SAMPLING AND ANALYSIS PLAN (SAP)
FOR THE
ASBESTOS ABATEMENT OF BUILDING 401
NIAGARA FALLS STORAGE SITE
LEWISTON, NEW YORK

PREPARED FOR:

DEPARTMENT OF THE ARMY
CORPS OF ENGINEERS, BUFFALO DISTRICT
BUFFALO, NEW YORK
CONTRACT DACW49-00-D-0007

Prepared by:

Jacobs Engineering Group, Inc. - Federal Operations
13723 Riverport Drive
Maryland Heights, MO. 63043

October 2001
Revision 1 – January 2002

ROUTING OF SHOP DRAWINGS, EQUIPMENT DATA, MATERIAL SAMPLES, OR MANUFACTURER'S CERTIFICATES OF COMPLIANCE FOR APPROVAL

(Used to route ENG Form 4025 with items attached. Not to become a part of the Contractor's Record.)

	TO:	FROM:	DATE
1	Stephen Yaksich	Todd Kufel	17-Jan-02

The attached items listed on ENG Form 4025 are forwarded for approval action.

CONTRACT NUMBER	CONTRACTOR
DACW49-00-D-0007	Jacobs Engineering
TRANSMITTAL NUMBERS	**PROJECT TITLE AND LOCATION**
Sampling and Analysis Plan (Rev. 1)	NFSS Building 401 Asbestos Abatement

COMMENTS (Attach additional sheet, if necessary.)

Revised work plan. All PDT comments have been resolved.

NO. OF INCL.	TYPED NAME AND TITLE	SIGNATURE
8	Todd Kufel, Project Engineer	Todd C. Kufel

	TO:	FROM:	DATE
2			

COMMENTS (Attach additional sheet, if necessary.)

NO. OF INCL.	TYPED NAME AND TITLE	SIGNATURE

	TO:	FROM:	DATE
3			

COMMENTS (Attach additional sheet, if necessary.)

NO. OF INCL.	TYPED NAME AND TITLE	SIGNATURE

	TO:	FROM:	DATE
4	Judith Leithner	Stephen Yaksich	1/18/02

The following action codes are given to items listed on ENG Form 4025:

ACTION CODES

A - APPROVED AS SUBMITTED.
B - APPROVED, EXCEPT AS NOTED ON DRAWINGS. RESUBMISSION NOT REQUIRED.
C - APPROVED, EXCEPT AS NOTED ON DRAWINGS. REFER TO ATTACHED SHEET, RESUBMISSION REQUIRED.
D - WILL BE RETURNED BY SEPARATE CORRESPONDENCE.
E - DISAPPROVED (SEE ATTACHED)
F - RECEIPT ACKNOWLEDGE
G - OTHER (specify).

ACTION CODES TO BE INSERTED IN COLUMN G, SECTION I, ENG FORM 4025 (Attach sheets, when required.)

ITEM NO. *(Taken from ENG Form 4025)*	1.7a							
CODE GIVEN	A							

REMARKS

Item No. 1.7a - Approved as submitted.

NO. OF INCL.	TYPED NAME AND TITLE	SIGNATURE
1	Stephen Yaksich, Chief, Engineering Division	S Yaksich

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JACOBS ENGINEERING
Engineers and Constructors

Jacobs Engineering Group Inc Federal Operations
13723 Riverport Drive
Maryland Heights MO 63043

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☒ APPROVAL RECOMMENDED _____ Initials _____ Date 1/16/02
☐ APPROVAL AMENDED SUBJECT COMMENTS INDICATED _____ Initials _____ Date
☐ DISAPPROVAL RECOMMENDED _____ Initials _____ Date
APPROVED/DISAPPROVED _____ Signature _____ Date

Distribution List:

Brian Moore (USACE)
Virgil Jansen (Program Manager)
Leo Mann III (Project Manager)
David Fleming (Site Safety Health Officer)
Jon Van Voorhees (EMSL)
Richard H. Mannz (STL)

COMPLETION OF INDEPENDENT TECHNICAL REVIEW

Jacobs Engineering Group, Inc. has completed the Sampling and Analysis Plan for the asbestos abatement of Building 401, Niagara Falls Storage Site, Lewiston, New York. Notice is hereby given that an independent technical review has been conducted that is appropriate to the level of risk and complexity inherent in the project, as defined in the Quality Control Plan. During the independent technical review, compliance with established policy principles and procedures, utilizing justified and valid assumptions, was verified. This included review of assumptions; methods, procedures, and material used in analyses; alternatives evaluated; the appropriateness of data used and level of data obtained; and reasonableness of the results, including whether the product meets the customer's needs consistent with law and existing Corps policy.

Aaron Ensor — 10/10/01
(Signature) (Date)
Study/Design Team Leader and Team Members

Virgil W. Jensen — 10/11/01
(Signature) (Date)
Independent Technical Review Team Leader and Team Members

Leo Mann III — 10/10/01
(Signature) (Date)
Independent Technical Review Team Leader and Team Members

Ethan Tutsey — 10/11/01
(Signature) (Date)
Independent Technical Review Team Leader and Team Members

CERTIFICATION OF INDEPENDENT TECHNICAL REVIEW

Significant concerns and the explanation of the resolution are as follows (Describe the major technical concerns, possible impact, and resolution):

- Make appropriate grammar and format consistency changes (Grammar changes made)

- Define methods and specifics of standards concerning air monitoring. (Methods defined)

- QAPP should be more specific to project in some areas. (QAPP modified)

- Under section 2 of the QAPP specifically list key personnel as they apply to data assessment. (Section 2 Modified)

All concerns resulting from independent technical review of the project have been considered.

Virgil W Jansen
(Signature)

10/11/01
(Date)

(Engineer of Record)

ABBREVIATIONS AND ACRONYMS

AAAP	Asbestos Assessment and Abatement Plan
AAP	Asbestos Abatement Plan
ACGIH	American Conference of Governmental Industrial Hygienists
ACM	Asbestos Containing Material
AHERA	Asbestos Hazard Emergency Response Act
AIHA	American Industrial Hygiene Association
ALARA	As-Low-As-Reasonably-Achievable
ALI	Annual Limit On Intake
APR	air-purifying respirator
ASHARA	Asbestos School Hazard Abatement Reauthorization Act
ASTM	American Society for Testing and Materials
BRA	Baseline Risk Assessment
BTEX	Benzene, Toluene, Ethylbenzene, Xylenes
C&D	construction and demolition
CAA	Clean Air Act
CAPE	Cape Environmental Management Inc
CEDE	Committed Effective Dose Equivalent
CERCLA	Comprehensive Environmental Response Compensation and Liability
cfm	cubic feet per minute
CFR	Code of Federal Regulations
CHSP	Corporate Health and Safety Procedure
CIH	Certified Industrial Hygienist
CMS	Corrective Measures Study
COC	Chain of Custody
COPC	Chemical of Potential Concern
COR	Contracting Officer Representative
CRs	Carcinogenic Risk
CRZ	Contamination Reduction Zone
CWA	Clean Water Act
DA	Department of the Army
DAC	Inhalation Derived Air Concentrations
DOD	U.S. Department of Defense
DOE	U.S. Department of Energy
DOP	dioctylphthalate
DOT	U.S. Department of Transportation
dpm	Disintegrations Per Minute
EDC	Economic Development Conveyance Area
EMSL	EMSL Analytical, Inc.
EPA	U.S. Environmental Protection Agency
EZ	Exclusion Zone
F	Fahrenheit
f/cc	Fibers per cubic centimeter of air
FMEA	Failure Mode and Effects Analysis
FSP	Field Sampling Plan
GAC	Granulated Activated Carbon
GERT	General Employee Radiological Training
GFCI	Ground Fault Circuit Interrupter
HAZOP	Hazard and Operability Study
HazWOPER	Hazardous Waste Operations and Emergency Response
HEPA	High Efficiency Particulate Air

