overseeing staff activities and evaluating staff performance

Responsible for QA/QC procedures, records and reports

Participate in Proficiency programs associated with field of expertise as may be required to maintain or institute laboratory accreditations and/or certifications

Interact with potential and current customer

Attend, as needed, pre-contract meetings with customers and contractors

Analyze samples using appropriate technology and instrumentation in his or her specialty.

Prepare reports of results

QA/QC OFFICER

RESPONSIBLE TO: Laboratory Director and Laboratory Manager

MINIMUM POSITION REQUIREMENTS:

BS in Life or Physical Science + two (2) years experience in laboratory work including QA/QC functions

One (1) year experience in charge of all QA/QC activities in an area of laboratory expertise (to include a demonstrated competency in statistics)

Accreditation/certification in a minimum of one discipline for which QA/QC functions are performed

DUTIES AND RESPONSIBILITIES:

Develop QA/QC programs in accordance with accepted practice and appropriate guidelines to assure that the laboratory produces and results are valid and within acceptable operational and analytical limits

Maintain all QA/QC records, prepare timely reports and submit required data to accrediting and/or certifying organizations

Distribute test materials to personnel for analysis in accordance with the QA/QC operational protocols and evaluate results

Monitor sample handling, storage and tracking

Identify deficiencies and initiate and monitor corrective procedures

Review and manage all analytical reports

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MICROSCOPY / OPERATIONS MANAGER

RESPONSIBLE TO: Laboratory Manager

MINIMUM POSITION REQUIREMENTS:

Ph.D. in Life or Physical Sciences + one (1) year experience in a similar laboratory environment, **OR**
MS in Life or Physical Science + four (4) years experience in an analytical laboratory of which at least two (2) years involved management experience, **OR**
BA/BS in Life or Physical Science + six (6) years experience in an analytical laboratory of which at least two (2) years involved management experience

Experience with analytical instrumentation (GC, AA, GC/MS)

Experience with and/or knowledge of electron and light microscopy

DUTIES AND RESPONSIBILITIES:

Develop and implement new analytical methods appropriate to the laboratory's and/or Customer's needs

Evaluate laboratory material needs and make recommendations

Manage laboratory staff by making task assignments, overseeing staff activities and evaluating staff performance

Responsible for QA/QC procedures, records, reports and duties as indicated in the QA/QC Officer section on previous page.

Participate in Proficiency programs associated with field of expertise that may be required to maintain or institute laboratory accreditations and/or certifications

Interact with potential and current Customer

Attend, as needed, pre-contract meetings with Customers and contractors

Analyze samples using chemical technology and instrumentation

Prepare reports of results

MICROSCOPIST

RESPONSIBLE TO: Laboratory Manager

MINIMUM POSITION REQUIREMENTS:

BS in Science/Mineralogy + two (2) years experience in asbestos analysis using PCM or PLM technologies, **OR**
AAS in Science/Mineralogy + three (3) years experience in asbestos analysis

DUTIES AND RESPONSIBILITIES:

Log in and prepare samples

Analysis of samples

Prepare report of results

TEM or PCM support

Other duties as assigned

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SAMPLE CUSTODIAN/DATA ANALYST

RESPONSIBLE TO: Laboratory Manager

MINIMUM POSITION REQUIREMENTS:

BS/BA Science + some experience in chemistry or microscopy, **OR**
AAS in Science + two (2) years experience in chemistry or microscopy laboratory, **OR**
High School Diploma + three (3) years experience in chemistry or microscopy laboratory

DUTIES AND RESPONSIBILITIES:

Receive samples from Customers to include but not be limited to:
- Building and maintaining Customer relations through proper Customer services practices
- Ensuring completeness of Customer chain of custody (should accurately represent samples submitted)
- Signing chain of custody

Log in of samples into Batta Laboratories sample database

- Release samples and related paperwork to laboratory for analysis

Enter analytical data into lab database for final reports to include but not be limited to:
- PLM Customer data and analyst observations (taken from analytical bench sheet)
- PCM Customer data and analyst observations (taken from field sheet or chain of custody)
- Chemistry Customer data and analyst results
- Maintain Customer database
- Enter new Customer project numbers into Batta Laboratory ----New Project Logbook for subsequent entry into database
- Maintain daily sample summary logbook
- Log-out Chemistry and TEM samples following completion of analytical report

Other duties are as assigned

I-VII: PROTOCOLS OF INTERNAL COMMUNICATION AND COMPLAINTS

Batta has established detailed procedures for administrative complaints in the employee manual book, which is available in the front office. Also, lab personnel are urged to bring issues (either related to quality, laboratory operations or client relations) in a timely manner to their upper management according to the organization chart, especially when these issues are related to the effectiveness of the management system and the quality of the operation. The purposes of establishing these protocols of communication and complaints are not only to improve the quality of the product, but also to improve the effectiveness of the management system.

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CHAPTER II

SAMPLE RECEIVING AND LOG IN

II-I: STATEMENT OF PURPOSE

Guidelines and procedures included in this chapter are developed to ensure the appropriateness in handling samples that are in the chain of custody and the correctness of documenting them in relevance to a regulation/SOP requested by the customer. A complete list of regulations and SOPs that have been utilized at customers' requests is shown in **Appendix A**. The Lab Administrator will assume full responsibility during this operation, although the analysts, QA/QC officer and the lab manager may be involved as well as part of the COC. Each person will strictly follow the procedures that are outlined in this chapter if his/her involvement in sample receiving process is inevitable or is duly assigned.

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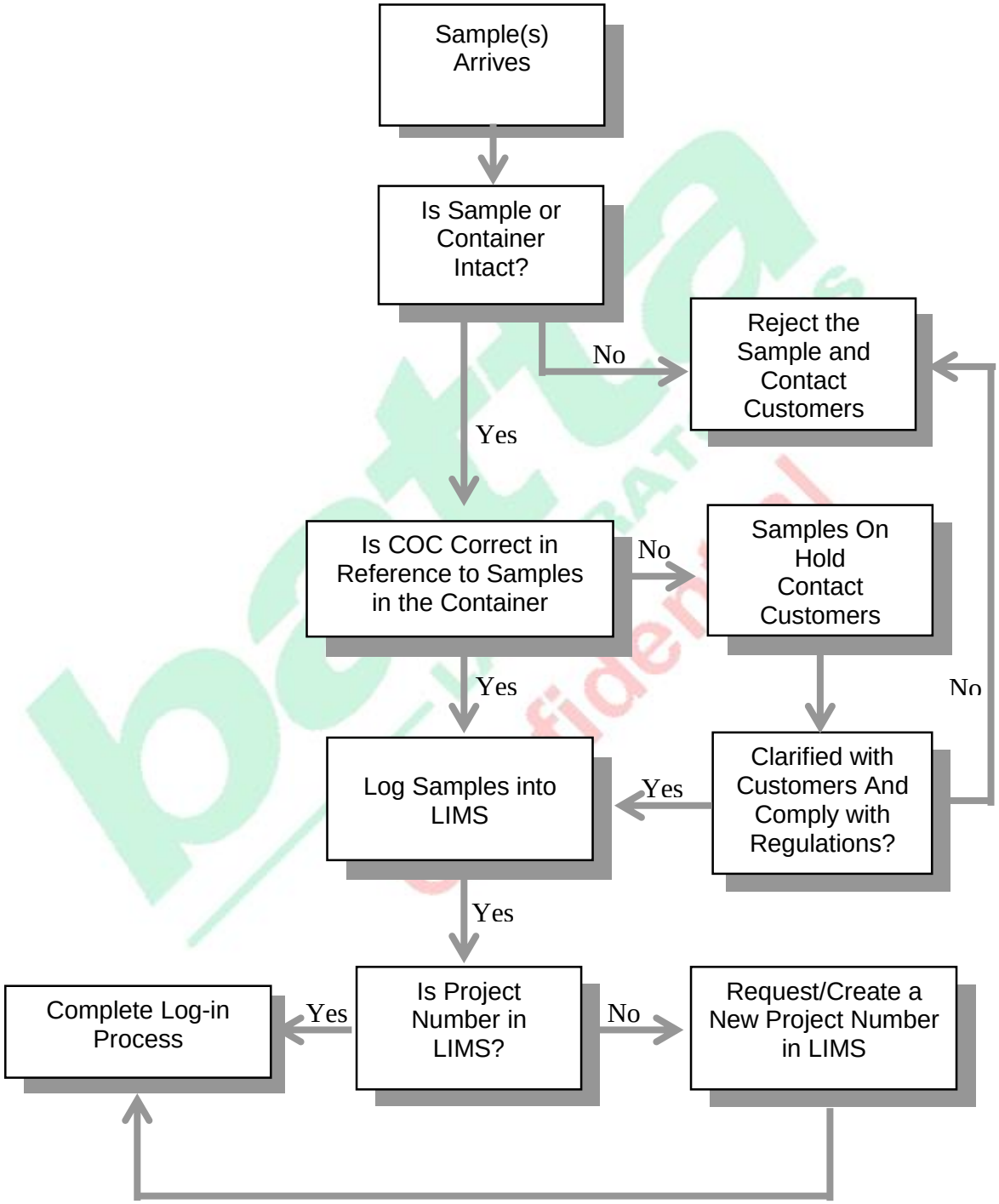
II-II: OPERATIONAL FLOW CHART



II-III: GUIDELINES

1. The Lab Administrator receives and logs in all samples for analysis. However, the analysts (Microscopists) or the receptionist may receive incoming samples. The COC (refer to **Page B-2** of **Appendix B** for an example) is checked to verify that correct amount of samples are present, the analysis types and turnaround times are recorded and that the delivering person has signed the form as "Samples Relinquished By". The samples are then placed in correspondent holding containers marked for specific sample types. Analysts will pull samples (for analysis) from these bins. There are five holding containers within the lab:
    a. "Sample Drop Box For All PLM Samples",
    b. "Sample Drop Box For All PCM Samples (<24 hour)
    c. "Sample Drop Box For All PCM Samples (>24 hour)
    d. "Sample Drop Box For All TEM Samples"
    e. "Sample Drop Box For All Chemistry Samples".

2. The receiving person will then sign, date and record the time on the COC in the section marked: "Samples Received By".

3. Sorting samples must be carried out in a negative HEPA hood (Hood #3 shown on Lab Layout, **Page D-3** of **Appendix D**). Samples that match the COC can be logged into the LIMS. Customers and the QA/QC officer will be notified if any sample does not match the COC The sample login program designates a unique number for each sample. This number is known as the Laboratory Sample Number and all the information about that sample in both the login program and the sample analysis program can be accessed by using this unique number. This Laboratory Sample Number is written on the COC and a sample range is written on the sample container/bag. The sample log report is printed out on as-needed basis by The Lab Administrator. Refer to **Page B-3** of **Appendix B** for an example of Asbestos Daily Login Summary Report. Information contained in the log report includes:

    a. Laboratory Sample Number
    b. BLI Project number
    c. Project name
    d. Date sample is logged
    e. Person's initials (who logged samples into database)
    f. Samples collected by (BEA technician or "CUSTOMER")
    g. Total number of samples in the group
    h. Walk-in? (Y or N)
    i. Testing and analysis to be performed (PCM, or PLM)
    j. Customer's field sample number

4. Batta lab sample numbers (for instance, 453790 through 453799) will be assigned to each field sample on the COC. On each page of the COC, the first lab sample number must be written as a complete lab number (453790 in this case) for the first sample, and the rest can be assigned with only the last three digits of the lab number for convenience and work efficiency. The written numbers must be legible and be consistent for the same COC. A Batta sample number range like 453790 – 799 shall be written on the sample bag or container.

5. BATTA will not accept any samples that are received in:
   - Broken bottles
   - Torn packages
   - Unsealed packages or packages unable to be sealed or made "air-tight"
   - No packages at all
   - Non-labeled containers when more than one sample is submitted
   - Or have container labeling not consistent with the client's chain of custody

   In cases where these issues are evident, the client is immediately contacted and the samples are either placed on analytical hold (until issues are resolved), the samples are returned to the client, or (with written permission from the client) disposed properly.
   This is to:
   - Protect the integrity of the samples as they were collected
   - Minimize cross contamination
   - Maximize the integrity of sample results
     Protect the health of BATTA employees.

6. Different customers may have different requests on how to keep the receiving records. Some customers may request the lab to keep all the communications, sample receipt and postal labels as part of the COC record keeping. Contact customers or the lab manager for a list of customers with particular requests. However, it is mandatory for all government related projects that all receipt, air bills and postal forms be kept as part of the COC.

7. All inquiries regarding sample receiving and log-in, if not resolved due to uncertainty of a regulation or a SOP requested by customers, should be forwarded to management. When samples that do not meet requirements, the client will be contacted, the samples may be labeled as "On Hold" or returned (with COC) to the customer at the written request of the client.

8. As a safeguard against sample contamination and health hazard, sample containers or bags must be opened inside the hood if opening a container or bag is necessary.

CHAPTER III

SAMPLE ANALYSIS

III-I: GENERAL SATEMENT

Guidelines and procedures included in this chapter are developed to ensure the quality and accuracy of analytical results regardless of a SOP. However, the analytical procedures defined by a specific SOP may be different from what will be outlined in this chapter, which is a generalization to meet NVLAP's requirements and common industrial standards. A complete list of regulations and SOPs that have been utilized at customers' requests is shown in **Appendix A**. The lab Analysts/Microscopists will assume full responsibility during this operation in reference to the analytical methods listed in Appendix A as requested, although the QC/QA officer and the lab manager may be involved as well as part of the responsibility. Each person must strictly follow the procedures that are outlined in this chapter. Any interruptions of or deviations from the procedures outlined in this manual due to unseen circumstances should be promptly directed to management. The areas of prep (Hood# 3, Hood# 4 or Hood # 5) and analyses (only Hood # 4 or Hood# 5) of bulk samples are shown on the Lab Layout plan on Page D-3 of Appendix D.

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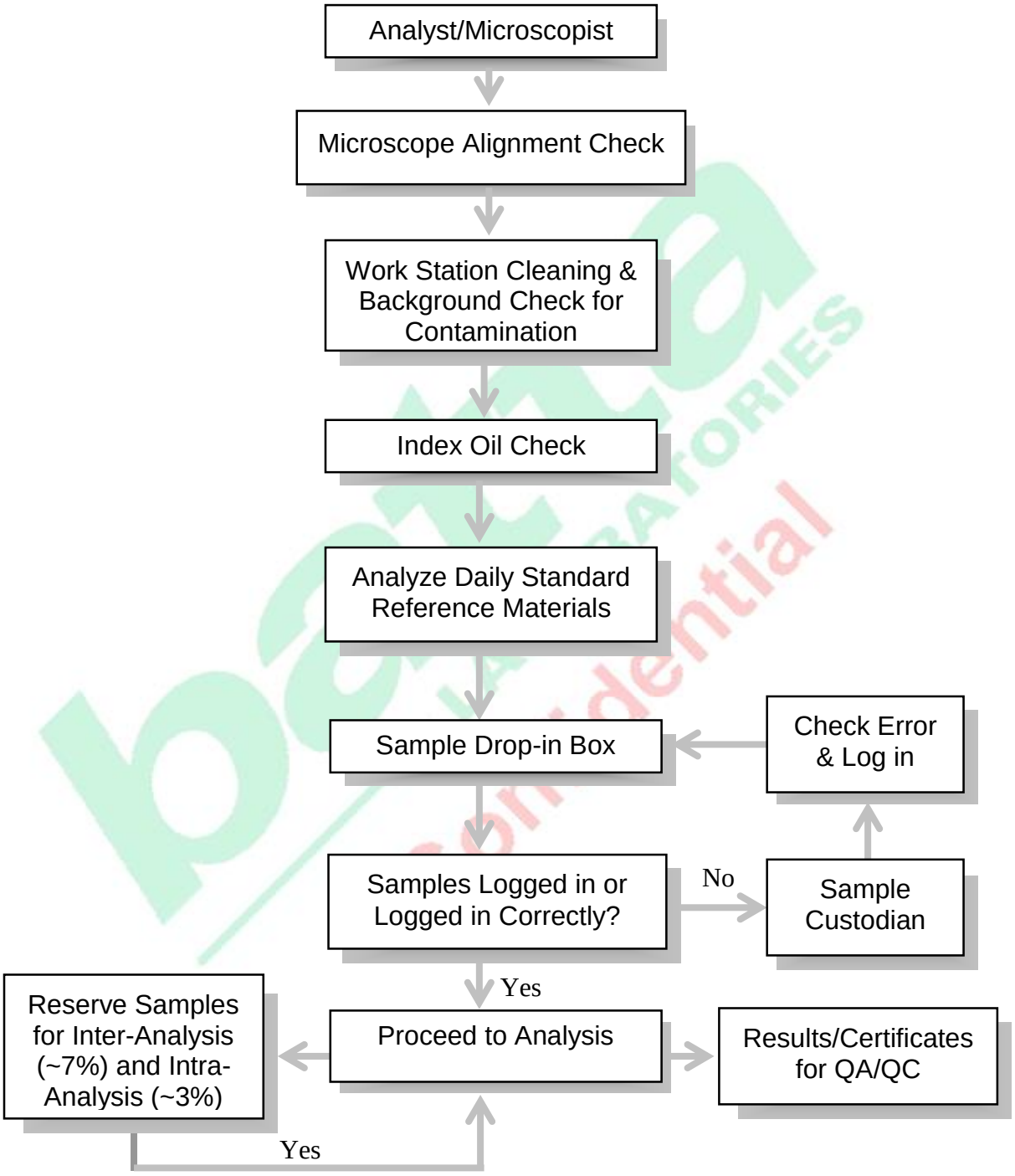
III-II: OPERATIONAL FLOW CHART



III-III: DAILY MICROSCOPE CHECK

The analyst should go through the Daily PLM Check List (see **Page B-4** of **Appendix B**) before proceeding to the analysis. The checklist includes PLM major components alignment, contamination blank checks and daily reference sample analysis. Any items in the list should be carried out during the process of analysis if needed. Refer to McCrone (1987)* for technical supports, such as illumination adjustment and stage centering.

III-IV: REFRACTIVE OIL CHECK (or Reagent Check)

Checking of the index oils used include:

1. The refractive index of the oil(s) to be utilized. This is done quarterly, and when new stocks are received. Analysts are instructed to fill their 4-ounce dropper bottles no more than half way when refilling the container.

2. Check for contamination by prepping a blank with or without SRM fiberglass.

3. The above results will be documented using the form on **Page B-4** and **Page B-5** of **Appendix B**.

III-V: ANALYTICAL GUIDELINES

A. Gross Examination
   1. Turn on the HEPA hood, open the sample container or bag and transfer a representative portion of the sample into a clean petri dish.
   2. Examine sample under stereomicroscope at 10x-30x to determine if there are any suspected asbestos fibers present.
   3. Sample may be manipulated with forceps and probes to find fibers.
   4. At this point, the sample type, homogeneity, texture and color are entered on the PLM Bench Sheet (see an example on **Page B-6** of **Appendix B**) for subsequent entry into the PLM Analysis Program found within selected computers throughout the building.

      Note to the analyst: Friability is NOT to be determined by the analyst. The customer (in writing) must provide determination of friability before it can be documented on the BATTA PLM bench sheet and C.O.A.

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* McCrone, W. C., 1987, Asbestos identification. The McCrone Research Institute. P199.

ANALYTICAL GUIDELINES (Continued)

5. If the sample contains two or more distinct heterogeneous layers, they must be treated as separate samples, as per NESHAP policy. The only possible exception would be wall systems containing joint compounds. In this case, the layers are composited. In all other cases, the samples will be layered according to the method unless specifically requested in writing by the customer. In such case, a statement will be made on the report that the samples were not analyzed in accordance with the EPA Method.
6. If the sample has heavy binder or coating on the fibers, the analyst may have a tough time determining optical properties, especially refractive index. Here are some suggestions to free fibers from tough matrices.
   a. There may be success in using your tweezers to "tease" fibers away from the matrix.
   b. After the sub-sample is in refractive fluid, it may help to use the curved end of tweezers to smash and break down the matrix.
   c. If the matrix is organic in composition, using organic solvents may help. Solvents such as tetrahydrafuran (THF), acetone, and chloroform work well with paints, tars, and vinyl. If organic solvents are used, ensure the exposed sub-sample has been given time to dry prior to adding refractive fluid. If not refractive index values will be affected.

      Note to the analyst: There are health and safety hazard involved in using organic solvents. Read the MSDS before using and always use organic solvents in a well-ventilated area and use gloves.

   d. If the matrix is inorganic in composition, hydrochloric acid (HCl) may be helpful. HCl (6N - 50% solution in distilled water) works well to break down calcites often found in floor tiles and pipe insulations. The sample should be quickly rinsed with distilled water to neutralize the sample. If not, there may be damage done to the PLM (etching of lenses) and the optical properties of the asbestos may be altered. Under no circumstances should the analyst use acids considered strong oxidizers, especially sulfuric acid (H_2SO_4). The strong water-absorbing properties of H_2SO_4 can easily destroy the crystalline structure of chrysotile, chemically altering the fiber.

      Note to the analyst: There are health and safety hazard involved in using acids. Read the MSDS before using and always use acids in a well-ventilated area and use gloves.

ANALYTICAL GUIDELINES (Continued)

e. If all else fails, the analyst can attempt to ash the sample. Refer to the section I-VI (Page III-7) on dealing with non-friable materials. Ashing the sample works well for samples bound with organic matrix, such as tar and vinyl. Care should be taken to not use the ashed sub-sample to determine quantity, since the matrix will likely be reduced in the ashing process. A percent determination of suspect fibers may be done prior to ashing or the analyst may take pre and post weights and perform an EPA point count. The point count calculation should take into account the loss of mass during matrix reduction.

B. Asbestos Identification

1. Check PLM for good alignment with the Nylon test slide (or anthophyllite or amosite) to assure properly crossed polars and crosshair alignment. Close down and focus the field iris using the sub-stage condenser.
2. Mount suspect fibers from sample onto a clean microscope slide in a corresponding refractive high dispersion oil and place a coverslip on top.
3. Place slide on the rotating stage and focus fibers.
4. Check for positive identification of asbestos by the determination of the following optical properties and mark your observations in the appropriately-labeled spaces on the PLM Bench Sheet:
   a. morphology - what do the fibers look like: kinky, straight, platy
   b. color (in plane light) and pleochroism - in plane polarized light, do the fibers exhibit colors that change with fiber orientation?
   c. refractive indices - both parallel and perpendicular to elongation; the dispersion staining color is observed with the central stop and this color is matched to the Color Chart from McCrone (1987). The corresponding wavelength from the chart is then cross-matched with a temperature and a refractive index is then found using Su's method (Su and Cooke, 1997)*.
   d. Birefringence - Measurement of the difference of the indices of refraction (the absolute value of alpha – gamma, or α-γ).
   e. extinction characteristics - behavior of fiber when observed between crossed polars: for five regulated asbestos types (all except for Actinolite), particle shows parallel extinction when a prominent direction (length) is oriented parallel to the polarizer and shows oblique extinction when the fiber is oblique to the polarizer.

* Su, S.C. and Cooke, P. M., 1997, References for asbestos identification by polarized light microscopy in bulk samples. 1997 NVLAP Asbestos Regional Meetings.

ANALYTICAL GUIDELINES (Continued)

f. sign of elongation - behavior of fiber when observed between crossed polars with the red compensator plate inserted. If fiber has elongated particles with n parallel > n perpendicular, it is said to have a positive sign of elongation: blue color when fiber aligned at 2 & 8 o'clock, negative sign of elongation when yellow color aligned at 2 & 8 o'clock

5. Enter the above-mentioned data on the PLM Bench Sheet (see **Page B-6** of **Appendix B**).
6. If no asbestos is present, percent and type of any non-asbestos fibers (with at least one corresponding optical characteristic) is noted on the bench sheet. Optical properties can be birefringence, sign of elongation, morphology, extinction, or isotropic. The property must be a distinguishing feature disqualifying it from being asbestos. Two tables are available in Appendix B to assist the analysis. The first is a list of common asbestos and non-asbestos fibers (**Page B-11** of **Appendix B**). McCrone as part of their course material provided this table. A second table (labeled Table XIV on **Page B-12** of **Appendix B**) was copied from the McCrone reference book, Asbestos Identification (McCrone, 1987). Two flow charts that are from Su and Cooke (1997) were added on **Appendix B** as references for a typical asbestos identification (**Pages B-13** and **B-14** of **Appendix B**).
7. A box is checked on the PLM Bench Sheet to indicate whether it is (or is not) asbestos-containing material (ACM).
8. Identification of the non-fibrous components may be made, along with the percentage and is entered onto the PLM Bench Sheet.
9. The Bench Sheet has shaded sections indicating (to the data entry person) which items are required to be entered into the PLM Analysis Database for the final C.O.A.
10. The Bulk Asbestos Program generates the report for customers.
11. Once printed, the analyst checks the C.O.A. for errors and submits a package (C.O.A. and C.O.C.) to the signatory for final review.

C. Quantification Guidelines

1. Calibrated Visual Estimates (CVE): Analysts are required to analyze at least one reference sample (randomly selected from the reference sample lot) daily before analysis. For samples containing greater than 10% asbestos, it is recommended to report the visual estimate in an increment of 5%. For samples containing asbestos equal to or greater than 1 % but less than 10%, integer numbers from 1 to 10 are recommended. For samples containing less than 1% asbestos, 0.1% to 1% can be used as long as a reference sample of similar

concentration is compared. However, 0.5% is recommended for PT reports (such as NVLAP PT and inter laboratory PTs) should a sample contains less than 1% asbestos.

2. Point Count: A numerical number with at least two significant digits after the decimal is minimum requirement. However, the precision of the number should never exceed the precision of the scale or balance used. The number of significant digits after the decimal shall be consistent across the report and shall be in compliance with customers request if sated in the COC or contract.

D. Sample Clean up
   1. Place used slides into waste bucket beneath lab counter.
   2. Return remaining sample contents back into original container and seal. Wastes like wipes, used petri dishes, debris, etc are placed in a Ziploc bag and kept sealed within the hood, which later goes to a designated PLM waste container for proper disposal.
   3. Place sample in a temporary bulk sample box (See III-VIII).
   4. Wipe off tools, stereomicroscope and hood.
   5. Put tools in beaker filled with water.

III-VI: STANDARD OPERATING PROCEDURES FOR ANALYZING NON-FRIABLE MATERIALS

A. A non-friable material is placed on the stereomicroscope stage in a HEPA-filtered hood.
B. The sample is examined for any fibers.
C. If the sample is hard, such as a floor tile or cement, scrapings can be done with the points of tweezers or other tools and small particles can be mounted in a refractive index oil.
D. If the sample is hard but appears to have the ability to crumble, grind the sample with a mortar and pestle and re-examine the particles under the stereomicroscope.
E. If the sample is hard but cannot be ground, ashing may be used:
   1. Break down material as much as possible and place in a covered ceramic crucible.
   2. Place crucible in muffle furnace at 450°C or less for between 2 and 4 hours.
   3. Cool to room temperature and mount resulting ash in refractive index oil.
      Note to the analyst: When samples are ashed, percent determination by slide mount examination cannot be considered accurate due to possible reduction of sample matrix.
F. If ashing is not desired, acid dissolution (HCl) is recommended using the following procedures:
   1. Break down material as much as possible and place in a small beaker.
   2. Place beaker in fume hood.

3. Add dilute (6N) HCI to sample, enough to soak and allow reaction to proceed for about 10 minutes.
4. Filter solution with D.I. water and a vacuum assembly and dry filter.
5. Analyze residue.

Note to the analyst: Be careful when using HCl. Acids are corrosive! Injury to skin, or damage to clothing or analytical equipment may result from mishandling

G. After having followed the above procedures, continue with the methodology to analyze the sample (see **section III-V**).
H. For quantification purposes, please follow the NY DOH ELAP 198-1 June 1995 Method listed in **Item 9 of Appendix A**. Refer to **Pages B-17** and **B-18** of **Appendix B** for analytical data sheets and certificates.

III-VII: STANDARD OPERATING PROCEDURE FOR ANALYZING DUST SAMPLES

1. Dust samples are collected in 25mm or 37mm cassettes, using low-volume pumps.
2. The cassette is placed on the stereomicroscope stage in a HEPA-hood.
3. The cassette is carefully opened and the dust is examined.
4. Any suspicious fibers are mounted in the appropriate refractive index oil.
5. Follow the SOP of bulk sample analysis for identification of fibers.
6. **Asbestos-containing** will be reported for fiber types that are positively identified and are estimated to be present in quantities greater than 1%.
7. **TR or Trace** will be reported for fiber types that are positively identified and are estimated to be present in a quantity of less than 0.2%.
8. **None-detected** will be reported for samples in which no asbestos is confirmed as present.

Note to Analysts: Dust samples are normally analyzed under TEM. The above procedures are carried out only when the customers understand and agree to do so with proper notification of the laboratory manager. Dust samples analyzed by PLM will receive a report disclaimer indicating that the analytical method was not designed for non-building materials. Refer to Section III-XII below.

III-VIII: POINT-COUNT METHODOLOGY

Point-counting can be done in this laboratory by following the EPA 600/R-93/ 116 Method using the Chalkley Point Array reticule in conjunction with the SOP for bulk samples in this chapter. A special report format has been designed for point counts. A copy of both the work sheets and the C.O.A. are featured on **Pages B-7** and **B-15** of **Appendix B**.

III-IX: DISPOSAL PROCEDURES FOR BULK SAMPLES

1. Analyzed samples are put into a box under the HEPA hood.
2. When the box is full, it is sealed, appropriately marked and dated.
3. After at least 3 months storage time, the box is sealed, double bagged, goose-necked and labeled with our name and date, then it is given to an asbestos abatement contractor who in turn disposes of it at a landfill along with his asbestos trash.
4. The contractor and the landfill (as the waste is received) complete a chain of custody.

III-X: HANDLING THE CHEMICALS AND REAGENTS

1. ACS-certified chemicals, purchased from reputable suppliers, are used to prepare all reagents, solutions and standards. Date of receipt is marked on the label of all chemical reagents.
2. All glassware and other apparatus are cleaned thoroughly with appropriate soaps, detergents, or cleaning chemicals.

All hazardous chemicals are to be used in the fume hoods. Appropriate protection (i.e. eye protection, gloves, lab coats, and/or chemical apron) is mandated in this lab.

III-XI: REPORT

All PLM reports in this lab are automated and generated electronically through the database. These standardized reports meet the need of most of the customers. Occasionally, customers may request a customized report according to their needs. In this case, the QA/QC officer reviews the custom report for its integrity to comply with all quality requirements, such as data accuracy, formats, and citations.

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laboratories for the limited purpose of announcing their accredited status, and for use on the reports that describe only testing or calibration within the scope of accreditation. The term NVLAP and the NVLAP logo, including the Batta NVLAP code is strictly excluded from any personal use or being shown on non-business related paper or digital documents. When referenced for the laboratory's accredited status, the term NVLAP shall be accompanied by the NVLAP Lab Code, which is 101032-0. Whenever not certain, the person who needs to cite the NVLAP term should either consult with the laboratory QA/QC officer, or directly to the NIST 150 handbook (2006 edition) Annex A for details.

III-XII: POLICY ON DEPARTURE FROM STANDARD SOPS AND METHODS

Under special circumstances when some conditions of a certain SOP or method cannot be met or there is a need for departure from its original SOP and method, a statement must be made together with the reported results to notify customers of such changes or departures. Should departure significantly alter the results from the standard methods or SOPs, the customer must be notified prior to analysis to obtain authorization from the client for the analysis. By the meantime, the laboratory QC/QA officer must be notified of such an event and departure.

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CHAPTER IV

QA/QC PROGRAMS

IV-I: GENERAL SATEMENT

BATTA Laboratories LLC has integrated various programs ensuring that the quality of the analytical procedures and results meet clients' requests. These programs include the government programs (such as NVLAP), inter-laboratory programs and intra-laboratory programs. The QA/QC Officer assumes full responsibility to monitor these programs to be conducted routinely based on given guidelines specified in each program. Documentation, such as forms, procedures and data, are kept electronically whenever feasible in a secured, but accessible location for ease of recall.

IV-II: GOVERNMENT QUALITY ASSURANCE PROGRAMS

1. Our laboratory participates in several Federal Quality Assurance Programs. Samples (with results known only to the Federal Authority) are submitted to us on a periodic basis. We analyze these for the indicated analytes and submit our data to the regulatory agency in a timely fashion. In some cases, the proficiency sample testing program is part of an accreditation process and results of samples must be within tolerances defined as acceptable by the issuing organization. In other cases, results are used to compare the laboratory's competence to other participating laboratories. These proficiency surveys are an important part of our overall quality assurance program. All analysts who participate in client sample analysis also participate in their respective Quality Assurance Program.

2. Our laboratory is participating in the following Federal Quality Assurance programs:

    a. NVLAP Proficiency Testing for bulk asbestos samples. Identification is conducted using Polarized Light Microscopy (PLM). (NIST Laboratory No. 101032). Refer to **Pages E-23** through **E-26** of **Appendix E** as reference.
    b. NVLAP Proficiency Testing for Airborne Asbestos Analysis. Utilizes Transmission Electron Microscopy (TEM). (NIST Laboratory No. 101032).
    c. EPA Water Study Performance Evaluation for the Drinking Water Certifications in Delaware, Pennsylvania, and Virginia. (Lab ID. No. DE004)

d. EPA Water Pollution Performance Evaluation Studies. (Lab ID No. DE004).
e. AIHA Proficiency Analytical Testing (PAT) Program for fiber counting using Phase Contrast Microscopy (PCM), metals and solvents. (AIHA Laboratory No. 100448).
f. AIHA Environmental Lead Proficiency Analytical Testing program (ELPAT) for analyzing soil, paint, and airborne lead content. (AIHA Laboratory No. 100448).

IV-III: INTER- AND INTRA-LABORATORY PROGRAM

BATTA LABORATORIES, LLC participates in an Inter-laboratory Round Robin Program with at least two other qualified laboratories (refer to **Pages E-28** and **E-29** of **Appendix E** for reference). The program consists of a set of samples being analyzed type and percent of asbestos fibers, after which the mean, standard deviations and coefficient of variation (CV) are calculated. The Round-Robin is done several times (3-4) a year and results are kept in each lab for NVLAP Accreditation. A copy of the documentation is also submitted as part of the PLM Monthly Summary for any month during which the round robins are completed (refer to **Appendix E** for examples).

Each microscopist is involved in the Intra-laboratory Quality Program. At the end of each batch of samples analyzed, the analyst removes at least 7% of the total number analyzed and sets those samples aside for a different analyst to examine. Approximately 3% of the total number of samples analyzed are reanalyzed by the same analyst. Percent reanalysis values are updated frequently by the QA/QC Officer, indicating to all analysts whether their percent QA is on track. A daily reanalysis form or "PLM QC Log" (refer to **Page B-10** of **Appendix B**) is completed including all samples submitted for reanalysis. These samples are then placed in that month's designated duplicate sample bag. On a real time basis, each analyst enters original and reanalysis results into the Excel spreadsheet setup in the company's database. All statistics are calculated automatically, indicating out of spec (OOS) results. OOS situations are addressed immediately, with corrective actions and client notifications documented as required. All data is summarized in calendar month units of time (refer to **Appendix E**).

The accuracy and precision of each analyst is determined live time from the data accrued by the inter-analyst and intra-analyst results, inter-laboratory results, proficiency results, and daily reference material analysis. Analysts have constant and immediate access to their precision and accuracy statistics, as well as the stats of other analysts, and the lab as a whole.

IV-IV: USE OF REFERENCE MATERIALS

Each microscopist examines a previous NIST PLM Proficiency Round sample on a daily basis (see Page **E-27** of **Appendix E**). The reference values and sample ID numbers are kept on file (see **Page B-16** of **Appendix B**). The results are used to help the analyst calibrate the analysts' visual estimation. There are many samples (currently >30) covering four concentration ranges:

- **0 - 7%** (ranges #1)
- **7.1% - 30%** (range #2)
- **30.1% - 75%** (range #3)
- **>75%** (range #4)

By examining these samples on a routine basis, the analyst should acquire a skill to discern the difference between low percent (Trace - 7%) and samples with trace amounts of asbestos. By definition, a trace concentration is below the analytical detection limit, yet still is observed by the analyst. The best way to explain this is in the case of point counting. The analyst may see asbestos on the slide mounts, but none of the fibers fall on a point. In this case, the analyst must report the presence of asbestos, but cannot include it in the calculation. The result should read "Trace" in this case. A trace amount is a non-quantifiable value and cannot be taken into account as a part of the 100% sample content. This value is used in the same way as samples deemed to have "<1% asbestos". Trace is reported as part of an EPA point-count, not visual area estimation. The actual amount of asbestos in a sample reported as "trace" is assumed to be <0.25%.

The asbestos standards mentioned above are analyzed for percent asbestos present by visual area estimation and via point count (EPA 400 point and NY Stratified Point Count). The analysts' observations are documented on an Excel spreadsheet (see **Page B-9** of **Appendix B**) daily and the results are tabulated immediately on a continuing basis for each analyst. CV values are determined using either historical data or current data. To explain, at the beginning of each month, historical CV data is used until 20 daily reference samples have been analyzed for that month. Once 20 counts have accrued for the month, a CV is calculated based on that month's numbers. Each set of samples has the mean and individual standard deviations calculated, (using upper and lower control limits). From this data, a coefficient of variation (CV) is calculated. CV is calculated as follows:

$$CV = \frac{S}{x_{mean}} \cdot 100$$

whereas CV is used to determine the window of acceptability for client samples reanalyzed. For intra-analyst reanalysis, that analyst's personal CV for that range is used. For inter-analyst reanalysis, the averaged lab CV for that range is used.

As recommended by NVLAP, RTI Calibration Standards of known, low percentage asbestos samples were purchased and are available to all analysts. There are eight standards and they contain asbestos in the following ranges:

Std. 1	0.2– 0.5% chrysotile
Std. 2	1.5 – 2.5% chrysotile
Std. 3	1.0 – 2.0% amosite
Std. 4	1.5 – 2.5% chrysotile **and** 0.2 - 0.75 % amosite
Std. 5	0.2 – 1.0% chrysotile
Std. 6	2.5 – 3.5% chrysotile
Std. 7	2.5 – 3.5% amosite **and** 2.5 – 3.5% crocidolite
Std. 8	0.25 – 0.75% amosite

These calibration standards help the microscopists to more accurately estimate amount of asbestos in samples and also serve as a great training tool. These standard materials have been permanently mounted and have been incorporated into the arsenal of daily reference materials.

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IV-V: CUSTOMER COMPLAINT PROCEDURE

All customer complaints should be directed to the laboratory director or manager for proper handling and actions. Corrective and preventive actions shall be taken if the complaints are directly related to laboratory operations and the quality system. Complaints related to billing and services that are not directly related to or belonging to the realm of laboratory services should be timely forwarded to responsible personnel or departments.

Laboratory management is required to monitor feedback from customers in order to improve the laboratory quality system. Questionnaires or e-mails should be sent to customers from time to time regarding the quality of services and the quality of data provided. Currently, Batta Labs uses information routinely gathered from our Sales and Marketing Department. Feedback data will be compiled by the QA/QC officer and presented to the board of the laboratory management system for review at least annually. Corrective or preventive actions will be taken in response to any actual or potential nonconformities, noncompliance or complaints reported by customers.

If a customer disputes a result from the laboratory, Batta to takes the following steps to resolve the dispute:

1. The original report will be checked against the customer's report to ensure there is not a mix-up of report sheets.
2. Raw data will be retrieved from the computer and/or hard copy files to see if there was a mistake in the reporting of the results.
3. If there are no mistakes on the report, the sample will be reanalyzed.
4. If it is found that there is an error, the information will be entered onto a Corrective Action form. Hard copies of Corrective Action documents are kept in both hard copy and electronically. All errors are documented in the PLM Monthly Summary and are used to calculate error rate. Corrective and preventive actions will be taken accordingly as well, to include follow up.

IV-VI: CORRECTIVE AND PREVENTIVE ACTIONS

Both corrective and preventive actions shall be taken when nonconformity, noncompliance, errors, customer complaints or procedural misconduct occurs.

Corrective actions refers to actions taken to correct or eliminate the cause of **an existing** nonconformity, noncompliance, errors in data produced, defects or complaints in customer service or other undesirable situation in order to prevent their reoccurrence. Case-specific procedures of corrective actions are in effect to

deal with aforementioned unconformities or errors. General procedures for all cases include:

1. Review all original C.O.Cs, reports and communications for errors.
2. Investigate/verify the nature of the error or nonconformity
3. Correct the errors and contact the customers for corrections
4. Revise report packages
5. Investigate sources of errors and causes
6. Conduct error or nonconformity analysis if deemed by the laboratory QA/QC officer
7. Report of the findings to the laboratory director or QA/QC officer
8. Decision or plan for preventive actions

Preventive actions refers to actions to correct or eliminate the causes of **a potential** nonconformity, noncompliance, errors in data produced, defects or complaints in customer service or other undesirable situation in order to prevent their occurrence. Preventive action is actually a proactive process to identify opportunities for improvement rather than a reaction to the identification of problems or complaints.

The preventive actions at BATTA currently consist of annual management review, internal audit, monthly lab meetings, proficiency testing (NVLAP and Round-Robins), review on customer feedbacks and complaints, QA/QC data analysis and control charting, annual staff training, SOP revision, etc. General procedure for the preventive action shall follow:

1. Compiling data or findings through one or more of the above-mentioned process.
2. Analyzing the data and charting the trend
3. Evaluate the risk of potential nonconformities
4. Developing a corrective action plan
5. Submit the plan for management review
6. Executing the approved corrective action plan
7.

See Document Control Item P4 for the complete SOP for corrective and preventative actions.

IV-VII: QUARTERLY MEETINGS AND MANAGEMENT REVIEW

BATTA management conducts a review of the laboratory's management system and testing and/or calibration activities to ensure continuing suitability and effectiveness, and to introduce necessary changes or improvement. This management review shall be conducted at least yearly as a supplement and summarization of quarterly lab meetings. This management review will include, but not limited to the following aspects of interests:

1. the suitability of policies and procedures
2. reports from managerial and supervisory personnel
3. the outcome of recent internal audits
4. corrective and preventive actions
5. assessments by external bodies
6. the results of inter-laboratory comparisons and proficiency tests
7. changes in workload/productivity and resource allocation
8. customer feedback
9. complaints (internal and external)
10. recommendations for improvement (from all staff)
11. corrective and preventive actions
12. other relevant factors, such as quality control activities and staff training

The above management review will be summarized in a written form and circulated among management. All lab personnel will have access to this report. Original copies will be filed on the shared network drive.

IV-VIII: PROCEDURES FOR REVIEW OF REQUESTS, TENDERS AND CONTRACTS

All laboratory contracts and tenders or proposals will be reviewed by the lab director or manger against the request for proposal (RFP). Copy of RFP, proposals and contracts are provided to accounting office. Most of contracts are presented in written format with lab's terms and conditions. In case of walk-in clients, the chain of custody is used as legal agreement. Following steps are taken in review of contacts and proposals:

1. Testing methods are adequately defined by the tenders.
2. The lab verified that it has the adequate resources in meeting the requirements.
3. Any differences between RFP or tenders and the final contracts shall be resolved before commencing any work.
4. Records of reviews including discussion with customer, any subcontract work and any significant changes shall be properly maintained.
5. The customer will be informed of any deviation from the contract.
6. A change order will be initiated if contract is amended after work has commenced.

IV-IX: MEASUREMENT TRACEABILITY AND CONTROL CHARTS

Control charts are constructed to show calibration values or analysts' performance values vs. time, the magnitude of their variation, and the allowable limits of variation. The magnitude of variation specified for many of the control charts used for calibrations and performances is usually defined as 2 times of the standard deviation of a test data set otherwise specified in a specific method defined. R-values from inter- and intra-analyst data are compiled by month to summarize analyst precision. To determine accuracy, R-values are calculated comparing known values of previous NIST proficiency test samples to analyst daily reference sample analysis. To minimize the opportunity for bias, analysts to not have immediate access to reference sample result data. Analysts can view their precision and accuracy data on a per incidence basis and in control charts that summarize points for that current month. Algorithms of these statistical methods are exemplified in the monthly sample summaries.

All calibration standard and instruments are either traceable to their original manufacturer data or to recently calibrated data/certificates issued by certified calibration personnel. These traceability data are stored inside the TEM lab. One should consult with the lab QC/QA officer or laboratory manager if requesting of these data/certificates is needed.

IV-X: STAFF TRAINING AND TRAINING GUIDELINES

PLM analysts are trained by supervision and designated instructors following the laboratory standard operation procedures and methodologies for various PLM analyses. This informal, practicum-based course will cover the principles of polarized light microscopy, crystallography on regulated asbestos and their look-likes, regulations and methodologies, standard operation procedures for related analysis and trainees' statistics. It is BATTA policy that no trainees will operate the PLM without supervision of a designated qualified PLM analyst.

SRM materials and NVLAP PT test samples of known concentration are used for new analyst training and staff refreshing training. Inter-laboratory or Round-Robin samples are sometimes used for training purposes as well. Staff training is more a self-reviewing training than hands-on training for a new trainee with questions given by the supervisor or trainer after the self-reviewing. A checklist of training (see **Appendix C**) is standardized for all new trainees and analysts and must be initialed by the analyst (and trainer, if applicable) after their training session.

IV-XI: UNCERTAINTY OF MEASUREMENT

Otherwise specified, the uncertainty of measurement for analytical results is based on analyst and lab CV values. These controls of uncertainty measurement are mostly automated in real-time for warning and correction.

CHAPTER V

DATA MANAGEMENT

V-I: DATA MANAGEMENT

The data management in this section includes data storage, records keeping, data archiving, and data handling. Refer to Chapter VII of the Main Lab QAM for a complete policy and procedural overview of lab data management.

1. The electronic data such as analytical data, certificates, e-mails, etc. are kept in the laboratory's server system. A tree system in the server was established for storage of different data categories. Analysts and other lab personnel will have to work with the lab data system administrator and contracted IT staff (currently SSD, Wilmington, DE) on questions on how to appropriately store or retrieve data from the central system.

2. Temporary storage of the above categories (listed in Section V-I) is permitted at individual workstations. These data are backed up daily to the central server system. SSD then backs up the above referenced data on off-site tapes, CDs or other data storage media on a daily basis.

3. PLM benchsheets produced during the PLM analysis are kept in binders. Binders will be clearly titled and a time period should be written on the front of the binder to indicate a time frame in which included data were generated. At least three months of benchsheet records are kept near the PLM workstations inside the lab. Periodically, binders are relocated to a lab storage area in the BATTA secured compound. To compliment hard copy storage of PLM benchsheets, complete data package scans (which include PLM benchsheets) are stored electronically on the shared network drive indefinitely. Training data and personnel QA/QC records are stored into binders under each analyst' name, however these receords are generated and/or scanned and stored electronically on the shared network whenever possible.

4. Data and documents related to participation of accreditation programs such as NVLAP proficiency test, AIHA PAT Round, etc. are kept in binders located throughout the lab. (Refer to the **Lab Layout** in **Appendix D**).

V-II: DATA REVIEW

1. The Laboratory Administrator, their designates and analysts are responsible for the data accuracy during the sample receiving, analytical and reporting process. These procedures are detailed in Chapter II for sample receiving and Chapter III for sample analysis.

2. Data entry is completed directly according to the analytical sheets or benchsheets. It is mandatory for each analyst or data entry person to review the data before it is submitted to a higher level for QC/QA purposes. Personnel entering analytical raw data will not alter or amend any analytical data. The only exception to this is when the technical who accomplished the analysis is also the data entry technician. When data is altered or amended, a simple line will be drawn through the data and the corrected information will be written in close proximity along with the initials of the person making the amendment.

3. The laboratory QA/QC officer and management will review submitted reports or other data forms and check them against original C.O.C, and analytical data sheets or benchsheets.

4. Discrepancies found during the data review or QA/QC process will be checked for sources of errors. Final laboratory results may only be submitted to the customer when all error sources are clarified and corrected.

5. Discrepancies found during the re-analysis or duplicate analysis process (refer to Chapter III for details), the manager and QA/QC officer will be informed immediately. Sources of error will be investigated and corrected. A customer notification letter will be issued if the discrepancy has altered the nature of the results, such as from positive to negative, or from positive to negative.

V-III: DATA PACKAGE ASSEMBLY

1. Data packages in hardcopy forms currently available in this laboratory include: customer report packages, analytical benchsheet packages, training records, monthly summary reports, communications and contacts, accreditation programs packages, and references.

2. The customer report package should include: the original analytical report, copies of original C.O.C, shipping records, communication and contacts history and

other addenda. The laboratory package will include a copy of the analytical report and originals of the above referenced addenda, such as original C.O.C, shipping records, etc.

3. The analytical benchsheet packages are separated from the analytical report packages, but are clearly referenced with data, BATTA lab sample numbers, field sample numbers (if provided by the customers), and BATTA job numbers. Refer to Section I for correctly handling the packages and archiving. Analytical data (e.g. benchsheets, EDX spectra, etc.) are made available to the client at their written request.

V-IV: DATA/RECORD DISPOSAL

Both electronic data and hardcopy data are kept for a minimum of five (5) years. At disposal, electronic data will be purged by SSD computer personal using adequate security protocols and methods to ensure the permanent data erase to ensure that the data record cannot be restored or reused after disposal. Hardcopy records will be disposed by shredding to protect client identity and data security. A data disposal manifesto has to be signed and released by the disposal personnel to the laboratory manager for record.