HHE	Human Health Evaluation
HHRA	Human Health Risk Assessment
HI	Hazard Index
HQ	Hazard Quotient
HVAC	heating, ventilation, and air conditioning
IDLH	Immediately Dangerous to Life or Health
IHT	Industrial Hygiene Technician
IS	Interim Standards
JE	Jacobs Engineering
JEG	Jacobs Engineering Group
LEL	Lower Explosive Limit
LOOW	Lake Ontario Ordnance Works
LPM	liters per minute
MAP	Model Accreditation Plan
MCE	mixed-cellulose ester
MCLGs	Maximum Contaminant Level Goals
MCLs	Maximum Contaminant Levels
MDA	Minimum Detectable Activity
MED	Manhattan Engineering District
MSDS's	Material Safety Data Sheets
MSL	Mean Sea Level
NAM	Negative Air Machine
NAWQC	National Ambient Water Quality Criteria
NCP	National Contingency Plan
NEPA	National Environmental Policy Act
NESHAPS	National Emissions Standards for Hazardous Air Pollutants
NFSS	Niagara Falls Storage Site
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards and Technology
NOB	Non-friable Organically Bound
NPDES	National Pollution Discharge Elimination System
NRC	Nuclear Regulatory Commission
NVLAP	National Voluntary Laboratory Accreditation Program
NYCRR	New York Code of Rules and Regulations
NYSDOL	New York State Department of Labor
ORISE	Oak Ridge Institute for Science and Education
OSHA	Occupational Safety and Health Administration
PACM	Presumed Asbestos Containing Materials
PAPR	Powered Air Purifying Respirator
PBC	Public Benefit Conveyance Area
PCM	Phase Contrast Microscopy
PDU	Personal Decontamination Unit
PEL	Permissible Exposure Limit
PHA	Process Hazard Analysis
PLHCP	Physician or other Licensed Health Care Professional
PLM	Polarized Light Microscopy
PPE	personal protective equipment
PRGs	Preliminary Remediation Goals
PVC	polyvinyl chloride
QAPP	Quality Assurance Plan
QC	quality control
QCR	Quality Control Reports
QLFT	Qualitative Fit Test Requirements

QNFI	Quantitative Fit Test Requirements
RA	Restricted Area
RAD	Radiation
RCA	Radiologically Controlled Areas
RCCP	Radiation Control Contingency Plan
RCRA	Resource Conservation and Recovery Act
RFI	RCRA Facility Investigation
RFP	Request For Proposal
RGOs	Remedial Goal Objectives
RME	Reasonable Maximum Exposure
RW II	Radiological Worker II
RWP	Radiological Work Permit
SAR	Supplied-Air Respirator
SCBA	Self-Contained Breathing Apparatus
SCS	Soil Conservation Service
SDWA	Safe Drinking Water Act
SEV	Screening Ecological Value
SHM	Safety and Health Manager
SHP	Safety and Health Plan
SMCLs	Secondary Maximum Contaminant Levels
SOP	Standard Operating Procedures
SOR	Safety Observation Report
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
SSL	Soil Screening Level
STL	Severn Trent Services Laboratories
SVOCs	Semi-volatile Organic Compounds
SWMU	Solid Waste Management Unit
SZ	Support Zone
TAL	Total Analyte List
TBC	To Be Considered
TCLP	Toxicity Characteristic Leaching Procedure
TDS	Total Dissolved Solids
TEDE	Total Effective Dose Equivalent
TEM	Transmission Electron Microscopy
TLV	Threshold Limit Value
TSI	Thermal System Insulation
TSS	Total Suspended Solids
TWA	Time-Weighted Average
UCL	Upper Confidence Level
UCS	Unconfined Compressive Strength
UEL	Upper Explosive Limit
USACE	United States Army Corps of Engineers
USAEC	United States Army Environmental Center (formerly USATHAMA)
USATHAMA	United States Army Toxic and Hazardous Materials Agency (now USAEC)
USDA	United States Department of Agriculture
VOCs	Volatile Organic Compounds
WA	Work Area (Asbestos Regulated Area)
WBGT	Wet Bulb Globe Temperature Index
WCS	Waste Containment Structure

TABLE OF CONTENTS

FIELD SAMPLING PLAN (FSP)
FOR THE
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LEWISTON, NEW YORK

PREPARED FOR:

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Prepared by:

Jacobs Engineering Group, Inc. - Federal Operations
13723 Riverport Drive
Maryland Heights, MO. 63043

October 2001
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TABLE OF CONTENTS
FIELD SAMPLING PLAN (FSP)

Appendices

1.0 PROJECT BACKGROUND

Jacobs Engineering, Inc. (JE) is under contract with the United States Army Corps of Engineers (USACE), Buffalo District, to provide Engineering, Procurement, and Construction services tasks including, but not limited to, the development of required work plans for the asbestos abatement of Building 401 at the Niagara Falls Storage Site. As a contract requirement, JE is tasked to develop a Sampling and Analysis Plan (SAP). This SAP has been developed as a written plan to address the analysis that will be performed during the asbestos assessment and abatement of Building 401 and to provide a quality remedial construction services.

1.1 SITE HISTORY

Niagara Falls Storage Site (NFSS) is located at 1397 Pletcher Road, Lewiston, New York. The site is owned by the U.S. Department of Energy (DOE). The site consists of an engineered Waste Containment Structure (WCS), various buildings, and open areas. The site was originally a part of the Lake Ontario Ordnance Works (LOOW). The primary use of the site from early 1940s through mid 1950s was for storage, trans-shipment, and disposal of radioactive wastes from various sources.

Building 401 was initially the powerhouse for the production of TNT at LOOW, and was used to store radioactive materials in support of Manhattan Engineering District (MED) activities during World War II. The building was used for the production of Boron-10 from 1953 to 1959 and from 1965 to 1971 and then became a waste storage facility by MED. In 1971, Building 401 was gutted and its instrumentation and hardware were disposed of as surplus materials. The building has been inactive since.

Building 401 is steel frame four story structure approximately 100,000 square feet of floor area. The main structural system of the building consists of steel and concrete load bearing walls supporting the roof. There are multiple floors that contain rooms and offices and building service areas. There is a tower area and high bay. The building floor is concrete slab on grade.