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CHAPTER VI

SAFETY AND HOUSEKEEPING

VI-I: GENERAL GUIDES

All personnel are trained and supervised in proper safety and health procedures in the laboratory and in the field. An individual's safety attitude and safety performance is taken into consideration for all hiring and promotion decisions or changes in assignment. Employees who have difficulty in understanding the importance of a good safety attitude receive additional counseling and training. An employee who continues to display a poor safety attitude may be dismissed from the laboratory.

Safety equipment is provided to employees and its use is required when performing the work that may be hazardous, or when working in or passing through the laboratory or certain field areas. This equipment includes safety glasses and/or goggles, laboratory coats or aprons, steel-toed safety shoes, air-purifying and powered-air respirators, and other equipment as appropriate.

Fire extinguisher, an eyewash station, and a comprehensive industrial first aid kit are available in the laboratory at all times (see **Lab Layout** in **Appendix D**). All of these items are inspected and replenished on a regular basis. All employees are trained in their use.

Concentrated acids and alkalis, organic solvents, and other hazardous chemicals and reagents are properly stored in cabinets. A safety carrier is available for carrying bottles of hazardous substances from one area of the laboratory to another or into field areas.

All samples (asbestos, wastewater effluent, etc.) and other chemicals and substances are properly treated and disposed of when they are no longer needed.

Broken glassware is swept up promptly. A special container is kept in the laboratory for broken glass and other sharp fragments of material.

BATTA maintains a high standard of cleanliness and housekeeping in our laboratory. Illumination, ventilation, bench space, storage facilities and other ambient factors are maintained at appropriate levels.

Only **BATTA** employees are permitted in the laboratory or in equipment storage areas. Representatives of customer companies, prospective customers, representatives of government regulatory agencies, and job applicants may be given brief tours of these areas provided they are accompanied by a BATTA employee at all times.

Activities such as safety inspections, lessons, or meetings are held for technical personnel or for all employees as designated by management. MSDS (Material Safety Data Sheets) for all hazardous reagents are available to the workers in the laboratory.

VI-II: DISPOSAL PROCEDURES

Analyzed samples are placed into dated boxes and stored in a common area near PLM Station #4. These boxes are kept in the main lab area for a period of 2-4 months, then are moved out to the lab storage shed located within the secure compound at the back of the main building. Boxes of samples are kept for a period of at least six months, then are periodically bagged and goose-necked, then disposed by a local contractor for regulated asbestos disposal. Used glass slides, coverslips and associated PLM sample prep waste is kept in sharps-style containers at each respective PLM station area. This waste is disposed as common refuse on an as-needed basis.

VI-III: COMMON REAGENTS FOR PLM ANALYSIS

There are several reagents that may be used in sample preparation: index oils, THF, Chloroform, Ethyl Alcohol and acetone. All of these reagents are kept in the chemical storage bin in the main lab. However, small quantities of dilute acids, acetone and ethyl alcohol in separate containers may also be kept around the each PLM HEPA hood area for convenience and will always be in ventilated locations for safety and health.

VI-IV: EMERGENCY RESPONSES

There are five lab fire extinguishers at the following locations:

1. At the entrance of the bulk asbestos and chemistry lab,
2. behind the door of the Metals Prep Room,
3. at the entrance of the TEM prep area,
4. next to the emergency shower/eyewash station (glassware cleaning section),
5. at the entrance to the break room.

6. Emergency gathering ground is the parking lot at both entrances to the BLI building. Safety shower is at the rear entrance of the bulk asbestos and chemistry lab. Contact the lab manager immediately if precursors of emergencies occur.

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APPENDIX A

LIST OF METHODS

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Note: documentations of methods listed in this appendix are available on the shelves inside the laboratory, and are within the reach of the analysts.

1. EPA/600/R-93/116, July 1993, Test Method: Method for the Determination of Asbestos in Bulk Building Materials.

2. NIOSH 9002: Asbestos (bulk) by PLM, NIOSH Manual of Analytical Methods (NMAM), Forth Edition, 8/15/94.

3. SOP-1988-02 Rev. 1: Analysis of Resilient Floor Tile. Chatfield Technical Consulting Limited – Standard Operating Procedure.

4. Libby Asbestos Project, Libby, Montana: Analytical Guidance Documents, August 2003.

5. McCrone, W. C., 1987, Asbestos Identification. McCrone Research Institute.

6. McCrone, W. C., 1980, The Asbestos Particle Atlas. McCrone Research Institute.

7. Su, S. C., 1996, Rapidly and Accurately Determining Refractive Indices of Asbestos Fibers By Using Dispersion Staining Method – A standard operation procedure for bulk asbestos analysis by polarized microscopy, Hercules Incorporated Research Center.

8. Su, S. C., 1996, Refractive Index Liquid calibration Using Optical Glass Standards– A standard operation procedure for bulk asbestos analysis by polarized microscopy, Hercules Incorporated Research Center.

9. NY DOH ELAP 198-1 June 1995, Polarized-Light Microscope Methods for Identifying and Quantitating Asbestos in Bulk Samples.

10. Analytical Guidance Documents, Libby Asbestos Project, Libby, Montana, August 2003.

APPENDIX B

FORMS, ANALYSTICAL SHEETS AND CERTIFICATES

Table of Contents

Note: The forms, analytical sheets and certificates included in this appendix are digital copies of the originals to fit the page, and therefore may not reflect the true size. The originals are kept on the counter table or in the laboratory database ready for prints.

Chain of Custody

Dedicated to a Cleaner Environment Since 1982

BATTA

BATTA LABORATORIES, INC.
A Certified MBE Company

Delaware Industrial Park - 6 Garfield Way - Newark, DE 19713-5817
(302) 737-3376 - Fax (302) 737-5764
Web: http://www.battaenv.com E-mail: battaenv@battaenv.com

E.P.A. LAB ID # DE 004
A.I.H.A./NLLAP #100448
NVLAP #101032

BLI Project Number (Lab Use Only)

Pick-up charge (Lab Use Only)

Prices quoted for analysis (Lab Use Only)

CHAIN OF CUSTODY

(for Non-BEA Projects)

Client Name and Address:

☐ Sample picked-up by BATTA

☐ Sample delivered by CLIENT

Phone FAX

Turnaround Time Required:

Sampling Location:

Sample Collected By: Date/Time Sample Collected:

BATTA Lab Sample # (Lab Use Only)	Client Sample Number and/or Sample Description	Sample Type (air, aqueous, bulk, wipe)	Analysis/Method Requested	Analytical Result(s) (Lab Use Only)	Analysis Date (Lab Use Only)

(Lab Use Only)
Condition of Sample

Samples Relinquished By (Print & Sign/Date & Time):

Samples Received By (Print & Sign/Date & Time):

Samples Relinquished By (Print & Sign/Date & Time):

Samples Received By (Print & Sign/Date & Time):

(Lab Use Only)
Final Results Reported By: Date of Final Report:

The EPA requires that all wipe samples of settled dust be collected onto wipes that meet all the requirements of ASTM E 1792. Batta Laboratories, Inc. does not accept any liability for samples not collected in accordance with this standard.

Bob C:\WINDOWS\Desktop\Everything\Chain of Custody\Latest Lab COC7.xls

PCM/PLM Daily Login Summary Report

BATTA

PCM PLM Daily Login Summary Report

Report Date 3/3/04

Sample#	Project#	Project Name	Date Loged	Loged By	Date Due	Time Due	Date/Time Submit	Sampled By	Sample Total	Walkin	Sample Type	Field Sample#
447062	L233702	G46200J-CAPITALS D-BOOKER TWA	1/2/04	FF				J DOTSON	3	N	PLM	1A
447063	L233702	G46200J-CAPITALS D-BOOKER TWA	1/2/04	FF				J DOTSON	3	N	PLM	1B
447064	L233702	G46200J-CAPITALS D-BOOKER TWA	1/2/04	FF				J DOTSON	3	N	PLM	1C
447065	L333503	509603-PLEASANTVILLE SCH-2004 R	1/2/04	FF				J DOTSON	3	N	PLM	1A
447066	L333503	509603-PLEASANTVILLE SCH-2004 R	1/2/04	FF				J DOTSON	3	N	PLM	1B
447067	L333503	509603-PLEASANTVILLE SCH-2004 R	1/2/04	FF				J DOTSON	3	N	PLM	1C
447068	L339003	512303-ANNA P MOTE ELEMENTARY	1/2/04	FF				L BAKER	12	N	PCM	123003-01
447069	L339003	512303-ANNA P MOTE ELEMENTARY	1/2/04	FF				L BAKER	12	N	PCM	123003-02
447070	L339003	512303-ANNA P MOTE ELEMENTARY	1/2/04	FF				L BAKER	12	N	PCM	123003-03
447071	L339003	512303-ANNA P MOTE ELEMENTARY	1/2/04	FF				L BAKER	12	N	PCM	123003-04
447072	L339003	512303-ANNA P MOTE ELEMENTARY	1/2/04	FF				L BAKER	12	N	PCM	123003-05
447073	L339003	512303-ANNA P MOTE ELEMENTARY	1/2/04	FF				L BAKER	12	N	PCM	123003-06
447074	L339003	512303-ANNA P MOTE ELEMENTARY	1/2/04	FF				L BAKER	12	N	PCM	123003-07
447075	L339003	512303-ANNA P MOTE ELEMENTARY	1/2/04	FF				L BAKER	12	N	PCM	123003-08
447076	L339003	512303-ANNA P MOTE ELEMENTARY	1/2/04	FF				L BAKER	12	N	PCM	123003-09
447077	L339003	512303-ANNA P MOTE ELEMENTARY	1/2/04	FF				L BAKER	12	N	PCM	123003-10
447078	L339003	512303-ANNA P MOTE ELEMENTARY	1/2/04	FF				L BAKER	12	N	PCM	123003-11
447079	L339003	512303-ANNA P MOTE ELEMENTARY	1/2/04	FF				L BAKER	12	N	PCM	123003-12

Sample Total : 18

Daily PLM Checklist

BATTA

Daily PLM Checklist

1. Check off each item as it is being completed.
2. Complete daily to ensure all QA/QC is done in a timely manner.
3. QA/QC is not to be put-off due to high workloads.
4. Supervision will periodically check forms to ensure compliance.

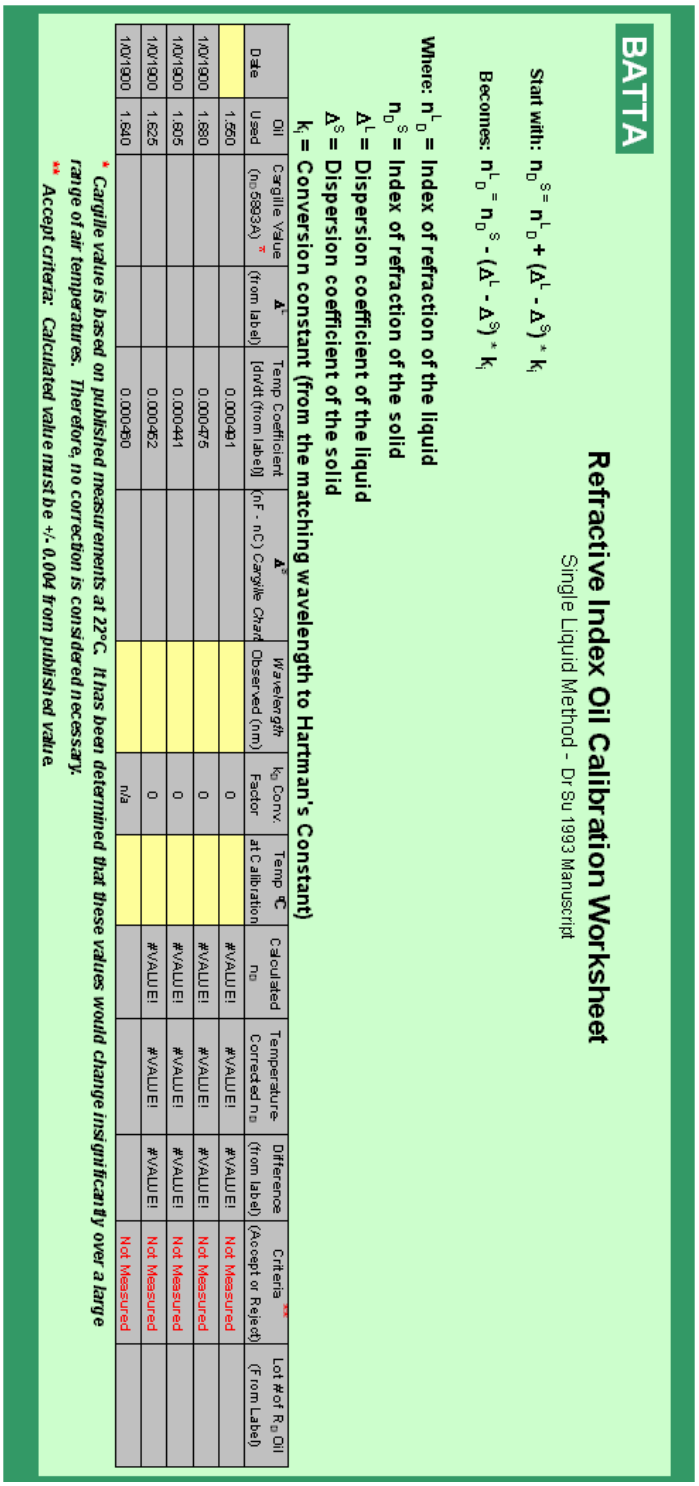
Asghar Keyvanfar December-04

PLM Station #1, Nikon Labophot-POL, Serial # 954996

Analyst Initials	Date	Align PLM 1. Stage Centered 2. Crosshairs (use Anthophyllite mount) 3. Polarizer (black background) 4. Field Iris (centered) 5. Central stop aligned (use Bertrand lens) 6. Check dispersion colors (use Amosite mount)						Contamination Blank (use SRM 1866 Fiberglass to check hood area, stereoscope, and tools)		
		1	2	3	4	5	6	Hood	Stereo	Tools
ak	12/1									
ak	12/2									
ak	12/3									
ak	12/4									
ak	12/5									
ak	12/6									
ak	12/7									
ak	12/8									
ak	12/9									
ak	12/10									
ak	12/11									
ak	12/12									
ak	12/13									
ak	12/14									
ak	12/15									
ak	12/16									
ak	12/17									
ak	12/18									
ak	12/19									
ak	12/20									
ak	12/21									
ak	12/22									
ak	12/23									
ak	12/24									
ak	12/25									
ak	12/26									
ak	12/27									
ak	12/28									
ak	12/29									
ak	12/30									
ak	12/31									

R. I. Oils Calibration Worksheet

(Also available in Appendix E of Monthly Summary)



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Refractive Index Oil Calibration Worksheet

Single Liquid Method - Dr Su 1993 Manuscript

Start with: $n_D^S = n_D^L + (\Delta^L - \Delta^S) * k_i$

Becomes: $n_D^L = n_D^S - (\Delta^L - \Delta^S) * k_i$

Where: n_D^L = Index of refraction of the liquid

n_D^S = Index of refraction of the solid

Δ^L = Dispersion coefficient of the liquid

Δ^S = Dispersion coefficient of the solid

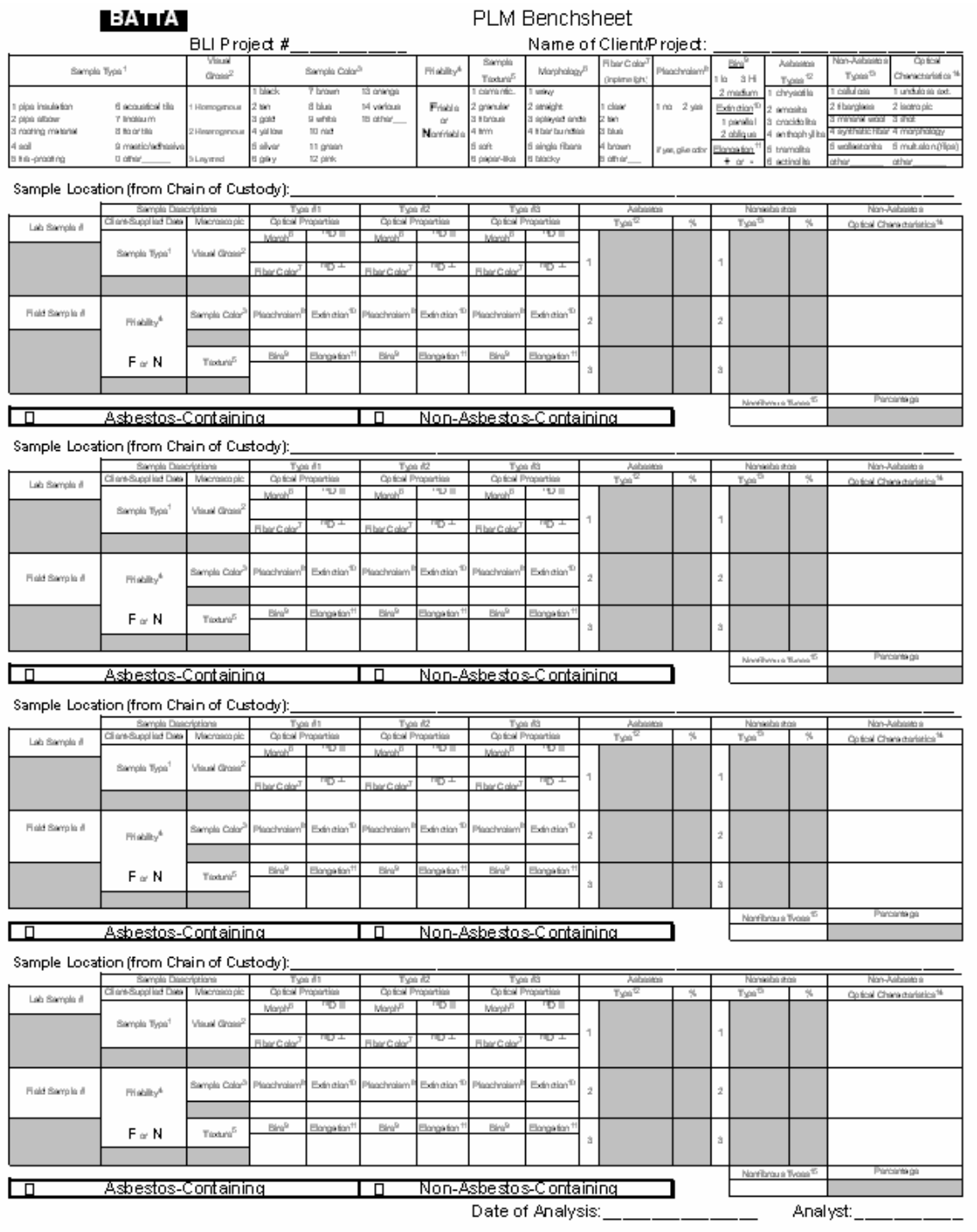
k_i = Conversion constant (from the matching wavelength to Hartman's Constant)

Date	Oil Used	Cargille Value (n_D 5893A) *	Δ^L (from label)	Temp Coefficient [dn/dt (from label)]	Δ^S (nF - nC) Cargille Chart	Wavelength Observed (nm)	k_i Conv. Factor	Temp °C at Calibration	Calculated n_D	Temperature Corrected n_D	Difference (from label)	Criteria ** (Accept or Reject)	Lot # of R_D Oil (From Label)
	1.560			0.00491			0		#VALUE!	#VALUE!	#VALUE!	Not Measured	
1/0/1900	1.680			0.00475			0		#VALUE!	#VALUE!	#VALUE!	Not Measured	
1/0/1900	1.605			0.00441			0		#VALUE!	#VALUE!	#VALUE!	Not Measured	
1/0/1900	1.625			0.00462			0		#VALUE!	#VALUE!	#VALUE!	Not Measured	
1/0/1900	1.640			0.00480			n/a					Not Measured	

* *Cargille value is based on published measurements at 22°C. It has been determined that these values would change insignificantly over a large range of air temperatures. Therefore, no correction is considered necessary.*

** *Accept criteria: Calculated value must be +/- 0.004 from published value*

PLM Benchsheet



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PLM Benchsheet

BLI Project #__________ Name of Client/Project: __________

Sample Type[1]	Visual Class[2]	Sample Color[3]	Friability[4]	Sample Texture[5]	Morphology[6]	Fiber Color[7] (in plane light)	Pleochroism[8]	Bire[9] 1 lo 3 Hi 2 medium	Asbestos Types[12]	Non-Asbestos Types[13] / Optical Characteristics[14]
1 pipe insulation 2 pipe elbow 3 roofing material 4 soil 5 fire-proofing 6 acoustical tile 7 linoleum 8 floor tile 9 mastic/adhesive 0 other_____	1 Homogenous 2 Heterogenous 3 Layered	1 black 2 tan 3 gold 4 yellow 5 silver 6 grey 7 brown 8 blue 9 white 10 red 11 green 12 pink 13 orange 14 various 15 other___	Friable or Nonfriable	1 cementitious 2 granular 3 fibrous 4 firm 5 soft 6 paper-like	1 wavy 2 straight 3 splayed ends 4 fiber bundles 5 single fibers 6 blocky	1 clear 2 tan 3 blue 4 brown 5 other___	1 no 2 yes If yes, give color	Extinction[10] 1 parallel 2 oblique Elongation[11] + or -	1 chrysotile 2 amosite 3 crocidolite 4 anthophyllite 5 tremolite 6 actinolite	1 cellulose / 1 undulose ext. 2 fiberglass / 2 isotropic 3 mineral wool / 3 shot 4 synthetic fiber / 4 morphology 5 wollastonite / 5 mutation (flips) other_____ / other_____

Sample Location (from Chain of Custody):__________

	Sample Descriptions		Type #1		Type #2		Type #3		Asbestos			Nonasbestos			Non-Asbestos
Lab Sample #	Client-Supplied Data	Macroscopic	Optical Properties		Optical Properties		Optical Properties			Type[12]	%		Type[13]	%	Optical Characteristics[14]
	Sample Type[1]	Visual Class[2]	Morph[6]	ⁿD ∥	Morph[6]	ⁿD ∥	Morph[6]	ⁿD ∥	1			1			
			Fiber Color[7]	ⁿD ⊥	Fiber Color[7]	ⁿD ⊥	Fiber Color[7]	ⁿD ⊥							
Field Sample #	Friability[4]	Sample Color[3]	Pleochroism[8]	Extinction[10]	Pleochroism[8]	Extinction[10]	Pleochroism[8]	Extinction[10]	2			2			
	F or N	Texture[5]	Bire[9]	Elongation[11]	Bire[9]	Elongation[11]	Bire[9]	Elongation[11]	3			3			

Nonfibrous Types[15]	Percentage

☐ Asbestos-Containing ☐ Non-Asbestos-Containing

Sample Location (from Chain of Custody):__________

	Sample Descriptions		Type #1		Type #2		Type #3		Asbestos			Nonasbestos			Non-Asbestos
Lab Sample #	Client-Supplied Data	Macroscopic	Optical Properties		Optical Properties		Optical Properties			Type[12]	%		Type[13]	%	Optical Characteristics[14]
	Sample Type[1]	Visual Class[2]	Morph[6]	ⁿD ∥	Morph[6]	ⁿD ∥	Morph[6]	ⁿD ∥	1			1			
			Fiber Color[7]	ⁿD ⊥	Fiber Color[7]	ⁿD ⊥	Fiber Color[7]	ⁿD ⊥							
Field Sample #	Friability[4]	Sample Color[3]	Pleochroism[8]	Extinction[10]	Pleochroism[8]	Extinction[10]	Pleochroism[8]	Extinction[10]	2			2			
	F or N	Texture[5]	Bire[9]	Elongation[11]	Bire[9]	Elongation[11]	Bire[9]	Elongation[11]	3			3			

Nonfibrous Types[15]	Percentage

☐ Asbestos-Containing ☐ Non-Asbestos-Containing

Sample Location (from Chain of Custody):__________

	Sample Descriptions		Type #1		Type #2		Type #3		Asbestos			Nonasbestos			Non-Asbestos
Lab Sample #	Client-Supplied Data	Macroscopic	Optical Properties		Optical Properties		Optical Properties			Type[12]	%		Type[13]	%	Optical Characteristics[14]
	Sample Type[1]	Visual Class[2]	Morph[6]	ⁿD ∥	Morph[6]	ⁿD ∥	Morph[6]	ⁿD ∥	1			1			
			Fiber Color[7]	ⁿD ⊥	Fiber Color[7]	ⁿD ⊥	Fiber Color[7]	ⁿD ⊥							
Field Sample #	Friability[4]	Sample Color[3]	Pleochroism[8]	Extinction[10]	Pleochroism[8]	Extinction[10]	Pleochroism[8]	Extinction[10]	2			2			
	F or N	Texture[5]	Bire[9]	Elongation[11]	Bire[9]	Elongation[11]	Bire[9]	Elongation[11]	3			3			

Nonfibrous Types[15]	Percentage

☐ Asbestos-Containing ☐ Non-Asbestos-Containing

Sample Location (from Chain of Custody):__________

	Sample Descriptions		Type #1		Type #2		Type #3		Asbestos			Nonasbestos			Non-Asbestos
Lab Sample #	Client-Supplied Data	Macroscopic	Optical Properties		Optical Properties		Optical Properties			Type[12]	%		Type[13]	%	Optical Characteristics[14]
	Sample Type[1]	Visual Class[2]	Morph[6]	ⁿD ∥	Morph[6]	ⁿD ∥	Morph[6]	ⁿD ∥	1			1			
			Fiber Color[7]	ⁿD ⊥	Fiber Color[7]	ⁿD ⊥	Fiber Color[7]	ⁿD ⊥							
Field Sample #	Friability[4]	Sample Color[3]	Pleochroism[8]	Extinction[10]	Pleochroism[8]	Extinction[10]	Pleochroism[8]	Extinction[10]	2			2			
	F or N	Texture[5]	Bire[9]	Elongation[11]	Bire[9]	Elongation[11]	Bire[9]	Elongation[11]	3			3			

Nonfibrous Types[15]	Percentage

☐ Asbestos-Containing ☐ Non-Asbestos-Containing

Date of Analysis:__________ Analyst:__________

PLM Point Count Worksheet

BATTA

POINT COUNT WORKSHEET

Client Name: ____________________ Date Analyzed: ___/___/_____ BL Project #: **BL**______________

Sample:

PREP #	Asbestos	Cellulose	Glass	Other Fib	Non Fib	Empty
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
Total						

Results: ______________________

Sample:

PREP #	Asbestos	Cellulose	Glass	Other Fib	Non Fib	Empty
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
Total						

Results: ______________________

Sample:

PREP #	Asbestos	Cellulose	Glass	Other Fib	Non Fib	Empty
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
Total						

Results: ______________________

Sample:

PREP #	Asbestos	Cellulose	Glass	Other Fib	Non Fib	Empty
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
Total						

Results: ______________________

CALCULATIONS: P = (AP * 100)/TP

Analyst ______________________

P = PERCENTAGE OF ,TP = TOTAL NUMBER OF NON-EMPTY POINTS

AP = NUMBER OF ASBIIF AP < 4 AFTER 400 NON-EMPTY POINTS; REPORT < 1% ASBESTOS

NOTE: IF AP = 0 AFTER 400 NON-EMPTY POINTS, REPORT NO ASBESTOS. REPORT TRACE IF ASBESTOS SEEN, BUT NOT ON A POINT.

PLM Certificate

Dedicated to a Cleaner Environment Since 1982

BATTA

BATTA LABORATORIES, INC.

A Certified MBE Company
Delaware Industrial Park, 6 Garfield Way
Newark, DE 19713-5817
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Web : http://www.battaenv.com E-mail : battaenv@battaenv.com

E.P.A. LAB ID #DE004
A.I.H.A. /NLLAP #100448
NVLAP #101032

CERTIFICATE OF PLM ANALYSIS

Page 1 of 1

Test Method: BATTA SOP in conjuction with EPA 600/R-93/116 Report Date: 3/3/04

Sampling Data

BLI Project # :
Project Name:

Date Sampled: 02/25/04
Sampled By: J KUNTZ
Date Analyzed: 02/27/04

Sample ID		Client-Supplied Data			Analytical Data		Reported Results		
Lab Sample#	Client Sample #	Sample Location	Material Type	Friable?	Texture	Color	Non-Asbestiform Components	Asbestiform Components	
450490	1A	ROOF- 11 MIDDLE, FIELD	Roofing material	No	Firm	BLACK	30 % Cellulose	N/A	Non-Asbestos Containing
			SEE NOTE 1				70 % Non-fibrous		
450491	1B	ROOF 11 EAST END, FIELD	Roofing material	No	Firm	BLACK	30 % Cellulose	N/A	Non-Asbestos Containing
			SEE NOTE 1				70 % Non-fibrous		
450492	2A	ROOF-11, FLASHING	Roofing material	No	Firm	BLACK	40 % Cellulose	N/A	Non-Asbestos Containing
			SEE NOTE 1				60 % Non-fibrous		
450493	2B	ROOF-11, FLASHING	Roofing material	No	Firm	BLACK	40 % Cellulose	N/A	Non-Asbestos Containing
			SEE NOTE 1				60 % Non-fibrous		
450494	2C	ROOF-11, FLASHING	Roofing material	No	Firm	BLACK	40 % Cellulose	N/A	Non-Asbestos Containing
			SEE NOTE 1				60 % Non-fibrous		

NOTE 1 *Organically-bound, nonfriable material may interfere with the accurate quantification of asbestos. In these cases, the EPA recommends more definitive analysis by a matrix-reduction method (i.e. Chatfield SOP-1988-02, Rev.1).*

NOTE 2 *Due to limitations of the EPA PLM method, floor tiles may yield false negative (<1%) results by this method. Due to this, the EPA recommends more definitive analysis using analytical electron microscopy.*

ANALYST: Asghar Keyvanfar REVIEWED BY: _____

* This report does not constitute endorsement by NVLAP and/or any other U.S. goverment agencies.
* The test data pertain only to the items tested. No assumptions or conclusions should be made to materials or samples not analyzed. Furthermore, Batta Laboratories, Inc. assumes no responsibility for the accuracy of results influenced by the use of improper collection techniques or equipment.
* Due to the general inhomogeneity of asbestos containing materials (ACM), EPA and OSHA have recommended submission of at least three samples of each type of materials for PLM analysis. Submission of fewer samples may compromise the accuracy of ACM determination.
* Floor tile samples may yield "false negative" (<1%) asbestos results. There is no EPA approved PLM-analytical method for floor tiles. Definitive results can be obtained by TEM analysis.

Individual Analyst Daily Reference Sample Analysis

Asghar Keyvanfar Daily PLM Reference Sample Analysis -- December-04

DATE	Analyst Initials	Reference Sample #	Analyst Result						Analyst Result						Entered in Computer
			Ch	Am	Cr	Ant	Tr	Act	Ch	Am	Cr	Ant	Tr	Act	
12/1	ak														
12/2	ak														
12/3	ak														
12/4	ak														
12/5	ak														
12/6	ak														
12/7	ak														
12/8	ak														
12/9	ak														
12/10	ak														
12/11	ak														
12/12	ak														
12/13	ak														
12/14	ak														
12/15	ak														
12/16	ak														
12/17	ak														
12/18	ak														
12/19	ak														
12/20	ak														
12/21	ak														
12/22	ak														
12/23	ak														
12/24	ak														
12/25	ak														
12/26	ak														
12/27	ak														
12/28	ak														
12/29	ak														
12/30	ak														
12/31	ak														

Individual Analyst Duplicate Analysis

COMPLETE THIS SECTION EVERY DAY DUPES ARE DONE !!!

# of samples analyzed:_____ **# of samples submitted for reanalysis:**_____

Note to the analyst: When sample has more than one asbestos type, enter each asbestos type on a separate line.

Analyst: Robert Shumate *PLM QC Log* **Date:**_____

Original Analysis

			Original Results	
Date of Analysis	**Lab Sample #**	**Client Sample #**	% **Asbestos**	**Asbestos Type (Chys, Amos, Croc, Trem, Actin, Anth)**

Reanalysis

				Reanalysis Results	
Date Reanalyzed	**Second Analyst**	**Lab Sample #**	**Client Sample #**	% **Asbestos**	**Asbestos Type (Chys, Amos, Croc, Trem, Actin, Anth)**

Identification Chart For Common Asbestos and Non-asbestos Fibers

FIBERS	SHAPE	SIZE (µm)	COLOR (Pleochroism)	REFRACTIVE INDICES
• Chrysotile #1	meandering path; easily deformed curly fibers	fibrils < 1; bundles any size	NA	n⊥ 1.54 n∥ 1.55 +
• Lizardite #13	rounded aggregates of tiny thin flakes or plates	10 - 100	NA	sl > chrysotile
• Antigorite #13	usually short fiber aggregates; rod-like	0.1 - 1	NA	sl > lizardite
• Anthophyllite #6	straight fibers bend easily; spring action like amosite	like chrysotile	NA	ca. 1.60 n⊥ ca. 1.63 n∥
• Tremolite #4	may be like chrysotile or amosite	" "	NA	ca. 1.61 n⊥ ca. 1.63 n∥
• Actinolite #5	may be like chrysotile but usually like amosite	" "	NA	ca. 1.62 n⊥ ca. 1.64 n∥
• Amosite #2	straight fibers bend easily with spring action	" "	NA	ca. 1.67 n⊥ ca. 1.70 n∥
• heated #8	more brittle on heating therefore shorter fibers	" "	orange ⊥ red brown ∥	1.7 – 1.8 n⊥ 1.75 - 1.95 n∥
• Crocidolite #3	like amosite	" "	grey - blue ⊥; blue ∥	ca. 1.695 n∥ ca. 1.705 n⊥
• Brucite (Nemalite) #9	like tremolite	" "	NA	ca. 1.59 n⊥ ca. 1.58 n∥
• Wollastonite #7	like tremolite	usually non-fibrous rods	NA	ca. 1.62 n⊥ ca. 1.64 n∥
Mineral wool #52	exotic shapes, tear drops, slugs, round crossection	0.1 - 5	NA	1.52 - 1.70
Wood Fibers #26	flat ribbons often pitted + cotton-like fibers	0.1 - 2	NA	ca. 1.53 n⊥ ca. 1.57 n∥
• Talc #11	flat flakes and thin ribbons; some fibers	5 - 50	NA	ca. 1.54 n⊥ ca. 1.59 n∥
Linen (Flax) #31	smooth except for nodes	1 -5	NA	ca. 1.53 n⊥ ca. 1.59 n∥
Hair (Wool) #34	curly, scales, usually a medulla	1 - 5	NA	ca. 1.545 n⊥ ca. 1.555 n∥
Straw #25	long narrow, tapered fibers; serrated + baggy cells	5 - 500	lignin yellow	ca. 1.53 n; ca. 1.57 n∥
Cotton #30	twisted fibers	0.1 - 5	NA	ca. 1.53 n⊥ ca. 1.57 n∥
Kevlar #48	long, circular-crossection filament, may be shredded, crossmarks	uniform	lt. yellow ⊥; yellow ∥	1.62 n⊥ – 2.35 n∥
Dacron #49	rounded crossection; long	"	NA	1.53 n⊥ -- 1.70 n∥
Nylon #47	circular crossection; long	"	NA	1.52 n⊥ -- 1.575 n∥
Viscose rayon #43	longitudinal striations; long	"	NA	1.52 n⊥ - 1.55 n∥
Acetate rayon #44	longitudinal striations; long	"	NA	ca. 1.48 - ca. 1.485 n∥
• Vermiculite #10	flat sheets; mica-like	100 - 5000	yellow	ca. 1.55 n⊥ - 1.57 n∥
Muscovite mica #61	"	10 - 1000	NA	ca. 1.565 n⊥ – ca. 1.60 n∥
Biotite mica #62	"	20 - 2000	yellow to brown	ca. 1.58 n⊥ - ca. 1.62 n∥
Quartz #56	"glass" chips and flakes	10 - 200	NA	1.544 (ω) - 1.553 (ε)
Calcite #58	excellent rhombohedral cleavage; twin bands	10 - 200	NA	1.486 (ε) - 1.525 (ε') - 1.658 (ω)
Diatoms #1	one-celled plant skeletons, honeycombs	10 - 200	NA	ca. 1.43
• Perlite #12	foamed glass	10 - 500	NA	ca. 1.50
• Ceramic fiber #14	like mineral wool but smaller diameter	0.1 - 200	NA	1.54 to 1.70
Polyethylene	filaments or shredded, like chrysotile	variable	NA	ca. 1.52 n⊥ ca. 1.57 n∥

• White plastic box; others in Particle Reference Set

Optical Properties of Asbestos and Asbestos-like Substances (McCrone, 1987)

TABLE XIV. Optical Properties of Asbestos and Other Asbestiform Substances

Mineral Fibers	Refractive Indices α	γ	Biref. γ-α	Color	Extcn.	Sign Elongn.	n_D(liq)	C.S. Dispersion Staining ∥lg.	⊥lg.
Chrysotile	1.532 - 1.549	1.545 - 1.556	0.015	white		+	1.550	red-purple	blue
Antigorite[1]	1.558 - 1.567	1.562 - 1.574	0.01	white		+	1.550	gol. yel.	red-purple
Lizardite	1.545	1.558	0.013	white	undulose	+(edge)	1.550	red-purple	p. blue
Amosite {C.	1.633 - 1.664	1.654 - 1.687	0.02	white		+	1.680	blue	p. blue
Amosite {G.	1.664 - 1.686	1.687 - 1.729	0.03	p. green		+	1.680	gold	blue
Crocidolite	1.654 - 1.698	1.666 - 1.712	0.01	blue	∥& obl.	-	1.680	p.yellow	yellow
Tremolite	1.603 - 1.620	1.627 - 1.642	0.02	white	∥& obl.	+	1.605	yellow	blue[2]
Actinolite	1.620 - 1.667	1.642 - 1.686	0.02	white	∥& obl.	+	1.605	v. p. yel.	p.yellow
Ferroactinolite	1.667 - 1.683	1.686 - 1.702	0.02	p. green	∥& obl.	+	1.680	gol. yel.	p.blue
Anthophyllite	1.606 - 1.648	1.626 - 1.670	0.02	white		+	1.605	yellow	red-purple
Byssolite	fibrous tremolite/actinolite			white	∥& obl.	+	1.605		
Richterite	1.590	1.611	0.02	white	∥& obl.	+	1.605	red-purple	p. blue
Brucite[3]	1.560 - 1.590(ω)	1.580 - 1.600(ε)	0.025	white		-,+	1.550	yellow	p.yellow
Talc	1.539 - 1.550	1.589 - 1.600	0.05	white		+	1.550	p.yellow	blue-gr.
Palygorskite[4]	ca. 1.52	ca. 1.53	ca. 0.01	white		+,-	1.550	v.p.blue	v.p.blue
Epsomite[5]	1.433	1.461	0.03	white		+,-	1.550	white	white
Prieskite[6]	1.665	1.679	0.014	p.green	∥& obl.	+,-	1.680	blue	p. blue
Bastite[7]	1.676	1.689	0.013	brown		+	1.680	p.yellow	blue
Amosite(heated)[8]	ca. 1.74	ca. 1.94	0.2	red		+	1.680	white	white
Wollastonite[9]	1.612	1.632	0.02	white	tricl.	+,-	1.605	p.yellow	p.yel.-purple
Szaibelyite[10]	1.581	1.666	0.085	white		-	1.605	v.p.blue	p.yellow

1. fibrous antigorite = picrolite
2. ⊥lg. blue (obl. extcn.) magenta (∥ extcn.)
3. $Mg(OH)_2$, (when fibrous, called nemalite)
4. a clay, $Mg_2Si_3O_8 \cdot 4H_2O$ in attapulgite group
5. $MgSO_4 \cdot 7H_2O$
6. a ferroactinolite
7. a ferromagnesium silicate (orthopyroxenes altered to serpentines) pseudomorphs show bronze-like lustre
8. heated amosite
9. $CaSiO_3$
10. $(Mg,Mn)(BO_2)(OH)$

Abbreviations

∥ = parallel, ⊥ = perpendicular
gol. = golden
yel. = yellow
gr. = green
C.S. = central stop
obl. = oblique
p. = pale
ca. = about
v.p. = very pale
bl. = blue
C. = cummingtonite
G. = grunerite

Methodology of Bulk Asbestos Sample Analysis

BULK SAMPLE ANALYSIS

MOUNT SAMPLE IN LIQUID 1.550HD OR 1.680HD
VIEW BETWEEN CROSSED (OR SLIGHTLY UNCROSSED) POLARS

Isotropic

Fibrous
Mineral wool
Glass fiber
Ceramic

Nonfibrous (Appendix 26)
Groundglass
Diatoms
Perlite
Pumice

Anisotropic

Fibrous

Curly
1.550 HD
Chrysotile
Paper
Cotton
Polyethylene
Polypropylene
Synthetics

Straight
1.680 HD
Amosite
Crocidolite

1.605 HD
Anthophyllite
Tremolite
Actinolite
Brucite
Wollastonite

Nonfibrous (Appendix 26)

1.550 HD
Chrysotile
|| Magenta
⊥ Blue
(+) Sign Elong.
|| Ext

1.680 HD
Amosite
|| Yellow-golden
⊥ Blue
(+) Sign Elong.
0-5° Ext

1.550 HD
Talc Fibers
|| Yellow
⊥ Blue/yellow ⊥
(+) Sign of Elong.
Oblique or Ext

Paper
|| Pale yellow
Pale blue
(+) Sign Elong.
Ext absent
(usually) to
complete

Crocidolite
Blue
⊥ Golden-yellow
(–) Sign Elong.
0-3° Ext
Pleochroic: blue (||), gray-blue (⊥)

Polypropylene
Polyethylene
Polyolefin
} Δ melt

Synthetics (Appendix 18)

1.605 HD

(+)
Anthophyllite
Tremolite
Actinolite

(–)
Brucite

(+,–)
Wollastonite

|| Ext Only
Anthophyllite
|| Pale yellow
⊥ Golden-yellow to
Blue-magenta

Oblique Ext and || Ext
Tremolite (Low Fe)
|| Yellow
⊥ Blue
} oblique view

|| Yellow
⊥ Magenta
} parallel view

Actinolite
|| Yellow (pale)
⊥ Yellow

Remount in 1.63 HD
If all $n_{||}$ > 1.63 = actinolite
If all $n_{||}$ < 1.63 = tremolite
If $n_{||}$'s > and < 1.63 = both

Methodology of Bulk Insulation Sample Analysis

Identification of Asbestos in Insulation

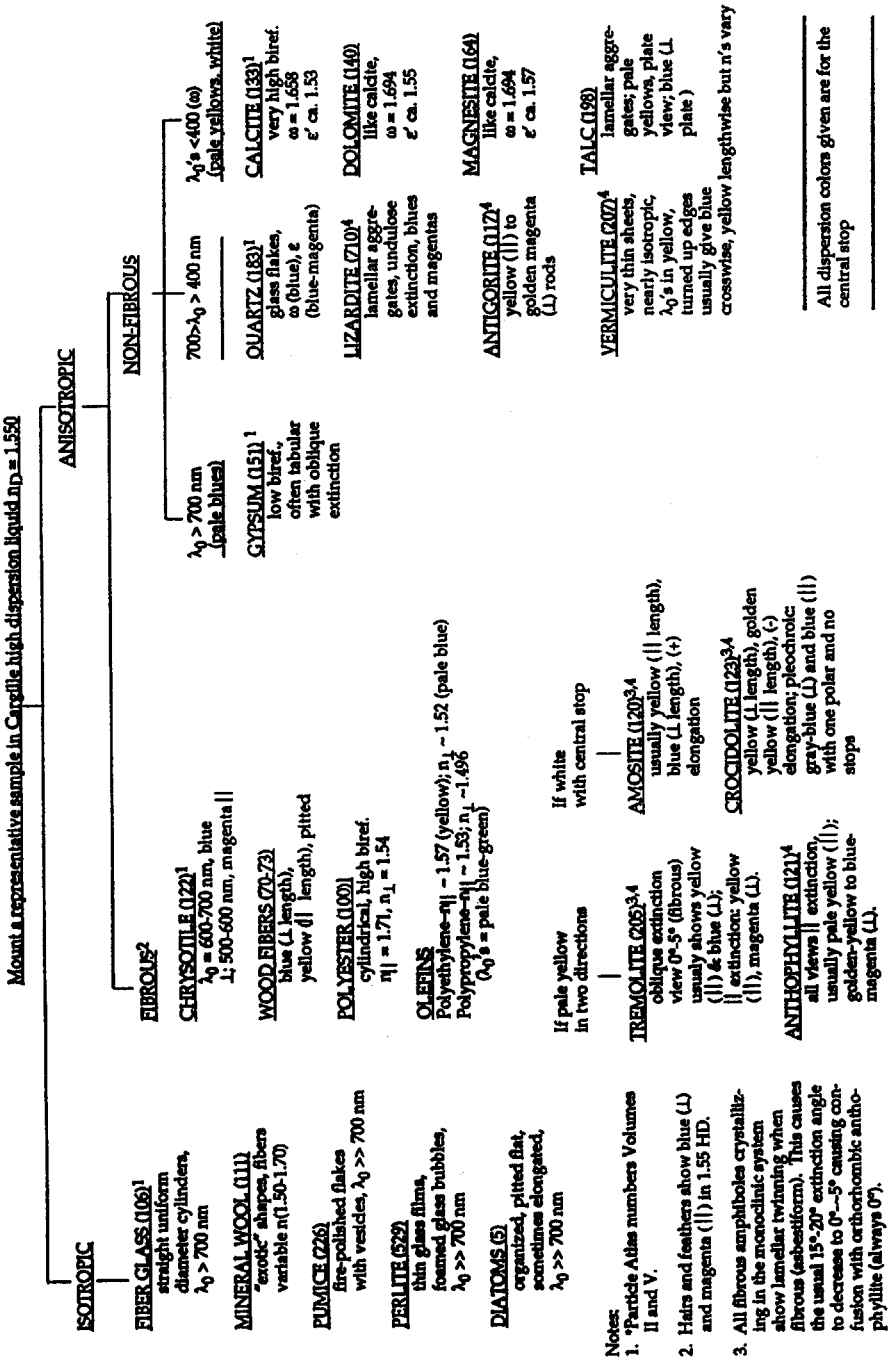

Table 7. Procedure for analysis of asbestos materials

Certificate For Point Count Analysis

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E.P.A. LAB ID# DE004
A.I.H.A./NLLAP #100448
NVLAP #101032

CERTIFICATE OF PLM ANALYSIS
(Quantitation using EPA point-count

Test Method: BATTA SOP in conjunction with EPA 600/R-93/116

Page 1 of 1
Report Date: 9/12/2000

Sampling Data
BLI Project #: N/A
Project Name: N/A

Date Sampled: 9/8/2000
Sampled By: D Brown
Date Analyzed: 9/8/2000

	Client-Supplied Data				Analytical Data			Results	
Lab Sample #	Client Sample #	Sample Location	Material Type	Friable?	Texture	Visual	Color	Asbestiform Components	Other Components
356999	1a	I/S room 123	pipe ins	y	fibrous	Homo	grey	0.25 % Chrysotile	40% Cellulose 30% Fiberglass 29.75% Non-fibrous Other
357000	1b	I/S room 123	pipe ins	y	fibrous	Homo	grey	0.50 % Crocidolite	20% Cellulose 10% Fiberglass 69.50% Non-fibrous Other
357001	1c	I/S room 123	pipe ins	y	fibrous	Homo	grey	0.75 % Amosite	60% Cellulose 30% Fiberglass 9.25% Non-fibrous Other
357002	1d	I/S room 123	pipe ins	y	fibrous	Homo	grey	0.00 None Detected	30% Cellulose 15% Fiberglass 55.00% Non-fibrous Other
357003	1e	I/S room 123	pipe ins	y	fibrous	Homo	grey	0.00 None Detected	10% Cellulose 70% Fiberglass 20.00% Non-fibrous Other

ANALYST: Asghar Keywanfar **REVIEWED BY:** _____

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* Due to the general inhomogeneity of asbestos-containing materials (ACM), EPA and OSHA have recommended submission of at least three samples of each type of materials for PLM analysis. Submission of fewer samples may compromise the accuracy of ACM determination.
* Floor tiles may yield "false nagative" (<1%) asbestos results. There is no EPA approved PLM-analytical method for floor tiles. Definitive results can be obtained by TEM analysis.

List of PLM Reference Samples

PLM Reference Samples

Standard Daily Reference Material

Lab ID#	NVLAP ID#	Reference Value	
		Type 1	Type 2
1	M22001-4	4% Amosite	5% Chrysotile
2	M22001-2	2% Actinolite	
3	M22001-1	4% Crocidolite	Trace Chrysotile
4	M12003-2	9.9% Amosite	1.1% Chrysotile
5	M22003-1	0.5% Crocidolite	
6	M12007-1	10% Amosite	2% Crocidolite
7	M12006-1	8% Amosite	4% Chrysotile
8	M12006-2	8% Amosite	
9	M12007-4	2.5% Amosite	0.5% Chrysotile
10	M12007-3	2.5% Tremolite	
11	M1992-1	83% Chrysotile	
12	M21998-4	0.5% Anthophyllite	
13	M21996-2	13% Chrysotile	
14	M11993-1	35% Chrysotile	

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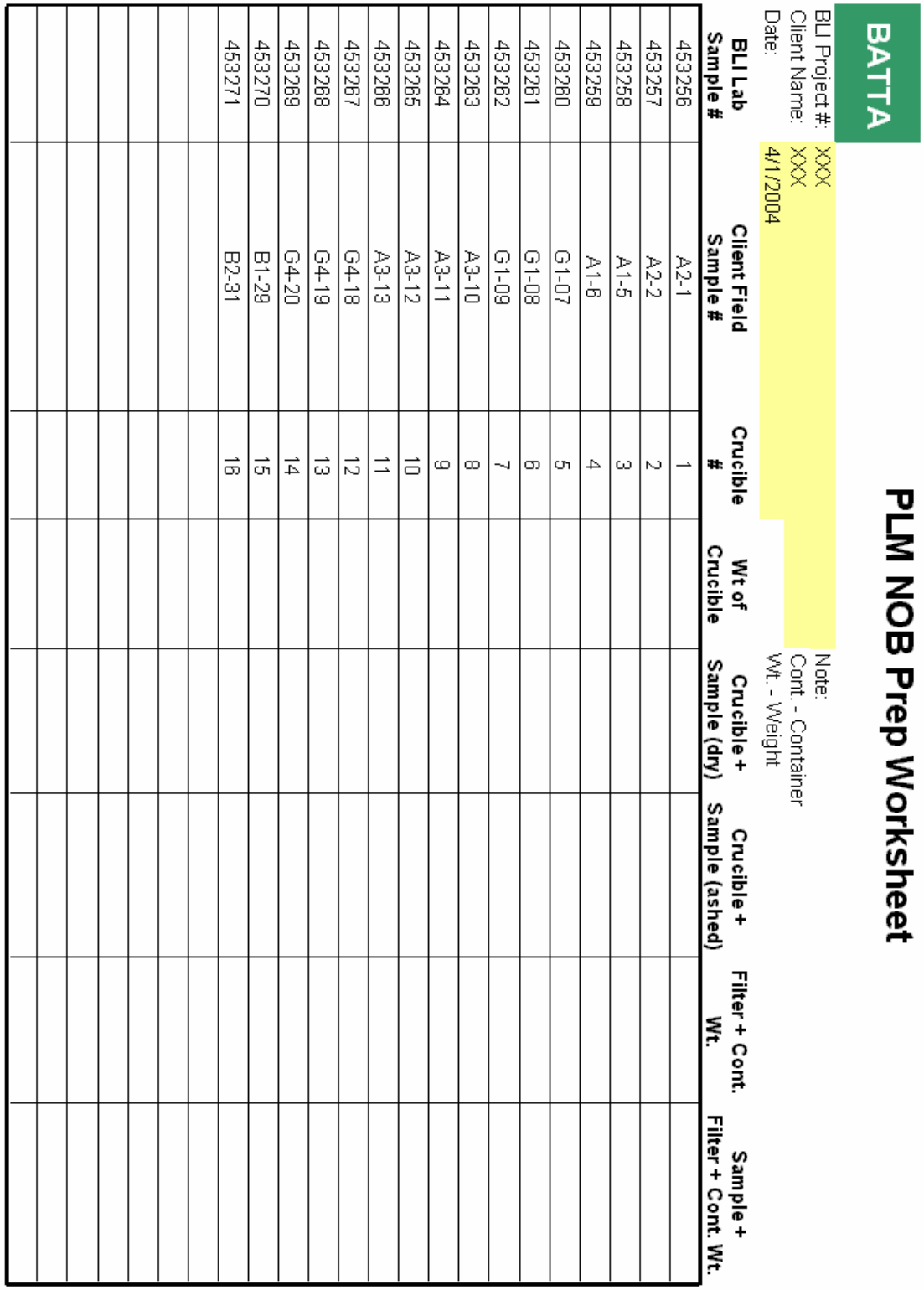
Sample Prep Sheet for PLM NOB Bulks



BATTA

BLI Project #: XXX
Client Name: XXX
Date: 4/1/2004

PLM NOB Prep Worksheet

Note:
Cont. - Container
Wt. - Weight

BLI Lab Sample #	Client Field Sample #	Crucible #	Wt of Crucible	Crucible + Sample (dry)	Crucible + Sample (ashed)	Filter + Cont. Wt.	Sample + Filter + Cont. Wt.
453256	A2-1	1					
453257	A2-2	2					
453258	A1-5	3					
453259	A1-6	4					
453260	G1-07	5					
453261	G1-08	6					
453262	G1-09	7					
453263	A3-10	8					
453264	A3-11	9					
453265	A3-12	10					
453266	A3-13	11					
453267	G4-18	12					
453268	G4-19	13					
453269	G4-20	14					
453270	B1-29	15					
453271	B2-31	16					

Certificate of PLM NOB Analysis

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A.I.H.A./NLLAP #100448
NVLAP #101032

CERTIFICATE OF PLM ANALYSIS

Page 1 of 6

Test Method: NYDOH ELAP 198.1 (Modified) - PLM analysis by matrix reduction

Report Date: #######

Sampling Data

BLI Project #: N/A
Project Name: N/A
Project Location: N/A

Date Sampled: 3/8/2004
Sampled By: Client
Analyzed By:: 3/16/2004

Analytical Parameters

Effective Area (mm²) 962 Media: MCE Pore Size (um): 0.80

Sample ID		Client-Supplied Data				Results			
							Non-Asbestos Content		Asbestos Content
Lab Sample #	Client Sample #	Sample Location	Material Type	Sample Color	Friable? (y or n)	Inorganic Residue, %	Non-fibrous (Percent, Type)	Fibrous (Percent, Type)	Asbestos (Percent, Type)
451346	C28-02	Not Given	Floor Tile	Grey	n/a	9.58%	100% Other	N/A	None Detected
451347	C28-03	Not Given	Mastic	Black	n/a	14.99%	100% Other	N/A	None Detected
451348	C28-04	Not Given	Floor Tile	Grey	n/a	7.41%	100% Other	N/A	None Detected
451349	C28-05	Not Given	Mastic	Tan	n/a	12.50%	100% Other	N/A	None Detected
451350	C28-14	Not Given	Bulk	Grey	n/a	10.31%	100% Other	N/A	None Detected
451351	C28-17	Not Given	Bulk	Black	n/a	24.45%	100% Other	N/A	None Detected
451352	C28-18	Not Given	Bulk	White	n/a	43.85%	100% Other	N/A	None Detected
451353	C28-19	Not Given	Bulk	Brown	n/a	10.75%	100% Other	N/A	None Detected

ANALYST: Asghar Keyvanfar **REVIEWED BY:** _____

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Form of Request for SOP modification

BATTA

Request for Modification to Analytical Methodology or Laboratory Standard Operating Procedures

Instructions to Requester: *Submit to contact(s) at bottom of form for review and approval. File the approved copy with QA-QC Officer for submission and incorporation into appropriate BATTA SOP or QA Manual.*

Name and/or Method Number Affected: _____

Requester:_____Job Title: _____
Microscopy or Chemistry Lab:_____Date of Request:_____

Description of Modification:
_____
_____

Reason for Modification:
_____
_____

Potential Implications of this Modification:
_____
_____

Laboratory Applicability (circle one): All Individual Sections) _____
_____

Duration of Modification (circle one):
Temporary Date(s): _____
Laboratory Sample Numbers & Clients Affected: _____
_____

Permanent (Complete Proposed Modification Section) Effective Date: _____

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):
_____
_____
_____
_____

Technical Review: _____Date: _____
(Laboratory Manager or designate)

Laboratory Review and Approval: _____Date:_____
(QA-QC Officer or designate)

Approved By:_____Date: _____

Title: Laboratory Director, BATTA Laboratories, Inc.

APPENDIX C

IN HOUSE TRAINING PROGRAM

Table of Contents

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IN-HOUSE TRAINING PROGRAM

1. The trainee will be tutored by a competent PLM microscopist in order to be qualified to analyze samples.

2. The trainee shall become familiar with the written regulations: EPA 600, OSHA 29 CFR 1910.1001, OSHA 29 CRF 1926.1101.

3. The trainee must become knowledgeable in the optical properties of asbestos and non-asbestos fibers. This is best achieved by attending the McCrone PLM Asbestos Identification Course of equivalent. (See Training Course Outline). We currently have Dr. Su Chun Su on staff as a Technical Consultant. He is used as a primary trainer for PLM and TEM analysis.

4. When viewed on PLM slides, each field of view should be described by both people.

5. A series of 20 standard reference samples (either NIST standards or previous rounds of NVLAP proficiency samples) is analyzed by each trainee. The trainee's conclusions of type, percent and optical properties of asbestos must be in 100% agreement with the published results before the trainee may analyze samples for the laboratory. At this point, the trainee is considered proficient. After this, NVLAP samples are analyzed and monitored from round to round and the error should be less than 5%. Statistics of the trainee will be documented as shown on the **Pages C-5** and **C-6** of this appendix.

6. After trainees have met the above requirements and statistics, they may start analyzing samples for the lab and qualified as PLM analysts and will participate required routine QC/QA programs as documented in this SOP.

7. Competency of the trainee is checked through the use of the Quality Assurance Program.

IN-HOUSE ASBESTOS IDENTIFICATION TRAINING OUTLINE

I. Proper Use of Microscopes
   - A. Stereoscope
   - B. Polarized Light Microscope
   - C. Kohler Illumination

II. Properties of Asbestos
   - A. Serpentines
     1. Asbestos Types
     2. Look-Alikes
   - B. Amphiboles
     1. Asbestos Types
     2. Look-Alikes
   - C. Crystallography
     1. Anisotropic
     2. Isotropic
   - D. Optical Properties w/Plain Polarized Light
     1. Color/Pleochroism
     2. Morphology
     3. Refractive Index
        - a. Becke Line
        - b. Dispersion Staining Color
   - E. Optical Properties w/Crossed Polarized Light
     1. Interference Color
     2. Birefringence
     3. Extinction Characteristics
     4. Sign of Elongation
   - F. Conoscopic Polarized Light
     1. Interference Figure
     2. Optic Sign

III. Immersion Method
   - A. Temperature Coefficient
   - B. Dispersion Coefficient

IV. Becke Line Method
   - A. Using Optical Glass
   - B. Practical Use Versus Dispersion Staining

V. Dispersion Staining Color Method
   - A. Central Stop
   - B. Annular Stop
   - C. Measuring Wavelengths, XO
   - D. Calibrating Refractive Fluids

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IN-HOUSE ASBESTOS IDENTIFICATIO TRAINING OUTLINE
(continued)

VI. Uncommon Asbestos Types
- A. Actinolite
- B. Anthophyllite
- C. Tremolite

VII. Non-Asbestos Particles
- A. Fibrous Glasses
- B. Refractory Ceramics
- C. Synthetics
- D. Cellulose
- E. Wollastonite

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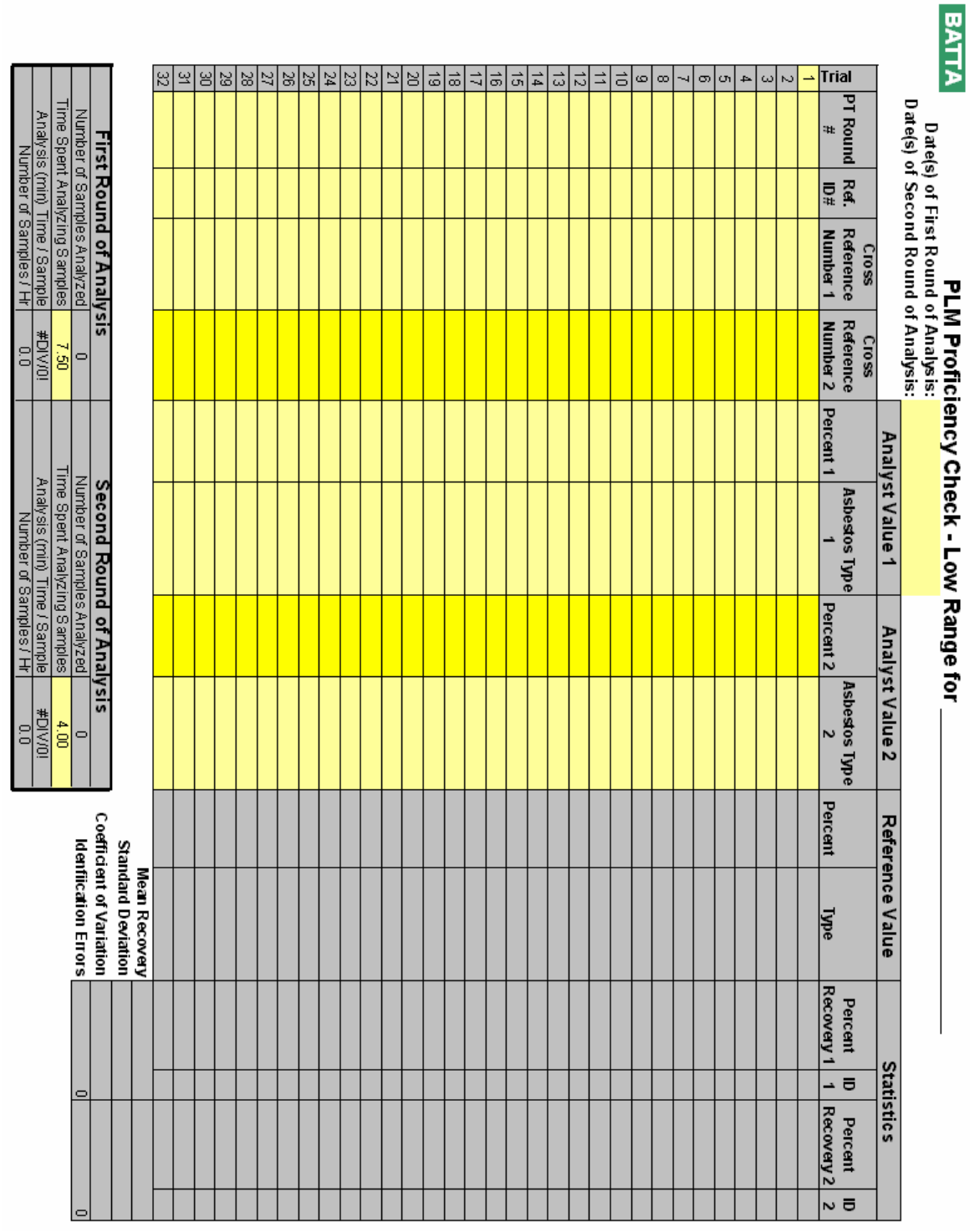
PLM Training Statistics



BATTA

PLM Proficiency Check - Low Range for _____

Date(s) of First Round of Analysis:
Date(s) of Second Round of Analysis:

Trial	PT Round #	Ref. ID#	Cross Reference Number 1	Cross Reference Number 2	Analyst Value 1 Percent 1	Analyst Value 1 Asbestos Type 1	Analyst Value 2 Percent 2	Analyst Value 2 Asbestos Type 2	Reference Value Percent	Reference Value Type	Statistics Percent Recovery 1	Statistics ID 1	Statistics Percent Recovery 2	Statistics ID 2
1														
2														
3														
4														
5														
6														
7														
8														
9														
10														
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21														
22														
23														
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25														
26														
27														
28														
29														
30														
31														
32														
									Mean Recovery					
									Standard Deviation					
									Coefficient of Variation					
									Identification Errors		0		0	

First Round of Analysis		Second Round of Analysis	
Number of Samples Analyzed	0	Number of Samples Analyzed	0
Time Spent Analyzing Samples	7.50	Time Spent Analyzing Samples	4.00
Analysis (min) Time / Sample	#DIV/0!	Analysis (min) Time / Sample	#DIV/0!
Number of Samples / Hr	0.0	Number of Samples / Hr	0.0

PLM Training Statistics (continued)

Page 1of 4

BATTA

Analyst Accuracy

NVLAP PT Round

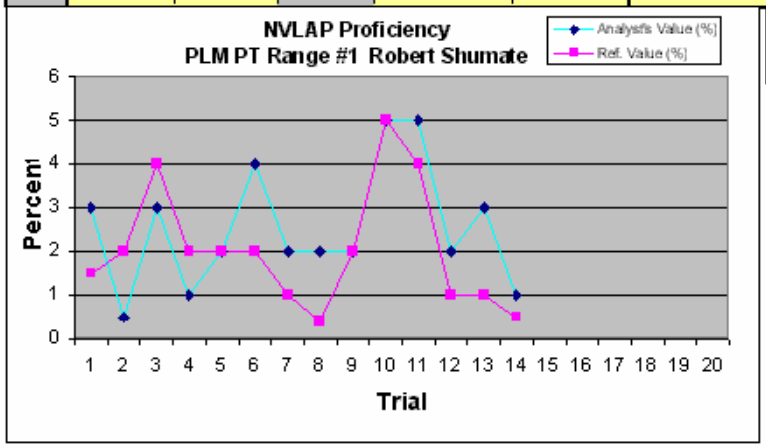
Range #1	(<1 - <5%)
Range #2	(5 - 20%)
Range #3	(21 - 50%)
Range #4	(51 - 100%)

RGS Proficiency Misidentifications		
# of Mis-IDs	# of Samples Analyzed	% of Total
0	25	0.00

RGS - PT Range #1 (<1 - <5%) Analyst Results | *Reference Results*

Trial #	PT Round #	Ref. ID#	Analyst	Asbestos Type	Analyst's Value (%)	Asbestos Type	Ref. Value (%)	Percent Recovery	ID
1	M21999	1-1	RGS	Amosite	3	Amosite	1.5	200.00	
2	M21999	1-2	RGS	Chrysotile	0.5	Chrysotile	2.0	25.00	
3	M21999	2	RGS	Chrysotile	3	Chrysotile	4.0	75.00	
4	M12000	4-3	RGS	Crocidolite	1	Crocidolite	2.0	50.00	
5	M22000	1	RGS	Chrysotile	2	Chrysotile	2.0	100.00	
6	M22000	2-1	RGS	Amosite	4	Amosite	2.0	200.00	
7	M22000	2-2	RGS	Chrysotile	2	Chrysotile	1.0	200.00	
8	M12001	2	RGS	Chrysotile	2	Chrysotile	0.4	500.00	
9	M22001	2	RGS	Actinolite	2	Actinolite	2.0	100.00	
10	M22001	3	RGS	Crocidolite	5	Crocidolite	5.0	100.00	
11	M22001	4-1	RGS	Amosite	5	Amosite	4.0	125.00	
12	M12002	1-1	RGS	Amosite	2	Amosite	1.0	200.00	
13	M12002	1-2	RGS	Chrysotile	3	Chrysotile	1.0	300.00	
14	M22002	3	RGS	Actinolite	1	Actinolite	0.5	200.00	
15			RGS						
16			RGS						
17			RGS						
18			RGS						
19			RGS						
20			RGS						

Mean (Asb %) =	2.54
Std Dev (Asb %) =	1.39
Mean % Recovery =	169.64



APPENDIX D

EQUIPMENT AND LAB LAYOUT

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LIST OF EQUIPMENT

Quantity	Item
3	Hepa-Hood – McCrone HEPA - Nuaire NU-119300 - AirFiltronics
4	Microscope – (2) Fisher Stereomaster 2 -Olympus BHT-001 (McCrone, Ser. #205314, 3/4/86) -Nikon Labophot POL (Optical Apparatus, Ser. #954996, 4/91)
3	Mortar and Pestles
1	Muffle Furnace
1	Low Temperature Oven
	Various Tools (Tweezers, forceps, blades, needles, etc.)
	Refractive Index Oils (Cargille Series B and E)
	Solvents (HCl, THF, etc.)
	Petri Dishes
	Filters (from 0.22 µm to 5 µm)
1	Filtration Device
	Graduated Cylinders
	Funnels
	Crucibles
	Slides and Cover Glasses
	Sample Waste Container

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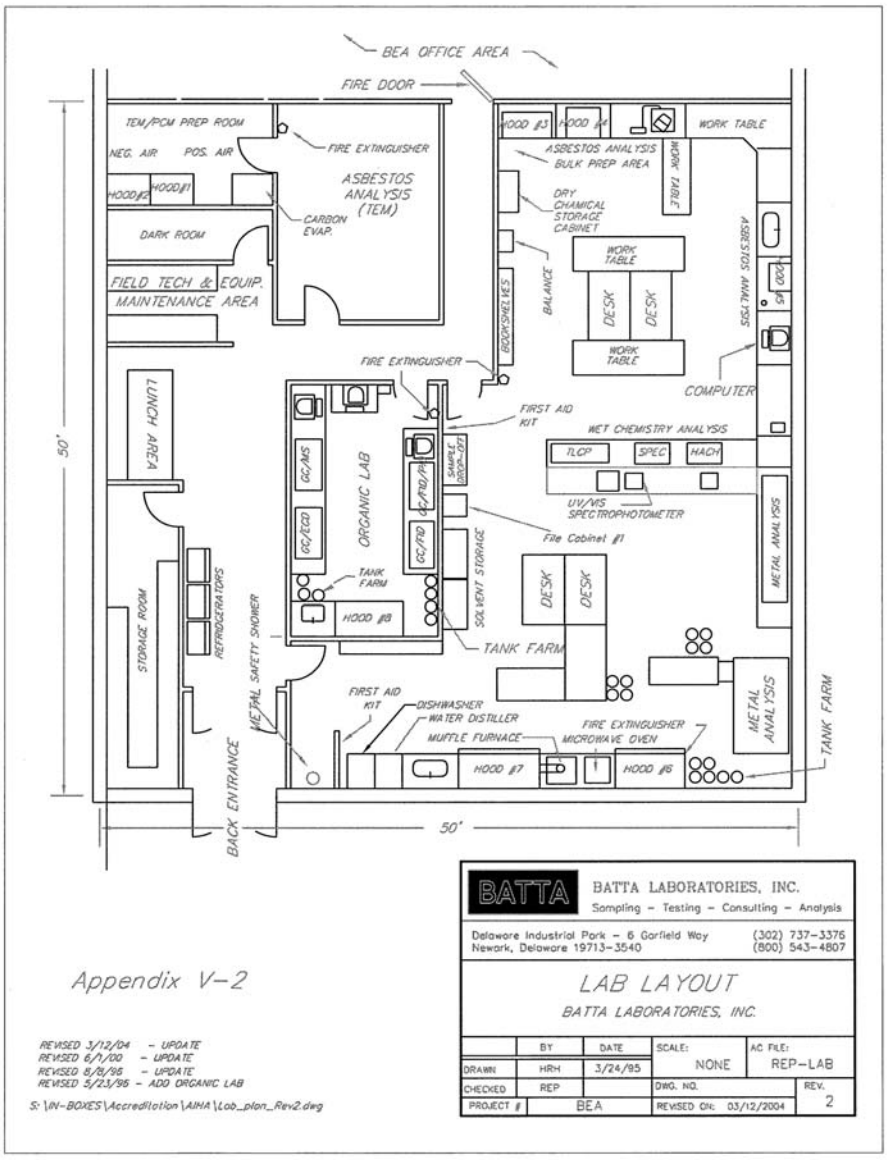
LAB LAYOUT


BEA OFFICE AREA
FIRE DOOR
TEM/PCM PREP ROOM
NEG. AIR
POS. AIR
FIRE EXTINGUISHER
ASBESTOS ANALYSIS (TEM)
HOOD #2
HOOD #1
CARBON EVAP.
DARK ROOM
FIELD TECH & EQUIP. MAINTENANCE AREA
HOOD #3
HOOD #4
WORK TABLE
ASBESTOS ANALYSIS BULK PREP AREA
WORK TABLE
DRY CHEMICAL STORAGE CABINET
BOOKSHELVES
BALANCE
WORK TABLE
DESK
DESK
WORK TABLE
ASBESTOS ANALYSIS
FILES
COMPUTER
FIRE EXTINGUISHER
FIRST AID KIT
WET CHEMISTRY ANALYSIS
TLCP
SPEC
HACH
UV/VIS SPECTROPHOTOMETER
File Cabinet #1
METAL ANALYSIS
LUNCH AREA
GC/MS
GC/ECD
ORGANIC LAB
GC/PID
GC/PID
SAMPLE PREP
SOLVENT STORAGE
TANK FARM
HOOD #8
DESK
DESK
TANK FARM
STORAGE ROOM
REFRIGERATORS
METAL SAFETY SHOWER
FIRST AID KIT
DISHWASHER
WATER DISTILLER
MUFFLE FURNACE
FIRE EXTINGUISHER
MICROWAVE OVEN
HOOD #7
HOOD #6
METAL ANALYSIS
TANK FARM
BACK ENTRANCE
50'
50'
BATTA
BATTA LABORATORIES, INC.
Sampling – Testing – Consulting – Analysis
Delaware Industrial Park – 6 Garfield Way
Newark, Delaware 19713-3540
(302) 737-3376
(800) 543-4807
LAB LAYOUT
BATTA LABORATORIES, INC.
BY
DATE
SCALE:
AC FILE:
DRAWN
HRH
3/24/95
NONE
REP-LAB
CHECKED
REP
DWG. NO.
REV.
PROJECT #
BEA
REVISED ON: 03/12/2004
2
Appendix V-2
REVISED 3/12/04 – UPDATE
REVISED 6/1/00 – UPDATE
REVISED 8/8/96 – UPDATE
REVISED 5/23/95 – ADD ORGANIC LAB
S:\IN-BOXES\Accreditation\AIHA\Lab_plan_Rev2.dwg

APPENDIX E

PLM MONTHLY SUMMARIES

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Note: the summaries listed in the appendix are examples. For a complete list of originals with raw data sheets please refer to the manual on the shelf located in the TEM office.

Examples of A Month PLM Summary

BATTA

BATTA Monthly PLM Summary

Manager: Naresh Batta Month/Year: December-03

PLM Analytical QC

- ☑ Monthly PLM Summary Page
- ☑ Monthly QC Summary
- ☑ Daily QC Charts (control limits)
- ☑ PLM Contamination / Alignment Worksheet
- ☑ NVLAP Proficiencies (copy of sumittal) NVLAP Round # M22003
- ☑ Daily Reference Samples & Round Robin RR Round # 26
- ☑ Refractive Oil Calibrations
- ☑ Deficiencies & Corrections

PLM QC Program

Lab Name: BATTA
Month/Year: Dec-03

Monthly PLM Summary Page

Last Revision: Monday, January 26, 2004

= indicates possible error or omission.

Lab Accuracy

Monthly QC Summary	Total Samples Analyzed	Total Samples QC'd**	# of Ref Samples Analyzed	# of Round Robin Samples Analyzed	# of NVLAP Proficiency Samples Analyzed	% QC'd	Total # of Errors *	Quantitative Error Rate (should be <5%)
	499	44	21	4	0	13.83	0	0.00

*See Analytical **and** Non-Analytical Deficiencies and Corrections notebooks for December-03
**Should match total in *Analyst Accuracy* Section below.

Analyst Accuracy

Analyst	# PLM Samples Analyzed	# Samples Reanalyzed	Classification Errors (From Analysis)	Classification Errors (From Reanalysis)	Total Analyst Classification Error Rate (%)
AK	499	0	0	0	0.00
RGS	0	44	0	0	0.00
Total # of Samples Reanalyzed		44			

Laboratory and Analyst Precision

	PLM Analyst CVs			
Analyst Initials	Range #1 (0-<5%)	Range #2 (5-20%)	Range #3 (21-50%)	Range #4 (51-100%)
AK	0.342	0.148	0.128	0.057
RGS	0.510	0.402	0.301	0.122
Lab Avg.	0.426	0.275	0.214	0.090

Comments:

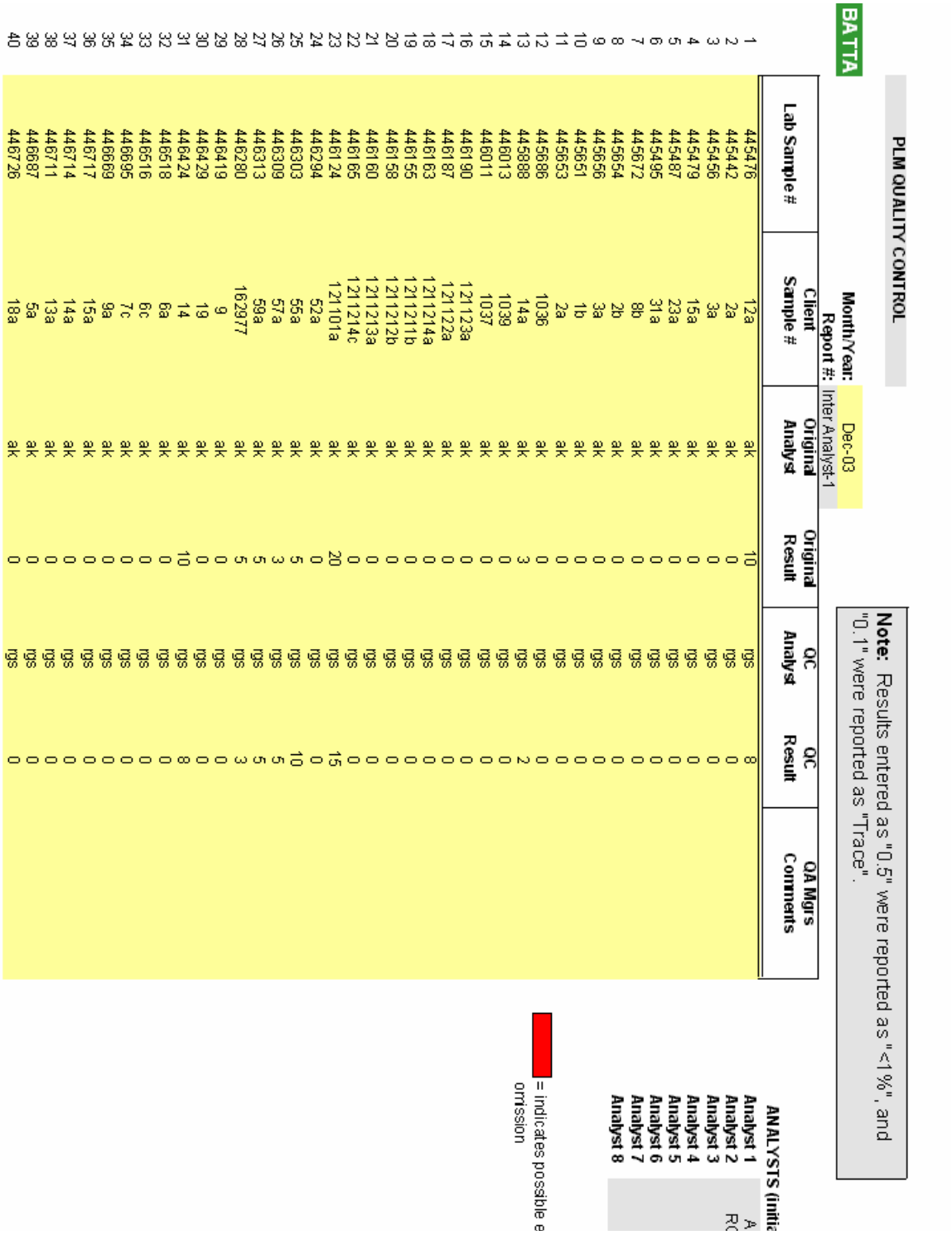
All calibrations and QC done in timely manner. Oil calibrations done 12/03/03; all ok. Starting this month, I have changed the Oil Cal. spreadsheet to adjust for temperature. Calculation used is from Dr. Su publication. Final results for PLM NVLAP Round M22003 received on Dec 30th. PLM Round Robin #26 samples analyzed by RGS in Dec. Results received on Dec 23rd. - END

Robert Shumate

Monthly PLM QC Summary

Lab: BATTA **Report#:** Inter Analyst-1 **Month/Yea**

	Lab Sample#	Client Sample #	Original Analyst	Original Result	QC Analyst	QC Result	Variance Original	Variance QC	Conclusio (from CV
1	445476	12a	ak	10	rgs	8	0.22	-0.22	Pass
2	445442	2a	ak	0	rgs	0	0	0	Pass
3	445456	3a	ak	0	rgs	0	0	0	Pass
4	445479	15a	ak	0	rgs	0	0	0	Pass
5	445487	23a	ak	0	rgs	0	0	0	Pass
6	445495	31a	ak	0	rgs	0	0	0	Pass
7	445672	8b	ak	0	rgs	0	0	0	Pass
8	445654	2b	ak	0	rgs	0	0	0	Pass
9	445656	3a	ak	0	rgs	0	0	0	Pass
10	445651	1b	ak	0	rgs	0	0	0	Pass
11	445653	2a	ak	0	rgs	0	0	0	Pass
12	445686	1036	ak	0	rgs	0	0	0	Pass
13	445888	14a	ak	3	rgs	2	0.40	-0.40	Pass
14	446013	1039	ak	0	rgs	0	0	0	Pass
15	446011	1037	ak	0	rgs	0	0	0	Pass
16	446190	121123a	ak	0	rgs	0	0	0	Pass
17	446187	121122a	ak	0	rgs	0	0	0	Pass
18	446163	1211214a	ak	0	rgs	0	0	0	Pass
19	446155	1211211b	ak	0	rgs	0	0	0	Pass
20	446158	1211212b	ak	0	rgs	0	0	0	Pass
21	446160	1211213a	ak	0	rgs	0	0	0	Pass
22	446165	1211214c	ak	0	rgs	0	0	0	Pass
23	446124	121101a	ak	20	rgs	15	0.29	-0.29	Pass
24	446294	52a	ak	0	rgs	0	0	0	Pass
25	446303	55a	ak	5	rgs	10	-0.67	0.67	Pass
26	446309	57a	ak	3	rgs	5	-0.50	0.50	Pass
27	446313	59a	ak	5	rgs	5	0.00	0.00	Pass
28	446280	162977	ak	5	rgs	3	0.50	-0.50	Pass
29	446419	9	ak	0	rgs	0	0	0	Pass
30	446429	19	ak	0	rgs	0	0	0	Pass
31	446424	14	ak	10	rgs	8	0.22	-0.22	Pass
32	446518	6a	ak	0	rgs	0	0	0	Pass
33	446516	6c	ak	0	rgs	0	0	0	Pass
34	446695	7c	ak	0	rgs	0	0	0	Pass
35	446669	9a	ak	0	rgs	0	0	0	Pass
36	446717	15a	ak	0	rgs	0	0	0	Pass
37	446714	14a	ak	0	rgs	0	0	0	Pass
38	446711	13a	ak	0	rgs	0	0	0	Pass
39	446687	5a	ak	0	rgs	0	0	0	Pass
40	446726	18a	ak	0	rgs	0	0	0	Pass
41	446720	16a	ak	0	rgs	0	0	0	Pass
42	446740	22a	ak	0	rgs	0	0	0	Pass
43	446958	1	ak	0	rgs	0	0	0	Pass



PLM QUALITY CONTROL

BATTA

Month/Year: Dec-03
Report #: Inter Analyst-1

Note: Results entered as "0.5" were reported as "<1%", and "0.1" were reported as "Trace".

	Lab Sample #	Client Sample #	Original Analyst	Original Result	QC Analyst	QC Result	QA Mgrs Comments
1	445476	12a	ak	10	rgs	8	
2	445442	2a	ak	0	rgs	0	
3	445456	3a	ak	0	rgs	0	
4	445479	15a	ak	0	rgs	0	
5	445487	23a	ak	0	rgs	0	
6	445495	31a	ak	0	rgs	0	
7	445672	8b	ak	0	rgs	0	
8	445654	2b	ak	0	rgs	0	
9	445656	3a	ak	0	rgs	0	
10	445651	1b	ak	0	rgs	0	
11	445653	2a	ak	0	rgs	0	
12	445686	1036	ak	0	rgs	0	
13	445888	14a	ak	3	rgs	2	
14	446013	1039	ak	0	rgs	0	
15	446011	1037	ak	0	rgs	0	
16	446190	121123a	ak	0	rgs	0	
17	446187	121122a	ak	0	rgs	0	
18	446163	1211214a	ak	0	rgs	0	
19	446155	1211211b	ak	0	rgs	0	
20	446158	1211212b	ak	0	rgs	0	
21	446160	1211213a	ak	0	rgs	0	
22	446165	1211214c	ak	0	rgs	0	
23	446124	121101a	ak	20	rgs	15	
24	446294	52a	ak	0	rgs	0	
25	446303	55a	ak	5	rgs	10	
26	446309	57a	ak	3	rgs	5	
27	446313	59a	ak	5	rgs	5	
28	446280	162977	ak	5	rgs	3	
29	446419	9	ak	0	rgs	0	
30	446429	19	ak	0	rgs	0	
31	446424	14	ak	10	rgs	8	
32	446518	6a	ak	0	rgs	0	
33	446516	6c	ak	0	rgs	0	
34	446695	7c	ak	0	rgs	0	
35	446669	9a	ak	0	rgs	0	
36	446717	15a	ak	0	rgs	0	
37	446714	14a	ak	0	rgs	0	
38	446711	13a	ak	0	rgs	0	
39	446687	5a	ak	0	rgs	0	
40	44672	18a	ak	0	rgs	0	

= indicates possible e
omission

ANALYSTS (initia
Analyst 1 A
Analyst 2 R
Analyst 3
Analyst 4
Analyst 5
Analyst 6
Analyst 7
Analyst 8

PLM CV
BATTA

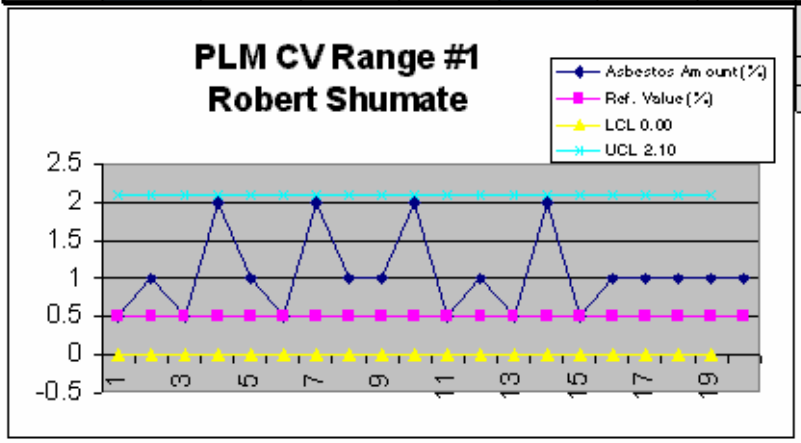
2001
2002
2003
2004

Page 1of 4

PLM Analyst CVs				
Analyst Initials	Range #1 (0-<5%)	Range #2 (5-20%)	Range #3 (21-50%)	Range #4 (51-100%)
AK	0.342	0.148	0.128	0.057
RGS	0.510	0.402	0.301	0.122
Lab Avg.	0.426	0.275	0.214	0.090

RGS - CV Range #1 Analyst Results						*Reference Results*				
Trial #	Date (2002)	Ref. ID #	Analyst	Asbestos Type	Asbestos Amount (%)	Asbestos Type	Ref. Value (%)	LCL	UCL	Percent Recovery
1	8/26	2	RGS	Anth.	0.5	Anth.	0.5	0.00	2.10	100.00
2	8/30	2	RGS	Anth.	1	Anth.	0.5	0.00	2.10	200.00
3	9/12	2	RGS	Anth.	0.5	Anth.	0.5	0.00	2.10	100.00
4	10/3	2	RGS	Anth.	2	Anth.	0.5	0.00	2.10	400.00
5	11/2	2	RGS	Anth.	1	Anth.	0.5	0.00	2.10	200.00
6	11/25	2	RGS	Anth.	0.5	Anth.	0.5	0.00	2.10	100.00
7	12/10	2	RGS	Anth.	2	Anth.	0.5	0.00	2.10	400.00
8	5/25	2	RGS	Anth.	1	Anth.	0.5	0.00	2.10	200.00
9	6/26	2	RGS	Anth.	1	Anth.	0.5	0.00	2.10	200.00
10	8/14	2	RGS	Anth.	2	Anth.	0.5	0.00	2.10	400.00
11	9/6	2	RGS	Anth.	0.5	Anth.	0.5	0.00	2.10	100.00
12	10/31	2	RGS	Anth.	1	Anth.	0.5	0.00	2.10	200.00
13	11/5	2	RGS	Anth.	0.5	Anth.	0.5	0.00	2.10	100.00
14	12/12	2	RGS	Anth.	2	Anth.	0.5	0.00	2.10	400.00
15	3/14	2	RGS	Anth.	0.5	Anth.	0.5	0.00	2.10	100.00
16	5/23	2	RGS	Anth.	1	Anth.	0.5	0.00	2.10	200.00
17	6/29	2	RGS	Anth.	1	Anth.	0.5	0.00	2.10	200.00
18	9/12	2	RGS	Anth.	1	Anth.	0.5	0.00	2.10	200.00
19	10/16	2	RGS	Anth.	1	Anth.	0.5	0.00	2.10	200.00
20	8/20	2	RGS	Anth.	1	Anth.	0.5	0.00	2.10	200.00

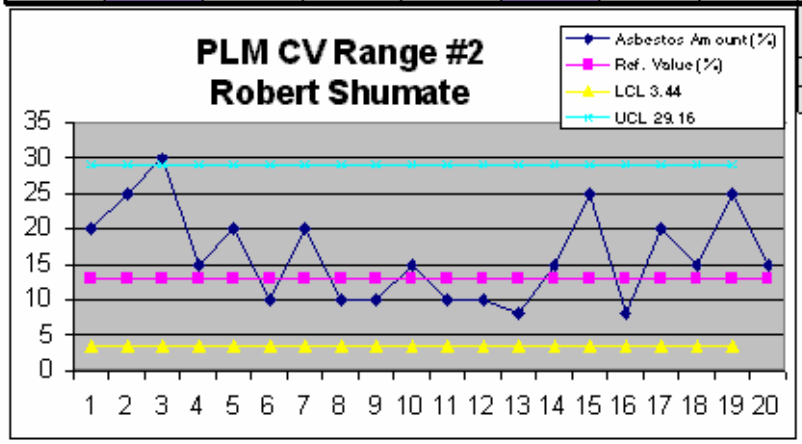
Mean (Asb %) =	1.05
Std Dev (Asb %) =	0.54
Mean % Recovery =	210.00
Analyst RSD or CV =	0.510



Page 2of 4

RGS - CV Range #2 Analyst Results						*Reference Results*				
Trial #	Date (2002)	Ref. ID #	Analyst	Asbestos Type	Asbestos Amount (%)	Asbestos Type	Ref. Value (%)	LCL	UCL	Percent Recovery
1	10/17	3	RGS	Chrys.	20	Chrys.	13	3.44	29.16	153.85
2	1/24	3	RGS	Chrys.	25	Chrys.	13	3.44	29.16	192.31
3	3/5	3	RGS	Chrys.	30	Chrys.	13	3.44	29.16	230.77
4	3/20	3	RGS	Chrys.	15	Chrys.	13	3.44	29.16	115.38
5	4/19	3	RGS	Chrys.	20	Chrys.	13	3.44	29.16	153.85
6	5/16	3	RGS	Chrys.	10	Chrys.	13	3.44	29.16	76.92
7	5/31	3	RGS	Chrys.	20	Chrys.	13	3.44	29.16	153.85
8	8/21	3	RGS	Chrys.	10	Chrys.	13	3.44	29.16	76.92
9	8/27	3	RGS	Chrys.	10	Chrys.	13	3.44	29.16	76.92
10	9/5	3	RGS	Chrys.	15	Chrys.	13	3.44	29.16	115.38
11	9/19	3	RGS	Chrys.	10	Chrys.	13	3.44	29.16	76.92
12	10/4	3	RGS	Chrys.	10	Chrys.	13	3.44	29.16	76.92
13	11/4	3	RGS	Chrys.	8	Chrys.	13	3.44	29.16	61.54
14	12/4	3	RGS	Chrys.	15	Chrys.	13	3.44	29.16	115.38
15	1/17	3	RGS	Chrys.	25	Chrys.	13	3.44	29.16	192.31
16	3/21	3	RGS	Chrys.	8	Chrys.	13	3.44	29.16	61.54
17	5/27	3	RGS	Chrys.	20	Chrys.	13	3.44	29.16	153.85
18	7/1	3	RGS	Chrys.	15	Chrys.	13	3.44	29.16	115.38
19	8/7	3	RGS	Chrys.	25	Chrys.	13	3.44	29.16	192.31
20	9/25	3	RGS	Chrys.	15	Chrys.	13	3.44	29.16	115.38

Mean (Asb %) =	16.30
Std Dev (Asb %) =	6.55
Mean % Recovery =	125.38
Analyst RSD or CV =	0.402



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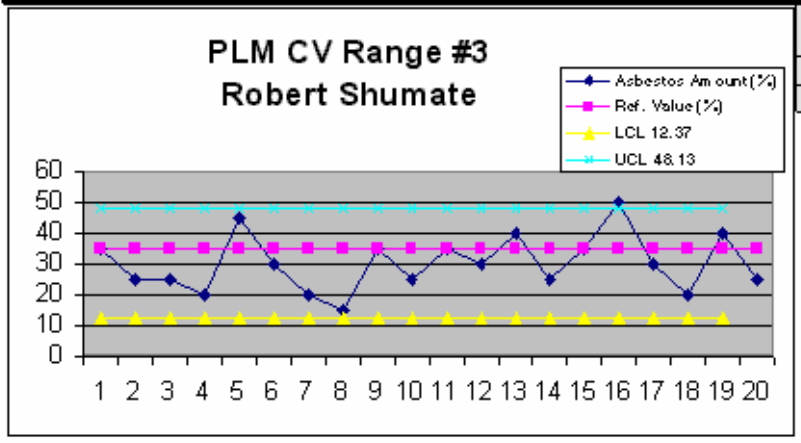
RGS - CV Range #3 Analyst Results						*Reference Results*				
Trial #	Date (2002)	Ref. ID #	Analyst	Asbestos Type	Asbestos Amount (%)	Asbestos Type	Ref. Value (%)	LCL	UCL	Percent Recovery
1	10/1	4	RGS	Chrys.	35	Chrys.	35	12.37	48.13	100.00
2	11/3	4	RGS	Chrys.	25	Chrys.	35	12.37	48.13	71.43
3	1/29	4	RGS	Chrys.	25	Chrys.	35	12.37	48.13	71.43
4	3/8	4	RGS	Chrys.	20	Chrys.	35	12.37	48.13	57.14
5	3/21	4	RGS	Chrys.	45	Chrys.	35	12.37	48.13	128.57
6	4/22	4	RGS	Chrys.	30	Chrys.	35	12.37	48.13	85.71
7	5/28	4	RGS	Chrys.	20	Chrys.	35	12.37	48.13	57.14
8	6/13	4	RGS	Chrys.	15	Chrys.	35	12.37	48.13	42.86
9	8/22	4	RGS	Chrys.	35	Chrys.	35	12.37	48.13	100.00
10	8/28	4	RGS	Chrys.	25	Chrys.	35	12.37	48.13	71.43
11	9/9	4	RGS	Chrys.	35	Chrys.	35	12.37	48.13	100.00
12	9/20	4	RGS	Chrys.	30	Chrys.	35	12.37	48.13	85.71
13	10/7	4	RGS	Chrys.	40	Chrys.	35	12.37	48.13	114.29
14	11/8	4	RGS	Chrys.	25	Chrys.	35	12.37	48.13	71.43
15	12/5	4	RGS	Chrys.	35	Chrys.	35	12.37	48.13	100.00
16	1/20	4	RGS	Chrys.	50	Chrys.	35	12.37	48.13	142.86
17	3/25	4	RGS	Chrys.	30	Chrys.	35	12.37	48.13	85.71
18	6/18	4	RGS	Chrys.	20	Chrys.	35	12.37	48.13	57.14
19	7/5	4	RGS	Chrys.	40	Chrys.	35	12.37	48.13	114.29
20	9/2	4	RGS	Chrys.	25	Chrys.	35	12.37	48.13	71.43

Mean (Asb %) =	30.25
Std Dev (Asb %) =	9.10
Mean % Recovery =	86.43
Analyst RSD or CV =	0.301



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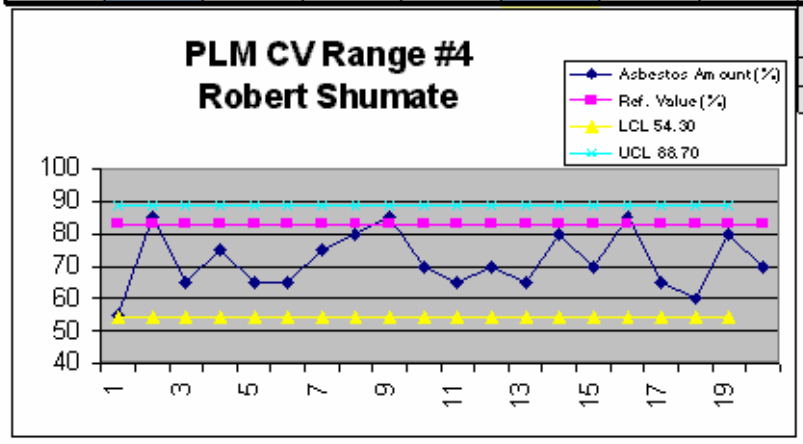
RGS - CV Range #4 Analyst Results						*Reference Results*				
Trial #	Date (2002)	Ref. ID #	Analyst	Asbestos Type	Asbestos Amount (%)	Asbestos Type	Ref. Value (%)	LCL	UCL	Percent Recovery
1	2/22	1	RGS	Chrys.	55	Chrys.	83	54.30	88.70	66.27
2	3/16	1	RGS	Chrys.	85	Chrys.	83	54.30	88.70	102.41
3	3/22	1	RGS	Chrys.	65	Chrys.	83	54.30	88.70	78.31
4	4/23	1	RGS	Chrys.	75	Chrys.	83	54.30	88.70	90.36
5	5/29	1	RGS	Chrys.	65	Chrys.	83	54.30	88.70	78.31
6	8/19	1	RGS	Chrys.	65	Chrys.	83	54.30	88.70	78.31
7	8/23	1	RGS	Chrys.	75	Chrys.	83	54.30	88.70	90.36
8	8/29	1	RGS	Chrys.	80	Chrys.	83	54.30	88.70	96.39
9	9/11	1	RGS	Chrys.	85	Chrys.	83	54.30	88.70	102.41
10	10/2	1	RGS	Chrys.	70	Chrys.	83	54.30	88.70	84.34
11	10/8	1	RGS	Chrys.	65	Chrys.	83	54.30	88.70	78.31
12	11/13	1	RGS	Chrys.	70	Chrys.	83	54.30	88.70	84.34
13	12/6	1	RGS	Chrys.	65	Chrys.	83	54.30	88.70	78.31
14	1/21	1	RGS	Chrys.	80	Chrys.	83	54.30	88.70	96.39
15	5/15	1	RGS	Chrys.	70	Chrys.	83	54.30	88.70	84.34
16	6/19	1	RGS	Chrys.	85	Chrys.	83	54.30	88.70	102.41
17	9/11	1	RGS	Chrys.	65	Chrys.	83	54.30	88.70	78.31
18	10/2	1	RGS	Chrys.	60	Chrys.	83	54.30	88.70	72.29
19	11/20	1	RGS	Chrys.	80	Chrys.	83	54.30	88.70	96.39
20	1/4	1	RGS	Chrys.	70	Chrys.	83	54.30	88.70	84.34