1.2 SUMMARY OF EXISTING SITE DATA

An extensive radiological survey was performed in September 1994 by Oak Ridge Institute for Science and Education (ORISE) (*Radiological Survey of Buildings 401, 403, and the Hittman Building, Niagara Falls Storage Site, Lewiston, NY*, Oak Ridge Institute for Science and Education (ORISE), Tennessee, March 1995; BNI-FUSRAP CCN 128541). This data is also summarized in Attachment 2 of the RFP DACW49-01-R-0023, Asbestos Abatement of Building 401, NFSS, Youngstown, NY (RFP). The following narrative summarizes the findings contained in that document.

1.2.1 Affected Area Survey Unit Results

Building 401 has been divided into affected and unaffected areas based on the results of previous radiological surveys. These areas are identified in Table 4a of Attachment 2 of the RFP.

In all affected areas, ORISE performed surface scans for total alpha and beta activity on 100% of the accessible floor and lower wall (i.e. up to 2 m) surfaces; and on approximately 25% of the upper walls (i.e. above 2 m), ceilings, overhead pipe runs, beams, and equipment. ORISE also established a 1 m x 1 m grid on the floor and lower walls, and collected fixed point direct measurements and smears for alpha and beta surface activity in the center of each floor and lower wall grid block. Additional measurements were made to determine the average residual surface activity in 1 m^2 areas around locations of elevated direct radiation detected by scans. On ungridded surfaces, ORISE also performed fixed point direct measurements and smears for alpha and beta surface activity every 5 to 10 m^2 and at the locations of elevated direct radiation.

Area A1

In affected area A1, which includes Rooms 117 and 119 (estimated floor area 290 m^2), ORISE reported the following data on fixed point direct measurements and smears for alpha and beta surface activity:

Survey Area	Floor	Lower Walls	Upper Walls & Ceiling
# of Locations	306	156	52
Direct alpha range[1]	<39 to 84	<39 to 53	<47 to 1,600
Direct beta range[1]	<470 to 3,600	<470	<430 to 44,000
Removable alpha range[1]	<12	<12	<12
Removable beta range[1]	<16 to 20	<16	<16

1: units are dpm/100 cm^2

ORISE found 3 locations exceeding NRC Reg Guide 1.86 surface contamination guidelines: on an I-beam, within 1 floor grid block, and within a floor drain. Results for these 3 locations are found in Table 4a of Attachment 2 of the RFP.

Table 5a of RFP Attachment 2 indicates the contaminated sections of the I-beam and the floor drain (to a 1-ft depth) were removed. No residual contamination was found around the floor drain post-removal. Residual contamination on the remaining portion of the I-beam (direct beta results) ranges from 20,000 to 28,000 dpm/100 cm^2.

Area A2

In affected area A2, which includes Room 102 (estimated floor area 30 m^2), ORISE reported the following data on fixed point direct measurements and smears for alpha and beta surface activity:

Survey Area	Floor	Lower Walls	Upper Walls & Ceiling
# of Locations	30	72	7
Direct alpha range[1]	<37 to 60	<37 to 40	<37 to 580
Direct beta range[1]	<430	<430	<430 to 2,700
Removable alpha range[1]	<12	<12	<12
Removable beta range[1]	<16	<16	<16

1: units are dpm/100 cm^2

ORISE found 1 location exceeding NRC Reg Guide 1.86 surface contamination guidelines: within a pipe protruding through the ceiling into Room 102. Results for this location are found in Table 4a of Attachment 2 of the RFP.

Table 5a of RFP Attachment 2 indicates this pipe was removed and prepared for disposal. The floor area underneath the former pipe was decontaminated, and post-decontamination survey results were reported to be 65 dpm/100 cm^2 average alpha activity, and 136 dpm/100 cm^2 average beta activity.

Area A3

In affected area A3, which includes Room 121 (estimated floor area 238 m^2), ORISE reported the following data on fixed point direct measurements and smears for alpha and beta surface activity:

Survey Area	Floor	Lower Walls	Upper Walls & Ceiling
# of Locations	172	98	33
Direct alpha range[1]	<37 to 85	<47 to 810	<47 to 1,400
Direct beta range[1]	<460 to 34,000	<460 to 6,200	<460 to 240,000
Removable alpha range[1]	<12	<12	<12
Removable beta range[1]	<16	<16	<16

1: units are dpm/100 cm^2

ORISE found the most extensive radiological contamination in this survey unit. The following locations exceeded NRC Reg Guide 1.86 surface contamination guidelines:

- 1 individual floor measurement and 19 floor grid blocks (1m x 1m); these are all on the west side of Room 121
- 1 location on a lower ledge on the north wall
- 6 locations on I-beams
- 1 location on a mezzanine
- 1 location on an air duct.

Results for these locations are found in Table 4a of Attachment 2 of the RFP.

Table 5a of RFP Attachment 2 indicates surface grinding was performed on the floor areas identified by ORISE. However the areas did not receive post-decontamination surveys. It also indicates the air duct was removed, and some of the I-beam contamination was removed via stripping.

Area A4

In affected area A4, which includes Room 122 (estimated floor area 300 m^2), ORISE reported the following data on fixed point direct measurements and smears for alpha and beta surface activity:

Survey Area	Floor	Lower Walls	Upper Walls & Ceiling
# of Locations	296	150	52
Direct alpha range[1]	<37 to 480	<37 to 75	<47 to 140
Direct beta range[1]	<430 to 2,700	<430 to 6,200	<460 to 1,200
Removable alpha range[1]	<12 to 29	<12	<12
Removable beta range[1]	<16 to 42	<16	<16

1: units are dpm/100 cm^2

ORISE found the following locations exceeding NRC Reg Guide 1.86 surface contamination guidelines: 1 individual floor measurement, 1 floor grid block (1m x 1m), a lower wall ledge extending within 2 contiguous grid blocks, and an I-beam. All contaminated areas were found in the southeast section of Room 122. Results for this location are found in Table 4a of Attachment 2 of the RFP.

Table 5a of RFP Attachment 2 indicates that surface grinding was performed on the contaminated floor areas, that contamination levels need to be determined on 1-ft of asbestos-containing material (ACM) pipe lagging, and that residual radioactivity was identified on a 10-ft section of I-beam and an asbestos-lagging pipe.

Area A5

In affected area A5, which includes the eastern portion of Room 217 (estimated floor area 96 m^2), ORISE reported the following data on fixed point direct measurements and smears for alpha and beta surface activity:

Survey Area	Floor	Lower Walls	Upper Walls & Ceiling
# of Locations	106	74	25
Direct alpha range[1]	<39 to 79	<39 to 65	<39 to 180
Direct beta range[1]	<470 to 6,600	<430 to 59,000	<470 to 110,000
Removable alpha range[1]	<12	<12	<12
Removable beta range[1]	<16	<16	<16

1: units are dpm/100 cm^2

ORISE found the following locations exceeding NRC Reg Guide 1.86 surface contamination guidelines: 5 floor locations (3 individual measurements and 2 floor grid blocks) and 2 lower wall ledge locations on the east side of Room 217, and an I-beam plate along the entire upper east wall. Results for these locations are found in Table 4a of Attachment 2 of the RFP.

Table 5a of RFP Attachment 2 indicates a portion of a 6-in I-beam along the east wall was cut out to access a 12-in beam in close proximity. This was performed to determine contamination levels on the 12-in beam. The 12-in beam was decontaminated via stripping, but there is no mention of post-decontamination survey results. Table 5a also reports that a wooden deck underneath Room 217 needs vacuuming of loose radiological contamination, but it does not include removable contamination survey results. Table 5a also discusses the following items in Room 217 which are not discussed in the ORISE report:

- equipment stands and adjacent floor area where surface grinding was performed, no post-decontamination surveys
- wooden wall support frames where decontamination is needed
- asbestos pipe insulation along the east wall where decontamination and removal is needed
- floor area along east edge of the room where decontamination is complete but post-decontamination survey still required
- additional I-beams, pipe hangars, and diagonals where contamination surveys are required

Area A6

In affected area A6, which includes Room 115 (estimated floor area 42 m^2), ORISE reported the following data on fixed point direct measurements and smears for alpha and beta surface activity:

Survey Area	Floor	Lower Walls	Upper Walls & Ceiling
# of Locations	56	60	9
Direct alpha range[1]	<35 to 2,900	<35 to 38	<35
Direct beta range[1]	<450 to 6,000	<450	<450
Removable alpha range[1]	<12 to 22	<12	<12
Removable beta range[1]	<16 to 23	<16	<16

1: units are dpm/100 cm^2

ORISE found 1 location exceeding NRC Reg Guide 1.86 surface contamination guidelines: a single floor measurement near a floor drain. Results for this location are found in Table 4a of Attachment 2 of the RFP.

Table 5a of RFP Attachment 2 indicates the drain was removed. Post-decontamination alpha survey results (average values for direct surveys) are reported as 229 dpm/100 cm^2 in the drain and 2,250 dpm/100 cm^2on the concrete floor. Beta contamination was reported to be below the guidelines.

Area A7 & A8

In affected areas A7 & A8, which includes the lockers in Rooms 108 and 211, ORISE reported the following data on fixed point direct measurements and smears for alpha and beta surface activity:

Survey Area	Lockers in Room 108	Lockers in Room 211
# of Locations	70	9
Direct alpha range[1]	<39 to 240	<39 to 280
Direct beta range[1]	<470 to 13,000	2,300 to 14,000
Removable alpha range[1]	<12	<12
Removable beta range[1]	<16	<16

1: units are dpm/100 cm^2

ORISE found the floors of most of the lockers, and other areas within several of the lockers to exceed NRC Reg Guide 1.86 surface contamination guidelines. Results for this location are found in Table 4a of Attachment 2 of the RFP.

Table 5a of RFP Attachment 2 indicates the lockers in Rooms 108 and 211 were removed. The lockers were mounted on pedestals. The pedestal surfaces were decontaminated but post-decontamination surveys were not performed.

In Room 203, ORISE found another blower which exceeded the NRC Reg Guide 1.86 guideline for total alpha contamination (average value). Reported results on this blower are: direct alpha 120 dpm/100 cm^2 (average exceeds 100 dpm/100 cm^2), direct beta <470 dpm/100 cm^2, removable alpha <12 dpm/100 cm^2, and removable beta <16 dpm/100 cm^2. Table 5a of RFP Attachment 2 indicates the contaminated area on the blower was decontaminated, but post-decontamination survey results are not provided.

1.2.2 Unaffected Area Survey Unit Results

In all unaffected areas, ORISE performed surface scans for total alpha and beta activity on 25% to 50% of the accessible floor and lower wall (i.e. up to 2 m) surfaces; and on 2% to 5% of the upper walls (i.e. above 2 m), ceilings, overhead pipe runs, beams, and equipment. Locations of elevated direct radiation were further investigated, and areas found to exceed 75% of the guidelines were reclassified as affected areas. Within each unaffected survey unit ORISE also collected a minimum of 30 fixed point direct measurements and smears for alpha and beta surface activity at randomly selected locations.

Survey Unit U1

In survey unit U1 (which includes Rooms 111, 113, 114, 116, 118, 125, and 133), ORISE reported the following data on fixed point direct measurements and smears for alpha and beta surface activity:

Survey Area	Floor	Lower Walls	Upper Walls & Ceiling
# of Locations	11	9	10
Direct alpha range[1]	<39	<39	<35
Direct beta range[1]	<470	<470	<450
Removable alpha range[1]	<12	<12	<12
Removable beta range[1]	<16	<16	<16

1: units are dpm/100 cm^2

Survey Unit U2

In survey unit U2 (which includes Rooms 101, 102, 103, 104, 105, 106, 107, 108, 109, and 112), ORISE reported the following data on fixed point direct measurements and smears for alpha and beta surface activity:

Survey Area	Floor	Lower Walls	Upper Walls & Ceiling
# of Locations	13	7	10
Direct alpha range[1]	<35	<35 to 130	<35
Direct beta range[1]	<450 to 1,500	<450 to 2,200	<450
Removable alpha range[1]	<12	<12	<12
Removable beta range[1]	<16	<16	<16

1: units are dpm/100 cm^2

ORISE reports that all elevated alpha and beta readings in survey unit U2 are associated with ceramic wall tile, which is known to have higher concentrations of naturally-occurring radioactivity present within it.

Survey Unit U3

In survey unit U3 (which includes Rooms 120, 128, 129 and 134), ORISE reported the following data on fixed point direct measurements and smears for alpha and beta surface activity:

Survey Area	Floor	Lower Walls	Upper Walls & Ceiling
# of Locations	12	8	10
Direct alpha range[1]	<35	<35	<35
Direct beta range[1]	<450 to 610	<450	<450
Removable alpha range[1]	<12	<12	<12
Removable beta range[1]	<16	<16	<16

1: units are dpm/100 cm^2

Survey Unit U4

In survey unit U4 (which includes Rooms 127, 131 and 132), ORISE reported the following data on fixed point direct measurements and smears for alpha and beta surface activity:

Survey Area	Floor	Lower Walls	Upper Walls & Ceiling
# of Locations	12	8	10
Direct alpha range[1]	<35	<35	<35
Direct beta range[1]	<450 to 550	<450	<450
Removable alpha range[1]	<12	<12	<12
Removable beta range[1]	<16	<16	<16

1: units are dpm/100 cm^2

Survey Unit U5

In survey unit U5 (which includes Room 122), ORISE reported the following data on fixed point direct measurements and smears for alpha and beta surface activity:

Survey Area	Floor	Lower Walls	Upper Walls & Ceiling
# of Locations	11	9	10
Direct alpha range[1]	<35	<35	<35
Direct beta range[1]	<450	<450	<450
Removable alpha range[1]	<12	<12	<12
Removable beta range[1]	<16	<16	<16

1: units are dpm/100 cm^2

Survey Unit U6

In survey unit U6 (which includes the north part of Room 217), ORISE reported the following data on fixed point direct measurements and smears for alpha and beta surface activity:

Survey Area	Floor	Lower Walls	Upper Walls & Ceiling
# of Locations	11	5	6
Direct alpha range[1]	<39 to 84	<39	<39 to 52
Direct beta range[1]	<470 to 2,600	<470	<470
Removable alpha range[1]	<12	<12	<12
Removable beta range[1]	<16	<16	<16

1: units are dpm/100 cm^2

A direct beta measurement on the floor exceeded the guideline for average beta activity. ORISE reports that the 1 m^2 area surrounding the measurement was resurveyed and the average beta activity was found to be 700 dpm/100 cm^2 (i.e. below the average guideline).