Mean (Asb %) =	71.50
Std Dev (Asb %) =	8.75
Mean % Recovery =	86.14
Analyst RSD or CV =	0.122



BATTA

Analyst Accuracy

NVLAP PT Round

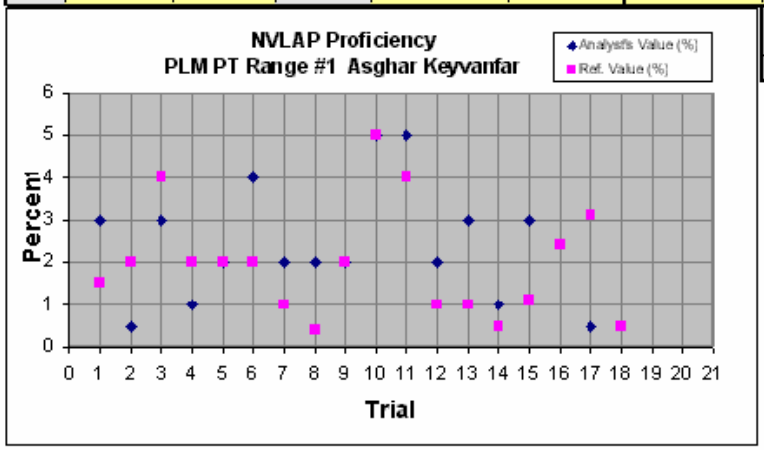
Range #1	(<1 - <5%)
Range #2	(5 - 20%)
Range #3	(21 - 50%)
Range #4	(51 - 100%)

AK Proficiency Misidentifications		
# of Mis-IDs	# of Samples Analyzed	% of Total
1	32	3.13

AK - PT Range #1 (<1 - <5%) Analyst Results | *Reference Results*

Trial #	PT Round #	Ref. ID#	Analyst	Asbestos Type	Analyst's Value (%)	Asbestos Type	Ref. Value (%)	Percent Recovery	ID
1	M21999	1-1	AK	Amosite	3	Amosite	1.5	200.00	
2	M21999	1-2	AK	Chrysotile	0.5	Chrysotile	2.0	25.00	
3	M21999	2	AK	Chrysotile	3	Chrysotile	4.0	75.00	
4	M12000	4-3	AK	Crocidolite	0	Crocidolite	2.0	0.00	
5	M22000	1	AK	Chrysotile	3	Chrysotile	2.0	150.00	
6	M22000	2-1	AK	Amosite	2	Amosite	2.0	100.00	
7	M22000	2-2	AK	Chrysotile	3	Chrysotile	1.0	300.00	
8	M12001	2	AK	Chrysotile	5	Chrysotile	0.4	1250.00	
9	M22001	2	AK	Anthophyllite	3	Actinolite	2.0	150.00	1
10	M22001	3	AK	Crocidolite	5	Crocidolite	5.0	100.00	
11	M22001	4-1	AK	Amosite	8	Amosite	4.0	200.00	
12	M12002	1-1	AK	Amosite	3	Amosite	1.0	300.00	
13	M12002	1-2	AK	Chrysotile	1	Chrysotile	1.0	100.00	
14	M22002	3	AK	Actinolite	2	Actinolite	0.5	400.00	
15	M12003	2-2	AK	Chrysotile	2	Chrysotile	1.1	181.82	
16	M12003	3-2	AK	Chrysotile	3	Chrysotile	2.4	125.00	
17	M12003	4	AK	Chrysotile	3	Chrysotile	3.1	96.77	
18	M22003	1	AK	Crocidolite	3	Crocidolite	0.5	600.00	
19			AK						
20			AK						

Mean (Asb %) =	2.92
Std Dev (Asb %) =	1.80
Mean % Recovery =	241.87



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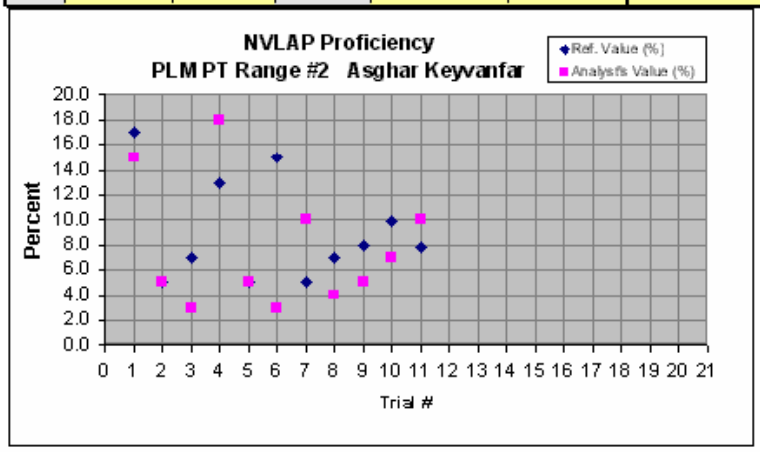
AK - PT Range #2 (5 - 20%) Analyst Results						Reference Results			
Trial #	PT Round #	Ref. ID#	Analyst	Asbestos Type	Analyst's Value (%)	Asbestos Type	Ref. Value (%)	Percent Recovery	ID
1	M21999	3	AK	Chrysotile	20	Chrysotile	17.0	117.65	
2	M21999	4	AK	Chrysotile	5	Chrysotile	5.0	100.00	
3	M12000	2	AK	Chrysotile	3	Chrysotile	7.0	42.86	
4	M12000	3	AK	Anthophyllite	30	Anthophyllite	13.0	230.77	
5	M12000	4-1	AK	Chrysotile	3	Chrysotile	5.0	60.00	
6	M22000	3	AK	Chrysotile	5	Chrysotile	15.0	33.33	
7	M22001	4-2	AK	Chrysotile	3	Chrysotile	5.0	60.00	
8	M12002	2	AK	Amosite	5	Amosite	7.0	71.43	
9	M12002	4	AK	Chrysotile	5	Chrysotile	8.0	62.50	
10	M12003	2-1	AK	Amosite	5	Amosite	9.9	50.51	
11	M12003	3-1	AK	Crocidolite	5	Crocidolite	7.8	64.10	
12			AK						
13			AK						
14			AK						
15			AK						
16			AK						
17			AK						
18			AK						
19			AK						
20			AK						

Mean (Asb %) =	8.09
Std Dev (Asb %) =	8.70
Mean % Recovery =	81.19



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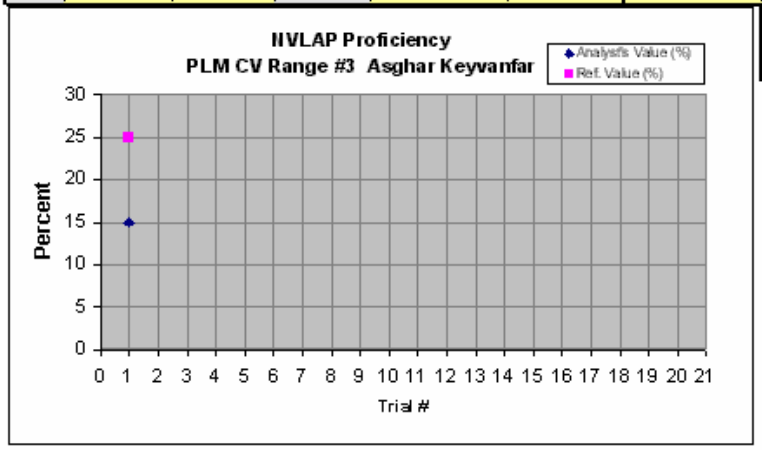
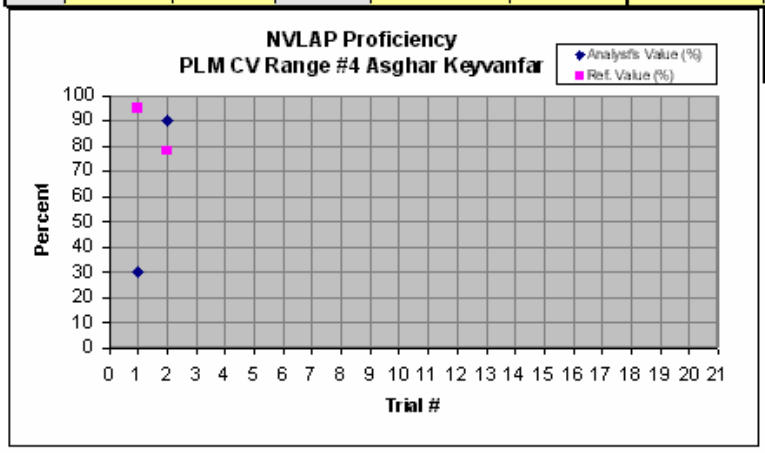
AK - PT Range #3 (21 - 50%) Analyst Results						Reference Results			
Trial #	PT Round #	Ref. ID#	Analyst	Asbestos Type	Analyst's Value (%)	Asbestos Type	Ref. Value (%)	Percent Recovery	ID
1	M22003	4	AK	Anthophyllite	20	Anthophyllite	25	80.00	
2			AK						
3			AK						
4			AK						
5			AK						
6			AK						
7			AK						
8			AK						
9			AK						
10			AK						
11			AK						
12			AK						
13			AK						
14			AK						
15			AK						
16			AK						
17			AK						
18			AK						
19			AK						
20			AK						

Mean (Asb %) =	20.00
Std Dev (Asb %) =	#DIV/0!
Mean % Recovery =	80.00



AK - PT Range #4 (51 - 100%) Analyst Results						Reference Results			
Trial #	PT Round #	Ref. ID#	Analyst	Asbestos Type	Analyst's Value (%)	Asbestos Type	Ref. Value (%)	Percent Recovery	ID
1	M12001	1	AK	Chrysotile	45	Chrysotile	95	47.37	
2	M22002	3	AK	Chrysotile	95	Chrysotile	78	121.79	
3			AK						
4			AK						
5			AK						
6			AK						
7			AK						
8			AK						
9			AK						
10			AK						
11			AK						
12			AK						
13			AK						
14			AK						
15			AK						
16			AK						
17			AK						
18			AK						
19			AK						
20			AK						

Mean (Asb %) =	70.00
Std Dev (Asb %) =	35.36
Mean % Recovery =	84.58



BATTA

Analyst Accuracy

NVLAP PT Round

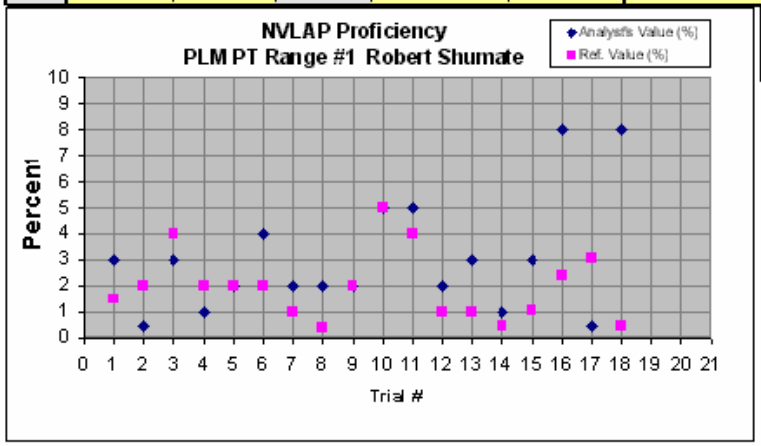
Range #1	(<1 - <5%)
Range #2	(5 - 20%)
Range #3	(21 - 50%)
Range #4	(51 - 100%)

RGS Proficiency Misidentifications		
# of Mis-IDs	# of Samples Analyzed	% of Total
0	32	0.00

RGS - PT Range #1 (<1 - <5%) Analyst Results | *Reference Results*

Trial #	PT Round #	Ref. ID#	Analyst	Asbestos Type	Analyst's Value (%)	Asbestos Type	Ref. Value (%)	Percent Recovery	ID
1	M21999	1-1	RGS	Amosite	3	Amosite	1.5	200.00	
2	M21999	1-2	RGS	Chrysotile	0.5	Chrysotile	2.0	25.00	
3	M21999	2	RGS	Chrysotile	3	Chrysotile	4.0	75.00	
4	M12000	4-3	RGS	Crocidolite	1	Crocidolite	2.0	50.00	
5	M22000	1	RGS	Chrysotile	2	Chrysotile	2.0	100.00	
6	M22000	2-1	RGS	Amosite	4	Amosite	2.0	200.00	
7	M22000	2-2	RGS	Chrysotile	2	Chrysotile	1.0	200.00	
8	M12001	2	RGS	Chrysotile	2	Chrysotile	0.4	500.00	
9	M22001	2	RGS	Actinolite	2	Actinolite	2.0	100.00	
10	M22001	3	RGS	Crocidolite	5	Crocidolite	5.0	100.00	
11	M22001	4-1	RGS	Amosite	5	Amosite	4.0	125.00	
12	M12002	1-1	RGS	Amosite	2	Amosite	1.0	200.00	
13	M12002	1-2	RGS	Chrysotile	3	Chrysotile	1.0	300.00	
14	M22002	3	RGS	Actinolite	1	Actinolite	0.5	200.00	
15	M12003	2-2	RGS	Chrysotile	3	Chrysotile	1.1	272.73	
16	M12003	3-2	RGS	Chrysotile	8	Chrysotile	2.4	333.33	
17	M12003	4	RGS	Chrysotile	0.5	Chrysotile	3.1	16.13	
18	M22003	1	RGS	Crocidolite	8	Crocidolite	0.5	1600.00	
19			RGS						
20			RGS						

Mean (Asb %) =	3.06
Std Dev (Asb %) =	2.23
Mean % Recovery =	255.40



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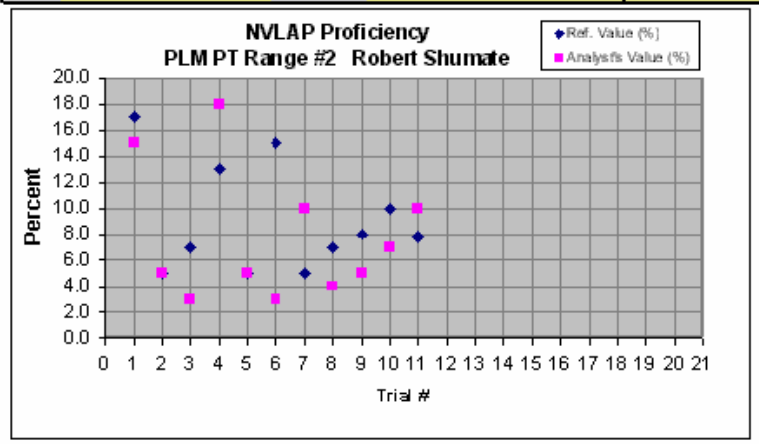
RGS - PT Range #2 (5 - 20%) Analyst Results						Reference Results			
Trial #	PT Round #	Ref. ID#	Analyst	Asbestos Type	Analyst's Value (%)	Asbestos Type	Ref. Value (%)	Percent Recovery	ID
1	M21999	3	RGS	Chrysotile	15	Chrysotile	17.0	88.24	
2	M21999	4	RGS	Chrysotile	5	Chrysotile	5.0	100.00	
3	M12000	2	RGS	Chrysotile	3	Chrysotile	7.0	42.86	
4	M12000	3	RGS	Anthophyllite	18	Anthophyllite	13.0	138.46	
5	M12000	4-1	RGS	Chrysotile	5	Chrysotile	5.0	100.00	
6	M22000	3	RGS	Chrysotile	3	Chrysotile	15.0	20.00	
7	M22001	4-2	RGS	Chrysotile	10	Chrysotile	5.0	200.00	
8	M12002	2	RGS	Amosite	4	Amosite	7.0	57.14	
9	M12002	4	RGS	Chrysotile	5	Chrysotile	8.0	62.50	
10	M12003	2-1	RGS	Amosite	7	Amosite	9.9	70.71	
11	M12003	3-1	RGS	Crocidolite	10	Crocidolite	7.8	128.21	
12			RGS						
13			RGS						
14			RGS						
15			RGS						
16			RGS						
17			RGS						
18			RGS						
19			RGS						
20			RGS						

Mean (Asb %) =	7.73
Std Dev (Asb %) =	5.00
Mean % Recovery =	91.65



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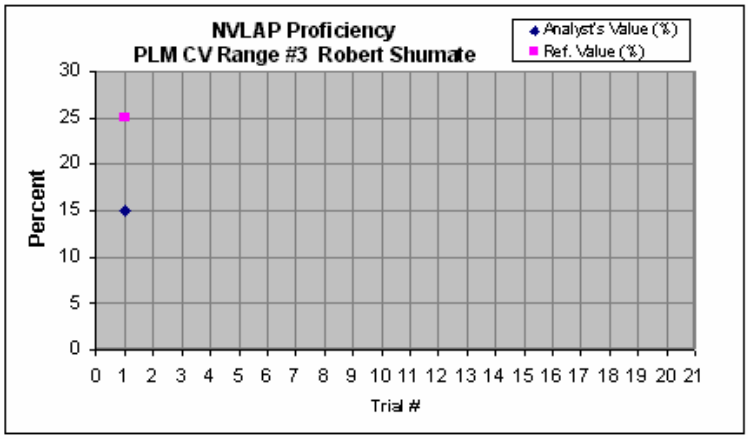
RGS - PT Range #3 (21 - 50%) Analyst Results						Reference Results			
Trial #	PT Round #	Ref. ID#	Analyst	Asbestos Type	Analyst's Value (%)	Asbestos Type	Ref. Value (%)	Percent Recovery	ID
1	M22003	4	RGS	Anthophyllite	15	Anthophyllite	25	60.00	
2			RGS						
3			RGS						
4			RGS						
5			RGS						
6			RGS						
7			RGS						
8			RGS						
9			RGS						
10			RGS						
11			RGS						
12			RGS						
13			RGS						
14			RGS						
15			RGS						
16			RGS						
17			RGS						
18			RGS						
19			RGS						
20			RGS						

Mean (Asb %) =	15.00
Std Dev (Asb %) =	#DIV/0!
Mean % Recovery =	60.00



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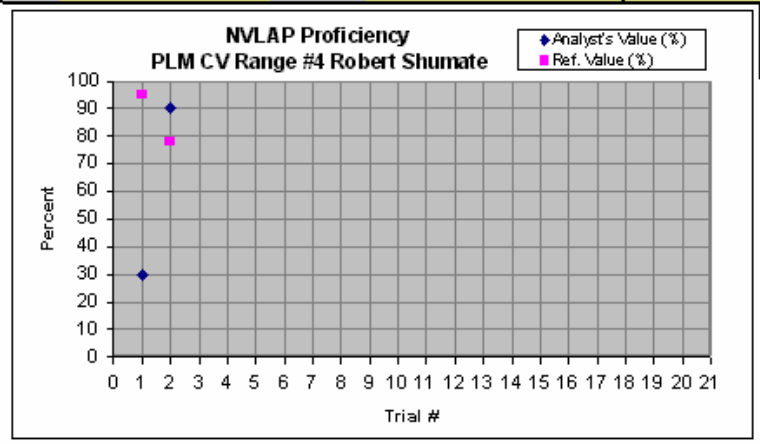
RGS - PT Range #4 (51 - 100%) Analyst Results						Reference Results			
Trial #	PT Round #	Ref. ID#	Analyst	Asbestos Type	Analyst's Value (%)	Asbestos Type	Ref. Value (%)	Percent Recovery	ID
1	M12001	1	RGS	Chrysotile	30	Chrysotile	95	31.58	
2	M22002	1	RGS	Chrysotile	90	Chrysotile	78	115.38	
3			RGS						
4			RGS						
5			RGS						
6			RGS						
7			RGS						
8			RGS						
9			RGS						
10			RGS						
11			RGS						
12			RGS						
13			RGS						
14			RGS						
15			RGS						
16			RGS						
17			RGS						
18			RGS						
19			RGS						
20			RGS						

Mean (Asb %) =	60.00
Std Dev (Asb %) =	42.43
Mean % Recovery =	73.48



BATTA

Daily PLM Checklist

1. Check off each item as it is being completed.
2. Complete daily to ensure all QA/QC is done in a timely manner.
3. QA/QC is not to be put-off due to high workloads.
4. Supervision will periodically check forms to ensure compliance.

Asghar Keyvanfar December-03

PLM Station #1, Nikon Labophot-POL, Serial # 954996

Analyst Initials	Date	Align PLM 1. Stage Centered 2. Crosshairs (use Anthophyllite mount) 3. Polarizer (black background) 4. Field Iris (centered) 5. Central stop aligned (use Bertrand lens) 6. Check dispersion colors (use Amosite mount)						Contamination Blank (use SRM 1866 Fiberglass to check hood area, stereoscope, and tools)		
		1	2	3	4	5	6	Hood	Stereo	Tools
ak	12/1	✓	✓	✓	✓	✓	✓	✓	✓	✓
ak	12/2	✓	✓	✓	✓	✓	✓	✓	✓	✓
ak	12/3	✓	✓	✓	✓	✓	✓	✓	✓	✓
ak	12/4	✓	✓	✓	✓	✓	✓	✓	✓	✓
ak	12/5	✓	✓	✓	✓	✓	✓	✓	✓	✓
ak	12/6									
ak	12/7									
ak	12/8	✓	✓	✓	✓	✓	✓	✓	✓	✓
ak	12/9	✓	✓	✓	✓	✓	✓	✓	✓	✓
ak	12/10	✓	✓	✓	✓	✓	✓	✓	✓	✓
ak	12/11	✓	✓	✓	✓	✓	✓	✓	✓	✓
ak	12/12	✓	✓	✓	✓	✓	✓	✓	✓	✓
ak	12/13									
ak	12/14									
ak	12/15	✓	✓	✓	✓	✓	✓	✓	✓	✓
ak	12/16	✓	✓	✓	✓	✓	✓	✓	✓	✓
ak	12/17	✓	✓	✓	✓	✓	✓	✓	✓	✓
ak	12/18	✓	✓	✓	✓	✓	✓	✓	✓	✓
ak	12/19	✓	✓	✓	✓	✓	✓	✓	✓	✓
ak	12/20									
ak	12/21									
ak	12/22	✓	✓	✓	✓	✓	✓	✓	✓	✓
ak	12/23	✓	✓	✓	✓	✓	✓	✓	✓	✓
ak	12/24	✓	✓	✓	✓	✓	✓	✓	✓	✓
ak	12/25									
ak	12/26									
ak	12/27									
ak	12/28									
ak	12/29	✓	✓	✓	✓	✓	✓	✓	✓	✓
ak	12/30	✓	✓	✓	✓	✓	✓	✓	✓	✓
ak	12/31	✓	✓	✓	✓	✓	✓	✓	✓	✓

Memo

To: Naresh Batta

From: Bob Shumate

Date: Tuesday, December 30, 2003

Ref: PLM Proficiency Results

On December 24, 2003, I received from you a copy of the M22003 NVLAP PLM Proficiency results. While we achieved an overall passing score, there were error points (130) assessed. Since our PLM lab normally has less than 50 error points assessed for any one PLM round, it is understandable that you found 130 excessive. Acquiring 150 error points in any one NVLAP Proficiency round indicates a round failure. You have requested that I investigate the problem. Below are my findings.

Problem:
In comparing the raw data to the copy sent to NVLAP, I realized that the 80 error points assessed to Sample #4 were due to administrative error. I did not transcribe any of the refractive index values to the final report form from the raw data. Both Asghar and I did measure those properties. Upon closer examination, I realized that had we reported those values, we would have been out of range for the alpha (α) direction. Values for both Asghar and I were too high and 40 error points would have been assessed instead of 80.

Corrective Action:
In the past, it had been standard procedure to have you look at the forms and compare the raw data to the report forms as a QC measure. In this instance you were not available to perform the QC before it was mailed. If you are not available in the future, I can have either Prasad or Bo look at the data before it is reported. Asghar and I will re-examine the optical properties of Sample #4 and resolve the "α – direction" issue. The issue will be documented in the Analytical Deficiency & Corrections Log.

Problem:
There was also 50 error points assessed to Sample #1. This was due to our reported concentration being out of range on the high end. The sample contained (according to NVLAP) 0.5% Crocidolite asbestos. The acceptable range is one-tenth percent to four percent. We reported six percent. Asghar's raw data shows three percent, while mine was eight percent. I simply reported close to the average of the two. It is relatively common to bias high on quantifying percentages for Crocidolite. The fiber color against the white background of the matrix offers high color contrast, causing subconscious high-end bias. According to the NVLAP results, 143 of the 238 (60%) participating labs reported concentrations of Crocidolite outside the acceptable range. All 143 of these labs over-estimated the quantity.

Corrective Action:
Under different conditions, we may be able to "recalibrate" ourselves easily by viewing standards of known concentrations at different levels (<1, 1, 5, 20 and above). Unfortunately, we have only one RTI standard with Crocidolite (3% Crocidolite, with 3% Amosite and 94% Gypsum). Additionally, we can go back and train visually with previously-analyzed NVLAP samples. Documentation of this training will be annotated in our QA records and in Asghar's and my lab personnel file. As part of our everyday QC, the analyst examines (macroscopically) one reference standard to "calibrate his eyes" for visual area estimation. Currently, there are no Crocidolite-containing standards. With Bo coming into the PLM program, I will be introducing four different standards. One of them will contain low-percent Crocidolite.

I will keep you updated on any problems or concerns regarding this issue. Thank you for offering me the opportunity to resolve this issue.

SIGNED

Robert G. Shumate, Jr.
Microscopy Manager

BATTA LABORATORIES, INC.

Deficiencies and Corrective Actions

Month: January 2004

1. **Analyst(s)**: Robert Shumate + Asghan Keyvanfar **Date**: 1/8/04

Reference #: NVLAP PLM M22003 **Sample #**: Samples [redacted] 4

#4 was submitted c/o optical (RI) data. It was a transcription error. In addition, the data would have been out of range. AK and I reevaluated the α values. We may have been observing beta directions.

2. **Analyst(s)**: Robert Shumate **Date**: 1/8/04

Reference #: NVLAP M22003 **Sample #**: Sample #1

The quantified value reported for Oxacholin was too high. I pulled some standards and "refreshed" myself on VAE. It had been a while, and I was out of practice.

3. **Analyst(s)**: Robert Shumate **Date**:_____

Reference #:_____ **Sample #**:_____

4. **Analyst(s)**: Robert Shumate **Date**:_____

Reference #:_____ **Sample #**:_____

UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, Maryland 20899-

December 22, 2003

Mr. Naresh C. Batta
Batta Laboratories, Inc.
Delaware Industrial Park
6 Garfield Way
Newark, DE 19713-5817

NVLAP Lab Code: 101032 - 0

Dear Mr. Batta,

Congratulations! Your laboratory has passed the September 2003 round of proficiency testing (PLMM22003) required by the National Voluntary Laboratory Accreditation Program (NVLAP) for Bulk Asbestos Analysis.

If your laboratory is accredited, your status remains unchanged. If your laboratory is not yet accredited, or if your laboratory's accreditation has been suspended, you will be notified of any requirements your laboratory must meet to complete the accreditation / reaccreditation process.

Enclosed you will find the Summary of Analysis and your laboratory's results.

If you have any questions, please call me at 301-975-6499, or Hazel M. Richmond at 301-975-3024.

Sincerely,

Thomas R. Davis

Thomas R. Davis, Senior Program Manager
Laboratory Accreditation Program

Enclosure(s)

NIST

NIST Bulk Asbestos Proficiency Test
September 2003, Round M22003

NVLAP Lab Code 101032-0

PROFICIENCY TEST M22003 SUBTOTALS

Sample 1	50
Sample 2	0
Sample 3	0
Sample 4	80

TOTAL POINTS 130

Failure = 150 or more total points

Note: for details and raw data sheet for this round of NVLAP analysis, please refer to the volume titled " PLM Monthly Summaries Oct 2003 – Present" on the shelf.

Asghar Keyvanfar Daily PLM Reference Sample Analysis -- December-03

DATE	Analyst Initials	Reference Sample #	Analyst Result Ch	Am	Cr	Ant	Tr	Act	Analyst Result Ch	Am	Cr	Ant	Tr	Act	Entered in Computer
12/1	ak	1	60												
12/2	ak	2				0.5									
12/3	ak	3	15												
12/4	ak	4	40												
12/5	ak	1	75												
12/6	ak														
12/7	ak														
12/8	ak	2				1.0									
12/9	ak	3	20												
12/10	ak	4	35												
12/11	ak	1	75												
12/12	ak	2				0.50									
12/13	ak														
12/14	ak														
12/15	ak	3	20												
12/16	ak	4	40												
12/17	ak	1	70												
12/18	ak	2				1.0									
12/19	ak	3	15												
12/20	ak														
12/21	ak														
12/22	ak	4	30												
12/23	ak	1	80												
12/24	ak	2				0.50									
12/25	ak														
12/26	ak														
12/27	ak														
12/28	ak														
12/29	ak	3	15												
12/30	ak	4	35												
12/31	ak	1	75												

EMSL

Memo

To: Richard Harding, EMSL Analytical, Inc. – Indianapolis
Gary Swanson, Carolina Environmental, Inc.
Robert G. Shumate Jr., Batta Laboratories, Inc.

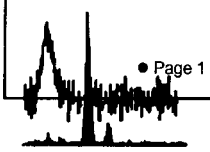
From: Sandra Sobrino, EMSL Analytical, Inc.-Chicago

Date: 12/19/03

Re: PLM Round Robin Program-Round #26 Results

Please find results of round #26 for PLM Round Robins. Carolina Environmental will host round #27. Thank you for your participation in this round. If you have any questions, please contact me at 773-313-0099.

Sandra Sobrino
EMSL Analytical, Inc. – Chicago
2444 West George Street
Chicago, IL 60618
Fax: 773-313-0139
Email: ssobrino@emsl.com



EMSL Analytical, Inc.-Chicago

PLM Round Robin Results - #26

Laboratory	Analyst	R26-1 Chrysotile	R26-2 Floor Tile Chrysotile	R26-2 Floor Tile Tremolite	R26-2 Mastic Chrysotile	R26-3 Chrysotile	R26-4 None Detected
EMSL Chicago	S. Sobrino	15	5	0	5	10	0
	S. Folz	15	10	0	10	8	0
BATTA	RGS	15	10	0	10	3	0
	AK	5	3	0	5	3	0
EMSL Indianapolis	R.Harding	25	3	0	2	10	0
	S.Harding	30	3	0	4	10	0
	MEP	20	3	0	8	5	0
	JCN	12	5	0.5	8	5	0
Carolina Environmental	EJ	25	8	0	8	15	0
	GS	15	10	0.5	10	15	0
	ES	20	15	0	10	15	0
	AM	15	10	0	10	5	0
	Greg	20	12	0.5	10	15	0
	SH	30	10	0	10	2	0
Mean		18.71428571	7.642857143	0.107142857	7.857142857	8.642857143	0
Standard Deviation		7.021161107	3.934211735	0.212907657	2.7416206	4.924149952	0
%RSD or CV		266.5411807	194.2665433	50.32362797	286.5875335	175.5197796	#DIV/0!

BATTA

Refractive Index Oil Calibration Worksheet

Single Liquid Method - Dr Su 1993 Manuscript

Start with: $n_D^S = n^L_D + (\Delta^L - \Delta^S) * k_i$

Becomes: $n^L_D = n_D^S - (\Delta^L - \Delta^S) * k_i$

Where: n^L_D = Index of refraction of the liquid
n_D^S = Index of refraction of the solid
Δ^L = Dispersion coefficient of the liquid
Δ^S = Dispersion coefficient of the solid
k_i = Conversion constant (from the matching wavelength to Hartman's Constant)

Date	Oil Used	Cargille Value (n_D 5893A) *	Δ^L (from label)	Temp Coefficient [dn/dt (from label)]	Δ^S (nF - nC) *Cargille Chart*	*Wavelength* Observed (nm)	k_D Conv. Factor	Temp °C at Calibration	Calculated n_D	Temperature-Corrected n_D	Difference (from label)	Criteria ** (Accept or Reject)	Lot # of R_D Oil (From Label)
12/3/03	1.550	1.55158	0.0267	0.000491	0.01112	580	0.05	23.0	1.550801	1.551783	0.00178	ACCEPTABLE	0902
12/3/03	1.680	1.67827	0.0348	0.000475	0.01226	600	-0.05	23.0	1.679397	1.680347	0.00035	ACCEPTABLE	0696
12/3/03	1.605	1.60585	0.0243	0.000441	0.01570	630	-0.19	23.0	1.6074845	1.6083665	0.00337	ACCEPTABLE	1095
12/3/03	1.625	1.62564	0.0275	0.000452	0.01759	570	0.10	23.0	1.624649	1.625553	0.00055	ACCEPTABLE	0902
12/3/03	1.640			0.000460			n/a					Not Measured	

** Cargille value is based on published measurements at 22°C. It has been determined that these values would change insignificantly over a large range of air temperatures. Therefore, no correction is considered necessary.*

*** Accept criteria: Calculated value must be +/- 0.004 from published value.*

BATTA

Refractive Index Oil Calibration Worksheet

Single Liquid Method - Dr Su 1993 Manuscript

Month December Year 2003

Start with: $n_D^S = n_D^L + (\Delta^L - \Delta^S) * k_i$

Becomes: $n_D^L = n_D^S - (\Delta^L - \Delta^S) * k_i$

Where: n_D^L = Index of refraction of the liquid
n_D^S = Index of refraction of the solid
Δ^L = Dispersion coefficient of the liquid
Δ^S = Dispersion coefficient of the solid
k_i = Conversion constant (from the matching wavelength to Hartman's Constant)

Date	Oil Used	Cargille Value (n_D 5893A) *	Δ^L (from label)	Temp Coefficient [dn/dt (from label)]	Δ^S (nF - nC) Cargille Chart	Wavelength Observed	k_D Conv. Factor	Temp (C) at Calibration	Calculated n_D	Lot # of R_D Oil (from label)
12-3-03	1.550	1.55158	0.0267	0.000491	0.01112	548	0.05	23	1.550801	0902
	1.680	1.67827	0.0348	0.000475	0.01226	600	-0.05	23	1.679397	0696
	1.605	1.60585	0.0243	0.000441	0.01570	630	-0.19	23	1.607845	1095
↓	1.625	1.62564	0.0275	0.000452	0.01759	570	0.10	23	1.627649	0902
	1.640	1.64333	0.0299	0.000460	0.01343					

Space for Calculations:

$\lambda_0 = 589$ $K_0 = 0.05$

$n_{LD} = 1.55158 - (0.02667 - 0.01112) \times 0.05$

$n_{LD} = 1.550801$

$\lambda_0 = 680$ $K_0 = -0.05$

$n_{LD} = 1.67827 - (0.0348 - 0.01226) \times -0.05$

$n_{LD} = 1.679397$

$\lambda_0 = 630$ $K_0 = -0.19$

$n_{LD} = 1.60585 - (0.0243 - 0.01570) \times -0.19$

$n_{LD} = 1.607845$

$\lambda_0 = 570$ $K_0 = 0.10$

$n_{LD} = 1.62564 - (0.0275 - 0.01759) \times 0.10$ $n_{LD} = 1.627649$

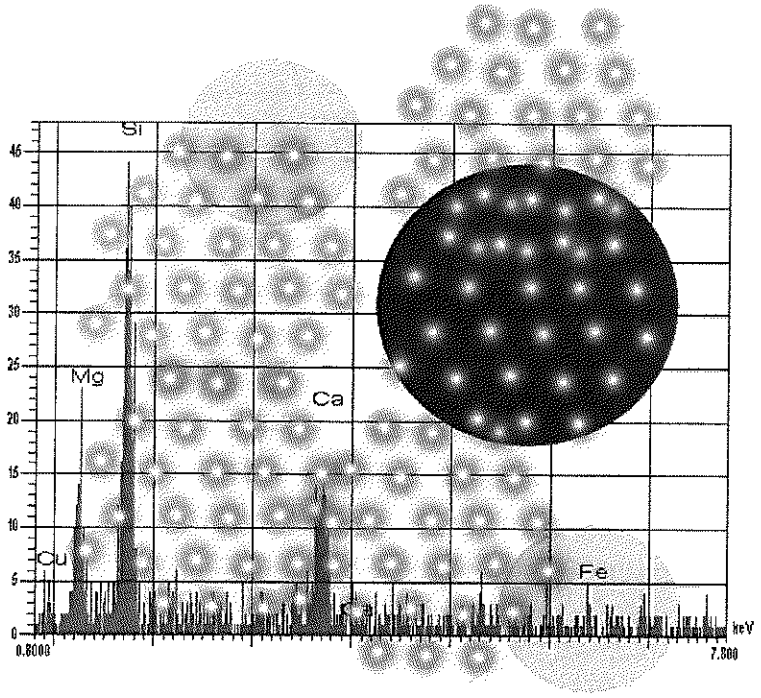
Analyst Signature

QA/QC Mgr. Signature

ATTACHMENT D
Batta Laboratories QM3
UFP-QAPP for Riverside Avenue Asbestos ER
June 2017

batta
LABORATORIES

QUALITY CONTROL/QUALITY ASSURANCE MANUAL FOR TEM ASBESTOS ANALYSIS



7th Edition

Jan 9, 2015

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2015 Asbestos QC/QA Manual Signature File

All laboratory personnel authorized to perform accredited analytes are to read, understand the QC/QA manual on an annual basis. Signatures of this document states that signing employees has read and understand the manual.

Name (Print)	Signature	Date
BRUCE FAULSEIT		1/13/15
Angela R. Yohn		1/13/15
Judy XU	Judy XU	1/13/15

Note: Changes made to all manuals you had reviewed were laboratory organization chart and a few typos. No other significant changes were made to previous versions.

BATTA LABORATORIES, INC.
Delaware Industrial Park
6 Garfield Way
Newark, DE 19713 - 5817

batta LABORATORIES

A CERTIFIED MBE COMPANY
EPA LAB ID# DE004
AIHA# 100448
NVLAP# 101032

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Table of Contents (Cont'd)

Appendices (8)

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CHAPTER I

ORGANIZATION

I-I: GENERAL STATEMENT

BATTA LABORATORIES, INC. (BLI) has developed a comprehensive management system of quality assurance and quality control practices to ensure the consistent accuracy and validity of the data/results that BLI produces. This management system, designed to optimize the quality of the laboratory operations for quality products, consists of quality, administrative and technical systems that govern the proper operations of this laboratory in fulfilling the requirement of the international standard ISO/IEC 17025 2005, and in agreement with the NVLAP procedures and general requirements provided in NIST handbook 150, the 2006 edition. The BLI management, through rigorous internal and external reviews and audits, is fully committed to continual improvement of the current QA system for better services.

BLI has been striving to deliver its customers reliable results by developing, improving and maintaining a rigorous quality assurance and quality control system. As such, BLI has constructed a viable organizational system for the general laboratory operation, including QA/QC procedures and practices of conducting routine customer analysis. As part of the quality control system, BLI is willing to cooperate with customers or their representatives in clarifying the customer's request and in monitoring the laboratory's performance in relation to the work performed while the confidentiality of the customers are protected.

In BLI, well established polices, documentation and quality control and quality assurance programs assure that the laboratory's management and personnel are free from any undue internal and external commercial, financial and other pressures and influences that may adversely affect the quality of our work. While data quality and clients' need are vital to BLI's success as a regional leading industrial laboratory, BLI's quality management system assures none-involvement in any activities that would diminish our confidence in laboratory's competence, impartiality, judgment or operational integrity.

BLI's management is committed to good professional practice and to the quality of its testing in serving its customers. The laboratory management is committed to comply with ISO requirements and NVLAP guidelines, and to continually improve the effectiveness of its management system.

I-II: MODIFICATION HISTORY AND REVISION POLICY

This laboratory QA/QC manual for Transmission Electron Microscope (TEM) is the seventh (7th) edition of its previous versions dated back in June 2011. This edition is intended to reflect the current laboratory operational practices and procedures that meet the requirements for the following: the NVLAP 2006 150 handbook, the ISO/IEC 17025, and the NY ELAP Programs, and the specifications related to the NELAC 2003 and 2009.

The QA/QC manual is revised on a bi-annual basis unless requested otherwise by the lab QA/QC officer in pursuant to the improvement and assurance of the quality of the laboratory operation and quality systems The need of revision, however, is reviewed annually by the management. Modifications/revisions may include: changes in specific laboratory procedures reflecting current practices, responses to changes in accreditation programs (NVAP, EPA, AIHA, etc.), responses to inspections (accreditation agency site visits and comments) and internal audit, methodological changes, and editorial revisions. However, a revision of the laboratory standard operational procedures (SOP) and changes in QC/QA protocols must be approved by the laboratory director and a revision request form shown in **Appendix III** must be filed prior to any changes to be applied. A proved record of this edition is also attached in **Appendix III**. Upon its completion, the manual has to be cosigned by the laboratory director, the laboratory manager, and the laboratory QA/QC officer. Laboratory staff will also have to sign a form of acknowledgement of their having reviewed and/or read through the revision.

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I-III: MODIFICATIONS MADE IN THIS EDITION

Organization Chart

I-IV: OBJECTIVES OF THE QUALITY ASSURANCE PROGRAM

1. To establish quality control procedures that will ensure that data and results generated in the laboratory are within known limits of accuracy and precision.

2. To establish procedures to document and verify that these quality control measures are, in fact, being carried out properly and consistently.

3. To establish procedures which ensure that, at any future date, any results reported to a customer or to a government regulatory agency can be traced to:

   - The date and procedures of sampling
   - The date the analysis was performed
   - The analyst who performed the tests
   - The raw data generated during the performance of the tests
   - The condition of any instrument, reagent, or equipment used in the analysis, at the time it was run
   - The status of the quality control system at the time the test was run

4. To establish procedures that minimize the possibility of erroneous data, or the deliberate falsification of data. Observing the limits derived from control charts and reference standards will do this.

5. To comply with the ISO/IEC 17025 2005 requirements

6. To comply with the NVLAP requirements

7. To establish standards for inter-laboratory comparison

8. To maintain leadership in the industry in terms of quality management system and quality of customer services.

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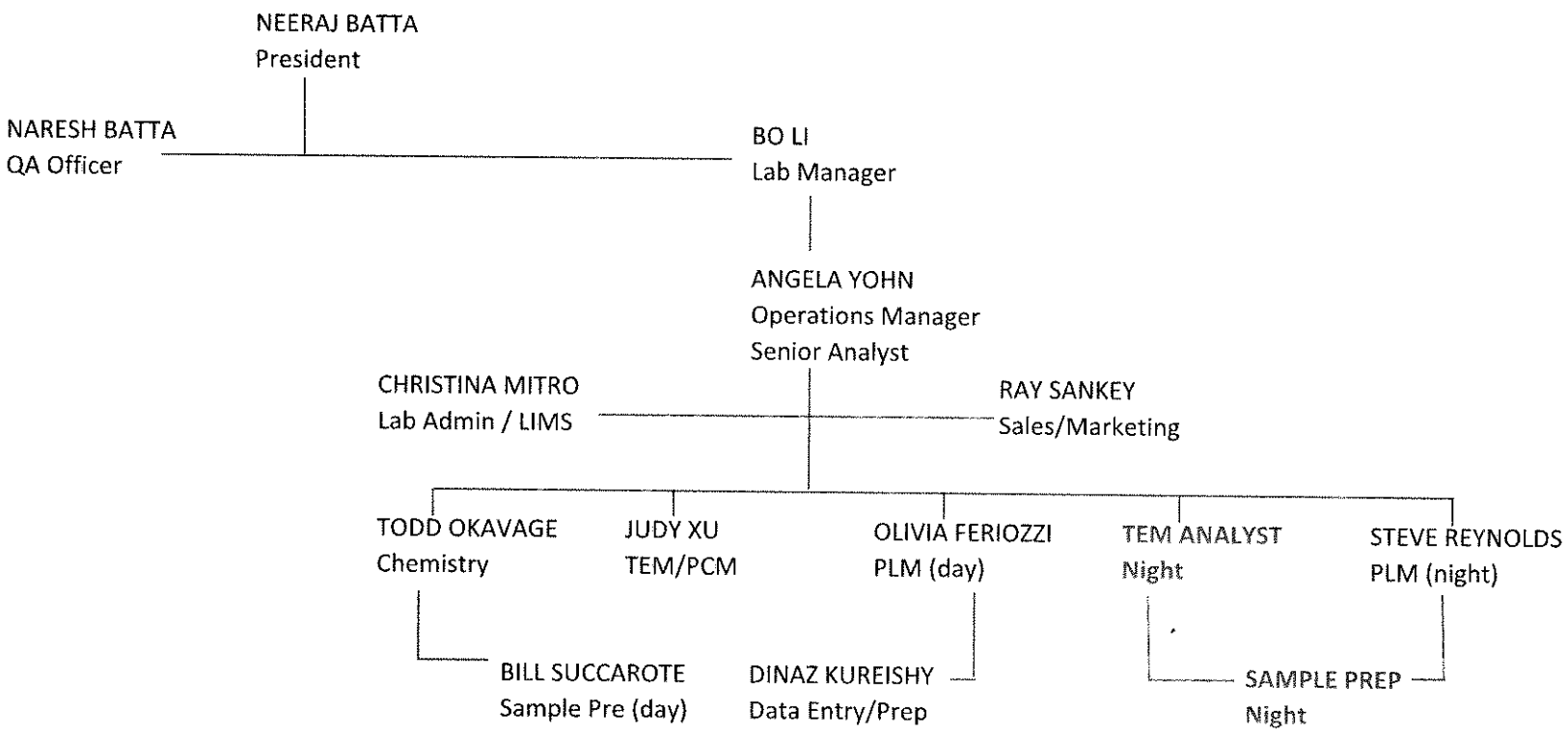
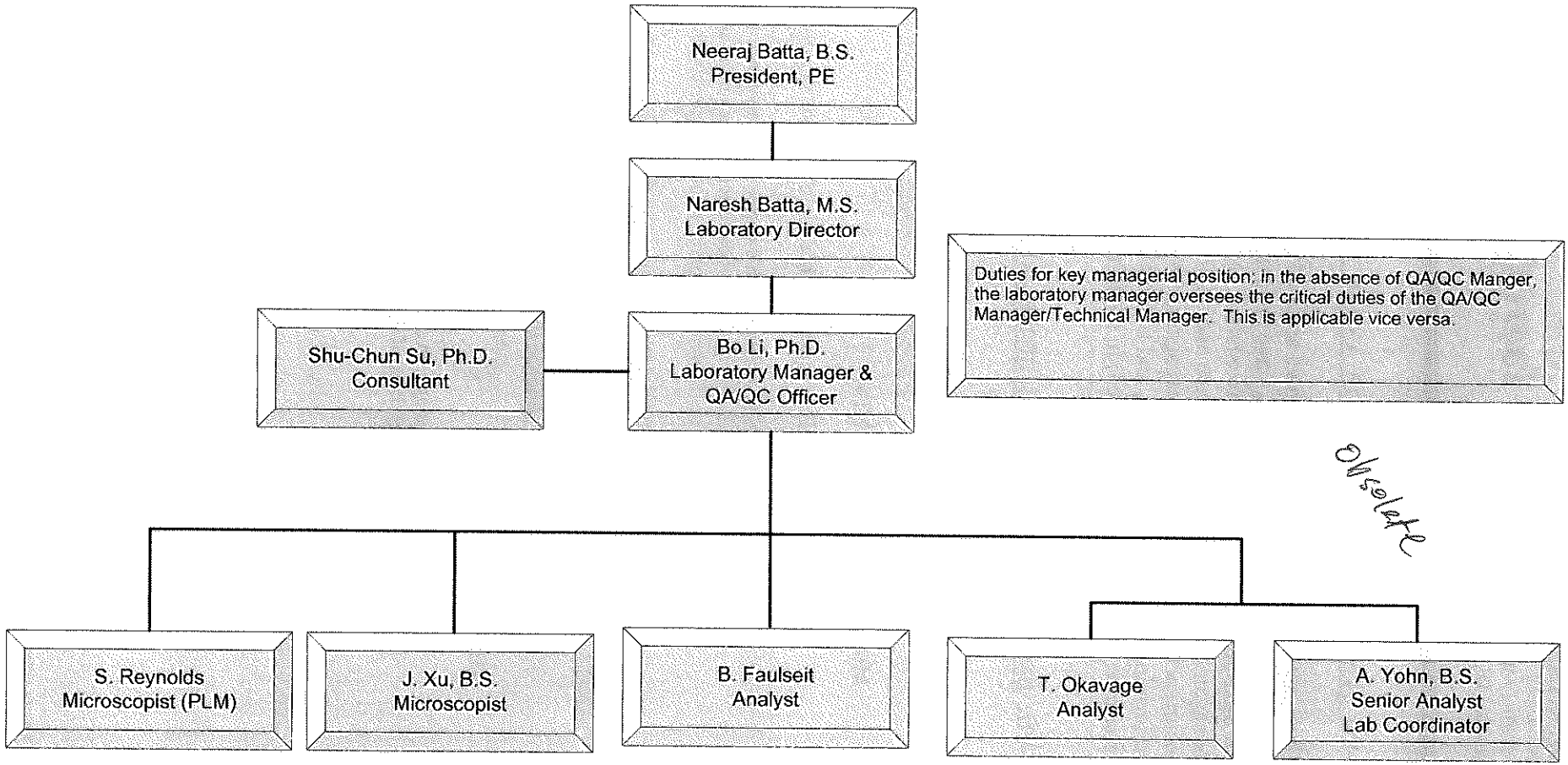
Batta Laboratories, Inc.



Current

Document Control Item QM3

Batta Laboratories, Inc. Organization Chart



I-VI: STATEMENT OF AUTHORITY AND DUTIES

The managerial and technical personnel as listed in the above organization chart, irrespective of other responsibilities, have the authority and due resources needed to carry out their duties, including the implementation, maintenance and improvement of the management system (quality assurance and quality control in specific), and to identify the occurrence of departures from the management system or from the procedures for performing tests and/or calibrations, and to initiate actions to prevent or minimize such departures, as defined (but not limited) in the following section.

I-VII: DECLARATION OF TITLES, DUTIES AND QUALIFICATIONS

LABORATORY DIRECTOR

MINIMUM REQUIREMENTS:

- PhD in science with a major in chemistry or associated science

OR

- MS in Science with a major in chemistry or associated science with six (6) years of experience in a laboratory of which at least three (3) years involved management responsibilities

OR

- BS in science with a major in chemistry or associated science plus eight (8) years of experience in a laboratory of which at least four (4) years involved management responsibilities
- Experience with and/or knowledge of environmental chemistry
- Experience with and/or knowledge of TEM and light microscopy

RESPONSIBILITIES:

- Manage, in conjunction with the Laboratory Manager, the activities of the laboratory

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LABORATORY MANAGER

RESPONSIBLE TO: Laboratory Director

MINIMUM POSITION REQUIREMENTS:

- PhD in Analytical Chemistry, Environmental Science or Geological Sciences plus one (1) year experience in a similar laboratory environment.

OR

- MS in Analytical Chemistry or Environmental Science + four (4) years of experience in an analytical laboratory of which at least two (2) years involved management experience
- Experience with analytical instrumentation (GC, AA, GC/MS)
- Experience with and/or knowledge of electron and light microscopy

DUTIES AND RESPONSIBILITIES:

- Develop and implement new analytical methods appropriate to the laboratory's and/or customer's needs
- Evaluate laboratory materials needs and make recommendations
- Manage laboratory staff by making task assignments, overseeing staff activities and evaluating staff performance
- Responsible for QA/QC procedures, records and reports
- Participate in Proficiency programs associated with field of expertise as may be required to maintain or institute laboratory accreditations and/or certifications
- Interact with potential and current customer
- Attend, as needed, pre-contract meetings with customers and contractors
- Analyze samples using appropriate technology and instrumentation in his or her specialty.
- Prepare reports of results

QA/QC OFFICER

RESPONSIBLE TO: Laboratory Director and Laboratory Manager

MINIMUM POSITION REQUIREMENTS:

- BS in environmental or associated science plus two (2) years of experience in laboratory work including QA/QC functions
- One (1) year experience in all QA/QC activities in an area of laboratory expertise
- Accreditation/certification in a minimum of one discipline for which QA/QC functions are performed

DUTIES AND RESPONSIBILITIES:

- Develop QA/QC programs in accordance with accepted practice and appropriate guidelines to assure that the laboratory produces and results are valid and within acceptable operational and analytical limits
- Maintain all QA/QC records, prepare timely reports and submit required data to accrediting and/or certifying organizations
- Distribute test materials to personnel for analysis in accordance with the QA/QC operational protocols and evaluate results
- Monitor sample handling, storage and tracking
- Identify deficiencies and initiate and monitor corrective procedures
- Review and manage all analytical reports

Controlled

TEM MICROSCOPIST

RESPONSIBLE TO: Laboratory Manager

MINIMUM POSITION REQUIREMENTS:

- Ph.D./MS in Mineralogy/Geology or material science plus 1 year experience in TEM/SEM analysis

- BS in Mineralogy/Geology or material science plus two (2) years of experience in TEM/SEM analysis

OR

- High School Diploma w/minimum 5 years TEM experience plus three (3) years light microscopy experience with proper training or certification

DUTIES AND RESPONSIBILITIES:

- Log in and prepare samples
- Analysis of samples
- Prepare report of results
- PLM or PCM support
- Other duties as assigned

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COORDINATOR/DEPUTY

RESPONSIBLE TO: Laboratory Manager

MINIMUM POSITION REQUIREMENTS:

- At least one year experience in Batta Laboratory that include duties of sample custodian, sample prep, sample analysis, data mining and analysis, and client contacts, etc.
- Good communication skills
- Minimum high school diploma

DUTIES AND RESPONSIBILITIES:

- Client contacts
- Task coordination
- Liaison between lab staff and management
- Support to sample custodian
- Other duties as assigned

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SAMPLE CUSTODIAN

RESPONSIBLE TO: Laboratory Manager

MINIMUM POSITION REQUIREMENTS:

- BS/BA Science + some experience in chemistry or microscopy

OR

- AAS in Science + two (2) years experience in chemistry or microscopy laboratory

OR

- High School Diploma + three (3) years experience in chemistry or microscopy laboratory or experiences that can be counted toward laboratory custodian position

DUTIES AND RESPONSIBILITIES:

Receive samples from customers to include but not be limited to:

- Building and maintaining customer relations through proper customer services practices
- Ensuring completeness of customer chain of custody (should accurately represent samples submitted)
- Signing chain of custody
- Log in of samples into Batta Laboratories sample database
- Release samples and related paperwork to laboratory for analysis
- Enter analytical data into lab database for final reports to include but not be limited to:
- PLM customer data and analyst observations (taken from analytical bench sheet)
- PCM customer data and analyst observations (taken from field sheet or chain of custody)
- Chemistry customer data and analyst results
- Maintain customer database

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- Enter new customer project numbers into Batta Laboratory ---- New Project Logbook for subsequent entry into database
- Maintain daily sample summary logbook
- Log-out Chemistry and TEM samples following completion of analytical report
- Other duties as assigned

I-VIII: PROTOCOLS OF INTERNAL COMMUNICATION AND COMPLAINTS

The company has established detailed procedures for administrative complaints in the employee manual book, which is available in the front office. Also, lab personnel are urged to bring issues (either related to quality, laboratory operations or client relations) in a timely manner to their upper management according to the organization chart, especially when these issues are related to the effectiveness of the management system and the quality of the operation. The purposes of establishing these protocols of communication and complaints are not only to improve the quality of the service, but also to improve the effectiveness of the management system.

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CHAPTER II

SAMPLE RECEIVING AND LOG-IN

II-I: STATEMENT OF PURPOSE

This chapter is to assure the proper handling of the customers' samples upon receipt. The Laboratory Sample Custodian (LSC) is the primary person who will be responsible for the quality of receiving and logging samples into the database by following the procedures listed below.

II-II: STANDARD OPERATION PROCEDURES

I. Check integrity of sample package: The LSC or his (or her) designee will open the original sample package in the negative air HEPA hood (see **Appendix V** for lab layout), which is free of contamination.

Rejection Criteria for Sample Packages

1. Bulk samples and air samples delivered to the analytical laboratory in the same container shall be rejected, or shipped with Styrofoam.
2. If the condition of the package indicates abnormal abuse, wear and/or damage, missing or mislabeled samples the LSC shall contact the customer and record the condition onto the C.O.C. form in the comments section.
3. If the package contains no or incomplete documentation then the LSC shall contact the customer and make record of the absence of documentation onto a C.O.C. form initiated by the LSC. Also reject samples mixed with bulks samples in the same container.

II. Rejection Criteria for AHERA TEM Air Samples

A. Check the conditions of each and every sample (cassette). Reject if any of the following occurs:
   1. Cassette(s) that are obviously damaged
   2. Cassette(s) that have been obviously tampered with
   3. Dislodgment of the filter(s)
   4. Lack of filter(s) in cassette(s)
   5. Lack of cap(s) on cassette(s)
   6. Lack of inlet tabs on cassette(s)

B. Check the customer documentation for each and every sample (cassette). Reject if any of the following occurs:
   1. No customer identification sample number(s) on the cassette(s)
   2. Less than 5 samples identified as inside samples
   3. Less than 5 samples identified as outside samples

4. Less than 3 blanks supplied by the customer:
   a. An identified laboratory blank
   b. An identified inside the work area blank
   c. An identified outside the work area blank
5. Less than 560 liters per air sample(s)

If the previously mentioned are not met, the LSC shall contact the customer and record the condition onto the customer's C.O.C form in the comments section.

III. Chain of Custody Form

A. Have the person who delivered the shipment sign the chain of custody.

A. If the number differs from that on the customer's authorization or data sheet, notify the LSC to contact the customer.

B. If samples are received through the postal services, the postal receipt, air bills, forms and other related documents might be kept for records if requested by the customers. A formal Batta Laboratories' COC is still needed to be completed as usual regardless of the aforementioned postal records. For state and federal related projects, it is mandatory to keep and document all postal records as part of the COC.

C. The person who signs off the COC must be alert on the information provided on the COC. Information that is related to customer's request, tenders or contract may be shown on the COC. Differences or discrepancies noted on the COC must be notified to laboratory Manager or Director for further clarification. Refer to **Section IV-XVI** of **Chapter IV** for further information.

IV. Log In

A. Check the sampling data on each cassette and the customer's data sheet for consistency.

B. Log in all sampling data into the BEA main lab database.

C. BLI lab sample numbers (for instance, 453790 through 453799) will be assigned to each field sample on the COC. On each page of the COC, the first lab sample number must be written as an entire lab number (453790 in this case) for the first customers' sample and the rest on the same page will be assigned with at least the last three digits of the lab number for convenience and work efficiency. However, the written numbers must be legible and consistent for the same COC, and the ink color must be different from customers' identification numbers. However, on the sample container, a BLI TEM job number (a four digits number) shall be written on the sample bag or container.

D. Enter "NA" on the C.O.C for information that is not given by the customer. However, the information on the C.O.C replaced with "NA" should not constitute part of the rejection criteria stated elsewhere in this section.

V. Initiate Project Folder and Fill in Lab Transmittal Sheets

A. Initiate project folder by signing a sequential number with reference to a prior job. Lab L-number, BEA number (if available), customer name, type of analysis, date of receipt and project location have to be shown on the folder tab for future references.

B. There are two transmittal sheets: COC transmittal sheet and sample prep transmittal sheet (see **Appendix III**). Both have to be filed within the project folder together with customer data/records and lab sheets.

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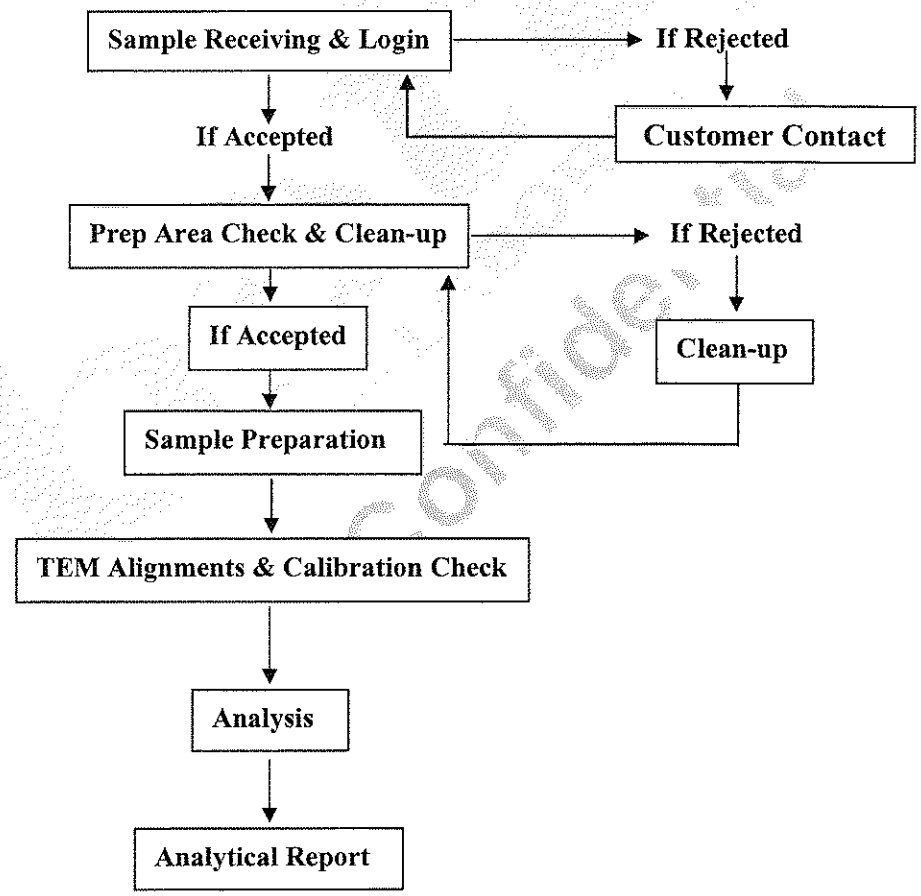
CHAPTER III

TEM ANALYSIS STANDARD OPERATION PROCEDURE

III-I: STATEMENT OF PURPOSE

This chapter is meant to assist analysts or TEM operators to better comply with all accreditation programs in which this lab has been participating, and to assure the consistency among analysts for higher achievement in data quality.

III-II: ANALYTICAL PARADIGM



III-III: SAMPLE RECEIVING AND LOGIN

Although sample receiving and login are the primary tasks by the sample custodian, analysts may have to carry out these tasks if necessary. Follow the procedures outlined in **Chapter II** to complete these tasks.

III-IV: PREP AREA CHECK AND CLEAN-UP

The positive air HEPA hood (see **Appendix V**) needs to be checked and cleaned for air sample preparation. The purpose of this step is to assure a free-of-contamination environment during sample preparation. Do not occupy the prep area with unnecessary items, and items that are present in the hood with samples to be prepped have to be safeguarded so as cross-contamination is kept at the allowable minimum. The following check list has to be brainstormed by the analyst before preparation:

1. Have the TEM lab contamination checks been done recently?
2. Have the reagent purity checks been done recently?
3. Have the tools that may have direct contact with the sample/filters been cleaned?

If any of the above listed occur aversively, corrective actions have to be taken prior to proceeding sample preparation and analysis. Items 1 and 2 are conducted twice (2) a year and updated information is available via database, or by consulting the QC/QA officer or the laboratory supervisor. Item 3 is enforced prior to each individual customer sample preparation.

III-V: SAMPLE PREPARATION

Methods of sample preparation vary with the analytical types, such as AHERA, bulk, soil and water. The following procedures are general steps regardless of analytical types. However, analysts are asked to abide by a chosen analytical method or SOP whenever and wherever conflict occurs (refer to Appendix IV for common analytical methods and SOPs).

I. Open sample packages inside a negative air HEPA hood.
II. Prep the bulk samples in the designated bulk prep area, and air samples inside the designated air-prep area (refer to lab layout in **Appendix V**). Follow the standard operation procedure of an analytical method in **Appendix IV** if a secondary filter is needed prior to analysis.
III. For AHERA and other air or vacuum samples, wet-wipe the cassettes to be analyzed, and return them into a separate clean bag after sample preparation. ID the new bag the same way as the original bag (refer to **Chapter II**). Keep both bags together as one package.
IV. Prep the sample filter substrate on a glass slide inside a positive air HEPA hood. Follow the SOP for MCE filter preparation if necessary (**Section VI-II** of **Appendix VI**).
V. Ash the sample substrate by referring to the SOP in **Section VI-I of Appendix VI** if required by the analytical method. If the Plasma Asher is not calibrated on

schedule, calibrate the etching time before using it on customers' samples. Refer to **Section VII-IV** of **Appendix VII** for instructions on the etching time calibration.

VI. Carbon-coat the sample substrate following the SOP in **Section VI-IV of Appendix VI** if required by the analytical method.

VII. Prep TEM grids for final analyses. Follow the SOP in **Section VI-IX of Appendix VI** if only MCE filters are used, together with **Section VI-X of Appendix VI** if PC filters used.

III-VI: TEM SCOPE ALIGNMENTS AND CALIBRATION CHECK

Before the analysis of customers' samples, the readiness of the TEM scope has to be checked. The following routines need to be verified and actions must be taken prior to analysis if any of these is not carried out according to schedules outlined in **Chapter VI: Quality Control and Quality Assurances Programs**. Records of these routines are currently maintained by the laboratory QA/QC officer.

I. Daily Routines:
   - A. Electron Beam Generation (**JEOL Method A, Section 5.2.5**, P5-15).
   - B. Condenser Lens Alignment (**JEOL Method A, Section 5.2.5**, P5-17).
   - C. Beam Centering Alignment (**JEOL Method B, Section 5.3.6**, P5-49).
   - D. Electron Gun Alignment (**JEOL Method B, Section 5.3.3**, P5-42).
   - E. Checking Condenser Lens Astigmation (**JEOL Method B, Section 5.3.2**, P5-40).
   - F. Checking Objective Lens Displacement (**JEOL Method B, Section 5.3.4**, P5-45).
   - G. Energy dispersive X-Ray Cu line check (**Section VII-X** of **Appendix VII**).

II. Weekly Routines: Energy Dispersive X-Ray Cu and Al Calibration (**Section VII-XI** of **Appendix VII**).

III. Monthly Routines:
   - A. TEM Spot Size Calibration (**Section VII-III** of **Appendix VII**).
   - B. D-space Measurements from the TEM Fluorescent Screen Calibration (**Section VII-VI** of **Appendix VII**).
   - C. Camera Constant Calibration (**Section VII-VII** of **Appendix VII**).
   - D. EDXA Mn K Alpha Peak Resolution Measurement (**Section VII-VIII** of **Appendix VII**).

IV. Quarterly Routines: Not available for the time being.

V. Semi-annual Routines: Chrysotile Beam Dose Calibration (**Section VII-V** of **Appendix VII**).

VI. Annual Routines: Not available for the time being.

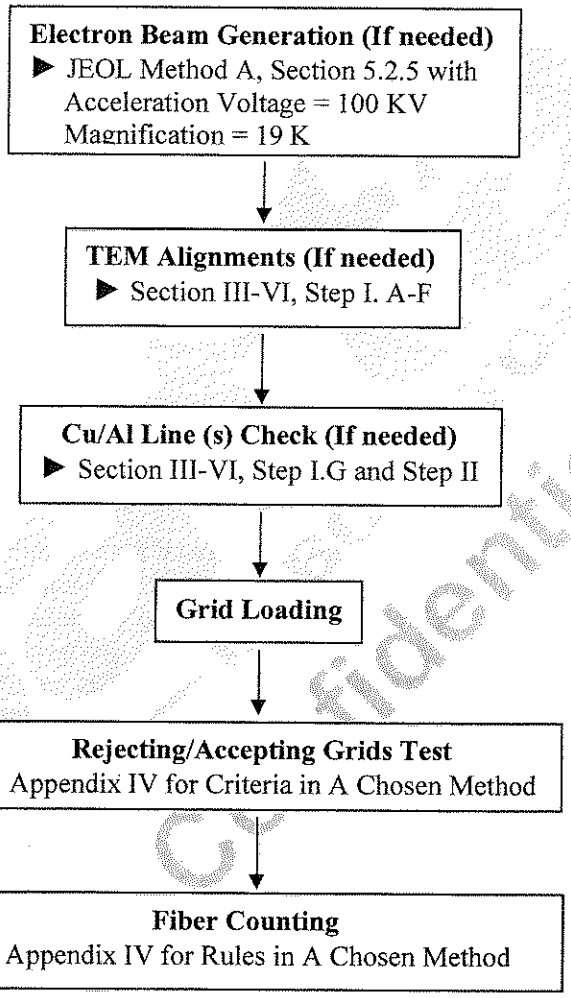
VII. Bi-annual Routines: EDXA Relative Sensitivity K-factors for Mg, Si, Ca, And Fe (**Section VII-IX** of **Appendix VII**).

III-VII: ANALYSIS

Analytical methods currently adopted by BLI and procedures of carrying out these analyses are detailed in their respective methods listed in **Appendix IV**. The following

procedures are general guidelines to be used in critical reference to a pertinent methodology shown in **Appendix IV**. The analysts are strongly recommended to refer to a referenced method or SOP whenever and wherever necessary.

A. The Paradigm



B. Rejecting/Accepting Grids Test

Particulate loading and carbon replica quality are evaluated to either reject or accept a grid (or a grid opening). The following rules have to be followed unless specified otherwise in a specific analytical method or by a certain customer.

I. Particulate Loading:
   1. If the entire grids are unevenly loaded, reject the grid and load the next grid sample.
   2. If the grid opening (G.O.) is unevenly loaded, proceed to next G.O.
   3. If the particulate loading is greater than (>) 10%, reject the G.O. and move to next G.O. (**NVLAP Lab Bulletin# LB-7-2002**).
   4. If the entire grid sample loading is greater than (>) 10%, reject the grid and load next grid.

II. Carbon replica quality:
   A. Reject if grid replica coverage is ≤ 50%. If it is a G.O., move to next G.O. and repeat the same.
   B. Reject if un-dissolved filter area ≥ 10%. If it is a G.O., move to next G.O. and repeat the same.
   C. Reject if opaque area ≥ 5%. If it is a G.O., move to next one and repeat the same.
   E. Reject if folded/overlapping replica is ≥ 50%. If it is a G.O., move to next one and repeat the same.

C. Fiber Counting

TEM fiber counting is conducted based on specific counting rules that are outlined in a pertinent method such as AHERA. Refer to **Appendix IV** for counting rules of a specific method. However, the following has to be decided before counting begins:

1. Counting Rules: Refer to **Appendix IV** for a specific method.
2. Analytical Sensitivity and Number of Grid Openings to Be Analyzed: Each method has requirements on how the grid should be analyzed. There are several analytical templates available to help analysts to calculate the above parameters before analyzing samples. These templates are report-writing templates that include AHERA, Philly Regs, Water, etc. Please consult the microscopy supervisor for locating these templates. Although the aforementioned templates were designed to automatically calculate the analytical sensitivity and number of GOs to read, analysts are encouraged to manually calculate them if needed.
3. Rules on Fiber Identification: These include special requirements on SAED, EDS, morphology, etc.
4. Fiber Documentation: A TEM countsheet (▶ **Appendix III**) has been developed to include physical (length, width, morphology, SAED, etc.) and chemical properties (EDS). Refer to a pertinent method for detailed rules on fiber counting.
5. Stopping Rules: Refer to a pertinent method for details (▶ **Appendix IV**).

III-VIII: ANALYTICAL REPORT

The report writing has been automated through an Excel application for various TEM analytical methods, such as AHERA. These report templates are accessible to all TEM analysts, and currently located at: T:\TEM Analysis. Each method is included into a

subfolder prefixed with the method name. Seek the microscopy supervisor's guidance if necessary.
The template will generate three types of files: countsheets, TEM alignment sheets, and a report certificate. The alignment sheets will stay with the database and a hardcopy may be released with the report certificate upon request.

The primary report delivered to customers includes: a certificate, all countsheets, original C.O.C., and attachments. Attachments are documents to be released only upon customers' request and may include: communication document, postal receipt, analysts' notes, a copy of methods, etc. For quality control and assurances on these documents, please refer to **Chapter IV** and **Chapter V** for sections on Data Review. Refer to **Appendix III** for examples of TEM certificates and countsheets.

III-IX: USING OR 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 the reports that describe only testing or calibration within the scope of accreditation. The term NVLAP and the NVLAP logo, including the BLI NVLAP code is strictly excluded from any personal use or being shown on non-business related paper or digital documents. When referenced for the laboratory's accredited status, the term NVLAP shall be accompanied by the NVLAP Lab Code, which is 101032-0. Whenever not certain, the person who needs to cite the NVLAP term should either consult with the laboratory QC/QA officer, or directly to the NIST 150 handbook (2006 edition) Annex A for details.

III-X: PROFICIENCY TESTING AND SAMPLE HANDLING

Treat proficiency testing (PT) samples as client samples except that log-in is optional. Recording of SAED and EDX is optional. All analysts have to participate (either prior to or after the official reporting date) in the same round of PT analysis; however, analysts will not postpone later than the next PT round. Analyst will not share results with others unless all participants completed and submitted their analytical results.

Lab may choose from past PT samples for standard reference analysis/verified analysis and round-robin analysis. When chosen as reference standard analysis, identity of the analysis shall be concealed from the analysts for known results or count ranges.

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CHAPTER IV

QUALITY CONTROL AND QUALITY ASSURANCE PROGRAMS

IV-I: GENERAL STATEMENT

BLI's data quality is assured through the following programs, operations and policies: national accreditation programs, staff training and monitoring program, proficiency test and inter-laboratory analysis, instrument maintenance, routine alignments and calibrations, protocol of customer contact, protocol of corrective and preventive actions, protocol of laboratory SOP revision, laboratory background contamination monitoring, internal reviews and QC/QA analyses. Data records of the above are kept in designated locations, some of which may be only accessed by the laboratory manager and the QC/QA officer. For record access, refer to each of the following pertinent sections or **Chapter V** on **Data Management**.

IV-II: ACCREDITATION PROGRAMS

BLI is accredited through the following agencies: NVLAP (NVLAP# 101032), AIHA (AIHA# 100448), and EPA (EPA ID# DE004). Although AIHA is not directly involved with BLI's TEM analysis, documentation of TEM QC/QA was often requested as part of the AIHA accreditation process. BLI started its participation for EPA inspection in year 2004 (February 10 and 11, 2004) for the laboratory's compliance with EPA regulations and BLI's compliance with NVLAP regulations. BLI has been accredited with NVLAP since 1986. This edition of TEM SOP (including QC/QA Programs) and sections provided in this chapter are reflections of BLI's compliance with all concurrent regulatory guidelines, including those described in NVLAP's NIST Handbook 150: Procedures and General Requirements (2001 Edition) and NIST Handbook 150-13: Airborne Asbestos Analysis (1995 Edition), whichever is up to date.

IV-III: PROFICIENCY TEST AND INTER-LABORATORY ANALYSIS

There are three proficiency tests currently participated by this lab: NIST proficiency test (through NVLAP), Round Robin (inter-laboratory), and Water Analysis (through NY ELAP). Round Robin is performed at minimum once (1) per year among participating laboratories (SanAir and several other labs that are randomly picked by a specific project manager of EPA or of EPA related agencies). Water Analysis is provided twice (2) a year by NY ELAP. All above proficiency analyses have their own separate record binders located in TEM lab archive shelf for references (see the lab layout view in **Appendix V**), and are also summarized in their perspective monthly sample summaries.

IV-IV: DATA REVIEW

1. Laboratory sample custodians and analysts are responsible for the data accuracy during the sample receiving and analytical process. These procedures are detailed in Chapter II for sample receiving and Chapter III for sample analysis.

2. Data entry person or the analysts have to enter the data as it is on the analytical sheets or benchsheets. It is mandatory for each analyst or data entry person to review the data before it is submitted to a higher level for QC/QA purposes.

3. The laboratory QC/QA officer, managers will review submitted reports or other data forms and check them against original C.O.C and original analytical data sheets or benchsheets.

4. Discrepancies found during the data review or QC/QA processes will be checked for sources of errors. Final laboratory results may be submitted to the customer when all error sources are clarified and corrected.

IV-V: STAFF TRAINING AND PERFORMANCE MONITORING

BLI's has developed a rigorous program to assure the quality of analysts' performance, as detailed in **Chapter VI**. This program includes semi-annual reference standard material analyses, verified analyses, re-analyses and recounts on previous analyzed grid openings and on re-prepped grid openings, staff training and QC/QA analysis on analysts' statistical data.

Discrepancies found during processes of data review and re-analyses are evaluated by the QC/QA officer who is to conduct the QC/QA statistical data analysis (currently by the microscopy supervisor). If the discrepancies or the results of the re-analyses alter the nature of previous analytical conclusions, such as fail or pass, positive or negative, the re-analysis will be conducted and verified by an analyst designated by the QC/QA officer. By all means of verification and clarification such as lab background blank check, re-prep, etc., the customer will be informed with explanation and a new certificate if the investigation still alters the nature of previous results. An internal memo will be issued and copied to the laboratory director with detailed explanation of the occurrence and measures to be taken to prevent future similar occurrences. Memos will be documented in corresponding TEM Monthly Summaries (see **Appendix VIII**).

IV-VI: ROUTINE ALIGNMENTS AND CALIBRATIONS

BLI's routine alignments and calibrations for TEM analysis include:

1. TEM scope alignments such as beam centering beam saturation, gun alignments, image wobbler, etc. These tasks are performed before each analysis.
2. Energy dispersive X-ray Cu line check (daily).
3. Energy dispersive X-ray Cu and Al calibration (weekly).
4. TEM spot size calibration (monthly).
5. D-space measurements from fluorescent screen (monthly).
6. Camera constant calibration (monthly).

7. EDXA Mon K alpha peak resolution measurement (monthly).
8. TEM beam dose calibration (every 6-month)
9. Plasma asher MCE filter etching time calibration (quarterly)
10. EDXA relative sensitivity K-factors for Mg, Si, Ca and Fe (bi-annually).

IV-VII: PROTOCOL OF CUSTOMER CONTACT AND CONFIDENTIALITY

The primary contact with the customer is directed to the laboratory manager or the lab supervisor. The sample custodian may contact the customer directly for matters defined in the custodian's duty section in **Chapter I**. Analysts may contact directly with the customer under the authorization by the upper level chain of command. All billing inquiries and price quotes should be directed to the lab manager or the accounting office. Under no circumstances should customers' information be released to a third party without authorization or consent of the parties involved.

IV-VIII: INTERNAL AUDIT

BLI's internal audit is conducted annually to review the laboratories compliance with QC/QA programs and regulations. Currently the NVLAP Handbook 150 and its attached checklist are the guidelines for BLI's TEM overall QC/QA compliance. A review report is kept within the folder under NVLAP Inspection located the TEM lab (see **Lab Layout** of **Appendix V**).

IV-IX: BACKGROUND CONTAMINATION MONITORING

BLI carries out two types of monitoring on background contamination: passive and active. The passive contamination check is done by placing TEM blank filters in three strategic areas – Prep Hood #1, Prep Hood #2 and TEM Prep Area (see **Appendix V** for a lab layout view); the active contamination check is done by setting three air pumps in the PLM analytical area, the bulk prep area and the air prep area. Filters are prepped for TEM analysis following the AHERA Method (**Appendix IV**). Documents related to the above analysis are complied with the monthly summary reports. The passive monitoring is conducted twice (2) a year, while the other one is quarterly. Refer to **Sections VI-XI & XII** of **Appendix VI** on how to conduct both types of contamination monitoring.

IV-X: REAGENT PURITY CHECK

There are three common reagents used in this lab: acetone, ethyl alcohol, and distilled water. These reagents are checked for contamination semi-annually. Analytical results are documented with monthly summaries. Refer to **Section VI-XIII** of **Appendix VI** for details.

IV-XI: QC/QA ANALYSIS

The following QC/QA analyses are currently conducted by BLI: re-analysis (including re-prep analysis) and laboratory blank analysis. Re-analyses are conducted for 10% of all AHERA samples analyzed for a period of time. Laboratory blank analysis, following the AHERA method on blank analysis, is conducted for 4% of the total AHERA samples

analyzed. Statistics on all the above mentioned analyses, including analyses on individual training purposes outlined in Chapter VI (such as verified analysis, standard reference analysis, etc.), is conducted monthly by the QC/QA officer or lab manager. All these QC/QA data are filed within the monthly reports.

IV-XII: MONTHLY SUMMARY

The BLI's monthly summary of TEM analysis will include all QC/QA data that occur within the same month. The monthly summary may contain the following items:

1. A factorial sheet or a checklist of contents
2. Monthly TEM QC report
3. Reanalysis data sheets
4. Sealed, set, failure and prep blank analysis
5. Verified asbestos analysis
6. NVLAP proficiency/Round Robins
7. Quarterly air monitoring (TEM and bulk prep areas)
8. Semi-annual passive contamination blanks
9. Annual standard reference material analysis
10. Monthly report for TEM calibrations
11. EDX calibration micrographs
12. Deficiencies & corrections

The monthly summary of Feb. 04, 2004 was attached as **Appendix VIII**.

IV-XIII: CUSTOMER COMPLAINTS AND FEEDBACKS

All customer complaints should be directed to the laboratory director or manger for proper handling and actions. Corrective and preventive actions shall be taken if the complaints are directly related to laboratory operations and the quality system. Complaints related to billing and services that are not directly related to or belonging to the realm of laboratory of services should be timely forwarded to responsible personnel or departments.

Laboratory director and mangers are required to monitor the feedbacks from customers in order to improve the laboratory quality system. Questionnaires or e-mails will be sent to customers on a regular basis regarding to the quality of services and the quality of data provided. These feedback data will be compiled by the QC/QA officer and presented to the board of the laboratory management system for review. Corrective or preventive actions will be taken in response to any actual or potential nonconformities, noncompliance or complaints reported by customers.

IV-XIV: CORRECTIVE AND PREVENTIVE ACTIONS

Both corrective and preventive actions shall be taken should nonconformity, noncompliance, errors, customer complaints or misconduct occur.

Corrective actions refers to actions taken to correct or eliminate the cause of **an existing** nonconformity, noncompliance, errors in data produced, defects or complaints in customer service or other undesirable situation in order to prevent their reoccurrence. Case-specific procedures of corrective actions are in effect to deal with aforementioned unconformities or errors. General procedures for all cases include:

1. Review all original C.O.Cs, reports and communications for errors.
2. Investigate/verify the nature of the error or nonconformity
3. Correct the errors and contact the customers for corrections
4. Revise report packages
5. Investigate sources of errors and causes
6. Conduct error or nonconformity analysis if deemed by the laboratory QC/QA officer
7. Report of the findings to the laboratory director or QC/QA officer
8. Decision or plan for preventive actions

Preventive actions refers to actions to correct or eliminate the causes of **a potential** nonconformity, noncompliance, errors in data produced, defects or complaints in customer service or other undesirable situation in order to prevent their occurrence. Preventive action is actually a proactive process to identify opportunities for improvement rather than a reaction to the identification of problems or complaints.

The preventive actions in BLI currently consists of annual management review, internal audit, monthly lab meetings, proficiency testing (NVLAP and Round-Robins), review on customer feedbacks and complaints, QC/QA data analysis and control charting, annual staff training, SOP revision, etc. General procedure for the preventive action shall follow:

1. Compiling data or findings through one or more of the above-mentioned process.
2. Analyzing the data and charting the trend
3. Evaluate the risk of potential nonconformities
4. Developing a corrective action plan
5. Submit the plan for management review
6. Executing the approved corrective action plan

IV-XV: LAB MEETINGS AND MANAGEMENT REVIEW

In accordance with NVLAP newly implemented requirement, BLI's top management will conduct a review of the laboratory's management system and testing and/or calibration activities to ensure BLI's continuing suitability and effectiveness, and to introduce necessary changes or improvement. This management review shall be conducted at least yearly besides regular lab meetings that have already been scheduled. This management review will include, but not be limited to the following aspects of interests:

1. the suitability of policies and procedures
2. reports from managerial and supervisory personal
3. the outcome of recent internal audits
4. corrective and preventive actions
5. assessments by external bodies
6. the results of inter-laboratory comparisons or proficiency tests
7. changes in the volume and type of the work

8. customer feedback
9. complains
10. recommendations for improvement
11. corrective and preventive actions
12. other relevant factors, such as quality control activities, resources and staff training

The above management review will be summarized in a written form and circulated among the top management board. Original copies will be filed with monthly summaries.

IV-XVI: PROCEDURES FOR REVIEW OF REQUESTS, TENDERS AND CONTRACTS

All laboratory contracts and tenders or proposals will be reviewed by the lab director or manger against the request for proposal (RFP). Copy of RFP, proposals and contracts are provided to accounting office. Most of contracts are presented in written format with lab's terms and conditions. In case of walk-in clients, the chain of custody is used as legal agreement. Following steps are taken in review of contacts and proposals:

1. Testing methods are adequately defined by the tenders.
2. The lab verified that it has the adequate resources in meeting the requirements.
3. Any differences between RFP or tenders and the final contracts shall be resolved before commencing any work.
4. Records of reviews including discussion with customer, any subcontract work and any significant changes shall be properly maintained.
5. The customer will be informed of any deviation from the contract.
6. A change order will be initiated if contract is amended after work has commenced.

IV-XVII: MEASUREMENT TRACEABILITY AND CONTROL CHARTS

Control charts are constructed to show calibration values or analysts' performance values vs. time, the magnitude of their variation, and the allowable limits of variation. The magnitude of variation specified for many of the control charts used for calibrations and performances is usually defined as 2 times of the standard deviation of a test data set otherwise specified in a specific calibration method defined in perspective calibration routines as shown in the QA database for instrument calibrations located in T: \\QA-QC Program\QAQC Data.

All calibration standard and instruments are either traceable to their original manufacturer data or to recently calibrated data/certificates issued by certified calibration personnel. These traceability data are stored inside the TEM lab. One should consult with the lab QC/QA officer or laboratory manager if requesting of these data/certificates is needed.

CHAPTER V

DATA MANAGEMENT

V-I: STATEMENT OF PURPOSE

To assure the quality of the analytical results and appropriate maintenance of the normal operation of the TEM and its accessories, good practices are needed to manage all the above related data and records. The QC/QA officer is the primary person of contact for record locating and retrieving.

V-II: DATA ARCHIVING

The data management in this section includes data storage, record keeping, data archiving, data handling and data/record disposal.

1. The electronic data such as analytical data, certificates, e-mails, etc. are kept in the laboratory's server system. A tree system in the server was established for storage of different data categories. Analysts and other lab personnel will have to work with the company's system administrator on questions on how to appropriately store or retrieve data from the central system.

2. Temporary storage of the above categories (listed in **Section V-I**) is permitted at individual workstations. These data are backed up daily to the central server system. The System Administrator then backs up the above referenced data on types, CDs or other data storage media on a daily basis.

3. Training data and personal QC/QA records will be documented into binders under each analyst' name, and are kept on the shelves in the TEM lab. Records older than three years are kept in the designated storage room outside the laboratory. These older records are positioned in the storage room based on the year. The lab QC/QA officer or mangers are in charge of appropriate storage of old data in the storage room.

4. Data and documents related to participation of accreditation programs such as NVLAP proficiency tests, Round Robins, etc. are kept in the monthly summaries file binders. These binders are located on the backroom shelves in the TEM lab.

5. References for analytical methods, SOPs and technical manuals are kept on the rear shelves in the TEM lab.

6. The laboratory computing network and central server are under a maintenance contract with SSD, Inc. Electronic data are backed up daily by the central server on a daily basis. Data restoration in emergency cases is carried out by SSD personel upon request.

V-III: DATA PACKAGE ASSEMBLY

1. Data packages in hardcopy forms currently available in this laboratory include: client report packages, TEM logbooks, training records, monthly summary reports, communications and contacts, accreditation programs packages, and references.

2. The client report package normally contains: the original analytical report, copies of original C.O.C, analytical count sheets, shipping records, communication and contacts history and other addenda. The laboratory package will include a copy of the analytical report and originals of the above referenced addenda, such as original C.O.C, shipping records, etc. In some cases, lab may only retain a copy of the COC when clients request to have their original COC returned. All hardcopies are manually scanned and stored in the central server system.

3. Monthly Summaries (see **Chapter IV** or **Appendix VIII**):
   TEM monthly summaries may include:
   1. A factorial sheet or a checklist of the contents
   2. Monthly TEM QC report
   3. Reanalysis data sheets
   4. Sealed, set, failure and prep blank analysis
   5. Verified asbestos analysis
   6. NVLAP proficiency/Round Robins
   7. Quarterly air monitoring (bulk and air prep areas)
   8. Semi-annual passive contamination blanks
   9. Annual standard reference material analysis
   10. Monthly report for TEM calibrations
   11. EDX calibration micrographs

V-IV: DATA/RECORD DISPOSAL

Both electronic data and hardcopy data are kept for a minimum of three (3) years. At disposal, electronic data will be purged by SSD computer personal using adequate security protocols and methods to ensure the permanent data erase to ensure that the data record cannot be restored or reused after disposal. Hardcopy records will be disposed by shredding to protect client identity and data security. A data disposal manifesto has to be signed and released by the disposal personnel to the laboratory manager for record.

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CHAPTER VI

QUALITY CONTROL ON ANALYSTS' PERFORMANCE

VI-I: STATEMENT OF PURPOSE

BLI maintains a rigorous program to monitor the analysts' performance in properly following the SOP for a certain analysis and delivering the results within allowable accuracy. The following are conducted at regular basis to assurance the quality of each individual's performance.

VI-II: STANDARD REFERENCE MATERIAL ANALYSIS (SEMI-ANNUAL)

The following standard reference materials are analyzed and charted on a semi-annual basis. Each analyst will participate in the analysis of the standard materials using verified asbestos analysis protocol outlined in Airborne Asbestos Method: Standard Method for Verified Analysis of Asbestos by Transmission Electron Microscopy – V2.0 (**Appendix IV**). Results are compiled by the lab manager or QC/QA officer and kept in the designated database location for future retrieval.

SRM 1876a

The laboratory obtains mean analytical results on SRM 1876a, which fall within 80% of the 95% confidence limits as published on the certificate.

SRM 1876a is analyzed to determine fiber density (s/mm^2). It is also used to train new analysts in identifying fibrous structures that consist mainly of single fibers.

SRM 1866

The laboratory's analytical criteria and instrumental/operator analysis conditions for asbestos identification will correctly classify $\geq$ 90% of asbestos structures to ensure that false negatives are not resulting from misidentification of asbestos as non-asbestos structures (both bundles and individual fibers.)

SRM 1866 is analyzed to determine the number of structures identified and fiber density (s/mm^2). It is also used to train new analysts in identifying fibrous structures that consist mainly of bundles and single fibers.

SRM 1866 is retained with the PLM and PCM standards within the laboratory. This material is an optional source of TEM analytical reference. A material safety data sheet and a certificate of materials composition is located in the folder labeled as "Laboratory Standard Materials" in the file storage cabinet (marked as Bin# 2 in the lab layout in Appendix V)

Four types of fibrous materials are contained in NIST 1866.

1. Chrysotile
2. Grunerite (Amosite)
3. Riebeckite (Crocidolite).
4. Glass Fiber

VI-III: TECHNICAL REVIEW

TEM analysts are recommended to constantly review the SOPs to assure that their routine analyses follow the guidelines described in a pertinent SOP. Although this is only a recommended option, the QC/QA officer or the lab manager may ask a specific question any time regarding a certain analysis to assure the consistency of the analysts' procedure with those written in the SOP. Corrective actions will be taken if discrepancies occur.

V-IV: ANNUAL INTERNAL REVIEW

In order to fully comply with the NVLAP programs and to assure the best analytical quantity, the internal review has been conducted annually. The major task of this review is to check the laboratory's current practice against the BLI's SOPs to assure quality appliance with the most current regulations and participating quality control/quality assurance programs. During this review, analysts will be checked and examined for compliance with the SOPs in effect. Documentation of such a review is available in the current file storage bin (Bin #2 on the Lab Layout View in Appendix V). A revision of previous edition of SOPs will take palace if necessary. A request form of SOP revision is shown in Appendix III and this form has to be approved by the laboratory director or the QC/QA officer prior to any SOP revision.

VI-V: STAFF TRAINING

Analysts are encouraged to keep their knowledge current with the newest advances in the technology. The laboratory director, QC/QA and laboratory managers are obligated to circulate the most updated information among the analysts if they are available. A new analyst will follow the guidelines described in the next section.

VII-VI: TRAINING GUIDELINES FOR A NEW ANALYST

TEM Analysts are trained by supervision and designated instructors following the laboratory standard operation procedures and methodologies for various TEM analyses. The course will cover the principles of transmission electron microscopy, crystallography on regulated asbestos and their look-likes, regulations and methodologies, standard operation procedures for related analysis and trainees' statistics build-up. It is BLI's policy that no trainees will operate the TEM without supervision of a designated qualified TEM analyst.

The accompanying supervisor will document each training session and its progress in the individual's trainees' logbook. The following shall appear on the logbook:

- Name of person being trained
- Date
- Time on
- Time off
- Sample number (if applicable)
- Task performed
- Comments/progress
- Supervisor's initials
- Trainee's initials

Qualification of Instructors and Supervisors

BLI requires that an instructor must have at least 2 years experience in the asbestos industries of TEM analysis. The supervisor can be the laboratory manager, QC/QA officer, or their deputies who are familiar with the laboratory organization, regulatory policies, QC/QA procedures and laboratory SOPs. Currently, the BLI's TEM training team includes Dr. Bo Li and Dr. Shu-chun Su. Dr. Li has a geology degree and has at least 5 years in the asbestos industries of both TEM and PLM analyses, and is currently the PLM and TEM section manager. Dr. Su is the company consultant for TEM, PLM and related asbestos analysis programs. Refer to Appendix I for their respective biographies.

Training Tasks

The following were included in the training program.

A. Calibration, alignment and micrograph processing using the JEOL JEM-100CXII Transmission Electron Microscope.
B. Acquisition and interpretation of X-ray analysis using the ATEM and the Kevex Delta 1 Analyzer.
C. Measurement and interpretation of electron diffraction patterns.
D. Asbestos counting rules for single fibers and complex structures.
E. Asbestos counting methodology using the following:
    - Grid and grid opening nonadjacent and semi-random selection.
    - X - Y stage translation and parallel traverses
    - Stage positioning and repositioning
F. Asbestos identifier requirements for morphological, SAED and EDXA.
G. Asbestos mineralogy encompassing its chemical composition, crystallography of the six regulated asbestos mineral and those minerals that closely resemble asbestos.
H. Acceptable and non-acceptable sample preparations.
I. Recognition of sample and instrumental artifacts.
J. Verified Analysis

Verified Asbestos Analysis

Verified Asbestos Analysis is performed on Standard Reference Materials, 1876b, 8411 and/or SRM 8410 (refer Section VI-II) and on in-house samples that meet the following criteria:

- Working standards which require a range approximately 1000-5000 s/mm^2
- Samples, which have a range of approximately 24 - 500 s/mm2 At least 20% of the samples, must have a range of 1000-5000 s/mm^2

Multiple analysts will independently analyze the same grid opening, and grid openings are analyzed with

- Same grid orientation
- Same starting point
- Same initial traverse direction and pattern
- Structures recorded by:
  - Size
  - Number of structures
  - Identifier data
  - Orientation and sketch of structure

The results are compared for multiple independent analyses. Any questionable structures will be reanalyzed and confirmed.

The method used for verified analysis is by Turner and Steel (1994) on Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy – Version 2. (see **Appendix IV**).

VI-VII: RE-ANALYSIS

Re-analyses are performed currently only on AHERA samples and these analyses are conducted in two ways: reanalysis among different analysts on the same grid openings (not a verified analysis) and on the sample grid openings for the same analysts following the same AHERA rules. These re-analyses are performed at 10% of total AHERA samples analyzed. Results of re-analyses then are documented and charted. The lab manager or the QC/QA officer will investigate and correct with the analyst if his or her variance exceeds the allowable range established for a given analyst.

VI-VIII: CHECKLIST FOR NEW TRAINEES TO IDENTIFY AND QUANTIFY ASBESTOS FIBERS BY TEM

1. Transmission Electron Microscope

1.1 Electronic system

1.2 Vacuum system

1.3 Chiller system

1.4 Alignment

1.5 Specimen holder

1.6 Filament change

1.7 Eucentricity and focus

1.8 Brightfield and darkfield imaging

1.9 Film camera system

1.10 Calibrations

1.10.1 Magnification - Film

1.10.2 Magnification - Phosphor viewing screen 1.10.3 Diffraction camera constant - Film

1.10.4 Beam dosage

1.10.5 Spot size

2. Energy Dispersive X-Ray Spectrometer

2.1 Characteristic X-ray

2.2 Detector and geometry

2.3 LN system

2.4 Calibrations

2.4.1 Energy channel (Al, Cu)

2.4.2 Manganese Ka peak resolution

2.4.3 Relative sensitivity (K-factor) factor: Mg, Fe, Ca, Na, Al

2.4.4 Na Ka peak detection

2.4.5 Grid area where abnormal x-ray spectra are generated

3. Sample Preparation

3.1 Plasma asher

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3.2 Condensation washer/Jaffe wick

3.3 Carbon coater

3.4 Polycarbonate filter preparation

3.5 Mixed cellulose ether filter preparation

3.6 AHERA rule of blank filter preparation

4. Asbestos Analysis

4.1 Grid quality evaluation 4.2 Grid scan scheme

4.3 Morphological criteria structure classification

4.3.1 Fiber

4.3.2 Cluster 4.3.3 13undle 4.3.4 Matrix

4.4 Structure sizing

4.5 Compositional criteria by EDX

4.5.1 Chrysotile

4.5.2 Amosite

4.5.3 Crocidolite 4.5.4 Tremolite 4.5.5 Actinolite 4.5.6 Anthophyllite

4.5.7 Non-asbestos interferences

4.6 Structural criteria by SAED (Selected Area Electron Diffraction)

4.6.1 Chrysotile

4.6.2 Amphiboles

4.6.2.1 Layer line spacing criteria 4.6.2.2 Zone axis pattern indexing 4.7 Count sheet

5. AHERA Protocol

5.1 Minimum volume requirement

5.2 Analytical sensitivity requirement and calculation 5.3 Clearance test

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5.3.1 Screening test 5.3.2 l3lank analysis 5.3.3 Z-test

6. Other Protocols

6.1 Yarnate
6.2 NIOSH
6.3 ELAP
6.4 Water
6.4 Dust
6.3 Soil

A similar checklist can be found in Appendix VIII at the time of completion and will be filled with the trainee's personal file.