Survey Unit U7

In survey unit U7 (which includes the west part of Room 217), ORISE reported the following data on fixed point direct measurements and smears for alpha and beta surface activity:

Survey Area	Floor	Lower Walls	Upper Walls & Ceiling
# of Locations	10	10	10
Direct alpha range[1]	<39 to 95	<39	<39 to 190
Direct beta range[1]	<470	<470 to 49,000	<470
Removable alpha range[1]	<12	<12	<12
Removable beta range[1]	<16	<16	<16

1: units are dpm/100 cm^2

ORISE reports that the elevated alpha and beta direct readings found on the floor were determined to be coming from a ducting elbow of a blower housing. However they had no access points into the elbow to investigate further. Results for this location are found in Table 4a of Attachment 2 of the RFP. Table 5a of RFP Attachment 2 indicates the blower and ducting were removed and prepared for disposal. Additional remaining ducting has not been radiologically characterized. No mention is made regarding post-decontamination surveys.

Survey Unit U8

In survey unit U8 (which includes Rooms 203, 210, 211, 212, 213, 214, 215, 219, and 221), ORISE reported the following data on fixed point direct measurements and smears for alpha and beta surface activity:

Survey Area	Floor	Lower Walls & Equipment	Upper Walls
# of Locations	10	10	10
Direct alpha range[1]	<39 to 63	<39 to 84	<39 to 63
Direct beta range[1]	<470 to 1,300	<470 to 1,300	<470
Removable alpha range[1]	<12	<12	<12
Removable beta range[1]	<16	<16	<16

1: units are dpm/100 cm^2

ORISE found only 1 item which exceeded NRC Reg Guide 1.86 surface contamination guidelines: a blower in the southeast corner of Room 217. Results for this location are found in Table 4a of Attachment 2 of the RFP.

Table 5a of RFP Attachment 2 indicates the blower and ducting were removed and prepared for disposal. Additional remaining ducting has not been radiologically characterized. No mention is made regarding post-decontamination surveys.

Survey Unit U9

In survey unit U9 (which includes Rooms 201, 202, 204, 205, 206, 207, 208, 209, and 222), ORISE reported the following data on fixed point direct measurements and smears for alpha and beta surface activity:

Survey Area	Floor	Lower Walls & Equipment	Upper Walls
# of Locations	12	8	10
Direct alpha range[1]	<39	<39	<39
Direct beta range[1]	<470	<470	<470
Removable alpha range[1]	<12	<12	<12
Removable beta range[1]	<16	<16	<16

1: units are dpm/100 cm^2

1.3 SITE-SPECIFIC DEFINITION OF PROBLEMS

The building has largely been inactive since 1971 and that has caused the interior and exterior to significantly erode. This has led to the release of asbestos containing materials from the walls, roofing materials, wall petitions, floor coverings, pipe runs and boiler systems. Friable asbestos is being released continually and has contaminated the interior of the building. In addition to the asbestos contamination the building also contains areas of radiological contamination (Radium 226 and Thorium 232) and bird and animal debris that must be dealt with during remediation.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

2.1 KEY PROJECT PERSONNEL

Resumes of Jacobs key project personnel working on this project are presented in Appendix I

2.1.1 Program Manager

At Jacobs, the program manager is the senior Jacobs representative on a project, and is management's primary contact for the client. The programs manager's responsibilities include:

- negotiating and executing contracts and modifications,
- approving criteria and procedures manuals for the project,
- ensuring that Jacobs QA/QC program is applied,
- supporting the project manager in executing a successful project, and
- maintaining contact with the Buffalo District throughout the work.

Virgil Jansen is the Jacobs Program Manager on this project.

2.1.2 Project Manager

Jacobs Project Manager will be responsible for overall project direction and performance, including;

- principal project contact and liaison with the Buffalo District Project Manager and Contracting Officer,
- quality and timeliness of deliverables,
- schedule and budget tracking,
- progress reporting,
- work performed by subcontractors, and
- problem resolution.

Leo Mann III will serve as the Project Manager.

2.1.3 Site Manager

The Site Manager will be responsible for coordinating all site activities including those of the on-site subcontractors and all laboratory activities. Specific Site Coordinator duties include:

- providing overall direction and supervision of sampling activities,
- assuring that appropriate field logs are maintained for project activities,
- providing overall supervision of the collection, handling and shipping of all samples, and
- monitoring all sampling operations to ensure that all project site personnel adhere to the provisions of the Sampling and Analysis Plan (SAP) and Site-Specific Safety and Health Plan (SSHP).

Leo Mann III will serve as the Site Manager.

2.1.4 QA Manager

The Jacobs QA Manager is responsible for assuring that the Jacobs QA program is implemented in all project activities including:

- QA protocols and procedures,
- audits to see that all deliverables are properly reviewed and checked, and
- documentation of quality objectives

Virgil Jansen will serve as the Project QA Manager.

2.1.5 Contractor's Quality Control Site Manager

The Contractor's Quality Control Site Manager (CQCSM) will be responsible for the proper execution of field QC procedures. The CQCSM will also report to the QA Manager and/or the Program Manager outside the normal project chain of command on quality matters.

Leo Mann III will serve as the CQCSM.

2.1.6 Site Safety Health Officer

The Jacobs Site Safety and Health Officer is responsible for the overall implementation of the Safety and Health plan during onsite activities. The responsibilities of this position include:

- Assist in on-site training and the day to day on-site implementation and enforcement of the accepted safety and health plan.
- Be assigned to the site on a full time basis for the duration of field activities.
- Have authority to verify site compliance with specified safety and health requirements, Federal, state and OSHA regulations and all aspects of the safety and health plan.
- Have the authority to stop work if unacceptable health or safety conditions exist, and take necessary action to re-establish and maintain safe working conditions.
- Coordinate any modifications to the site-specific safety and health plan.
- In coordination with site management and the Safety and Health Manager, recommend corrective actions for identified deficiencies and oversee the corrective actions.
- The SSHO will also function as the Radiation Safety Officer.
- review and validation of radiological data and radiological laboratory results

David Fleming is the SSHO and reports to the Jacobs Health and Safety Manager.