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CHAPTER VII

SAFETY AND HOUSEKEEPING

VII-I: GENERAL GUIDES

All personnel are trained and supervised in proper safety and health procedures in the laboratory and in the field. An individual's safety attitude and safety performance is taken into consideration for all hiring and promotion decisions or changes in assignment. Employees who have difficulty in understanding the importance of a good safety attitude receive additional counseling and training. An employee who continues to display a poor safety attitude may be dismissed from the laboratory.

Safety equipment is provided to employees and its use is required when performing the work that may be hazardous, or when working in or passing through the laboratory or certain field areas. This equipment includes safety glasses and/or goggles, laboratory coats or aprons, steel-toed safety shoes, air-purifying and powered-air respirators, and other equipment as appropriate.

Fire extinguisher, an eyewash station, and a comprehensive industrial first aid kit are available in the laboratory at all times (see Lab Layout View in **Appendix V**). All of these items are inspected and replenished on a regular basis.

Concentrated acids and alkalis, organic solvents, and other hazardous chemicals and reagents are properly stored in cabinets. A safety container or carrier is available for carrying bottles of hazardous substances from one location to the other.

All samples (asbestos, wastewater effluent, etc.) and other chemicals and substances are properly treated and disposed of when they are no longer needed.

Broken glassware is swept up promptly. A special container is kept in the laboratory for broken glass and other sharp fragments of material.

BLI maintains a high standard of cleanliness and housekeeping in our laboratory. Illumination, ventilation, bench space, storage facilities and other ambient factors are maintained at appropriate levels.

Only **BATTA** employees are permitted in the laboratory or in equipment storage areas. Representatives of client companies, prospective clients, representatives of government regulatory agencies, and job applicants may be given brief tours of these areas provided they are accompanied by a BATTA employee at all times.

Activities such as safety inspections, lessons, or meetings are held for technical personnel or for all employees as designated by senior management. MSDS (Material Safety Data Sheets) for all hazardous reagents are available to the workers in the laboratory.

VII-II: DISPOSAL PROCEDURES

Analyzed samples are put into a box in the darkroom. Bulk samples are kept in a box in the main lab area.

When the box is full, it is sealed, appropriately marked and dated.

After at least 3-month storage time, the cassettes (or bulks) are emptied and the samples are double-bagged, goose-necked and labeled with our name and date, then it is given to an asbestos abatement contractor for disposal.

VII-III: COMMON REAGENTS FOR TEM ANALYSIS

Currently there are three reagents used in TEM grid preparation: distilled water, ethyl alcohol and acetone. All of these reagents are kept in the chemical storage bin in the main lab (see Lab Layout View of **Appendix V**). However, acetone and ethyl alcohol may also be in separate containers and kept inside the TEM prep area for convenience and will always be in ventilated locations for safety and health. Note: the DI water for NOBs and EPA 100.2 must be filtered through filters of 0.1 micron porse size.

VII-IV: EMERGENCY RESPONSES

There are three fire extinguishers. One is on the right at the entrance of the bulk asbestos and chemistry lab, one is behind the door of the GC lab, and the other is at the entrance of the TEM prep area (see Lab Layout View in **Appendix V**). Emergency gathering ground is the parking lot at both entrances to the BLI building. Safety shower is at the rear entrance of the bulk asbestos and chemistry lab. Contact the lab manager immediately if precursors of emergencies occur. Exit the building if the fire alarm is on.

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APPENDIX I

PROFESSIONAL PROFILES

Table of Contents

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Naresh C. Batta, MS, RPIH
Laboratory Director, BATTA LABORATORIES, INC.

Education:

University of Delaware, Newark, DE, M.S. Chemistry.

Punjab University, India, B.S. Physics/Math.

Environmental Training, Inc., Cinnaminson, NJ, PA Asbestos Project Designer Ref., June 1994.

MidAtlantic Environmental Hygiene Resource Center, Phila., PA "Building Air Quality", October, 1993.

Leadtec Services, Inc., Mapleshade, NJ, "Lead Abatement Training Course" and "Lead Paint Abatement HUD Guidelines Course", June, 1991.

University of Delaware, Newark, DE, "Hazardous Material Training Course".

EPA Air Pollution Training Institute, Georgia Institute of Technology, Atlanta, GA, "AHERA Building Inspector Course", Nov. 1987.

Georgia Institute of Technology, Atlanta, GA, "Supervision of Asbestos Abatement Projects".

Georgia Institute of Technology, Atlanta, GA, "Project Designers Course", June 1988.

Temple University, Phila., PA, "AHERA Management Planner Course", April, 1988.

Niton Spectrum Analyzer.

Safety for Small Business.

Sick Building Syndrome.

Source Sampling for Particle Pollutants.

Asbestos Contract Specifications and Contract Document.

Position:

Laboratory Director: Responsibilities include: oversee all laboratory operations under the Laboratory Manager's supervision, evaluate laboratory activities for efficiency and productivity, offer technical assistance.

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Naresh C. Batta, MS, RPIH
(Continued)

Experience:

Mr. Batta has over twenty-eight (28) years of professional experience in the Air Pollution/Industrial Hygiene Testing / Monitoring/Analysis field. His experience includes:

- Sampling for lead and vinyl chloride in the plant atmosphere
- Sampling for polychlorinated biphenols (PCBs) from manufacturing plants
- Air monitoring during asbestos work
- Asbestos surveys in manufacturing plants, schools, private homes
- Indoor air monitoring
- Emission inventories
- Particle size measurement in the laboratory
- "In-situ" particle size measurement
- Nox emission measurement
- Sox emission measurement
- Particulate emission measurement
- Servicing of air pollution equipment
- Ambient air monitoring for particles

Mr. Batta has personally conducted air pollution/ industrial hygiene sampling and measurement work at the following locations:

- Basic Refractory, Gabbs, Nevada
- Upper Peninsula Generating Station, Marquette, Michigan
- Utah Power & Light Company, Huntington Canyon Station, Price, Utah
- City Services Oil Co., (CITGO), Lake Charles, Louisiana
- Pennsylvania Power & Light Co., Sunbury Station, Pennsylvania
- Puerto Rican Cement, Ponce, Puerto Rico
- Georgia Power, Berry Station, Alabama
- Cincinnati Gas & Electric Co., Cincinnati, Ohio
- Colorado Utilities, Colorado
- Monogahill Power, Albright Station, Morgantown, West Virginia
- Gulf Power, Lansing Smith Station, Panama City, Florida
- Betz Laboratories, Trevose Pennsylvania

Memberships:

- American Industrial Hygiene Association (AIHA)
- American Chemical Society (ACS)
- American Water Works Association
- Delaware Safety Council
- Environmental Information Association (EIA)
- Environmental Committee - DE State Chamber of Commerce
- International Right of Way Association
- The American Institute of Architects

Naresh C. Batta, MS, RPIH
(Continued)

- The society of American Military Engineers
- National Radon Association - charter member
- Environmental Export Assistance
- GPF Regional Export Assistance
- World Trade Center Institute

Summary:

Mr. Batta has had many years of diversified experience in the environmental field. He is responsible for establishing and managing the goals, directions and activities of the firm. He has developed the company from a one-man operation to a 32 plus persons business. He is also responsible for sampling, testing and characterization of hazardous waste materials, air monitoring, industrial hygiene testing, asbestos testing, water and waste water sampling and testing, smoke stack testing and consulting to industry for new and existing environmental regulations and their impacts to industry. Under Mr. Batta's leadership the company has earned the "Project Safety Excellence Award" from DuPont Co. for years 1999, 2000, 2001 and 2002. In addition, he has vast experience in industrial hygiene work, developing Safety & Health & Safety Plans (HASPs), asbestos and lead testing, water sampling, air pollution sampling and analysis in various locations throughout the United Sates. He is currently going into his second year as Treasurer for the Delaware Section of the American Industrial Hygiene Association (AIHA).

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Shu-Chun Su, Ph. D.

103 Chadd Road
Newark, DE 19711
302-995-3498 (w) scsu@delanet.com

Education:

1986 Ph.D. Geology/Mineralogy, Virginia Polytechnic Institute and State University. Dissertation: Alkali feldspars: Ordering, Composition and Optical Properties

1981 M.S. Mineralogy, University of Science and Technology of China, Beijing, China

1964 B.S. Geochemistry, Peking University, Beijing, China

Professional Employment:

03/99 - Present Senior Research Scientist and Lab Director

01/97 - Present Chairman, China Advisory Council, Hercules Incorporated

08/96 - Present Guest Research Professor, Chinese Academy of Sciences

03/96 - Present Research Scientist and Lab Director

03/91 - 02/96 Senior Research Microscopist and Lab Director

05/87 - 02/91 Research Microscopist and Lab Director
Light and Electron Microscopy Laboratory, Analytical Science Division
Hercules Incorporated, Research Center, Wilmington, Delaware

09/88 - Present - Technical Expert (Bulk Asbestos Analysis Program: Bulk asbestos samples analyzed by polarized light microscopy)
- Technical Expert (Airborne Asbestos Analysis Program: Airborne asbestos samples analyzed by transmission electron microscopy)
- National Voluntary Laboratory Accreditation Program (NVLAP)
- National Institute of Standards and Technology (NIST)
Gaithersburg, Maryland

01/86 - 04/87 Consultant in optical instrumentation and computer programming
Electro Tech, Inc., Blacksburg, Virginia

01/86 - 04/87 Post-doctoral Research Associate, Department of Geological Sciences, Virginia Polytechnic Institute and State University, Blacksburg, Virginia,

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Shu-Chun Su
(Continued)

08/81 - 12/85 Research Assistant, Department of Geological Sciences
Virginia Polytechnic Institute and State University, Blacksburg, Virginia

09/79 - 07/81 Research Assistant, Institute of Geology
Chinese Academy of Sciences, Beijing, China

08/77 - 08/79 Group Leader, the Central Laboratory of the 6th Geological Survey Team, Geological Survey of Gansu Province, Wuwei, Gansu, China

01/69 - 07/77 Assistant Research Associate, the Central Laboratory of Lanzhou Geological Survey of Gansu Province, Lanzhou, Gansu, China

09/64 - 12/69 Assistant Research Associate, the Central Laboratory of Lanzhou Northwestern Institute of Geological Sciences, Lanzhou, Gansu, China

Professional Organization:

Microscopy Society of America (former Electron Microscopy Society of America)
Mineralogical Society of America
Sigma Xi

Awards:

1983 Full Scholarship for attending the 3rd NATO Advanced Study Institute on Feldspars and Feldspathoids (June 26 - July 6, 1983, Rennes, France), CNRS (National Center of Scientific Researches), France

1984 Cunningham Fellowship for Academic Year 1984-85
Virginia Polytechnic Institute and State University

Publications:

Papers:

2003 Erazo-Majewicz, P. and Su, S.C., Cationic Conditioning Polymer Deposits On Hair. Society of Cosmetic Chemists Annual Scientific Meeting, New York, December 4, 2003.

2003 Su, S.C., A rapid and accurate procedure for the determination of refractive indices of regulated asbestos minerals. American Mineralogist, 88, 1979-1982.

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Publications:
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1999 Barbier, J., Grew, E.S., Moore, P.B., and Su, S.C., Khamaralite, a new beryllium-bearing mineral related to sapphirine: A superstructure resulting from partial ordering of Be, Al, and Si on tetrahedral sites. American Mineralogist, 84, 1650-1660.

1998 Su, S.C., Dispersion staining: Principles, Analytical Relationships and Practical Applications to the Determination of Refractive Index. The Microscope, 46, 123-146.

1997 Grew, E.S., Yates, M.G., Peacor, D. R., Rouse, R. C., and Su, S.C., Boralsilite (Al16B6Si2O37): A new mineral intermediate in composition and structure to silimanite and the aluminum borate Al4B2O9 and its paragenesis in pegmatites. American Mineralogist, 83, 638-651.

1996 Grew, E.S., Peacor, D. R., Rouse, R. C., Yates, M.G., Su, S.C., and Marquez, N., Hyttsjöite, a new, complex layered plumbosilicate with unique tetrahedral sheets from Långban, Sweden. American Mineralogist, 81, 743-753.

1994 Su, S.C., A revised dispersion method for determining the composition of olivine, orthopyroxene, augite, and plagioclase. American Mineralogist, 79, 1204-1206.

1994 Rouse, R. C., Peacor, D. R., Dunn, P. J., Su, S.C., Chi, P. H., and Yeates, H. Samfowlerite, a new Ca Mn Zn berrylosilicate mineral from Franklin, New Jersey: Its characterization and crystal structure. The Canadian Mineralogist, 32, 43-53.

1994 Grew, E. S., Yates, M. G., Belakovskiy, Rouse, R. C. Su, S.C., Marquez, N., Hyaloteckite from reedmergnerite-bearing per-alkaline pegmatite, Dara-i-Pioz, Tajikistan and from Mn skarn, Långban, Värmland, Sweden: a new look at an old mineral. Mineralogical Magazine, 58, 285-297.

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1992 Dunn, P.J., Peacor, D.R., and Su, S.C., Franklinphilite, the manganese analogue of stilpnomelane, from Franklin, New Jersey. The Mineralogical Record, 23, 465-468.

1992 Su, S.C., Calibration of refractive index liquids using optical glass standards with dispersion staining technique. The Microscope, 40, 95-108.

1991 Grew, E.S., Essene, E.J., Su, S.C., and Asami, M., Dissakisite-(Ce), a new member of the epidote group and the Mg-analogue of allanite-(Ce) from Antarctica. American Mineralogist, 76, 1990-1997.

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1988 Gunter, M.E., Bloss, F.D., and Su, S.C., EXCALIBR revisited. American Mineralogist, 73, 1481-1482.

1987 Peacor, D.R., Dunn, P.J., Su, S.C., and Innes, J., Ribbeite, a unit-cell twinned polymorph of alleghanyite and member of the leucophoenicite group from Kombat Mine, Namibia. American Mineralogist, 72, 213-216.

1987 Solie, D.N. and Su, S.C., An occurrence of barium-rich mica from Alaska Range. American Mineralogist, 72, 995-999.

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1986 Su, S.C., Ribbe, P.H., and Bloss, F.D., and Warner, J.K., Optical properties of the high albite (analbite)-high sanidine solid solution series. American Mineralogist, 71, 1393-1398.

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1986 Su, S.C., Ribbe, P.H., and Bloss, F.D., Alkali feldspars: Structural states determined from composition and optic axial angle 2V. American Mineralogist, 71, 1285-1296.

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1984 Su, S.C., Bloss, F.D., Extinction angles for amphiboles or pyroxenes: A cautionary note. American Mineralogist, 69, 399-403.

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1983 Bloss, F.D., Gunter, M., Su, S.C., and Wolfe, E.H., Gladstone-Dale constant: A new approach. Canadian Mineralogist, 21, 93-99.

1982 Ye, D.N. and Su, S.C., Structural-optical mineralogy: More on the optical anisotropy of carbonates. (in Chinese with English Abstract). Memoir of Institute of Geology, Academia Sinica, 215-221.

1981 Su, S.C. and Ye, D.N., X-ray powder method for simultaneous determination of composition and structural state of plagioclases. (in Chinese with English Abstract). Scientia Sinica (English Edition), No.5, 670-677

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1980 Su, S.C., Proposed optical classification scheme for alkali feldspars. (in Chinese). Geological Review, No.4, 349-356.

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1979 Su, S.C., A new method for determining acid plagioclases with universal stage. (in Chinese with English Abstract). Journal of Science, No.10, 459-460.

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Book Reviews:

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1986 Su, S.C., Optical Mineralogy, Second Edition, by David Shelley, Elsevier Science Publishing Co., Inc., New York, 1985, 321 p. American Mineralogist, 71, 1060.

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1984 (Contributor) English-Chinese Dictionary of Earth Sciences, 1142p. Geological Publishing House, Beijing, China.

Confidential

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1977 Optical determination table for non-opaque minerals using a semi-cylindrical coordinate system, 139 p. Geological Publishing House, Beijing, China.

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1980 Tsuboi, S., et al., 1977, Charts of plagioclase optics. Iwanami, Tokyo.

1976 Uytenbogaardt, W. and Burke, E.A.J., Tables for microscopic identification of ore-minerals (second revised edition). Elsevier, Amsterdam.

1975 Slemmons, D.B., 1962, Determination of volcanic and plutonic plagioclases using a three- or four-axis universal stage. Geological Society of America, Special Paper No.69, 64p.

1975 Cameron, E.N., 1961, Ore Microscopy. John Wiley and Sons, New York.

Presentations and Lectures:

2003 Instructor, Short Course on Spindle Stage. McCrone Research Institute (July 11-13, Chicago, Illinois)

1999 Instructor, Short Course on Spindle Stage. McCrone Research Institute (July 2-4, Chicago, Illinois)

1998 Su, S.C., Estimating the Refractive Index Difference between a Solid Particle and an Immersion Liquid. INTER/MICRO-98 (August 10-14, Chicago, Illinois)

1997 Su, S.C. and Cooke, P.M., Improve the Proficiency in Asbestos Identification by Polarized Light Microscopy. The 15th Annual Conference of the Environmental Information Association (former National Asbestos Council), March 22 - 25, Las Vegas, Nevada.

1996 Su, S.C., Understanding Detection Limit and Analytical Sensitivity in TEM Airborne Asbestos Analysis. NVLAP Annual Regional Meetings (East Region: October 4, Philadelphia, PA; Central Region: October 25, Minneapolis, MN; West Region: November 1, San Francisco, CA)

1996 Su, S.C. and Cooke, P.M., ?Back to Basics? for Bulk Asbestos Analysis. NVLAP Annual Regional Meetings (East Region: October 4, Philadelphia, PA; Central Region: October 25, Minneapolis, MN; West Region: November 1, San Francisco, CA)

1995 Su, S.C., Improving Accuracy in Refractive Index Measurement in Bulk Asbestos Analysis. NVLAP Annual Regional Meetings (East Region: September 15, Cincinnati, OH; Central Region: October 6, Houston, TX; West Region: October 27, Los Angeles, CA)

Presentations and Lectures:
(Continued)

1995 Su, S.C. and Cooke, P.M., Identifying Tremolite, Actinolite, and Anthophyllite in Bulk Asbestos Samples. NVLAP Annual Regional Meetings (East Region: September 15, Cincinnati, OH; Central Region: October 6, Houston, TX; West Region: October 27, Los Angeles, CA)

1995 Su, S.C., Cooke, P.M., Perkins, R.L., and Harvey, B., Analysis of Bulk Materials for Asbestos: The Problems and Solutions. Professional Development Seminars, Environmental Management '95, the 12th Annual Conference of the Environmental Information Association (former National Asbestos Council), April 22-26, Tempa, Florida

1994 Su, S.C., Measuring/recording refractive indices of asbestos fibers in NVLAP accredited environmental laboratories. NVLAP Annual Regional Meetings (West Region: June 29, Seattle, WA; Central Region: July 22, Chicago, IL; East Region: August 24, Gaithersburg, MD)

1994 Su, S.C., Determination of refractive indices of Asbestos Minerals. INTER/MICRO- 94 (July 18-21, Chicago, Illinois)

1993 Su, S.C., Determination of the refractive index of solids by dispersion staining method - An analytical approach. 51st Annual Meeting of the Microscopy Society of America (July 31 - August 4, Cincinnati, Ohio).

1990 Su, S.C., A computer program for rapidly and accurately orienting single crystals by X-ray precession method. Symposium in Honor of Professor F. Donald Bloss (July 22-25, Blacksburg, Virginia)

1989 Su, S.C., Application of Spindle Stage to determining the birefringence of Synthetic fibers. The 28th Annual Meeting of Eastern Analytical Symposium (September 24-29, New York City, New York).

1989 Instructor, Short Course on Spindle Stage and Computer Methods. August 14-18. Offered jointly by McCrone Research Institute and Virginia Polytechnic Institute and State University, Blacksburg, Virginia.

1989 Instructor, Short Course on Immersion Methods and Crystal Optics. August 7 - 11. Offered jointly by McCrone Research Institute and Virginia Polytechnic Institute and State University, Blacksburg, Virginia.

1986 Instructor, Short Course on Optical Identification of Crystals and Minerals. Virginia Polytechnic Institute and State University, Blacksburg, Virginia.

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Presentations and Lectures:
(Continued)

1986 Instructor, Short Course on Spindle Stage and Computer Methods. Virginia Polytechnic Institute and State University, Blacksburg, Virginia.

1986 Su, S.C., Ribbe, P.H., and Bloss, F.D., Optical, X-ray and microprobe study of low plagioclase single crystals: Discriminant analysis of discontinuities. The 14th General Meeting, the International Mineralogical Association (July 13-18, Stanford, California), Abstract with Programs, p.267.

1986 Su, S.C., Ribbe, P.H., and Bloss, F.D., Optical properties of alkali feldspars. Invited paper for the Symposium on Optical Properties of Minerals. The 14th General Meeting, the International Mineralogical Association (July 13-18, Stanford, California), Abstract with Programs, p.240.

1986 Su, S.C., Ribbe, P.H. and Bloss, F.D., Alkali feldspars: structural state determined from composition and optical angle 2V. The 99th Annual Meeting of Geological Society of America, Abstracts with Programs, 18, 766.

1985 Instructor, Short Course on Optical Identification of Crystals and Minerals. Virginia Polytechnic Institute and State University, Blacksburg, Virginia.

1985 Instructor, Short Course on Spindle Stage and Computer Methods. Virginia Polytechnic Institute and State University, Blacksburg, Virginia.

1985 Su, S.C., Ribbe, P.H., and Bloss, F.D., Structural states and properties of a low-high albite series of single crystal. The 98th Annual Meeting of the Geological Society of America, Abstract with Programs, 17, 729.

1984 Warner, J.K., Su, S.C., Ribbe, P.H., and Bloss, F.D., Optical properties of the analbite-high sanidine solid solution series. The 97th Annual Meeting of Geological Society of America, Abstract with Programs, 16, 687.

1983 Su, S.C., Bloss, F.D., Ribbe, P.H., and Stewart, D.B., Optical axial angle, a precise measure of Al,Si content of the T1 tetrahedral sites in K-rich alkali feldspars. The 96th Annual Meeting of Geological Society of America, Abstracts with Programs, 15, 701.

1983 Su, S.C., Bloss, F.D., Ribbe, P.H., and Stewart, D.B., Rapid and precise optical determination of Al,Si ordering in potassic feldspars. The 3rd NATO Advanced Study Institute on Feldspars, Feldspathoids and Their Paragenesis, June 26-July 6, Rennes, France.

1980 Su, S.C., Applications of the universal stage to the investigations of rock-forming minerals and structural geology. Short Course on the Universal Stage (August 14-19, Urumqi, Xinjiang, China)

1974 Su, S.C., Advances in the study of the optical orientation of plagioclases. First National Symposium on the Study of Placer Minerals (September 8-14, Guangzhou, China)

Presentations and Lectures:
(Continued)

1973 Su, S.C., Frequency of plagioclase twinning laws and its bearing on petrogenesis. Short course on Mineralogy and Petrology sponsored by jointly Geological Surveys of Gansu, Qinghai, Ningxia and Xinjiang Provinces (May 21-July 15, Xining, Qinghai, China)

1973 Su, S.C., Zoning in plagioclases and its bearing on petrogenesis. Ibid.

1973 Su, S.C., Feldspars and provenance of sedimentary rocks. Ibid.

Confidential

Delores Beard, B.S.

Microscopist, BATTA LABORATORIES, INC.

September 1979- May 1982 B.S. – Applied Physics , Stockton State College, Pomona, New Jersey

September 1977- May 1979 A.A. – Liberal Arts , Camden County College, Blackwood, New Jersey

Experience:

February 2000- September 2010: **Senior Asbestos Analyst,**
EMSL Analytical, Inc, Cinnaminson, New Jersey

Responsible for performing PLM asbestos identification for bulk and soils, PCM analysis on air samples, and TEM analysis on air & bulk samples. Maintaining QC for asbestos analysis and approved asbestos reports. Responsible for weekend coordination of all asbestos analyses.

February 2008- February 2009: **PLM Supervisor**

Responsible for training of new PLM and PCM analysts and maintenance of all training records.

November 1990- January 2000: **Asbestos Laboratory Director**

GA Environmental Services, Inc., Eddystone, Pennsylvania

Responsible for coordinating PCM, PLM analysis and maintaining all required documentation and QA/QC program (PCM/PLM); performing all microscopy (PLM,PCM) work and maintaining laboratory equipment and laboratory supply inventory.

Summary: Delores Beard has been working in the asbestos analytical field since year 200. Ms. Beard has both strong analytical and managerial experiences. Currently, she is assisting asbestos bulk sample analysis, report data previews and sample preps.

Confidential

Xiao-hong Xu (Judy), B. S., Microscopist

Education:

1995-1996 Delaware Technical Community College

1981-1985 Electronic Engineering College at Beijing, China
BS in Electronic Engineering

Term Paper: Microstructures and Characteristics of Semiconductors under Scanning Electron Microscopy and Transmission Electron Microscopy

Experience:

March 2006 to Nov 2006 Navy Recruiting District, Philadelphia.
Assistant System Administrator
March 200-March 2005 IBM of Wilmington, DE
Client Support Engineering
On site computing support
Instrumental Engineering
Client training and education
July 1985-October 1990 National Institute of Petroleum Research and Exploration Beijing, China
Assistant Laboratory Engineer
Transmission Electron Microscope Maintenance (TEM & SEM)
Sample Analysis Using Electron Microscope
National Technical Support and Training

Certification:

Microsoft Professional
Cisco and Internetworking Technology

Summary:

Ms. Xu acquired her B.S. degree in Electronic Engineering in 1985, and had been working as a TEM analyst for the national oil research facility in Beijing since then before moving to the Unite States. She is currently under training to be an asbestos TEM analyst.

Confidential

Daniel Pierce, Sample Custodian and Data Analyst

Education: High School
Newark High School graduated, High School Diploma

Summary: Mr. Pierce joined Batta Laboratories, Inc. in 2008 as a Sample Custodian and a Data analyst. He has been working within various industrial facilities as custodians and data analyst. Currently, Mr. Pierce is working as a principal sample custodian, soil particle size analyst, and deputy to the laboratory manager.

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Yeng-Chieh Tsai, Principle PLM Analyst

Education:

2002 National Taiwan University Taipei, Taiwan
Bachelor of Agriculture Chemistry.

2009-2010 University of Delaware Newark, DE, USA
Master of Civil Engineering (Environmental Engineering specialized)

Experience:

Current: Principle PLM Analyst, Batta Laboratories, Inc.
In charge of the Polarized Light Microscopes (PLM) operations using various EPA regulated methods including EPA 600/R-93-116 Method, NIOSH 9002 and EPA Libby Methods to analyze bulk samples.
Preparation and analysis of various environmental relevant fibers using NIOSH 7400 method by Phase-Contrast Microscopy.
Chemical analysis of aqueous samples using various instruments and related technology.
Compilation and analysis of various testing results to provide technical reports that meet the EPA regulatory requirements.
Perform metal analysis using Atomic Absorption (AA) spectrophotometers following the EPA approved Methods.
Providing assistance to sample custodian and data analyst in client data entrance and analysis.
Providing assistance to the lab manager and senior chemists in QA/QC software programs.
Routinely providing assistance in hazardous material disposal and treatment.
Assistance to TEM sample preparation and analysis using AHERA and ISO protocols.
Prepare and analyze volatile samples using EPA Method 24 by GC/MS.
Certificate: Asbestos identification with polarized light microscope

2005-2008 University of Delaware Newark, DE
Lab assistant/Master program (Participated in a NSF funded research projects that allowed me to gain the following experiences)

Summary: Having a PLM analytical certificate from an outside credited institute and proficient with asbestos bulk analysis through internal training programs, Mr. Tsai is currently working as a principle PLM analyst.

Bo Li, Ph.D., Laboratory Manager & QC/QA Officer
TEM/PLM Microscopist & Manger of Asbestos Services, BATTA LABORATORIES, INC.

Education: Ph. D. in Geology, College of Marine Studies and Earth Sciences, University of Delaware, Newark, DE.

1989 – 1991 Graduate School of Chinese Academy of Sciences, Beijing, China. M. S. in Geology.

1982 – 1986 Chinese University of Mining Technology, Xuzhou, China. B. S. in Mining Geology.

Position: TEM/PLM Microscopist

Responsibilities include: Comply with those required in **Section I-IV of Chapter I** for a Microscopist. The nature of this position is to assist the laboratory operation while the other key players are not available or in need of help. Duties include, but may not be limited to, samples log-in, preparation, and analysis using pertinent regulations and methods, such as EPA 600/R-93-116 Method, NIOSH 9002, and EPA Libby Methods by Polarized Light Microscopy.

Training: May 2003- February 2004 Batta Laboratories, Inc., Newark, DE: TEM analysis using various rules and regulations (AHERA, Philly Regs, ISO, NIOSH 7402, ASTM 5755, etc).

October 2003 – May 2004 Batta Laboratories, Inc., Newark, DE: PLM analysis on bulk asbestos samples.

June 1998 Batta Laboratories, Inc., Newark, DE: PCM analysis.

November 1998 Batta Laboratories, Inc., Newark, DE: PLM analysis on asbestos bulk samples.

Experience: TEM/PLM Microscopist, Batta Laboratories, Inc., Newark, DE, 2003-current.

Project Manager, Earth Resources Technology, Inc., Newark, DE, 2000-2003.

Teaching Assistant, University of Delware, DE, 1999-2000.

Microscopist, Batta Laboratories, Inc., Newark, DE, 1998-1999

Teaching Assistant, University of Delware, DE, 1996-1998.

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Experience:
(Continued)

Assistant Researcher, Geological Institute of Chinese Academy of Sciences, Beijing, China, 1991 – 1994.

Geologist, The Exploration Company of Shandong, Tai-an, China, 1986 – 1988.

Certification: PCM Analysis Certificate by S&L Limited, 1998.

PLM Analysis Certificate by S&L Limited, 1998.

OSHA 40 Hrs Certificate, 2001, 2003, and refreshers to current.

Affiliation: American Geophysical Union, Member since 1999.

Geological Society of America, Member since 1998.

Summary: Mr. Bo Li has been working in the field of geology since 1996, and has accumulated solid knowledge and experience in mineralogy and petrology for microscopic analyses, which are fundamentals for his geologic careers either in research institutes or industrial sectors.

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APPENDIX II

LIST OF EQUIPMENT

Confidential

Equip. ID	Batta Property ID	Quantity	Description	Location
JEM-100 CX II	02116	1	Transmission Electron Microscope	TEM Lab
JEM-100 CX II	00616	1	Power Supply	TEM Lab
HSKRIS Water Chiller	00617	1	Water Chiller System	TEM Lab
QUEST	00619	1	QUEST X-Ray Analyzer	TEM Lab
Kevex	01350	1	X-Ray Detector Model 3200-0173	TEM Lab
EMSL Plasma Asher	02120	1		TEM Lab
Denton Desk V Carbon Sputter	01872	1	Carbon Coating Equipment	TEM Lab
Computers	00413 00620 N/A	1 1 1		TEM Lab
File Cabinet	01357	1		TEM Lab
Storage Cabinet	00621	1		TEM Lab
Chairs	N/A	2		TEM Lab

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APPENDIX III

FORMS, SHEETS, AND CERTIFICATES

List of Contents

*TEM Training Package is not available in PDF format due to large file size

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SOP Revision Request Form

Template

BATTA

Request for Modification to Analytical Methodology or Laboratory Standard Operating Procedures

Instructions to Requester: *Submit to contact(s) at bottom of form for review and approval. File the approved copy with QA-QC Officer for submission and incorporation into appropriate BATTA SOP or QA Manual.*

Name and/or Method Number Affected: _____

Requester: _____ Job Title: _____
Microscopy or Chemistry Lab: _____ Date of Request: _____

Description of Modification:
_____
_____

Reason for Modification:
_____
_____

Potential Implications of this Modification:
_____
_____

Laboratory Applicability (circle one): All Individual Sections) _____
_____

Duration of Modification (circle one):
Temporary Date(s): _____
Laboratory Sample Numbers & Clients Affected: _____
_____

Permanent (Complete Proposed Modification Section) Effective Date: _____

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):
_____
_____
_____
_____

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Laboratory Review and Approval: _____ Date: _____
(QA-QC Officer or designate)

Approved By: _____ Date: _____

Title: Laboratory Director, BATTA Laboratories, Inc.

BATTA LABORATORIES, INC.
Delaware Industrial Park
6 Garfield Way
Newark, DE 19713 - 5817

APPENDIX III
Forms, Sheets, and Certificates

Appendix III-2 of 8

SOP Revision Request Form

Template

BATTA

Request for Modification to Analytical Methodology or Laboratory Standard Operating Procedures

Instructions to Requester: *Submit to contact(s) at bottom of form for review and approval. File the approved copy with QA-QC Officer for submission and incorporation into appropriate BATTA SOP or QA Manual.*

Name and/or Method Number Affected: TEM QA Manual

Requestor: Bo Li Job Title: QC/QA Officer

Microscopy or Chemistry Lab: Microscopy Date of Request: 05/04/11

Description of Modification: QA Manual Revisions: Revised Lab Organization chart; Plasma Etching protocol, Carbon Sputtering; Biosafety and types

Reason for Modification: See above

Potential Implications of this Modification: Permanent

Laboratory Applicability (circle one): All Individual Sections) Tem Microscopy Lab

Duration of Modification (circle one):

Temporary Date(s):

Laboratory Sample Numbers & Clients Affected:

(Permanent) (Complete Proposed Modification Section) Effective Date: 05/27/11

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

1> plasma etching protocol update: 2> Denton Carbon Sputtering operation procedures

Technical Review: Bo Li Date: 5/04/11
(Laboratory Manager or designate)

Laboratory Review and Approval: Bo Li Date: 5/04/11
(QA-QC Officer or designate)

Approved By: Date: 5/04/11

Title: Laboratory Director, BATTA Laboratories, Inc.

Appendix III- 2

C:\Accreditation\TEM SOP 2010 Rev\APPENDIX_III_Froms_2010 Rev.doc

BATTA LABORATORIES, INC.
Delaware Industrial Park
6 Garfield Way
Newark, DE 19713 - 5817

APPENDIX III
Forms, Sheets, and Certificates

Appendix III-2 of 8

SOP Revision Request Form

Template

BATTA

Request for Modification to Analytical Methodology or Laboratory Standard Operating Procedures

Instructions to Requester: *Submit to contact(s) at bottom of form for review and approval. File the approved copy with QA-QC Officer for submission and incorporation into appropriate BATTA SOP or QA Manual*

Name and/or Method Number Affected: Quality Control / Quality Assurance Manual for TEM

Requester: Bo Li Job Title: Manager

Microscopy or Chemistry Lab: Microscopy Date of Request: 2/4/2010

Description of Modification: 1> update Lab organization chart, 2> add TEM prep sheets to the manual appendices, 3> Annual Review and revisions if necessary

Reason for Modification: 1> Annual Modification review, 2> responses to recommendation from Delaware DNR

Potential Implications of this Modification: Annual updates

Laboratory Applicability (circle one): (All) Individual Sections) _____

Duration of Modification (circle one):
Temporary Date(s): Year 2010
Laboratory Sample Numbers & Clients Affected: N/A

Permanent (Complete Proposed Modification Section) Effective Date: 2/4/10

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):
N/A

Technical Review: _____ Date: 2/4/10
(Laboratory Manager or designate)

Laboratory Review and Approval: _____ Date: 2/4/10
(QA-QC Officer or designate)

Approved By: _____ Date: 2/4/10
Title: Laboratory Director, BATTA Laboratories, Inc.

2007 Request for Revision

BATTA

Request for Modification to Analytical Methodology or Laboratory Standard Operating Procedures

Instructions to Requester: *Submit to contact(s) at bottom of form for review and approval. File the approved copy with QA-QC Officer for submission and incorporation into appropriate BATTA SOP or QA Manual.*

Name and/or Method Number Affected: QA/QC Manual (TEM & plM)

Requester: Bo Li Job Title: Manager

Microscopy or Chemistry Lab: Microscopy Date of Request: 03/07/07

Description of Modification: Terminology and statements

Reason for Modification: In agreement with new ISO/IEC and NVLAP Requirements

Potential Implications of this Modification: In line with the new international standard for better quality control

Laboratory Applicability (circle one): All Individual Sections) Microscopy; May be useful for chemistry

Duration of Modification (circle one):

Temporary Date(s): _____

Laboratory Sample Numbers & Clients Affected: _____

(Permanent) (Complete Proposed Modification Section) Effective Date: 03/15/07

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable): Whereever appropriate

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

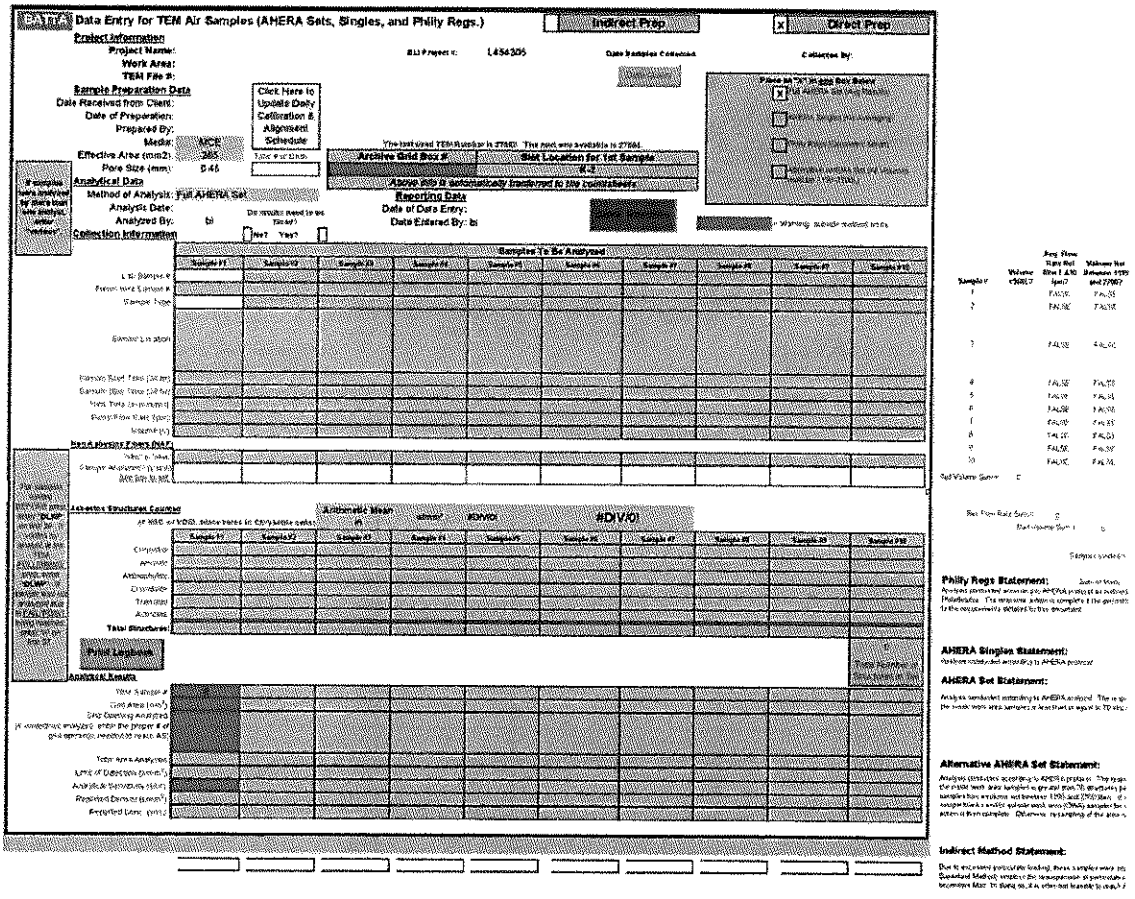
Laboratory Review and Approval: _____ Date: _____
(QA-QC Officer or designate)

Approved By: [signature] Date: 3/7/07

Title: Laboratory Director, BATTA Laboratories, Inc.

TEM Analytical Data Entry Template

Sample Sheet for AHERA Analysis



TEM Analytical Countsheet

Sample Sheet for AHERA Analysis

BATTA

TEM AHERA Analytical Countsheet

Daily TEM Alignments and EDX Calibrations Performed

	TEM File #	0 a	BLI Project#	L454305	
BL Sample #	0	TEM Sample #		Field ID#	0
	Grid #1 Box #	0	Grid #1 Location	K-2	
	Grid #2 Box #	0	Grid #2 Location	K-4	
	Grid #3 Box #	0	Grid #3 Location	K-6	
	Volume (liters)		Analytical Sensitivity (s/cc)		
	# of Grid Openings Analyzed	0	Grid Opening Avg Area (mm2)	0	
	Area of Sample Analyzed (mm2)	0	TEM Mag	19,000x	Acc.Voltage 100kV

GRID QUALITY EVALUATION - AHERA CRITERIA
(Check-off if criteria is met)

<5% of Grid Openings Have Holes ____ Grid Area has Even Loading ____
Grid Replica Coverage >50%____ Overall Particle Loading ≤ 10% ____
Undissolved Filter Area < 10% ____ Folded/Overlapping Replica <50% ____ <5% Grid Opening Area is Opaque ____

Sketch	Grid# - Sqsure ID (ex: 1 - E6)	Structure # and Type (ex: 1 - F)	Structure Length & Width (ex: 4.5 / 0.08)	SAED Pattern Identification (Chrys or Amph)	SAED Micrograph Plate #	Identification by EDXA (Type of Asbestos)

TEM Analyst _____ Date of Analysis _____

s/mm^2: _____ s/cc: _____

Arithmetic Average (s/mm^2): _____

Geometric Mean (s/cc): _____

Z-Test:_____ **PASS** **FAIL** Page # _____

NSD = No Structures Detected	
*B = Bundle	*F = Fiber
*C = Cluster	*M = Matrix

Totals	
	Total # of Structures
	# of Structures <5 um
	# of Structures >5 um
	# of Chrysotile Struct
	# of Amphibole Struct

C:\nv\Documents\Analytical Templates\TEM templates\TEM Air Templates (v 10).xls

Certificate of Analysis

Sample Sheet for AHERA Analysis

Dedicated to a Cleaner Environment Since 1982

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A Certified MBE Company

Delaware Industrial Park - 6 Garfield Way - Newark, DE 19713-5817
(302) 737-3375 - Fax (302) 737-5764
Web: www.battaenv.com E-mail: battaenv@battaenv.com

E.P.A. LAB ID# DE004
A.I.H.A./NLLAP #100448
NVLAP #101032

CERTIFICATE OF TEM ANALYSIS
Test Method: AHERA (40 CFR Part 763, Subpart E, Appendix A)

Page 1 of 1
Report Date: 10/31/2006

Sampling Data
BLI Project #: LXXXX
Project Name: XXXX-XXXX
Date Sampled: 10/30/2006
Sampling Location: XXXX-XXXX
Sampled By: CLIENT
Date Received: 10/30/2006

Analytical Data
Effective Filter Area (mm^2): 385 **Filter Media:** MCE **Filter Pore Size (µm):** 0.45 **Grid Opening Area (mm^2):** 0.0135
Date Samples Prepped: 10/30/2006 **Prepped By:** C. McDaniel **Date Analyzed:** 10/31/2006 **Analyzed By:** bl

	Client-Supplied Data				Analytical Data				Results		Comments
Lab Sample #	Client Sample #	Sample Type	Sample Location	Volume (L)	Area Analyzed (mm^2)	Number of Structures Detected	Asbestos Types Detected	Analytical Sensitivity (s/cc)	Limit of Detection (s/mm^2)	Reported Concentration (s/cc)	Reported Density (s/mm^2)	Notes From Analyst (see below)
544815	1	F-IWA	Work Area North	1200.00	0.0675	0	None Detected	0.0048	14.8	< 0.0048	< 14.8	None
544816	2	F-IWA	Work Area South	1200.00	0.0675	0	None Detected	0.0048	14.8	< 0.0048	< 14.8	None
544817	3	F-IWA	Work Area East	1200.00	0.0675	0	None Detected	0.0048	14.8	< 0.0048	< 14.8	None
544818	4	F-IWA	Work Area West	1200.00	0.0675	0	None Detected	0.0048	14.8	< 0.0048	< 14.8	None
544819	5	F-IWA	Work Area Center	1200.00	0.0675	0	None Detected	0.0048	14.8	< 0.0048	< 14.8	None

Analysis conducted according to AHERA protocol. The response action is complete if the arithmetic mean density of the six (6) work area samples is less than or equal to 70 structures per millimeter squared.

AHERA Set: Passes **Arithmetic Mean (s/mm^2):** 0.00

ANALYST: Bo Li **REVIEWED BY:** _____

Volumes provided by the client. Batta Laboratories does not accept liability for results reported in s/cc. This report pertains only to the items tested and does not constitute endorsement by NVLAP or other U.S. government agencies.

2260-544815-544819-(- Taj) xls

TEM Sample Prep Sheet

TEM SAMPLE PREP SHEET

Batch#/Proj#: L567708 **Total # of Samples:** 1
Sample #: 632597 **Prep Date:** 1/20/2010
TEM File#: 4187 **Prep By:** BL

Prep Type (Check One): Direct: ☐ Indirect: ☒ Dilution Series: ☒
Lab Blank (Check): ☒
Lot Blank: ☐
Method Blank: ☒

Effective Filter Area (mm^2): 346
(25 mm MCE filter)

Prep Method (Check One): AHERA: ☐ NIOSH 7402: ☐ ISO 13794: ☐
ASTM: ☐ ISO 10312: ☐
Libby-08: ☐ EPA 100.2: ☒ Other Methods (specify below):

Total Grids Prepped per Sample: 3
G.O. Size Used (mm^2): 0.009
Grid Box# and Loc# Assigned: 2785 & J

Sample #	Comments	Sample #		Comments
632597	5 → 100 → 25 (ml)			

Is the grid/filter prep acceptable according to the method? Yes: ☑ No: ☐

Note: Otherwise specified, each aliquot (ml) in a serial dilution shown in the comment column starts with a 100 ml solution. 15/100 or 15 means 15 ml taken from a 100 ml solution, and so forth.

ISO 10312 - ISO 10312:1995(E) Direct Transfer Method; ISO 13794 - ISO 13794:1999(E) Indirect Transfer Method; and ASTM: Dust or Microvac Sample Indirect Prep Method; EPA 100.2 - EPA600/R-94/134 (100.2)

TEM Training Packages

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APPENDIX IV

LIST OF METHODS AND REFERENCES

Confidential

SOPs and References	Location
JEM-CX100-II Instructions (with operations procedures and circuit diagrams)	TEM Lab
Federal Register 40 CFR part 763: Asbestos-containing materials in schools	TEM Lab
Guide for Quality Control on the Qualitative and Quantitative Analysis of Bulk Asbestos Samples: Version 1	TEM Lab
Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy – Version 2	TEM Lab
TEM Asbestos Analysis 407 B Vol. 1 with Chatfield Method, etc.	TEM Lab
NIOSH 7402	Chemistry Lab
TEM Analysis by Philly Regulations	TEM Lab
Water Analysis by TEM: EPA/600/R-94/134 and EPA/600/R-94/173	TEM Lab
CDM Documentation and SOPs	TEM Lab
State of New Jersey Method for Asbestos Bulk Analysis: 38 N.J.R. 2526	TEM Lab
EPA Test Method 600/R-93/116: Method for Determination of Asbestos in Bulk Building materials	TEM Lab
ASTM D5755-95: Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations	TEM Lab
ISO Protocols (Direct and Indirect Transfer Methods) for fiber counting: ISO 10312 & ISO 13794	TEM Lab

Confidential

SOPs and References	Location
TEM NOB Bulk Analysis: Chatfield Method and NY State Method (198.4)	TEM Lab
SRC-LIBBY-02 (Rev. 02): Quantification of Asbestos in Soil by SEM/EDS	TEM Lab
SRC-LIBBY-04 (Rev. 0): Measurement of Dust Loading	TEM Lab
Analytical Guidance Documents, Libby Asbestos Project	TEM Lab
ASTM D5755-03: Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations	TEM Lab
EPA-LIBBY-01 (Rev. 2): Asbestos Analysis of Soil by SEM/EDS	TEM Lab
EPA-LIBBY-08: Indirect Prep for Air and Dust Samples for TEM	TEM Lab
ISSI-LIBBY-01 (Rev.10): Soil Sample Preparation	TEM Lab
State of New York Methods of Bulk Asbestos Analysis	TEM Lab
Superfund Method	TEM Lab
EPA-LIBBY-03 (Rev. 1)	TEM Lab
CARB 435 (1991) (including Batta SOP-modified)	TEM Lab

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APPENDIX V

Maps, Figures and Charts

Contents:

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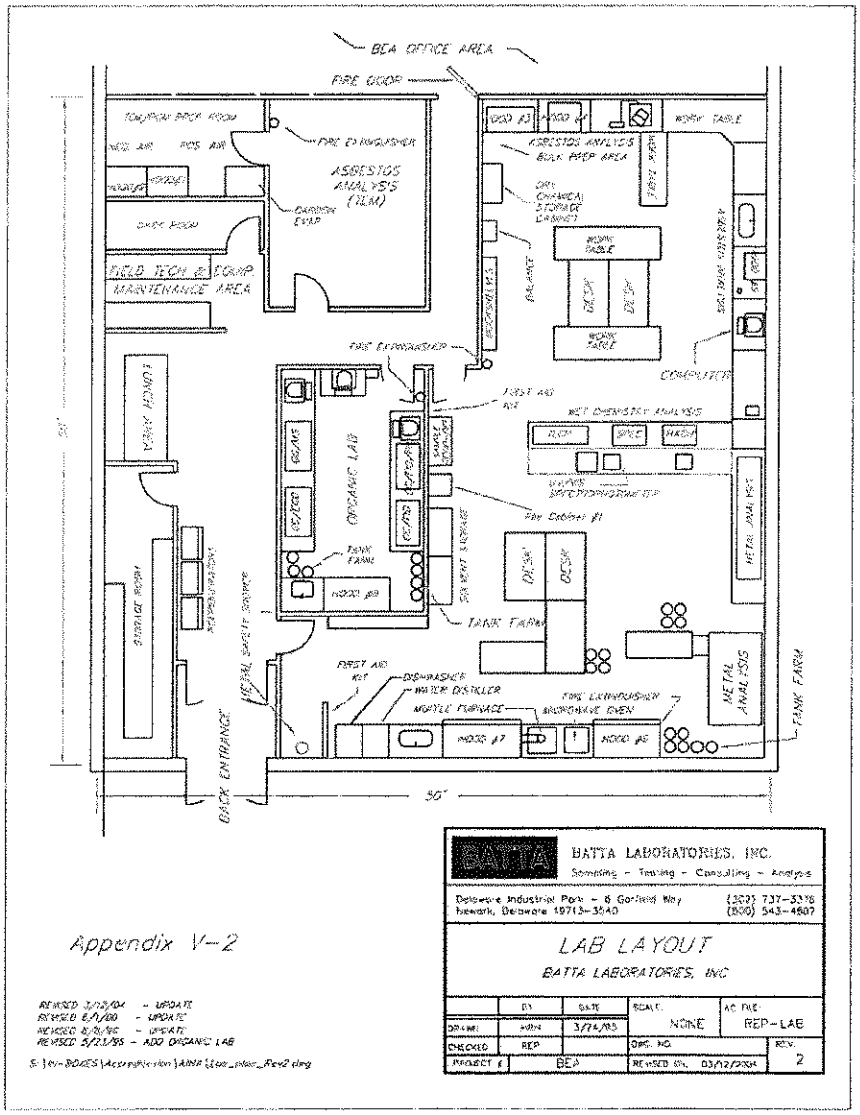
Lab Layout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FIRE DOOR
ASBESTOS ANALYSIS (TEM)
FIELD TECH & SUPP. MAINTENANCE AREA
ORGANIC LAB
METAL ANALYSIS
TANK FARM
BACK ENTRANCE
50'
Appendix V-2
BATTA LABORATORIES, INC.
Sampling - Testing - Consulting - Analysis
Delaware Industrial Park - 6 Garfield Way, Newark, Delaware 19713-3840
(302) 737-3376
(800) 543-4807
LAB LAYOUT
BATTA LABORATORIES, INC
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AC FILE: REP-LAB
DRAWN: MRM 3/24/95
CHECKED: REP
PROJECT #: BEA
REVISED: 03/12/2004
REV: 2

SpectraPlus Report

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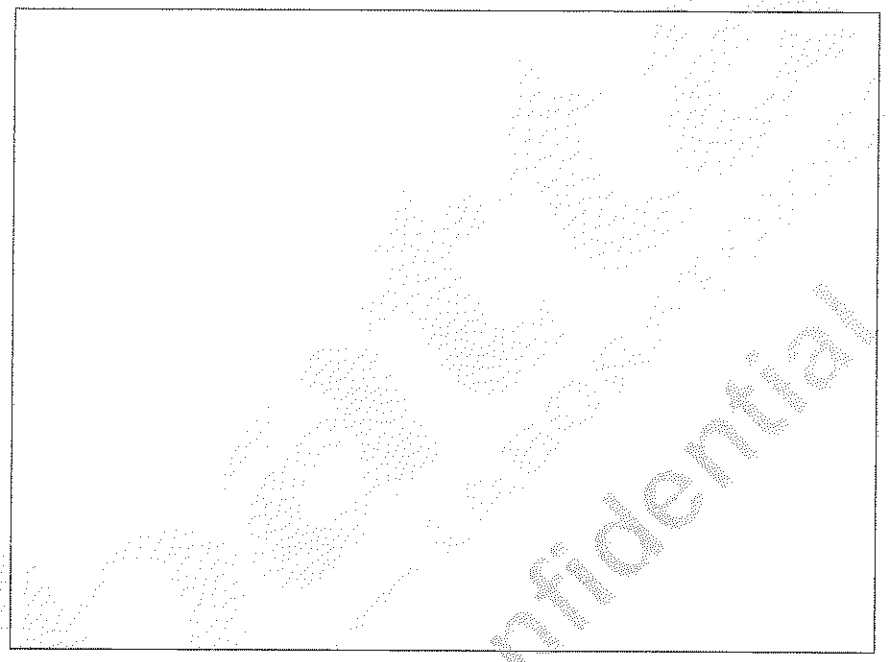

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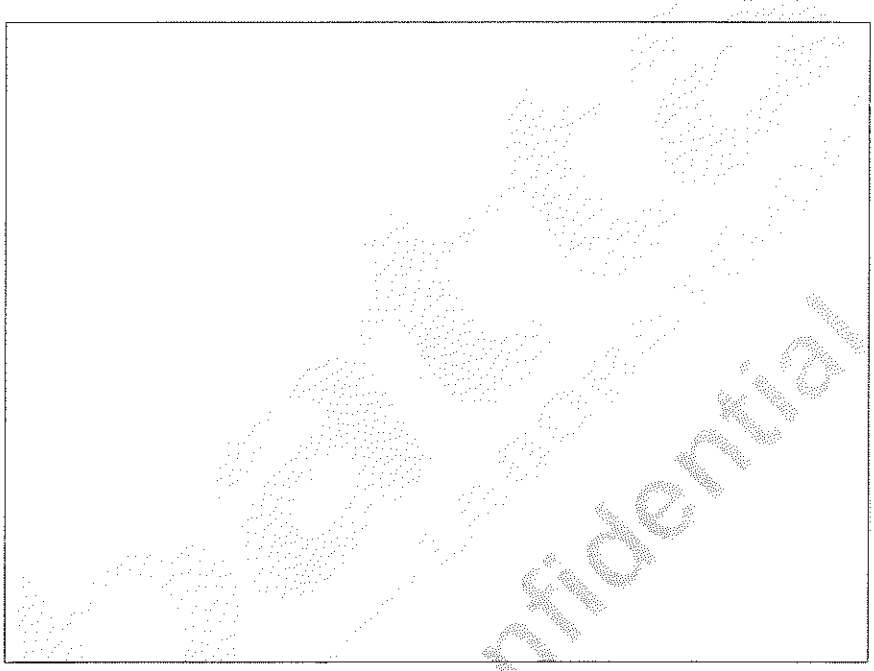

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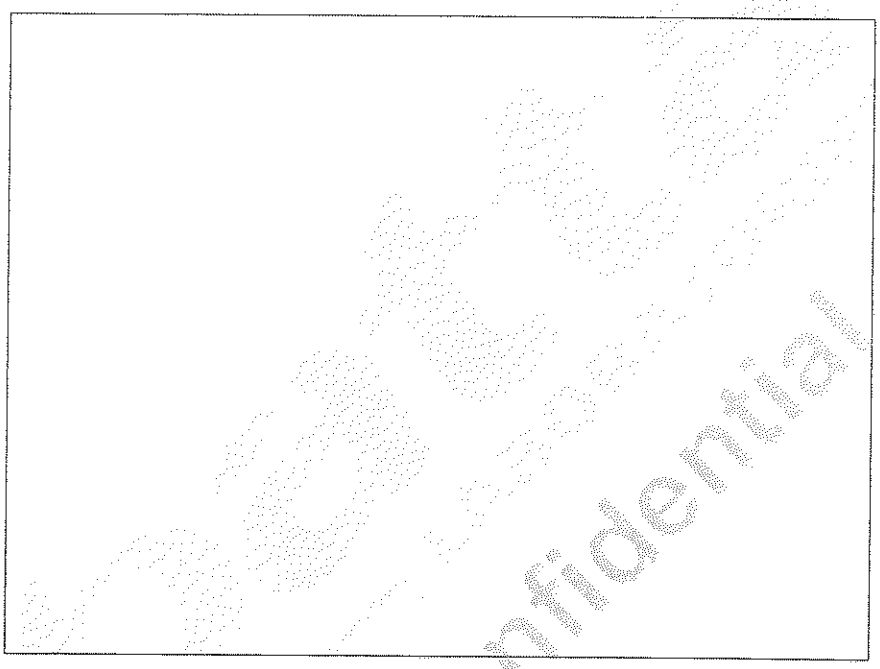

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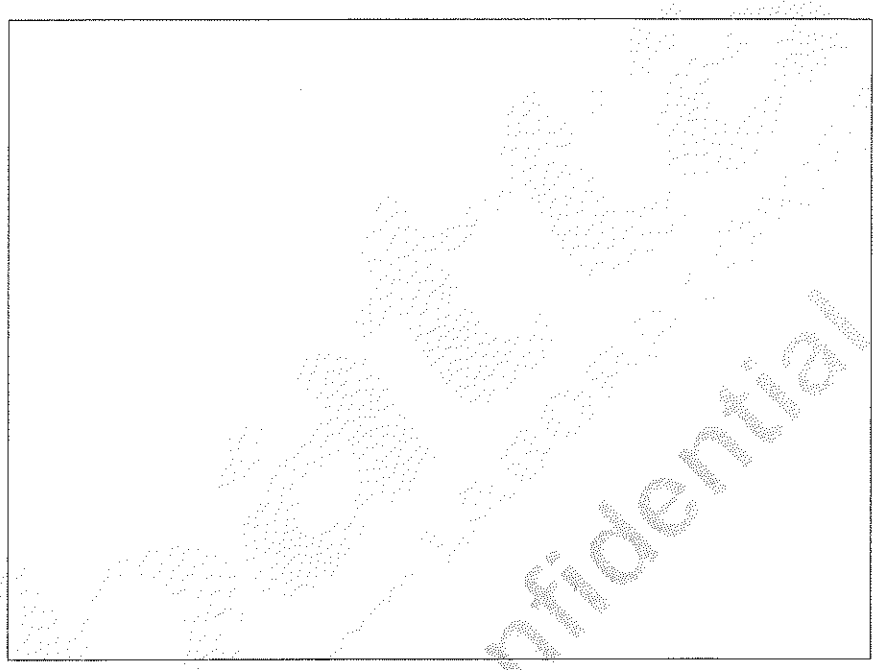

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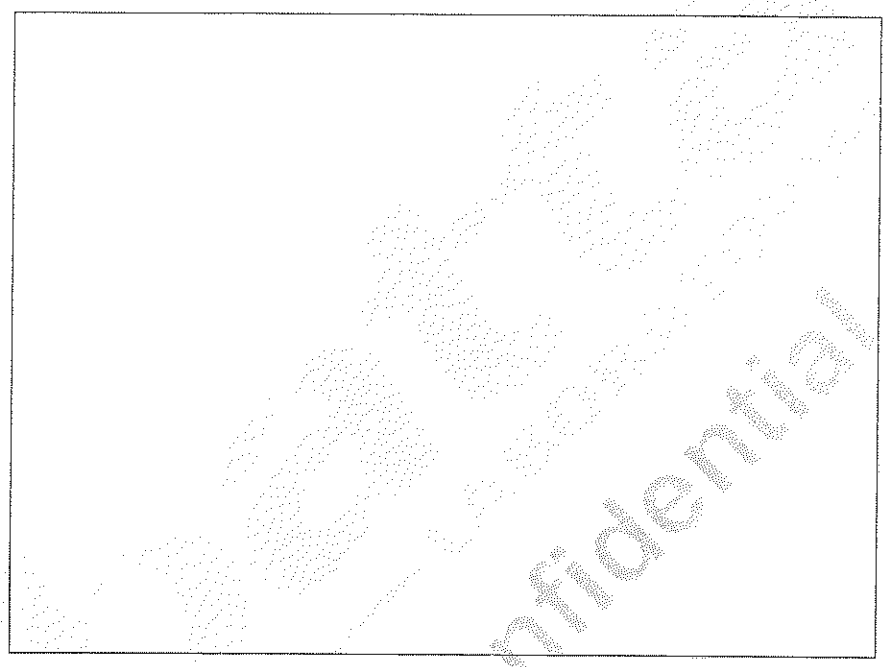

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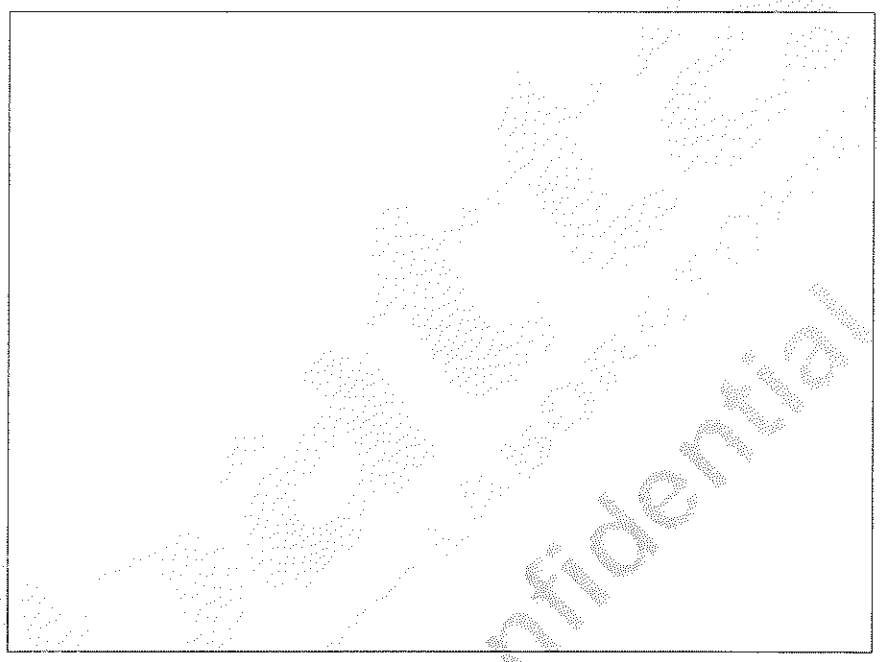

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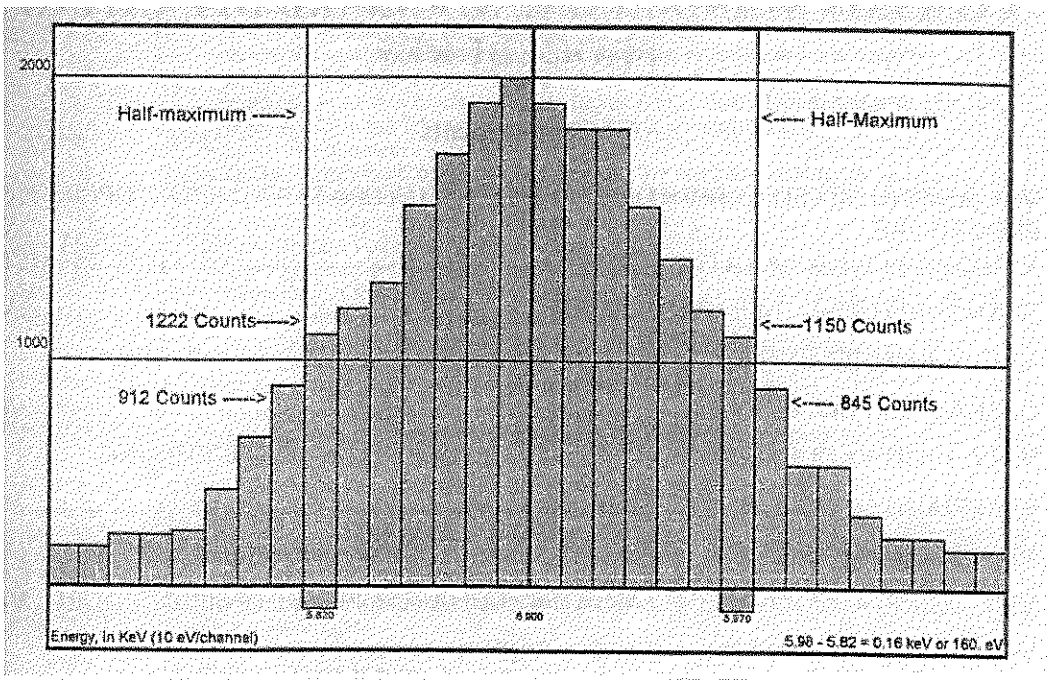
Mn K-alpha Peak with Typical Values of Counts in Channels Near the Half Maximum Point



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APPENDIX VI

COMMON STAND-ALONE STANDARD OPERATING PROCEDURES

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VI-I: ASHING MIXED ESTERS OF CELLULOSE FILTERS STANDARD OPERATION PROCEDURE

Warning: **1. BULK SAMPLES SHOULD NOT BE ASHED IN THE LOW TEMPERATURE PLASMA ASHER.**
2. DO NOT PLASMA ASH NIOSH 7402 SAMPLES

Applicable Analyses: AHERA, AHERA Singles, Philly Regs, ASTM (Soil, Dusts, and Vacuum), Water and CDM related samples.

I. Turn on the roughing pump.
II. Open the screen door to the Asher and pull out the glass cylinder.
III. Check the vacuum rubber seal for proper contact. Clean and apply sealant if needed.
III. Carefully place the glass slide(s) with the collapsed sample filter(s) into the glass cylinder traversing the length of the cylinder.
IV. Gently reinsert the cylinder back into its original position.
V. Turn on the AC power on the Plasma Asher.
VI. Turn the regulator of the oxygen tank to where 15 pounds per square inch is read.
VII. Turn on the vacuum switch to the Plasma Asher.
VIII. Do the following in order:
  A. Turn the power switch to the RF power on.
  B. Turn the RF meter switch on.
  C. Turn the power level knob 1/2 revolution clockwise.
  D. Adjust the tuning knob slowly until a bluish glow appears.
  E. Turn the power level knob slowly clockwise to the stop.
  F. Adjust the tuning knob until an optimum glow is achieved at the base of the cylinder where the slits are located towards the front.
IX. Ash the samples according to the most recent calibration time established.

►**Reference:** PLASMA ASHER MCE FILTER ETCHING TIME CALIBRATION STANDARD OPERATION PROCEDURE (Section VII-IV of Chapter VIII)

X. After a period of time (≥ calibration time), turn off all above-referenced switches in reserve order like the following:
  A. Turn the power level knob counter-clockwise to the top.
  B. Turn off the meter switch.
  C. Turn off the RF power switch.
  D. Turn off the vacuum switch.
  E. Turn off the Plasma Asher by turning off the red button.
  F. Turn off the oxygen supply.
  G. Turn off the power to the Plasma Asher and the roughing pump.
XII. Remove the sample slides and reset the Plasma Asher to its original status (off).

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VI-II: SAMPLE PREPARATION OF MIXED CELLULOSE OF ESTERS FILTERS (MCE) STANDARD OPERATION PROCEDURE

This method suits both **Dimethyl Formamide HCON $(CH_3)_2$ (DMF)** and acetone used as collapsing agents.

I. In the clean room, remove the requested sample cassette(s) to be prepped from the zip lock bag and arrange in an orderly, numerical order. Wet-wiping of cassettes should have already been accomplished before this point. Reject the sample if damaged prior the sample prep and notify the client or its representative and the lab manager or QC/QA officer.

II.
- A. On a clean glass slide or slides write with a solvent resistant marker the assigned TEM sample number (s) on the reverse side. The role of thumb is to assure the unique identification of each sample wedges on the slides. One recommendation would be using a sample range if more than one sample wedges are placed on one slide. For example, range 457898 – 903 is allowable. A TEM folder number is required to be marked on the slide regardless of whatever unique numbers (for instance, BLI lab number, client number, etc) were assigned on the slide (s). This number will be used to differentiate one slide from the other if more than one slide has to be prepped for two or more batches.
- B. With scotch tape, cover the number marked on the slide and cut away the excess with a straight razor blade. The tape will keep the numbers from being etched away in the Plasma Asher (step IV. D.)

III. For each sample cassette with the active filter in an upright position:
- A. Remove the plugs at the end of each cassette to relieve any pressure differential that may exist inside the cassette.
- B. Carefully cut through any labels, tape or jell bands with a razor blade at the cassette base.
- C. Determine to accept or reject the sample. Reject the sample if filters are damaged, overloaded or unevenly loaded. Inform the client or its representative and the lab manager or QC/QA officer.
- D. With as little disturbance as possible, disassemble the cassette at the cassette base.
- E. With forceps, carefully remove the 0.45µm filter from the cassette without touching the active portion of the filter, and transfer it to the pre-cleaned glass plate. Be sure that the 5.0 µm diffuser pad is not adhering to the filter.
- F. With a pre-cleaned razor or scalpel, cut a filter cross-section and remove it from the filter.
- G. Carefully place the rest of the filter back into the original cassette and reassemble.
- H. Place the cassette(s) back into the zip-lock bag and reserve them in a designated storage container.

IV. For each sample filter to be prepped using DMF, perform the following:
- A. On the glass slide with the pre-assigned I.D. number, place one to two drops of pre-mixed DMF solution. If the acetone method is employed, omit this step and go to step B.
- B. With a pair of forceps, carefully place, without touching the active part of the filter, the filter wedge, corresponding to the assigned I.D. number on the glass slide, onto the drop(s) of DMF solution. If acetone is used to replace DMF, apply filter wedges

directly over the slide in accordance with pre-assigned sample numbers. Then collapse the filter wedges with acetone vapor using a hot stage and move to step D.

C. With the drying oven in the hood at 60° to 70° centigrade, and the hood fan on, place the slide(s) onto a rack and dry for approximately 10 minutes or until the filter(s) clear.

D. Transfer the slide(s) to the Plasma Asher and follow the standard operation procedure for ashing mixed esters of cellulose filters.

►**Reference:** ASHING MIXED ESTERS OF CELLULOSE FILTERS STANDARD OPERATION PROCEDURE (Section VI-I of Appendix VI)

E. Transfer the slide(s) with the ashed filter(s) to the prepared High Vacuum Evaporator and carbon coat the filter(s).

►**Reference:** HIGH VACUUM EVAPORATOR CARBON COATING AND GOLD SPUTTERING STANDARD OPERATION PROCEDURE (Section VI-IV of Appendix VI)

V. For each carbon coated sample filter to be prepped, perform the following:

A. Place the carbon coated glass slide(s) onto the pre-cleaned glass plate in the hood. For filters to be washed by acetone go to step F.

B. Place pre-cleaned 100 mesh screen(s) onto the glass plate and assign the I.D. number from each carbon-coated filter to the relative mesh screen(s) by marking the glass plate relative to the mesh screen(s).

C. On custom designed 100 mesh screen(s), carefully place at least three pre-cleaned, quality controlled, naked finder grids for each carbon coated sample.

D. With a surgical knife, carefully cut the substrate for each sample and delicately place them on the grids, carbon side up, relative to their assigned I.D. number.

E. After at least 1.5 hours, remove the top to the prepared Jaffe Washer system.

F. With forceps, carefully pick up the assembly of grids and place into the Jaffe Washer system.

►**Reference:** **JAFFE WASH STANDARD OPERATION PROCEDURE** (Section VI-IX)

G. Slowly add straight DMF (or acetone) to the Jaffe Wash System until the wicking substrate is saturated. The level of the DMF or acetone should be just high enough to saturate the tissue paper.

H. Assign the grids inside the petri dish with numbers corresponding to those on the slide (s). This is normally done on the cover of the petri dish. However, a slice of paper with pencil marks placed inside the petri dish is recommended.

I. Set the count down timer to two (2) hours and allow the carbon coated filters to dissolve leaving the replicas intact. . **Note:** <u>If Acetone method is employed, grids can be removed from Jaffe Wick in no less than an hour. Once this step is completed for acetone-prepped grids, TEM analysis can begin and the following step is omitted</u>.

J. For each mesh sample screen in the Jaffe wash (DMF method only) perform the following:

A. The pre-cut 100 mesh bronze screens are shaped so they may be easily placed onto the cold finger in the condensation washer.
B. Only one custom shaped screen, which supports one sample of replicas, is allowed per cold finger.
C. After two (2) hours, remove the top on the Jaffe washer, and gently lay it to the side.
D. Gently loosen the cold finger to the condensation washer where the sample screen is to be placed.
E. With forceps, carefully remove the sample screen from the Jaffe wash.
F. Very carefully, place the sample screen on the flat surface of the cold finger and gently reinsert the cold finger into the condensation washer.
G. Replace the top on the Jaffe washer oriented to the original position relative to the sample screen.
H. Set the countdown timer for one (1) hour.
I. After one (1) hour in the condensation washer, gently loosen the cold finger (this must be done very carefully or the sample screen with the grid replicas can fall into the acetone reservoir).
J. With forceps, carefully remove the sample screen from the cold finger and lay it down on the pre-cleaned glass plate and allow for drying for five (5) minutes.
K. After allowing the grid replicas to dry, place the screen onto new filter paper and examine the grids at 30x power under the stereoscope.

▼Tips:

If the sample replica appears intact and is covering greater than approximately 50% of the grid openings, and also appears to have no noticeable undissolved filter remaining, with forceps, transfer the grids to their assigned grid box locations for future analysis. It is a strong indication that the filter is not fully dissolved if the grids stick to the tissue. In this case, the grids should be placed back into solvent dissolution (acetone or DMF).

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VI-III: GRID OPENING SIZE MEASUREMENTS (per lot of 100) STANDARD OPERATION PROCEDURE

I. With forceps place two copper finder grids from a lot (vial) of 100 onto a glass slide and assign a lot number to that lot by writing it on the vial with a solvent resistant pen (find the next assigned number in the TEM Grid Opening Measurements QC Book).

II. Take the glass slide with two grids and place it onto the stage of a calibrated Polarized Light Microscope and secure it into place.

III. With the X and Y stage knobs and rotating the 10x objective into place to achieve a magnification of 100x, locate one of the grids.

IV. Increase the magnification by rotating the 40X objective into place and locate a grid square (grid opening).

V. Using the 5 micron increment scale on the graticule determine the length and width of the grid opening in millimeters (1000 um = 1 mm).
   A. The 5-micron scale should be perpendicular to the straight sides of the grid opening (not corner to corner).
   B. Measure the diameter of the grid opening to a 1000th of a millimeter.
   C. Write the measurement in the appropriate GRID # column in the appropriate X column.
   D. Turn the graticule 90°, within the same grid opening, and measure the diameter of the straight sides.
   D. Write the measurement in the appropriate GRID # column in the appropriate Y column.
   E. Move the stage to a new grid opening.
   F. Do not measure a grid opening which has already been measured.
   G. Repeat steps A through G until 20 grid openings have been measured.

VI. Position the second grid on the glass slide and repeat steps III, IV and V (A through G).

VII. All of the measurements should be on one (1) page and under two (2) appropriate GRID # columns.

VI. Determine the average grid opening area for the two grids combined.

VIII. Enter the results onto the grid calibration Excel™ spreadsheet for calibrated results (Appendix V).

Note: The above procedure is only for grids that are purchased uncalibrated. For calibrated grids purchased with a certificated from a vendor (i.e 2SPI.com), recalibration in the lab is not required. Currently lab is using (prefrred) the calibrated grids from 2SPI, Inc.

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VI-IV: HIGH VACUUM EVAPORATOR CARBON AND GOLD SPUTTERING STANDARD OPERATION PROCEDURE

Material: Graphite or Carbon Rods (pre-sharpened); Ethanol Alcohol; Kimwipes; Safety Glasses; Screening Gargles; Vacuum Grease; Rubber Gloves
Safety: It is critical and mandatory that operator wear safety glasses during the vacuum operation and throughout the sputtering process

Operation Procedure (Revised According to Denton Desk V manual)

Warning: **Check and make sure all power switches are at "off" positions. Due to the defect safety design feature on the carbon accessory console, failure to do so may cause life threatening situations.**

Pre-sputtering Preparation:

1. Check and make sure all power sources are in "off" position for both Desk V and the carbon accessory console. The power switch of the Desk V at the very right bottom of its rear panel (operator facing the front); and the power switch of the carbon accessory console is on the front panel located at the lower right corner.
2. Install the carbon rods and fit well according the training demo (see Desk V manufacturer's manual). Just tightened enough so as not to break the carbon tip.
3. Gently clean the sealing surface of the carbon accessory head using ethanol alcohol.
4. Cleaning all carbon residues on all accessories if needed before operation. Gently clean all rubber gaskets well using ethanol alcohol if needed; and fit them back to their counterparts after cleaning. Use vacuum grease stingily if needed for better vacuum. Note: try not to apply or spread grease beyond the inner part of the rubber edges.
5. Load sample slides onto the rotational plate inside the Desk V vacuum chamber.
6. Place the carbon accessory glass tube above the Desk V vacuum chamber and fit well to get a reasonable seal.
7. Place the carbon accessory sputter head on top of the glass tube and fit well to get a reasonable seal.

Sputtering Operation:

8. Turn the Desk V power on. When the screen comes on, push/press "System Start" button. In the next screen, push/press the "Screens" button. When next screen comes up, press/push "Manual Sputter" button.
9. When a new screen comes up, push/press the "Rotation off" button which will turn on the power to the rotation stage inside the vacuum chamber. Looking through the view port, adjust the rotation knob on the right on top of the Desk V to achieve a desirable rotation speed.
10. Press/push the "Mech Pump Off" button to turn on the mechanical pump.
11. Wait until the chamber pressure drops below 0.15 Torr, then press "Turbo Pump Off" button to turn the turbo pump on.
12. After the message "Turbo @ speed" shows up below the "Turbo Pump On" button, the Desk V is ready for carbon sputtering.
13. Turn on the carbon accessory power.
14. Gradually raise the power until you can see a red glow for degassing and hold for a few seconds. Then gradually raise the power ~passing 40 A until moderate sparks occur.

Keep the spark constant by slightly increase the current until the tip of carbon is consumed.

15. Turn he power knob of the carbon accessory console down to zero; and then turn off the carbon accessory power.
16. If no further sputtering is needed, now turn off system using either one of the following methods:
    A (strongly recommended): Push/press "Auto Vent Selection" button and wait until the system automatically vents and shuts down. This will take between 10-15 minutes.
    B (not recommended for novice or rush prep): Push/press the "Turbo Pump On" to turn off the turbo pump (please note that the text on the button will change to "Turbo Pump Off"). **Warning: Do Not Turn Off the mechanical pump off until 15 minutes later.** After 15 minutes, press/push the "Mech Pump On" button to turn off the mechanic mump. You can immediately turn it on and off several times with 15 seconds interval to protect the turbo pump (note: this is optional).
17. After complete system degassing, turning off all powers (both the main system and the carbon accessory console). The touch screen of the main system is very sensitive and can be triggered by any contact during unloading of samples and can cause injury and damage to the pumps. **Check and make sure that the power of the carbon accessory console is in "off" position and the power dial was returned to zero**, then carefully remove the carbon accessory; and remove the coated samples.
18. Set and secure all parts at their original locations.

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VI-V: MICROGRAPH DEVELOPING STANDARD OPERATION PROCEDURE

All micrograph developing takes place in the small storage room next to the TEM lab. The door to the room has a tight seal with plastic covering the bottom edge of the door along the floor. This helps keep the excess light from entering the room while the door is closed. The room includes a basin-style sink holding three (tanks) containers: Developer, Fixer and tap water. The instructions on preparing the photographic solutions are below. If chemicals different from the ones listed below are used, follow the manufacturer's instructions.

VI-V.1: Preparation of Developer (Kodak D-19)

1. Warm two liters of tap water to 52°C.
2. Slowly empty the contents of a 595g package of developer crystals into the water, stirring constantly. Be careful not to add the powder too quickly.
3. Add the solution to the developer tank until the level is one inch from full. Let stand for at least 12 hours prior to using.
4. Time required for developing micrographs using fresh solution should be between one and two minutes. Required time increases as the solution ages. Solution should be good for use for at least one month following preparation. When developer reaches a caramel color, discard and prepare fresh solution.
5. When not in use, keep developer tank covered to cut down on light exposure and evaporation.

VI-V.2: Preparation of Fixer (Kodak General Purpose)

1. While stirring vigorously, pour the contents of a 680g package of fixer into two liters of water not exceeding 26°C. Continue stirring until all powder is dissolved.
2. Once dissolved, add the solution to the fixer tank until one inch from full. Fixer may be used immediately following preparation. If there is a small amount of undissolved powder remaining, there should be no problem. There may be a milky tinge to solution if water temperature was too high. The color should clear upon standing.
3. Fixer should be good for use for at least two months. Rule of thumb: prepare a fresh batch of fixer for every other batch of developer prepared.
4. When not in use, keep covered.

VI-V.3: Darkroom Procedure

1. With only the orange filtered safe light on and the door closed, open the film box containing exposed film. Load the exposed film into the slotted plastic tray. Each tray hold 18 negatives. This tray is used for holding the film in place while it is in the photographic solution.
2. Once loaded, take the slotted tray and submerge it into the developer solution. The solution need not be covered, however the darkroom MUST remain totally dark during this step. Keep the tray submerged for about 2 minutes (longer submersion may be required if the solution is not fresh).
3. Remove the tray from the developer and place into the container having tap water only. The container having tap water should be placed under the running tap, in effect cycling the water through. Keep the tray in the tap water for at least one

minute. Longer time periods in tap water will not adversely affect the development of the image. Once the tray is placed in water, lights may be turned on.

4. Remove the tray from the tap water and place in the container holding fixer. Leave the tray in the fixer for 10 minutes. The freshness of the solution does not greatly affect the time required to submersion.
5. Remove the tray from the fixer and place the tray back into the container holding tap water. With the tap running cool water into the container, let the tray stay for at least 20 minutes. This time should allow for most of the mineral deposits to be washed away from the film, allowing for a clear dry.
6. Remove the tray from the tap water.
7. Remove each film plate from the tray and hang on the line strung across the room using the clothes pins provided.
8. Once all water has dried from the film, the film may be taken for cataloging.

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VI-VI: MICROGRAPH IMAGE TAKING STANDARD OPERATION PROCEDURE

I. Magnification is 19,000x.
II. The fluorescent screen is illuminated.
III. Center the image to be micrographed within the square of the pop up screen with the specimen position knobs.
IV. Focus the image where the image is as sharp as possible with the medium and fine focus knobs located on the instrument panel on the right side of the column.
V. Exposure knobs are located on the instrument panel on the right side of the column.
Exposure specifications:
    A. Shutter Speed Knob = 11
    B. Sensitivity Knob = 6
VI. Shutter buttons should be set on manual that is lit up. The shutter buttons are located on the instrument panel on the right side of the column.
VII. Film Advance buttons should be on single that is lit up. Film Advance buttons are located on the instrument panel on the right side of the column.
VIII. With the condenser knobs, turn the illumination of the brightness of the image down.
    A. While turning the illumination on the screen, notice the exposure lights above the exposure knobs.
        1. There are three (3) exposure indication lights.
            a. Red
            b. Green
            c. Red
        2. The first (1st) red light, from the left, determines under exposure illumination.
        3. The second (2nd) light that is green indicates the proper illumination at which to take the micrograph of the image.
        4. The third (3rd) light, red, indicates over exposure of the image.
    B. With the condenser knobs, adjust the illumination on the screen until the 1st red light turns on.
    C. With the condenser knobs, slowly increase the illumination of the image until the green light comes on. This is the proper illumination of the image (for that particular shutter speed) in order to obtain a micrograph.
IX. Take a micrograph of the image.
    A. Be sure film is loaded in the TEM. The UNUSED FILM counter will indicate status of unused film.
    B. The CAMERA AIRLOCK OPEN light should be on.
    C. Push the FILM ADVANCE button to advance the film plate into position.
    D. Cover the VIEWING WINDOW with the window cover.
    E. Push the FLUORESCENT screen button (the button is not labeled, but it is located closest to the FILM ADVANCE button) that will light up.
    F. The EXP red light will come on during the assigned exposure time.
    G. AVOID TOUCHING THE TEM DURING THE EXPOSURE TIME.
    H. When the EXP red light turns off, remove the window cover.
X. Document the micrograph.

VI-VII: SAED MEASUREMENT AND INTERPRETATION OF CHRYSOTILE STANDARD OPERATION PROCEDURE

I. Place the micrograph negative of the suspect chrysotile diffraction pattern onto the light table.

II. Turn on the light to the light table.

III. If the diffraction pattern is asymmetrical, establish the rotated angle of the diffraction pattern relative to the long dimension of the negative.

IV. Chrysotile SAED patterns should have the following characteristics and the micrograph and observed on the screen:

- streaks on the layer lines other than the central line: (110) spot and (130) spot
- some streaking on the central line.
- spots of normal sharpness on the central line: (002) and (004)
- repeat of the layer lines (row spacing) should be approximately 5.3 A°.
- center doublet is 7.3Å. (incident beam to the (002) spot).

The diffraction spot streaking is not unique to chrysotile alone. Some clay minerals also exhibit streaking in the same manner, such as halloysite, and vermiculite where the structure has been altered.
With the current camera constant, make the following measurements from the micrograph negative:

A. Distance "r" between the rows in millimeters (mm) perpendicular to the row axis.

Formula:

$$CC = \frac{d \text{ (in angstroms)}}{r}$$

CC = Camera constant (in millimeter - angstroms).
r = Measured spacing in the rows or spots in millimeters.
d = Actual spacing of rows or spots in angstrom units.

Example: CC = 24.88 mm-angstroms.
r = 4.7 mm (spacing or rows/spots in mm).

$$\frac{24.88}{4.75} = 5.2 \text{ angstroms.}$$

►ACCEPTED ROW SPACING RANGE = 5.2 - 5.4 angstroms

CHRYSOTILE DIFFRACTION PATTERN ILLUSTRATION

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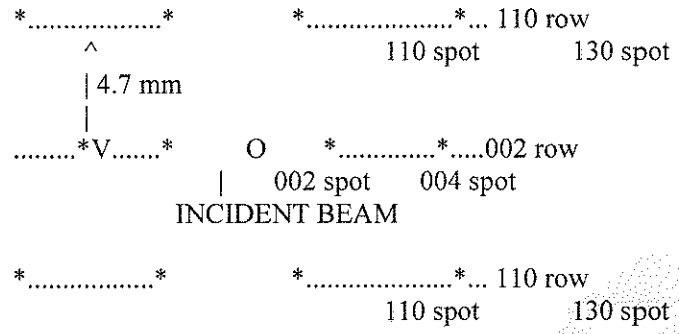
(make reference to a chrysotile standard SAED micrograph)



B. Measure the distance between the "110" spots. Calculate the "d" spacing using the camera constant and the formula in "A".

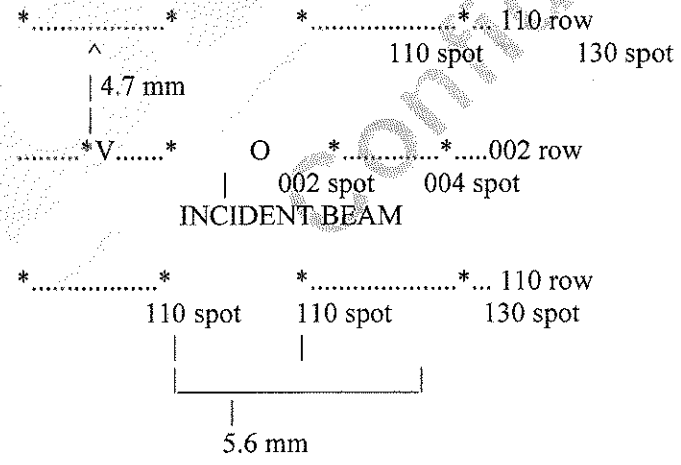
Example: CC = 24.88 mm-angstroms.
r = 5.6 mm (spacing or rows/spots in mm).

$\frac{24.88}{5.60}$ = 4.4 angstroms.

▶ACCEPTED DISTANCE BETWEEN 110 SPOTS RANGE = 4.0 - 5.0 angstroms.

CHRYSOTILE DIFFRACTION PATTERN ROW SPACING ILLUSTRATION

(Make reference to a chrysotile standard SAED micrograph)



C. Measure the distance between the incident beam (undiffracted beam) and the 002 spot. The spot may be difficult to see as well as identifying the incident beam. If the incident beam and the 1st order 002 spots are too

difficult to identify, then measure the distance between the next 002 spots (2nd order spots). Be sure to divide by 4 to get the correct value.

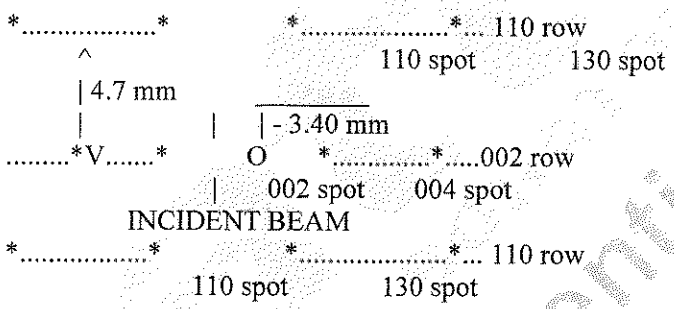
Calculate the "d" spacing using the camera constant and the formula in "A".

Example: CC = 24.88 mm-angstroms.
r = 3.40 mm (spacing or rows/spots in mm).

$$\frac{24.88}{3.40} = 7.3 \text{ angstroms.}$$

▶ **ACCEPTED DISTANCE BETWEEN 002 SPOTS RANGE = 7.3 angstroms.**

CHRYSOTILE DIFFRACTION PATTERN ROW SPACING ILLUSTRATION
(Make reference to a standard chrysotile SAED micrograph)



V. Record all measurements and calculations onto an index card and tape the negative, from which the measurements were made, onto the index card.

VI. Record the I.D. number of the negative onto the index card.

VII. If the previous measurements meet the criteria for chrysotile identification specifications, then record "CHRYSOTILE" on the index card.

VIII. For further confirmation of the chrysotile diffraction pattern, the angle measured from the 002 row (horizontal) starting at the beam of incident, to the 110 spot should approximately equal 60 degrees (+ or - 3 degrees).

IX. If the previous measurements do not meet the criteria for chrysotile, re-measure the diffraction pattern.

X. If the re-measurement still does not constitute a chrysotile diffraction pattern, then contact the technical supervisor.

XI. If the technical supervisor finds the measurements not to be a chrysotile diffraction pattern, and the structure has been reported as chrysotile, based upon the diffraction pattern solely and contributes to a change in the reported fiber concentrations, then the technical supervisor will contact the client. "AMBIGUOUS" shall be recorded on the index card.

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VI-VIII: SAED MEASUREMENT AND INTERPRETATION OF AMPHIBOLES STANDARD OPERATION PROCEDURE

Monoclinic amphiboles are in the category of minerals that can not be fully identified on the basis of diffraction alone. Although it may be possible to identify them as amphiboles, discrimination between them, e.g. between hornblende and a tremolite requires EDXA.

Amphibole SAED patterns have the following characteristics on the screen and micrographs:

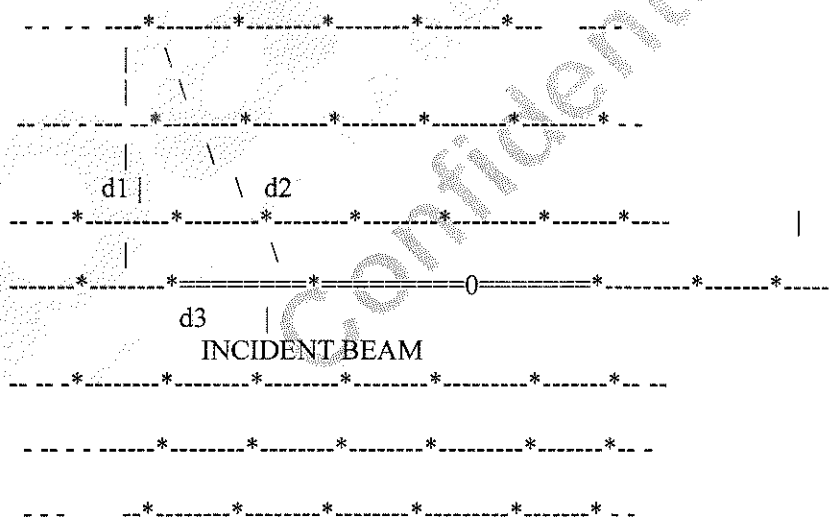
- layer lines (row spacing) from very closely spaced dots
- repeat distance between the layer lines (row spacing) are approximately 5.3Å.
- streaking in layer lines are occasionally present due to crystalline structure defects.

The pattern illustrated is particularly difficult to achieve and the angles will usually not equal 90 degrees.

This is an ideal monoclinic diffraction pattern for an amphibole illustration.

AMPHIBOLE DIFFRACTION PATTERN

▼Tips: Zone axis is parallel to beam of incident.
Make reference to a standard amphibole SAED micrograph.



I. Measure the distance "d1" between the rows in millimeters (mm) perpendicular to the row axis as indicated in the diffraction illustration.

Note: In the illustration, divide "d1" by 3 because the measurement is through 3 row spacings.

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d1 = Measured spacing of the rows or spots in millimeters.

d1 = 28.2 mm (which is the total distance of d1 over 3 row spacings. Note the illustration).

d1 = Average spacing distance between the rows or spots in mm.

Example:

$d1 = \frac{28.2}{3} = 9.4$ mm (average distance between the three rows)

II. Repeat step I for measurements and calculations for d2 and d3.
III. Measure Angle 1 and Angle 2 as accurately as possible.
IV. Record d1, d2, d3, Angle 1 and Angle 2 respectively onto an index card.
V. If an EDXA spectrum was taken of the same structure, determine the asbestos mineral which it may represent and look up the respective mineral in the Mineral Powder Diffraction File Data Book and record onto the same index card the following physical data: Lattice Parameters (a,b, and c). Inter-axial angle Beta.

Example: Grunerite (Amosite) - monoclinic crystal system

a = 9.57b = 18.22 c = 5.33
Beta = 102.1

▼**Tips:** Examples of standards are in the posted on the wall in the TEM lab.

VI. If an EDXA spectrum was not taken of the same structure, determine the asbestos mineral which it may represent by looking up the respective asbestos minerals in the Mineral Powder Diffraction File Data Book and record onto the same index card the following physical data for each regulated asbestos mineral:

Lattice Parameters (a,b and c).
Interaxial angle Beta.

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VI-IX: JAFEE WASH STANDARD OPERATION PROCEDURE

For MCE filters:

I. Custom cutting the wicking substrate for the Jaffee wash, perform the following:
   - A. Place one pre-cut piece of foam into a 5cm petri-dish.
   - B. Using scissors, cut (or use pre-cut) 2-4 pieces of paper tissue (Kimwipes™ work best) where they will fit over the without touching the sides of the petri-dish.
   - C. Fill the dish with Acetone just before the liquid puddles within the dish.
   - D. Grids may now be added to the dish.

For PC filters:

II. In a pre-cleaned, five (5) centimeter petri-dish:
   - A. Place one pre-cut piece of foam into a 5cm petri-dish.
   - B. Using scissors, cut (or use pre-cut) 2-4 pieces of paper tissue (Kimwipes™ work best) where they will fit over the without touching the sides of the petri-dish.
   - C. Be sure that the foam is not touching the sides of the petri-dish.
   - E. Be sure that the foam does not touch the cover of the petri-dish.
   - F. Fill the dish with Chloroform just before the liquid puddles within the dish.
   - G. Grids may now be added to the dish.

The above procedure has been recently adopted in lieu of DMF use.

▼**Tips:** It is important to ID the sample grids. Refer to Step II.A of Section VI-II of this appendix for method of assigning Ids. However, all marks should be on the cover of the petri-dish. It is also recommended to write Ids on a piece of paper with a pensile and carefully insert the paper inside the petri-dish in accordance with the sample order.

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1. If the sample replica appears intact and is covering greater than approximately 50% of the grid openings, and also appears to have no noticeable un-dissolved filter remaining, with forceps, transfer the grids to their assigned grid box locations for future analysis.
2. If the sample replica appears to have un-dissolved filter remaining, repeat step VI (F through L).
3. If the sample replica appears to be less than or equal to approximately 50 percent intact, repeat steps V (A through J) and VI (A through L) for that sample.

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VI-XI: TEM LAB CONTAMINATION CHECK (PASSIVE) STANDARD OPERATION PROCEDURES

I. With a pair of forceps, separate the 0.45□m, 25mm MCE filter from its 5.0μm diffuser and backing pad. Be careful not to touch the active area of the filter.
II. Lay the filter on filter paper in a petri dish identified with that day's date and location where the filter is to be placed.
III. Repeat steps I and II to prepare two (2) more filters.
IV. Lay the filters in strategic locations:
    A. Place one inside prep hood #1.
    B. Place one inside prep hood #2.
    C. Place one on a flat surface within three feet of the TEM analytical area.
    D. Leave the filters for 11-14 days undisturbed and open to the ambient air.
    E. Prep as a TEM AHERA sample in accordance with the SOP.
    F. Document in the QA/QC Program Excel™ spreadsheet for blanks.