2.2 ANALYTICAL LABORATORY

2.2.1 Asbestos

EMSL Analytical, Inc. (EMSL) will provide the primary analytical laboratory services for the asbestos assessment and abatement monitoring. EMSL is a full-service environmental analytical laboratory. The firm has fully equipped laboratories with the latest instrumentation and an advanced data management system. EMSL is accredited by the National Voluntary Laboratory Accreditation Program (NVLAP) for analysis of asbestos samples by Transmission Electron Microscopy (TEM) and Polarized Light Microscopy (PLM), by the American Industrial Hygiene Association (AIHA) for Phase Contrast Microscopy (PCM), and by the State of New York for asbestos analysis, refer to Appendix IX of the Quality Assurance Project Plan (QAPP) for copies of EMSL's certificates. EMSL's key laboratory personnel are discussed in EMSL's Quality Assurance Plan located in Appendix I of the QAPP. The point of contact is John VanVoorhees. The laboratory address is below:

EMSL Analytical, Inc.
107 Haddon Avenue
Westmont, NJ 08108
Office: (800) 220-3675
Fax: (856) 858-7141

2.2.2 Radiological

Severn Trent Laboratories, Inc. (STL) will provide the analytical laboratory services for the radiological water analysis. STL is a full-service environmental analytical laboratory that has been evaluated by the USACE Hazardous, Toxic and Radioactive Waste Mandatory Center of Expertise (USACE-HTRW-MCX) and is approved as a Hazardous Waste Testing Laboratory in support of their environmental investigations. The firm has fully equipped laboratories with the latest instrumentation and an advanced data management system. STL's key laboratory personnel are discussed in STL's Quality Assurance Plan (QAP) located in Appendix IV of the QAPP. The point of contact is Richard H. Mannz. The laboratory address is below:

STL St. Louis
13715 Rider Trail North
Earth City, MO 63045
Office: (314) 298-8566
Fax: (314) 298-8757

2.3 HEALTH AND SAFETY MANAGEMENT

The field activities described in this SAP will be conducted in accordance with the requirements of the Safety and Health Plan (SHP). Jacobs staff assignments for management of health and safety compliance are detailed in the SHP.

3.0 PROJECT SCOPE AND OBJECTIVES

3.1 TASK DESCRIPTION

3.1.1 Asbestos Assessment

Conduct a condensed asbestos assessment of Building 401. The assessment includes the following:

- Identify and locate building materials suspected to be asbestos containing materials (ACM).
- Delineate homogeneous areas for sampling.
- Sample suspect ACM and categorize material as friable or non-friable.
- Quantify amounts of ACM.
- Photograph sample locations, ACM, and various areas.
- Laboratory analysis of suspect ACM samples.
- Document findings.

3.1.2 Radiological Assessment

3.1.2.1 Pre-and Post-Construction Surveys

A pre-construction radiological survey will be performed concurrent with the asbestos assessment. The assessment includes the following:

- Resurvey of approximately 20% of work areas determined by ORISE to meet the NRC Reg Guide 1.86 criteria for Ra-226 and Th-232. Should any of these areas be found to contain activity exceeding NRC Reg Guide 1.86 criteria, they will be reclassified as MARSSIM Class 1 areas.

- Collection of direct alpha and beta measurements for use in determining estimated standard deviations (σ) for the various surfaces present within the building. This information will be necessary to apply MARRSIM Section 5.5.2.2 guidance to determine required number of measurements in the Class 1, 2, and 3 areas.

Post-construction radiological surveys will also be conducted (as required in Section 3.1 of the RFP) to ensure asbestos abatement activities did not radiologically contaminate the work area. Scope of these surveys will be similar in magnitude to MARSSIM Class 3 surveys.

3.1.2.2 Asbestos Radiological Status Determination

Final status surveys will be performed on all suspect asbestos during the assessment phase. These surveys will delineate asbestos which exceeds NRC Reg Guide 1.86 criteria. Asbestos removed from areas exceeding NRC Reg Guide 1.86 criteria will not be released from site.

The Scope of Work for the Asbestos Assessment/Abatement of Building 401 identifies the appropriate radiological release criteria to be the US Nuclear Regulatory Commission (NRC) Regulatory Guide 1.86 acceptable surface contamination levels. Attachment 2 of the RFP identifies the primary radiological contaminants as Ra-226 and Th-232. NRC Reg Guide 1.86 criteria for Ra-226 and Th-232 are:

Acceptable Surface Contamination Values

Contaminant	Average	Maximum	Removable
Ra-226	100 dpm/100 cm^2	300 dpm/100 cm^2	20 dpm/100 cm^2
Th-232	1,000 dpm/100 cm^2	3,000 dpm/100 cm^2	200 dpm/100 cm^2

Averages apply to areas not larger than 1 m^2. Maximums apply to areas not larger than 100 cm^2. Removable activity applies to 100 cm^2 areas. The Ra-226 values will be applied to alpha contamination, and the Th-232 values will be applied to beta-gamma contamination.

Work areas where asbestos abatement will occur will be included in the pre-construction radiological survey. Approximately 20% of such areas determined by ORISE to meet the NRC Reg Guide 1.86 criteria for Ra-226 and Th-232 will be resurveyed. Should any of these areas be found to contain activity exceeding NRC Reg Guide 1.86 criteria, they will be reclassified as MARSSIM Class 1 areas. Asbestos within such areas would be subject to all Class 1 final status survey requirements as described herein.

Any asbestos present in the locations found to exceed NRC Reg Guide 1.86 surface contamination guidelines will be considered radioactively-contaminated and will not be released from the site. This includes:

- areas identified by ORISE and not yet remediated
- areas identified during on-site operations to fail the MARSSIM final status surveys as described herein
- remediated areas (post-ORISE survey) as identified in Table 5a of Attachment 2 which fail MARSSIM Class 1 final status surveys, to be performed during on-site operations.

In addition, since NRC Reg Guide 1.86 applies only to surficial contamination and not volumetric contamination, asbestos-containing thermal systems insulation will not be released from site if: the exterior wrapping is not intact, or with intact wrapping, the wrapping fails the MARSSIM final status surveys as described herein. Asbestos-containing thermal systems insulation mud will also not be released if the hard, smooth surface has deteriorated and eroded to the point that an adequate total contamination survey can not be performed.

Areas exceeding NRC Reg Guide 1.86 criteria will be posted radioactive material areas. Remediated areas (post-ORISE survey) as identified in Table 5a of Attachment 2 requiring final status surveys are considered Class 1 survey units. Class 1 survey units will not exceed 100 m^2, and have been divided into survey units with relatively homogeneous characteristics (e.g., floor tile, ceiling tile).

All other areas requiring final status surveys are considered Class 2 survey units. Class 2 survey units will not exceed 1,000 m^2, and are divided into survey units with relatively homogeneous characteristics (e.g., floor tile, ceiling tile).

3.1.2.3 MARSSIM Criteria for Final Status Surveys

The following parameters are required inputs when determining the required number of data points in a final status survey unit: probability of making a Type I decision error (α), probability of making a Type II decision area (β), standard deviation (σ) of the contamination levels, derived concentration guideline ($DCGL_W$), and the lower bound of the gray region (LBGR). Type I and Type II decision area probabilities will initially both be set at 0.05. $DCGL_W$ values are the applicable NRC Reg Guide 1.86 values (average values for direct measurements). LBGR will initially be set to one-half the $DCGL_W$, as recommended in MARSSIM Section 5.5.2.2.

Not enough information is provided to estimate σ at this point. Individual data values are not provided in Attachment 2 of the RFP or in the ORISE report, and almost all individual data values reported by Batelle (Reference 1 of Attachment 2) are reported in a less than detection limit format. During the pre-construction radiological survey, direct alpha and beta measurements will be collected and used in determining estimated σ's for the various surfaces present within the building. MARRSIM Section 5.5.2.2 guidance will be used to determine required number of measurements in the Class 1, 2, and 3 survey units. Comparison of measurements from the reference areas and the Class 1, 2, or 3 survey units will be made using the Wilcoxon Rank Sum test. Reference area measurements will be collected in unaffected areas.

As recommended in MARSSIM Table 5.9, Class 1 survey units will receive 100% coverage for scanning surveys. Field instruments to be used for the scanning surveys include the following:

Detector Type	Radiation Detected	Nominal Background	Nominal Efficiency	Detector Area	Lc for 1-min count (dpm/100 cm^2)	Scan MDC (dpm/100 cm^2)
Gas Proportional	Alpha	5 cpm	20%	126 cm^2	21	NA
	Beta	400 cpm	20%	126 cm^2	185	842
Dual Phosphor Scintillation	Alpha	3 cpm	20%	100 cm^2	20	NA
	Beta	300 cpm	20%	100 cm^2	201	919

The critical levels (Lc) and the scan minimum detectable concentrations (MDCs) are below the applicable NRC Reg Guide 1.86 average and maximum contamination criteria. Any direct or scanning measurement within a Class 1 survey unit exceeding applicable NRC Reg Guide 1.86 average contamination criteria (i.e. $DCGL_W$) will be further investigated by determining the average and maximum contamination levels in the surrounding 1 m^2 area. Those areas exceeding NRC Reg Guide 1.86 criteria will not be released from the site.

Class 2 survey units will receive 50% coverage for scanning surveys, which is within the range recommended in MARSSIM Table 5.9. Any direct or scanning measurement within a Class 2 survey unit exceeding applicable NRC Reg Guide 1.86 average contamination criteria (i.e. $DCGL_W$) will be further investigated by determining the average and maximum contamination levels in the surrounding 1 m^2 area. Those areas exceeding NRC Reg Guide 1.86 criteria will not be released from the site.

3.1.3 Asbestos Abatement

Remove and properly dispose of asbestos containing materials from Building 401 as identified during the asbestos assessment. The abatement includes the following:

- Removal of friable ACM prior to removing non-friable ACM.
- Remove non-friable in such a manner as to not render it friable.
- Remove only debris that will hamper the removal and disposal of ACM.
- Remove asbestos in accordance with federal, state, and local regulation.
- Package and transport ACM debris in accordance with federal, state, and local regulations.
- Monitor work for radiation and segregate radioactive ACM waste from non-radioactive waste.
- Perform air monitoring during asbestos abatement for release of asbestos. Refer to Section 4.3 for air monitoring.

3.2 APPLICABLE REGULATIONS/STANDARDS

Work during the coarse of this projects shall be done in accordance with the following:

Code of Federal Regulations (CFR)

10 CFR

- Part 19 Notices, Instructions, and Reports to Workers
- Part 20 Standards for Protection Against Radiation
- Part 30 Rules of General Applicability to Domestic Licensing of Byproduct Material

29 CFR

- Part 1910 Occupational Safety and Health Regulations for the General Industry
- Part 1926 Occupational Safety and Health Regulations for the Construction Industry

40 CFR

- Part 61 National Emission Standards for Hazardous Air Pollutants.
- Part 763 Subpart E – Asbestos Hazard Emergency Response Action

State of New York

- 12 NYCRR Part 56 Asbestos

National Institute for Occupational Safety and Health (NIOSH)
- Method 7400: Fibers.
- Method 7402: Asbestos Fibers.

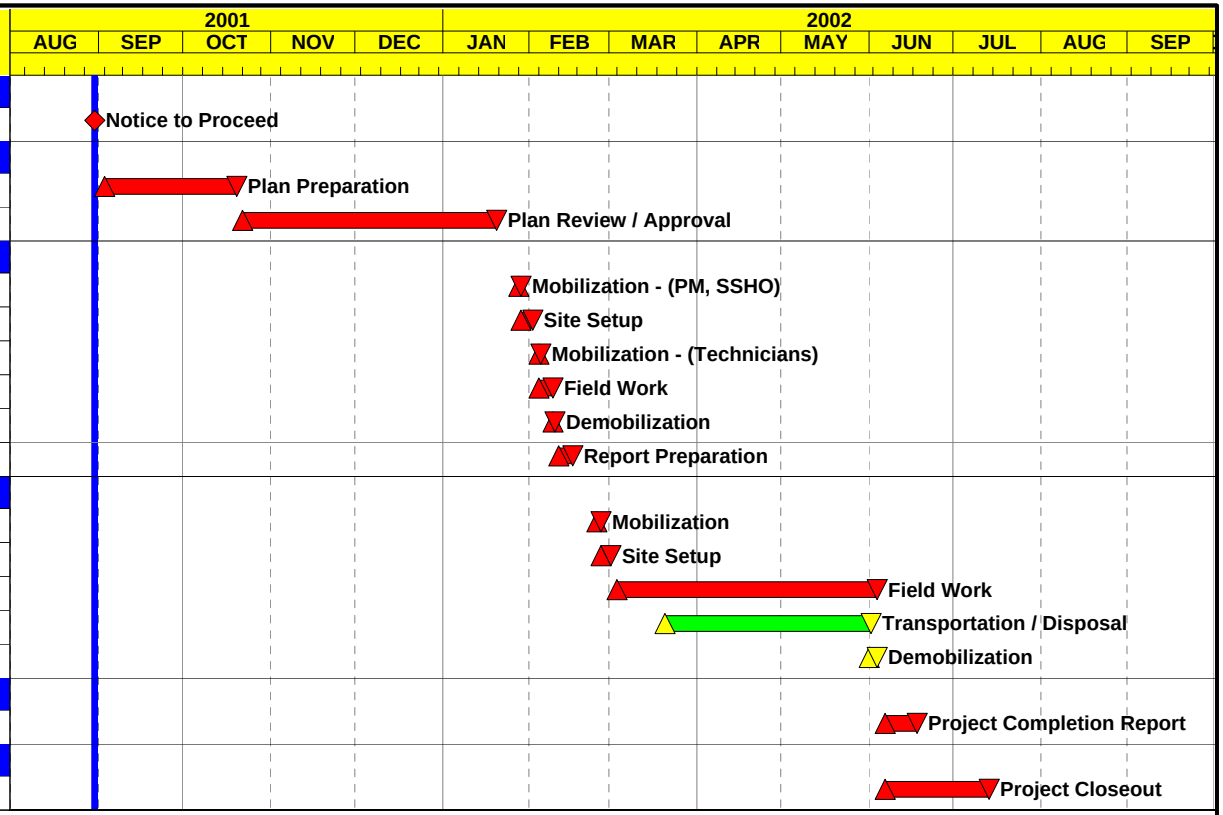
US Army Corps of Engineers
- Safety and Health Requirements Manual
- Safety and Occupational Health Requirements for Hazardous, Toxic, and Radioactive Waste (HTRW) Activities
- Radiation Protection Manual
- Ionizing Radiation Protection

US Nuclear Regulatory Commission (NRC) Regulatory Guide 1.86

3.3 PROJECT SCHEDULE

See next page for project schedule.