This is required to be accomplished once every six months, following any service to the equipment, any significant disturbance to the room (e.g. replacing of ceiling tiles) or cleaning.

IV. For each open sample laboratory (passive) blank:

    A. Analyze by AHERA method.
    B. On subsequent analysis, a sample of less than (<) 18 structures per millimeter squared (s/mm^2) must be maintained.
    C. If subsequent analyses exceed an average of greater than (>) 53 structures per millimeter squared (s/mm^2), then wet wipe all exposed areas.
    D. Repeat steps I through VIII (A and B).
    E. If the analysis of a sample exceeds 18 structures per millimeter squared (s/mm^2) for any single preparation, then wet wipe all exposed areas. Repeat steps I through VIII (A through D).

The documentation of this passive contamination check is complied with corresponding monthly summary reports.

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VI-XII: TEM LAB CONTAMINATION CHECK (ACTIVE) STANDARD OPERATION PROCEDURES

This is required to be accomplished quarterly, following any service to the equipment, any significant disturbance to the room (e.g. replacing of ceiling tiles) or cleaning. Air pumps are set at three strategic areas: Prep Hood #1, Prep Hood #2, and the PLM Prep Area (see Lab Layout View in **Appendix V**).

For each open sample laboratory (active) blank:

A. At least 1200 liters volume of air collected.
B. Analyze by AHERA method.
C. On subsequent analysis, a sample of less than (<) 18 structures per millimeter squared (s/mm^2) must be maintained.
D. If subsequent analyses exceed an average of greater than (>) 53 structures per millimeter squared (s/mm^2), then areas need to be cleaned.

The documentation of this air monitoring is complied with corresponding monthly summary reports.

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VI-XIII: REAGENT PURITY CHECK STANDAR OPERATION PROCEDURES

Type of Reagent	Preparation Method	Method of Testing	Frequency of Operation
Acetone	Evaporation, sonication and filter	AHERA	Semi-annually
Ethyl Alcohol	Filtration	AHREA	Semi-annually
Distilled Water	Filtration	AHERA	Semi-annually

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Appendix VII

ROUTINE ALIGNMENTS AND CALIBRATIONS FOR TEM ANALYSIS

Table of Contents

VII-I: STATEMENT OF PURPOSE

Alignments of TEM scope and calibrations of instruments involved in all TEM analyses are vital to the assurance of analytical accuracy and data quality. These alignments and calibration conducted periodically are required in various QC/QA programs that BLI have been participating, such as NVLAP, AIHA, etc. Routine alignments of TEM scope are conducted daily and each time before analysis of client's samples. Frequency of calibrations depends on the type of calibrations, ranging from daily to semi-annual. These calibrations are described in the following sections.

VII-II: TEM ALIGNMENTS (DAILY) STANDARD OPERATION PROCEDURE

This task is done daily and documented with clients' analytical datasheets (Appendix V). The following order of steps is performed in reference to JEOL 100CX II Instructions manual for standard operation procedures:

Electron beam generation (JEOL Method A, Section 5.2.5, P5-15)
Condenser Lens Alignment (JEOL Method A, Section 5.2.5, P5-17)
Beam Centering Alignment (JEOL Method B, Section 5.3.6, P5-49)
Electron Gun Alignment (JEO Method B, Section 5.3.3, P5-42)
Checking Condenser Lens Astigmation (JEOL Method B, Section 5.3.2, P5-40)
Checking Objetive Lens Displacement (JEOL Method B, Section 5.3.4, P5-45)

► **Reference: JEOL 100 CX II INSTRUCTIONS (Appendix IV)**

VII-III: ANALYTICAL TEM SPOT SIZE CALIBRATION STANDARD OPERATION PROCEDURE (MONTHLY)

This calibration is to be performed **MONTHLY.**

I. Have the TEM activated and aligned.

► **Reference: TRANSMISSION ELECTRON MICROSCOPE (TEM) ALIGNMENT STANDARD OPERATION PROCEDURE** (Section VII-II)

II. Filament emission is to the pre-designated stop.
III. Magnification = 19,000x.
IV. Acceleration Voltage = 100 KV.
V. Objective Lens Aperture - position 0.
VI. Field Limiting Aperture - position 0.
VII. Turn the spot size knob to three (3).
VIII. Condense the beam with the condenser to crossover.
IX. Center the beam with the alignment trans knobs.
X. Turn the spot size knob to four (4).
XI. Condense the beam with the condenser to crossover.
XII. Center the beam with the alignment trans knobs on the inner circle on the fluorescent screen.

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XIII. With the alignment trans knobs carefully position the beam within the radius of the inner circle. It may be necessary to use the X and Y condenser stigmator located in the right hand cabinet on the TEM to adjust the roundness of the spot. Be sure that the spot is adjusted to its maximum roundness.

XIV. Record a micrograph of the image. Create a sweeping motion in a singular, diagonal direction (by turning one of the alignment trans knobs) during exposure to create a "line" that can be measured from the film. Enter the width in millimeters of the "line" (as seen on the developed micrograph) into the Spot Size Measurement section of the Magnification worksheet for that month (as part of the **TEM Monthly Summary**). Record also the date of measurement and the plate number of the micrograph.

XV. If the spot size is less than or equal (≤) to 0.250 um, then the spot size is within acceptable limits.

XVI. If the spot size is greater than (>) 0.250 um, then re-align the TEM and repeat steps I through XV.

XVI. If the spot size is still greater than 0.250 um (250 nm), then contact the laboratory manager for further actions.

VII-IV: PLASMA ASHER MCE FILTER ETCHING TIME CALIBRATION (THREE MONTHS) STANDARD OPERATION PROCEDURE

This calibration is to be performed every three (3) months.

I. Take a clean glass slide. Clean both surfaces (top and bottom) if necessary to make sure that the slide is free of debris and dusts. Determine its weight in grams and place the value in the Plasma Asher Calibration Template located in T:\QA-QC Program\QAQC Data.

II.

III. Follow the standard operation procedures (see reference below) to prepare 3 pie-shaped quarters of laboratory grad blank TEM MCE filters (25mm diameter, 385 mm^2 area, and 0.45 μm pore size) and collapse the filters by acetone vapor.

▶ **Reference: SAMPLE PREPARATION OF MIXED CELLULOSE FILTERS STANDARD OPERATION PROCEDURE** (Section VI-II of Appendix VI)

IV. After collapsing by acetone, determine its weight and record in the template.

V. Ash the glass slides and filters following the standard operation procedures and ash the filters for ten (10) minutes or a period not to exceed twenty (20) minutes and not less than 5 minutes.

▶ **Reference:** ASHING MIXED ESTERS OF CELLULOSE FILTERS STANDARD OPERATION PROCEDURE (Section VI-I of Appendix VI)

VI. After ashing, wait for the temperature to equilibrate to ambient air temperature and determine the mass of the filters and glass slide in grams and record in on the template.

VII. Repeat Steps I through V as the second round of measurement, and make sure to record all data on the template.

Note: The calibration should take place over a 20-minute time period to yield roughly 10% etching; however, NVLAP mandates 5% etching, which visually gives an orange peel-like surface texture on the filter surface once placed under TEM. Due to various factors, this etching method along can yield either an under etched or over etched surface. Therefore, visual calibration after the quantitation method is mandated in plasma etcher calibration in this laboratory.

VIII. After the template calculates an etching time, prep three lab grad blank samples using the following three etching times: the template calculated time, and the times with 15 seconds off setting (+ and -) the template calculated time. Observer and compare the three preps under the TEM scope and determine which time gives a better or optimal orange-peel like surface texture. If none of the above is achievable, redo the calibration by repeating Steps I through VII. An optimal time can be empirically interpolated between the above three values if needed, and shall be documented on the template.

IX. Document the calibrated time on the template, and post the calibration chart with final calibrated time on the wall next to the etcher.

Note: 1. Depending upon the system stability (such as no breakdowns, no major part replacements or maintenance, etc.), quantitative calibration (Steps I through VI) can be omitted and a visual calibration based on the calibrated time of the last quarter is acceptable. However, this must be calibrated by prepping three lab blanks following Step VII above, and must be done on a monthly basis until the next quantitative calibration takes place. If the visual calibration cannot achieve an acceptable result, the quantitate method is mandated. **2.** The visual calibration only shall not exceed three consecutive months; and a quantitative approach must be done in the following quarter. **3.** If the desired etching cannot be achieved in the time range suggested in Step IV, adjust the plasma power level or oxygen flow/pressure and repeat Steps I through VII.

VII-V: CHRYSOTILE BEAM DOSE CALIBRATION STANDARD OPERATION PROCEDURE (6 MONTHS)

This calibration is to be performed every six months.

I. Activate and align the TEM scope.

► **Reference:** **TEM ALIGNMENT STANDARD OPERATION PROCEDURE FOR ASBESTOS ANALYSIS** (Section VII-II)

II. From a pre-prepared grid:

A. Perform SAED on at least 50 chrysotile unit fibrils at 100 KV which are ≤0.1μm.

1. For each chrysotile structure in which an SAED is performed, record them on to the CHRYSOTILE BEAM DOSE EXCEL™ WORKSHEET (Appendix V).
2. If the SAED for chrysotile is recognizable for ≥15 seconds, record a "1."

3. If the SAED for chrysotile is not recognizable for ≥15 seconds, record a "0."

B. Calculate the percentage of chrysotile structures that produced SAED patterns for 15 seconds or longer.

III. If the calculated percentage is ≥90%, then the requirement for beam dosage at 100 KV is met.

IV. If the calibrated percentage is <90%, then contact the lab manager or the technical supervisor. Gun bias and beam current adjustments may be needed to correct the problem. Repeat Steps II through IV if error sources are found and corrected.

V. Complete the BEAM DOSE EXCEL™ WORKSHEET with calibration data acquired.

VII-VI: D-SPACE MEASUREMENTS FROM THE TEM FLUORESCENT SCREEN CALIBRATION STANDARD OPERATION PROCEDURE (MONTHLY)

The fluorescent screen calibration for visual screen d-spacing is performed for every new camera constant calibration. These calibrations are performed on a monthly basis.

I. Load the gold grid standard into the sample specimen holder with forceps.

II. Observe the grid at LOW MAG by perform the following:
- A. Acceleration Voltage = 100 KV.
- B. Turn the Filament Emission knob to the pre-determined stop slowly.
- C. Objective Lens Aperture - Position 0
- D. Field Limiting Aperture - Position 0
- E. Turn the Condenser knob to spread the beam if needed.
- F. Turn the left Specimen shifting knob clockwise until it comes to the stops.
- G. Turn the Specimen selector to where the left side of the grid is approximately oriented in the center of the fluorescent screen to prevent from traversing beyond the stops on a grid opening at high magnification.

III. For polycrystalline diffraction ring analysis of the gold standard do the following:
- A. Acceleration Voltage = (must be) 100 KV.
- B. Push the MAG button on the TEM. This will zoom in on the grid opening selected.
- C. Turn the MAGNIFICATION knob to 19,000x magnification.
- D. Turn the CONDENSER knob to beam crossover.
- E. With the ALIGNMENT TRANS knobs, center the beam.
- F. Turn the CONDENSER knob to spread the beam.
- G. Focus the image with the MEDIUM FOCUS knobs.
- H. Objective Lens Aperture - Position 0

  Make sure the Goniometer is eucentric by doing:
  1. Center the image in the center of the fluorescent screen.
  2. The eucentric adjusting screw is located on the Goniometer just below the specimen holder port.
  3. With the Goniometer unlocked, turn the Goniometer counter-clockwise.
  4. The image will either move to the left or right.

5. Adjust the movement of the structure by turning the eucentric screw in either direction to compensate. The goal is to obtain a stationary image while the Goniometer is turned.

I. The Camera Length should be set on 22 cm.
J. Objective Lens Aperture - position 0.
K. Field Limiting Aperture - position 4.
L. Center the polycrystalline surface with the Field Limiting Aperture alignment knobs.
M. Press the pop-up screen button to elevate the screen.
N. Center the polycrystalline surface with the Field Limiting Aperture alignment knobs if needed.
O. Press the SA DIFF function button.
P. If the diffraction rings need focusing, use the camera length focus knob which is the smaller knob of the camera length knobs.

III. Confirming that the polycrystalline diffraction rings are illuminated on the fluorescent screen at a camera length of 22 centimeters, with the camera length outer knob turn the camera length to 55 centimeters, which will be displayed on the MAGNIFICATION/CAMERA LENGTH on the TEM.

IV. Record the following information for calibration:
A. Diameter of Inner Inscribed Circle
B. Radius of Au Ring #1
C. (d) Au Ring#1

To obtain select area electron diffraction patterns, use 55 cm to observe SAED patterns on the screen and leave the small pop-up screen flat in order to estimate d-spacing on the screen of a diffraction pattern.

Direct screen measurements are a rough estimation for d-spacing and visual measurements are difficult to determine. The best way to determine the actual d-space measurement is to take a micrograph of the diffraction pattern and perform measurements from the negative.

Measurements from Asbestos Standards

Chrysotile and amphibole standards, on this particular scope, typically show the d-spacing between the rows to be approximately one half the diameter of the inner inscribed circle (3.8 mm). This should be however checked each time a new camera constant is calculated on a monthly basis.

V. Enter recorded data to the D-Space Measurement Form in Excel spreadsheet format for automatic calculation.

VII-VII: CAMERA CONSTANT CALIBRATION (MONTHLY) STANDARD OPERATION PROCEDURES

The TEM camera constant calibration must be determined on a monthly basis.

I. Load the gold grid standard into the sample specimen holder with forceps.
II. To observe the grid at LOW MAG, perform the following:

A. Acceleration Voltage = 100 KV.
B. Turn the filament emission knob to the pre-determined stop slowly.
C. Objective Lens Aperture - Position 0
D. Field Limiting Aperture - Position 0
E. Turn the condenser knob to spread the beam if needed.
F. Turn the left specimen-shifting knob clockwise until it comes to the stops.
G. Turn the specimen selector to where the left side of the grid is approximately oriented in the center of the fluorescent screen (this step will prevent you from traversing beyond the stops on a grid opening at high magnification).

III. Polycrystalline diffraction ring analysis of the gold standard must be done at 19,000x magnification.
A. Acceleration Voltage = (must be) 100 KV.
B. Push the MAG button on the TEM that will zoom in on the grid opening selected.
C. Turn the MAGNIFICATION knob to 19,000x magnification.
D. Turn the CONDENSER knob to beam cross-over.
E. With the ALIGNMENT TRANS knobs, center the beam.
F. Turn the CONDENSER knob to spread the beam.
G. Focus the image with the MEDIUM FOCUS knobs.
H. Objective Lens Aperture - Position 0. Check the eucentricity of the Goniometer by following Step III-H in Section VIII-VI of this appendix.
I. The camera length should be set on 22 cm.
J. Objective Lens Aperture - position 0.
K. Field Limiting Aperture - position 4.
L. Center the polycrystalline surface with the Field Limiting Aperture alignment knobs.
M. Press the pop-up screen button to elevate the screen.
N. Center the polycrystalline surface with the Field Limiting Aperture alignment knobs if needed.
O. Press the SA DIFF function button.
P. If the diffraction rings need focusing, then use the camera length focus knob which is the smaller knob of the camera length knobs.

IV. With the polycrystalline diffraction rings illuminated on the fluorescent screen at a camera length at 22 centimeters:
A. With the camera length outer knob, turn to 55 centimeters which will be displayed on the MAGNIFICATION/CAMERA LENGTH on the TEM.
B. With the condenser knob, turn the brightness down to where the image of the SAED pattern of the structure is barely visible on the screen.
C. Be sure film is loaded in the TEM. The unused film counter will indicate status of unused film.
D. The camera airlock open light should be on.
E. Push the film advance button to advance the film plate into position.
F. Check to make sure that the exposure sensitivity is set at 7.5.
G. The exposure shutter speed should be set at 22 seconds.
H. Cover the viewing window with the window cover.

I. Push the fluorescent screen button (the button is not labeled, but it is located closest to the film advance button) which will light up.
J. The EXP red light will come on during the assigned exposure time.
K. Avoid touching the TEM during the exposure time.
L. When the EXP red light turns off, remove the window cover.

V. Remove the film-receiving magazine from the camera chamber.
A. Turn the filament emission knob off slowly.
B. Open the camera chamber door by turning the handle counter-clockwise.
1. The TEM column will decompress to atmosphere and will also make a hissing sound that is normal.
2. The camera chamber door will automatically open after two (2) to three (3) minutes.
C. Remove the film-receiving magazine from the camera chamber. Be careful not to let the lid to the film receiving magazine to even slightly open. This will expose (flash) the negative. You will then have to take another gold ring diffraction micrograph again!

VI. Immediately, take the film-receiving magazine to the Dark Room and develop following the micrograph developing procedures.

VII. After developing the gold ring diffraction negative, perform the following:
A. Initial and write the day's date of the calibration and the plate number of the negative to be measured on determination of the camera constant form.
B. Determine the average camera constant by measuring the multiple gold rings on the gold ring diffraction negative.
Equipment needed:
a. Clear straight edge with millimeter scale.
b. Marker pen or pencil.
c. White table surface or a light table.
d. The camera constant form (T:\\QA-QC Program\QAQC Data).

C. Lay the gold ring negative on a white surface. Use a light if possible.
D. The d-spacing of interest on the SAED patterns usually lie closest to the center spot.
E. With a marker pen draw three lines intersecting the graph center spot at 60 degree angle that pass the 1st and 3rd gold rings.
F. Measure the diameter of the first gold ring to a 10th of a millimeter (example: 21.2 mm) for all three angles (0, 50 and 120 degrees) and record the averaged result on the camera constant form.
G. Measure the diameter of the 3rd gold ring to a 10th of a millimeter (example: 21.2 mm) for all three angles (0, 50 and 120 degrees) and record the averaged result on the camera constant form.
H. Determine the camera constant for each gold ring measured and record the results on the camera constant form (automated).

VIII. After determining the camera constant, enter the camera constant data into the TEM computer on the Excel™ spreadsheet (T:\\QA-QC Program\QAQC Data).

IX. File the negatives with the corresponding monthly summary binder.

VII-VIII: EDXA Mn K ALPHA PEAK RESOLUTION MEASUREMENTS (MONTHLY) STANDARD OPERATION PROCEDURE

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

Resolution is measured as the width (horizontally), in energy (eV) of the manganese K-alpha (Mn k-alpha line). Actually, a gaussian distribution about the centroid) is at a point equal to one-half the height of this line (or "peak"). Counts (x-ray events from the detector) are accumulated in the multi-channel analyzer (MCA) until there are 2000 counts in the peak (centroid) channel. The width of the peak at the "half-maximum" point (1000 counts) is then measured (either in channels or in eV) to determine the resolution. Thus, resolution is expressed as, "146 eV FWHM" (Full Width Half Maximum).

Should a count other than 2000 in the centroid channel be used, one of two results would be expected. Counts less than 2000 will result in resolution measurements which vary considerably from one measured to the next (e.g., 146 eV, 135 eV, 150 eV, etc., FWHM for a given detector) since the total number of counts is too small to achieve a significant statistic basis. However, an average of a series of such measurements would be close to the actual resolution. The other extreme would be to use more than 2000 counts in the centroid channel. To do so introduces another factor - long term stability - into the resolution measurement. Amplifier stability (over a long period of time) will have an impact on the resolution measurement and therefore is not considered a part of the resolution measurement. An unstable amplifier will cause the energy of the x-ray line to shift about its true position and broaden the peak. A system that exhibits the same resolution at different counts in the centroid channel certainly has a stable amplifier.

For example:

By visualizing a curve which intersects the count and the lower energy limit of each channel (e.g. at 5.82 keV and 912 counts, the first point on the curve, then 5.83 keV and 1222 counts for the second point, etc.), the resolution determination is simplified. From this curve, there are 14 channels above the 1000-count level. At low-energy and high-energy half maximum points, the curve crosses the 1000-count level between channel boundaries (outside the whole 14 channels). In these regions, a linear interpolation may be used to determine the energy at which the curve crosses the half-maximum point. For a definitive mathematical determination, a worksheet program set up in Excel™ is used. See QA/QC manager for details.

At the low energy side of the curve, the channel beginning at 5.82 keV (covering 5.82 through 5.829 keV) contains 912 counts. The channel beginning at 5.83 keV contains 1222 counts. The curve must then cross the 1000-count level between 5.82 keV and 5.83 keV. The crossover point (in eV above 5.82 keV) would be:

$$\frac{(1000 \text{ counts} - 912 \text{ counts})}{(1222 \text{ counts} - 912 \text{ counts})} \quad (10 \text{ eV/channel}) = x$$

$$\frac{(88 \text{ counts})}{(310 \text{ counts})} \quad (10 \text{ eV/channel}) = x$$

$(0.28)\ (10 \text{ eV/channel}) = x \qquad x = 2.8 \text{ eV or } 0.0028 \text{ keV}$

The low-energy side crossover point is thus 5.82 keV + 0.0028 eV, or 5.823 keV.

At the high-energy side of the curve, the channel beginning at 5.97 keV (covering 5.97 to 5.979 keV) contains 1150 counts. The channel beginning at 5.98 keV contains 845 counts. The curve crosses the 1000-count level between 5.97 and 5.98 keV. The crossover point (in eV above 5.97 keV) would be:

$$\frac{(1150 \text{ counts} - 1000 \text{ counts})}{(1150 \text{ counts} - 845 \text{ counts})} \quad (10 \text{ eV/channel}) = y$$

$$\frac{(150 \text{ counts})}{(305 \text{ counts})} \quad (10 \text{ ev/channel}) = y$$

$(0.49)\ (10 \text{ eV/channel}) = y \qquad y = 4.9 \text{ eV or } 0.0049 \text{ keV}$

The high-energy side crossover point is thus 5.97 keV + 0.0049 eV, or 5.975 keV.

Resolution (the full width of the peak at the half-maximum point) is the difference in energy between the high-energy side crossover point (ex: 5.975 keV) and the low-energy side crossover point (ex: 5.823 keV). Thus, (5.975 keV – 5.823 keV), or 0.152 keV (152 eV FWHM).

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▶**Reference:** **MANGANESE K-ALPHA PEAK WITH THE TYPICAL VALUES OF COUNTS IN CHANNELS NEAR THE HALF MAXIMUM POINT (Appendix V)**

Perform the following with standard NIST MnO_2:
The EDXA system must be calibrated (Al/Cu) before executing the following.

▶**Reference:** **ENERGY DISPERSIVE X-RAY Cu & Al CALIBRATION STANDARD OPERATION PROCEDURE** (Section VII-XI)

Automatic Calibration (Mandatory):

I. Load the standard Mn (NIST MnO_2) grid into the sample specimen holder with forceps.
   - A. Carefully place the specimen holder, in the specimen holder magazine, point first (do NOT let the ruby, at the point of the specimen holder, touch any surface.
   - B. Gently push the handle of the specimen holder into the specimen holder magazine until you hear a click.
   - C. The specimen holder will now stay in place and the vacuum pumps will come on.
   - D. The red light on the X-tilt goniometer will come on.
   - E. When the specimen holder magazine equalizes pressure with the column of the TEM interior proper, the red light will turn off.
   - **F.** Holding the handle of the specimen holder firmly, slowly turn the handle counter-clockwise where the specimen holder will then be sucked into the specimen chamber.

II. To observe the grid at LOW MAG, perform the following:
   - A. Acceleration Voltage = 100 KV.
   - B. Turn the Filament Emission knob to the pre-determined stop SLOWLY.
   - C. Objective Lens Aperture – Position 0
   - D. Field Limiting Aperture – Position 0
   - E. Turn the Condenser knob to spread the beam if needed.
   - F. Turn the left Specimen shifting knob clockwise until it comes to the stops.
   - G. Turn the Specimen selector to where the left side of the grid is approximately oriented in the center of the fluorescent screen (this step will prevent you from traversing beyond the stops on a grid opening at high magnification).

III. For SPOT SIZE 3 perform the following steps.
   - A. Filament emission is to the pre-designated stop.
   - B. Magnification = 19,000x.
   - C. Acceleration Voltage = 100 KV.
   - D. Center the structure on the fluorescent screen.
   - E. Focus the image.
   - F. Objective Lens Aperture – position 0.
   - G. Field Limiting Aperture – position 0.
   - H. Turn the spot size knob to two (2).
   - I. Condense the beam with the condenser to crossover.
   - J. Center the beam with the alignment trans knobs.
   - K. Turn the spot size knob to three (3).
   - L. Condense the beam with the condenser to crossover.
   - M. Center the beam with the alignment trans knobs on the structure to be analyzed.

IV. Noran (Quest) X-ray Analyzer computers activated.

1. Refer to manufacturer's instructions for start-up procedure.

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2. To determine the resolution (FWHM and FWTM), the **Expert** menu must first be initiated. If it is visible (to the right of the Help menu), proceed to step 4. If not, proceed to step 3.
3. With the **Control-Alt-Shift** keys pressed, right click over the toolbar section (to the right of the last icon. This will make the **Expert** menu accessible.
4. Locate a Mn particle with the size and density sufficient to allow between 1000 and 2500 counts per second. This will ensure representative accumulation of counts without overloading the detector. Set spot size to 3 and bring the beam to crossover atop the particle to be tested.
5. Double-click on the monitor's keV scale to bring up the **Scale** window. Enter values for 'X', effectively spreading Mn peak (and sufficient low and high background channels) to fit the screen. Proper 'X' values to enter would be 5.50 and 6.25. Click **OK** and proceed to the **Expert** menu.
6. After clicking the **Expert** menu, choose **Measure FWHM**. If you are prompted to save the existing spectra, click No or Yes depending on if the current spectra needs to be kept. If No is chosen, the spectra displayed will be erased. If Yes is chosen, you will be prompted to decide where to save the spectra currently displayed.
7. Configuring the Mn Resolution Test: When the **FWHM Measurement** window pops up, choose Mn as the atomic symbol, K as the **Line**, **Livetime (s)** as either a minimum of 100 or a maximum of 200. It is also acceptable to choose **Peak Counts**. If **Peak Counts** are chosen, a value of 2000 is appropriate. The **No. trials** value chosen is based on the analyst's time available (more trials run, more time taken to execute the test), however the value should be a minimum of 3. For **Additional Measurements**, chose the Measure FWFM and FWTM option. While FWFM is not a concern, there are NVLAP guidelines on proper values for FWTM. Consult the latest NVLAP checklist to determine appropriate values of FWTM.
8. Click **Run** to begin the test.
9. The **FWHM Results** window will pop up as the test begins. Each trial's result will post inside this window as it is completed. The test will not be completed until all trials requested (in step 7) are executed. Should you wish to stop the test prior to completion, click the **Pause** or **Stop** button. Clicking **Pause** will permit you to resume the test whenever you are ready. Clicking **Stop** effectively ends the test session. After clicking **Stop**, you will have to restart the test from the beginning (Step 8) to complete the test.
10. During the test, keep an eye on the cps value at the bottom of the screen. If the value goes below 1000 or above 2500, reposition the particle or beam spot location to re-achieve the proper counts per second (see Step 4).
11. After all trials are completed and the results – statistics are posted inside the window, print out the values and keep for your records. Save spectra to the following location: **C:\Noran\Quest\Spectra\QA-QC Spectra\EDX Calibrations\Mn Resolution**. Inside the Mn Resolution subdirectory, there are additional locations for year and month. Ensure the final location is appropriate for the date performed.

Manual Calibration (optional: only when auto-calibration fails or in question):

12. Using a blank measurement form found on the TEM Laptop at the following location: **C:\Bob's Desktop\QA-QC and T-Drive\QA-QC Program\QAQC Data\Personnel Forms-Worksheets\Main Lab Worksheets\Wkshts-TEM\EDXA Forms\MnGRAFPAPR**, enter peak heights at the energy levels specified. For the high and low end background counts, enter the calculated average height between 5.50 keV and 5.65 keV (for the low end) and between 6.12 keV and 6.23 keV (for the high end). File this form (with a print-out of the spectra) as part of the TEM Monthly Summary for that month. Enter the Mn peak counts onto the Excel spreadsheet MnRes worksheet of that months' TEM Calibration file. The Calibration files (named Temcal*.xls) are located in the C drive of the TEM Laptop.

VII-IX: EDXA RELATIVE SENSITIVITY K-FACTORS FOR Mg, Si, Ca, AND Fe (Biennially)

This operation is performed bi-annually (once/2 years).
An x-ray detector's performance on a TEM can change dramatically as a function of time, often due to damage to the silicon detector or from oil and/or ice contamination of the detector and/or detector window. These problems can cause peak broadening across the spectrum and reduced sensitivity at lower x-ray energies. Since these changes in sensitivity are a function of x-ray energy, they can affect the identification criteria for asbestos. Calibration of the detector to check for these effects is with a reference material of known thickness and composition that yields both low and medium to high energy x-rays, such as found in SRM 2063a. The variation in k-factors over time is monitored.

Relative sensitivity factors and detector resolution are useful for monitoring detector performance. The parameters that are directly or indirectly monitored with these factors relate to quantitative analysis and general detector characteristics such as the detector efficiency, electronics, presence of absorbing layers on the detector window, etc. will be monitored by these measurements.

Standard Required: SRM 2063a

Elements for Sensitivity Measurement:

Mg (Magnesiun)	7.97% by wt
Si (Silicon)	25.35% by wt
Ca (Calcium)	11.82% by wt
Fe (Iron)	11.06% by wt

Perform the following for the standard SRM 2063a:

Load the grid into the sample specimen holder with forceps.
Carefully place the specimen holder, in the specimen holder magazine, point first (do not let the ruby, at the point of the specimen holder, touch any surface.)
Gently push the handle of the specimen holder into the specimen holder magazine until you hear a click.
The specimen holder will now stay in place and the vacuum pumps will come on.

The red light on the x-tilt goniometer will come on.
When the specimen holder magazine equalizes pressure with the column of the TEM interior proper, the red light will turn off.
Holding the handle of the specimen holder firmly, slowly turn the handle counter-clockwise where the specimen holder will then be sucked into the specimen chamber.

Relative Sensitivity (K-Factors) Data Collection – Using Quest Analyzer

1. Noran (Quest) X-ray Analyzer computers activated.
2. Refer to manufacturer's instructions for start-up procedure.
3. Locate a grid opening with the specimen film fully intact. If Relative Sensitivity Dead
4. Zone Analysis (see notations below) has been done, refer to the grid map for areas of the grid to avoid. General rule of thumb is to analyze close to the center of the grid.
5. Proceed to the center of a grid opening. There are no particles to find; the elements are "impregnated" into the film. Simply bring beam to crossover on spot size 3.
6. Collect spectral counts for at least 200 seconds, but not more than 300 seconds.
7. Save spectra to the following location: **C:\Noran\Quest\Spectra\QA-QC Spectra\EDX Calibrations\K-Factors**. Inside the K-Factors subdirectory, there are additional locations for year and month. Ensure the final location is appropriate for the date performed.
8. Using a blank measurement form found on the TEM Laptop at the following location: **C:\Bob's Desktop\QA-QC and T-Drive\QA-QC Program\QAQC Data\Personnel Forms-Worksheets\Main Lab Worksheets\Wkshts-TEM\EDXA Forms\ 2063aGRAFPAPR**, enter peak heights at the energy levels specified. There is no need to document the background counts. File this form (with a print-out of the spectra) as part of the TEM Monthly Summary for that month. Enter the peak counts onto the Excel spreadsheet K-Factor worksheet of that months' TEM Calibration file. The Calibration files (named Temcal*.xls) are located in the C drive of the TEM Laptop.

M. Calculations:
You may use the EDXA RELATIVE SENSITIVITY FACTOR FORM. Data gathered from the acquisition can be directly entered into the Excel™ spreadsheet designed for k-factors. See the QA/QC manager for details. The following formula is used to determine the relative peak intensities for each element to Silica.

$$\text{element K-factor} = \frac{(\text{element Wt.. \% / Si Wt.\%}) \text{ Si Intensity}}{\text{element Intensity}}$$ or

$$Ka = \frac{C_a \times Si_i}{Si_c \times I_a}$$

Where:
C_a = Concentration,element *a* (in wt percent)
Si_c = Concentration of Si (in wt percent)
I_a = Intensity of characteristic peak of element *a*

File the form and the printouts into the Excel™ spreadsheet. See QA/QC manager for details.

VII-X: ENERGY DISPERSIVE X-RAY Cu LINE CHECK (DAILY) STANDARD OPERATION PROCEDURE

Note: This calibration is done on a daily basis, assuming that the TEM was used that day. If the TEM is not used for client or QA analysis, no calibration is required.

I. Load a clean copper grid on to the specimen holder.

II. Be sure the grid is securely locked down in the specimen holder.

III. Insert the sample into the Transmission Electron Microscope (TEM) and check to see that the goniometer (X-tilt) is set on 0 degrees.

IV. Go to MAG 19,000x.

V. Traverse the edge of a grid bar of a grid opening.

VI. Transmission Electron Microscope (TEM) activated and aligned.

▶**Reference:** **TRANSMISSION ELECTRON MICROSCOPY (TEM) ALIGNMENT STANDARD OPERATION PROCEDURE** (Section VII-II)

A. Filament emission knob is at the pre-designated stop.
B. Magnification = 19,000x.
C. Acceleration Voltage = 100 KV.
D. Center the edge on the fluorescent screen.
E. Focus the image.
F. Objective Lens Aperture - position 0.
G. Field Limiting Aperture - position 0.
H. Turn the spot size knob to two (2).
I. Condense the beam with the condenser to crossover.
J. Center the beam with the alignment trans knobs.
K. Turn the spot size knob to three (3).
L. Condense the beam with the condenser to crossover.
M. Center the beam with the alignment trans knob on the edge to be analyzed.

VII. Noran (Quest) X-ray Analyzer computers activated.

1. Refer to manufacturer's instructions for start-up procedure.
2. Locate the grid bar edge and situate the beam (at spot size 3) such that it will allow between 1000 and 2500 counts per second. This will ensure representative accumulation of counts without overloading the detector. Set spot size to 3 and bring the beam to crossover at that location.
3. Double-click on the monitor's keV scale to bring up the **Scale** window. Enter values for 'X', effectively spreading Cu peak (and sufficient low and high background channels) to fit the screen. Proper 'X' values to

enter would be 7.50 and 8.50. Click OK and proceed to the **Acquisition** menu.

4. After clicking the **Acquisition** menu, choose Auto Calibration. The **Define Calibration Peak** window will pop up and prompt you to place your cursor on the calibration peak. Choose OK to continue. If you are prompted to save the existing spectrum, click No or Yes depending on if the current spectrum needs to be kept. If No is chosen, the spectrum displayed will be erased. If Yes is chosen, you will be prompted to decide where to save the spectrum currently displayed.
5. Configuring the Cu Peak Calibration: When the **Automatic Calibration** window pops up, choose Cu as the atomic symbol, K as the **Line**, **Maximum Iterations** should be either a minimum of 2 or a maximum of 10.
6. Click **Run** to begin the test.
7. Once the test is finished, the status window will pop up indicating the calibration is complete. No values will be given and no spectrum is displayed for printing. Simply enter the calibration onto the TEM Align-Cal Schedule (see below).
8. **Performed Weekly:** With the 'X' spread, print a spectrum of Cu following calibration. File the printout with the TEM Monthly Summary for that month. Save the spectrum to the following location: **C:\Noran\Quest\Spectra\QA-QC Spectra\EDX Calibrations\Cu Line Calibration**. Inside the Cu Line Calibration subdirectory, there are additional locations for year and month. Ensure the final location is appropriate for the date performed. The date it is performed is entered onto the TEM Calibration/Alignment Schedule worksheet found at the following location: **T:\TEM Analysis\AHERA\Templates\ Align-Cal Sched Template**. The template is constantly updated to the current date, and is saved (at the end of the month) to the following location: **T:\TEM Analysis\AHERA\Templates\Align-Cal Schedules.** The file name is given in regard to the month and year. For example, location and filename for the schedule for the month of March 2004 would be: **T:\TEM Analysis\AHERA\Templates\Align-Cal Schedules\Align-Cal Schedule 03-2004.**

NOTE: A. To cut back on the paper load, it is not required to print a spectra for the daily copper line check. There is a requirement to enter the calibration onto the TEM Alignment/Calibration log. This will give a daily log over the course of each month of all TEM cal/align activity. If a printout is needed in any case, the following procedure has been included.

B. If the Cu line check does not meet the previous specifications, then perform standard operation procedures for ENERGY DISPERSIVE X-RAY Cu and Al CALIBRATION.

9. Log the Cu line Check into the EDXA Cu Daily Schedule.

VII-XI: ENERGY DISPERSIVE X-RAY Cu & Al CALIBRATION (WEEKLY) STANDARD OPERATION PROCEDURE

This calibration is performed weekly.

I. Load a clean copper grid, on to the specimen holder.
II. On top of the grid, place an aluminum standard that is approximately 3 mm square where it covers approximately one half (1/2) of the grid openings.
III. Be sure the aluminum standard is securely locked down onto the grid.
IV. Insert the sample into the Transmission Electron Microscope (TEM) and check to see that the goniometer (X-tilt) is set on 0 degrees.
V. At LOW MAG, scan the grid and notice the orientation of the aluminum standard on the grid openings.
VI. Locate a grid opening in which the aluminum (Al) standard traverses approximately one half of a grid opening.
VII. Go to MAG 19,000x.
VIII. Locate the Al standard and notice its orientation to the grid opening.
IX. Traverse the edge of the Al standard and establish a mid-point of that edge in the grid opening.
X. Transmission Electron Microscope (TEM) activated and aligned.
    A. Filament emission knob is at the pre-designated stop.
    B. Magnification = 19,000x.
    C. Acceleration Voltage = 100 KV.
    D. Center the edge on the fluorescent screen.
    E. Focus the image.
    F. Objective Lens Aperture - position 0.
    G. Field Limiting Aperture - position 0.
    H. Turn the spot size knob to two (2).
    I. Condense the beam with the condenser to crossover.
    J. Center the beam with the alignment trans knobs.
    K. Turn the spot size knob to three (3).
    L. Condense the beam with the condenser to crossover.
    M. Center the beam with the alignment trans knobs on the edge to be analyzed.
XI. Activate SpectraPlus program on Quest, and follow the Cu line auto-calibration check in **VII-X** for both Cu and Al auto-calibrations. Quest will notify when both processes are done.
XIX. Log the calibration into the Daily TEM Cal/Alignment Schedule posted on the TEM Lab wall.

VII-XII: Confirmation of EDXA Sodium Peak Detection in Crocidolite and Mg-Si Peak Resolvability Check (Quarterly)

The presence of Na in EDXA spectrum is a crucial feature for the positive identification of crocidolite. NVLAP requires periodic check on EDXA system used in airborne asbestos analysis to ensure the capability of detection Na in crocidolite.

A peak is considered statistically significant if its minimum size is 3 times the standard deviation of the background at the peak position (Goldsten et al., 1989, p.286; also refer to the

method compiled in the Technical Report on the TEM shelf). The following procedure has been developed and implemented to confirm the detection of Na in Crocidolite:

1. Acquire an EDXA spectrum on a NIST crocidolite standard.

2. On the EDXA display, use the cursor to read the number of count of the background trace. Take 10 readings in the background to the left of Na *Kα* peak in the range of 0.60-0.78 KeV at 0.02 KeV intervals. Take another 10 readings in the background in-between Mg Ka and Si Ka peaks in the range of approximately 1.36 to 1.54 KeV at 0.02 KeV intervals.

3. On the EDXA Display, use the cursor to read the number of counts of Na *Kα* peak at 1.04 KeV.

4. Enter all above counts into the spread sheet called "NaGRAFPAPR.xls" following the file path: \\server1\temshare\QA-QC Program\QAQC Data\Personnel Forms-Worksheets\Main Lab Worksheets\Wkshts-TEM\EDXA Forms\NaGRAFPAPR.xls. The spreadsheet will calculate and perform linear regression analysis on the above count-KeV data set (see the example that follows) and the necessary statistics to validate the sodium peak detection. The math behind the calculation can be found in the Technical Report on the TEM shelf).

5. Save the file into the corresponding monthly QA folder.

A NIST Chrysotile standard is used to perform the Mg-Si peak resolvability check. Do the following:

1. Acquire a 100-200 second spectrum from a single chrysotile fibril (<0.05μm in width) from a NIST traceable standard (i.e. 1866, 8140, 1876b).
2. Print a copy of the spectrum showing the resolvable Mg-Si peaks and save to its appropriate subdirectory (i.e. corresponding monthly QA folder) as record.
3. If the peaks are apparently separable and significant, enter "y" on the monthly QA/QC spread sheet located in: \\server1\temshare\QA-QC Program\QAQC Data.

VII-XIII: Screen Magnification Calibration (Monthly)

I. Transmission Electron Microscope (TEM) activated and aligned.
To activate the TEM:

II. Load the grating replica, with 2160 lines per millimeter, into the sample specimen holder with forceps.

III. Load the specimen holder into the TEM.

A. Carefully place the specimen holder, in the specimen holder magazine, point first (do NOT let the ruby, at the point of the specimen holder, touch any surface.)

B. Gently push the handle of the specimen holder into the specimen holder magazine until you hear a click.

C. The specimen holder will not stay in place and the vacuum pumps will come on.

D. The red light on the X-tilt goniometer will come on.

E. When the specimen holder magazine equalizes pressure with the column of the Tem interior proper, the red light will turn off.

F. Holding the handle of the specimen holder firmly, slowly turn the handle counter-clockwise where the specimen holder will then be sucked into the specimen chamber.

IV. To observe the grid at LOW MAG, perform the following:

A. Acceleration Voltage = 100 KV.

B. Turn the Filament Emission knob to the pre-determined stop SLOWLY.

C. Objective Lens Aperture - Position 0

D. Field Limiting Aperture - Position 0

E. Turn the Condenser knob to spread the beam if needed.
Turn the left Specimen shifting knob clockwise until it comes to the stops.

F. Turn the Specimen selector to where the left side of the grid is approximately oriented in the center of the fluorescent screen (this step will prevent you from traversing beyond the stops on a grid opening at high magnification).

G. Push the MAG button on the TEM. The TEM will zoom in on the grid opening selected.

H. Turn the MAGNIFICATION knob to 10,000x magnification.

I. Turn the CONDENSER knob to beam crossover.

J. With the ALIGNMENT TRANS knobs, center the beam.

K. Turn the CONDENSER knob to spread the beam.

L. Focus the image with the MEDIUM FOCUS knobs.

M. Objective Lens Aperture - Position 0

V. Determination of the TEM magnification on the fluorescent screen.

A. Define a field of view on the fluorescent screen either by markings or physical boundaries.

B. The field of view is measurable with the inscribed scale and concentric circles (all scales are metric).

1. The inscribed outer circle = 76.0 mm.
2. The inscribed inner circle = 7.6 mm.

C. Goniometer stage tilt is set to 0 degrees.

D. Make sure the goniometer is eucentric.

1. Center an image in the center of the fluorescent screen.
2. The eucentric adjusting screw is located on the goniometer just below the specimen holder port.
3. With the goniometer unlocked, turn the goniometer counter-clockwise.
4. The image will either move to the left or right.
5. Adjust the movement of the image by turning the eucentric screw in either direction to compensate.
6. You want the image to be stationary while the goniometer is turned.

E. The distance (mm) is then measured between two widely separated lines on the grating replica.

F. Note the number of spaces between the lines along the diameter of the inscribed outer circle.

G. Care is then taken to measure between the lines of the same relative positions of the lines (e.g. between left edges of lines).

H. Count the number of lines along the diameter of the outer inscribed circle.

I. Record the measurement onto the TEM computer on the Excel™ spreadsheet.

J. Locate another line perpendicular to the one just measured.

K. Count the number of lines along the diameter of the outer inscribed circle perpendicular to the once previously measured.

L. Record the measurement onto the TEM computer on the Excel™ spreadsheet.

M. Perform the measurements five (5) times repeating steps F through M moving to a new location o the grating replica each time.

N. Determine the arithmetic mean of the diameters measured and record it onto the TEM computer on the Excel™ spreadsheet.

O. Perform the following calculations using the following formula and log them onto the TEM computer on the Excel™ spreadsheet.

TRUE MAGNIFICATION

$$Ms = \frac{Xo \times (2{,}160 \text{ lines/mm})}{Ns}$$

DIAMETER OF OUTER CIRCLE AT 19,000x

$$\frac{Xo \times (1{,}000)}{Ms}$$

DIAMETER OF INNER CIRCLE AT 19,000x

$$\frac{Xi \times (1{,}000)}{Ms}$$

VI. Sign and date the Magnification Calibration performed onto the TEM computer on the Excel™ spreadsheet.

VII. Remove the grating replica from the TEM and store it in its designated location. The Grating Replica is stored in the Storage Cabinet on the second shelf from the top, in the TEM Standards Box.

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APPENDIX VIII

SAMPLE MONTHLY SUMMARY

(Not available in PDF format due to large file size)

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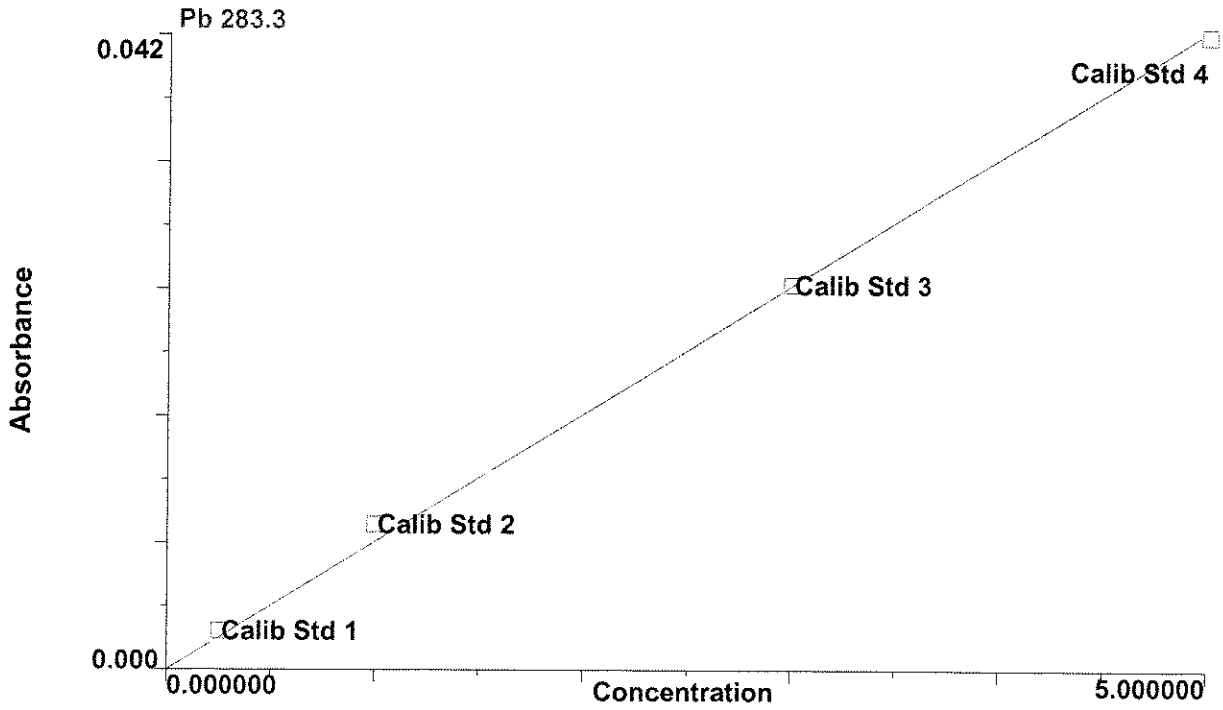
Edit Calibration

Result: 07-09-15A1-TOAA800

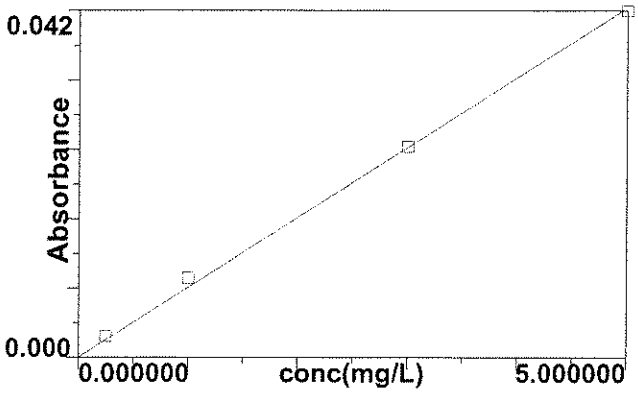


Calibration Curve Slope: 0.00853
Calibration Curve Intercept: 0.00000
Calibration Curve Correlation Coefficient: 0.999152
Calibration Curve Type: Linear Through Zero
Current Sample Concentration: 9.023 mg/L

Std #	Standard ID	Entered Conc.	Calculated Conc.	Action
Blank	Calib Blank 1	0	0.000	Include
1	Calib Std 1	0.2500	0.303	Include
2	Calib Std 2	1.0000	1.136	Include
3	Calib Std 3	3.0000	3.012	Include
4	Calib Std 4	5.0000	4.958	Include

Method: NIOSH 7082
Result: 07-09-15A1-TOAA800

Pb 283.3



Calib Eq'n: Lin Thru 0
Corr Coeff: 0.999152