Activity ID	Activity Description	Orig Dur	Rem Dur	%	Early Start	Early Finish
NOTICE TO PROCEED						
10000	Notice to Proceed	0	0	0	31AUG01	
PLAN PREPARATION / APPROVAL						
10010	Plan Preparation	47	47	0	03SEP01	19OCT01
10020	Plan Review / Approval	90	90	0	22OCT01	19JAN02
ASSESSMENT						
10030	Mobilization - (PM, SSHO)	1	1	0	28JAN02	28JAN02
10040	Site Setup	4	4	0	29JAN02	01FEB02
10050	Mobilization - (Technicians)	1	1	0	04FEB02	04FEB02
10060	Field Work	5	5	0	04FEB02	08FEB02
10070	Demobilization	1	1	0	09FEB02	09FEB02
10080	Report Preparation	5	5	0	11FEB02	15FEB02
ABATEMENT						
10090	Mobilization	1	1	0	25FEB02	25FEB02
10100	Site Setup	4	4	0	26FEB02	01MAR02
10110	Field Work	92	92	0	04MAR02	03JUN02
10120	Transportation / Disposal	73	73	0	21MAR02	01JUN02
10130	Demobilization	3	3	0	01JUN02	03JUN02
PROJECT COMPLETION REPORT						
10140	Project Completion Report	11	11	0	07JUN02	17JUN02
PROJECT CLOSEOUT						
10150	Project Closeout	37	37	0	07JUN02	13JUL02



Start Date 31AUG01
Finish Date 13JUL02
Data Date 31AUG01
Run Date 25SEP01 14:36

Early Bar
Progress Bar
Critical Activity

DO01 Sheet 1 of 1

Jacobs

D.O. #1 Asbestos Assess/Abatement

Date	Revision	Checked	Approved

4.0 FIELD ACTIVITIES BY AREA OF CONCERN (AOC)

4.1 ASBESTOS BULK SAMPLING

4.1.1 Rationale

Asbestos bulk sampling will be conducted during the asbestos assessment and abatement (as necessary) portion of the project to determine the asbestos content of the suspect building materials.

4.1.2 Method

Sampling and analysis of asbestos bulk sampling will be performed as specified herein. Bulk samples will be analyzed according to 12 NYCRR 56 asbestos regulations for bulk samples. Asbestos samples will be analyzed using Polarized Light Microscopy (PLM). Samples of Non-friable Organically Bound (NOB) materials found to contain less that 1% by PLM will then be analyzed using Transmission Electron Microscopy (TEM).

4.1.3 Field Procedures

4.1.3.1 Equipment

Tools
Small hand tools required to access and remove the suspect asbestos containing materials, including but not limited to:

- Razor knife
- 5 in 1 paint scraper
- hammer
- asbestos coring device

Bags
The bags for suspect asbestos bulk sample collection will be reclosable zip lock type, 4" x 6" in size, 2 mils thick, with white stripe for labeling.

4.1.3.2 Sample Collection

After preliminary assignment of homogeneous areas and a visual inspection of the building interior, a sampling scheme will be developed. AHERA protocols will be used. This will include random sampling of each homogeneous material and a collection of a minimum of three samples of each type of homogenous thermal system insulation, surfacing treatments, and miscellaneous materials. For a sampling area of less that 1,000 square feet, the inspector will collect at least 3 samples, between 1,000 and 5,000 square feet, the inspector will collect at least 5 samples and for a sampling area over 5,000 square feet, seven samples will be collected for analysis.

Sample locations will be determined in the field by the JE asbestos inspectors. Once the sample location is identified, a radiological survey at that location will occur, provided by a JE Health Physics Technician. This technician will be with the asbestos inspectors at all times while inside the building. If radiological survey results are below NRC Reg Guide 1.86 criteria, then a sample will be taken at that location. Further explanation of the radiological screening techniques as they would apply to the asbestos assessment are discussed in the Radiological Control Contingency Plan.

The asbestos bulk samples will be placed in to the zip lock type bag and the sampling equipment decontaminated with a damp asbestos free wipe. The sample will then be documented in the logbook. Upon completion of sampling the samples will be logged onto the chain-of-custody form and double bagged and placed in the shipping container. The bulk samples will be sent to the laboratory for analysis following 12 NYCRR Part 56 and EMSL's Standard Operating Procedures for asbestos bulk sample analysis.

4.2 ASBESTOS AIR MONITORING

4.2.1 Personal Monitoring

4.2.1.1 Rationale

Asbestos personal air monitoring samples will be collected during the asbestos assessment and during the asbestos abatement to determine the levels of airborne fibers in the employees breathing zone.

4.2.1.2 Method

Sampling and analysis of airborne concentrations of asbestos fibers will be performed as specified herein. Personal sampling required by Regulations 29 CFR 1910.1001 and 1926.1101, will be NIOSH Method 7400 Phase Contract Microscopy (PCM) analysis. The sampling cassettes will be archived for the duration of the project plus 6 months.

4.2.1.3 Field Procedures

4.2.1.3.1 Equipment

Filter
0.8 micrometer pore size, 25-mm diameter mixed cellulose ester filter with a back-up pad and a fully conductive cassette with conductive extension cowl.

Pump
Low volume personal sampling pumps capable of maintaining a flow of 0.5 L/min to 4 L/min over and eight hour period.

Rotameter
Precision rotameter (a secondary calibration device) capable of indicating flow rates from 0.5 L/min to 4 L/min.

4.2.1.3.2 Sample Collection

A minimum of 25 percent of the employees will be monitored for asbestos using the following method.

1. Assemble the sampling train and operate the pump for 5 to 10 minutes to equilibrate the equipment. Do not connect the cassette during the equilibration period.
2. Place the sampling equipment on the employee so that it does not interfere with work performance. Attach the cassette to the shirt collar or as close as practical to the nose and mouth of the employee, i.e., in a hemisphere forward of the shoulders with a radius of approximately six to nine inches. The inlet should always be in a downward vertical position to avoid gross contamination. Position the excess tubing so that it does not interfere with the work of the employee.
3. Calibrate the pump and record the starting time.
4. Observe the pump operation for a short time after starting to make sure it is operating correctly.
5. Record the information on the Air Sampling Form.
6. Check the sampling train regularly. More frequent checks may be necessary when heavy filter loading is possible. Verify the sampler is still assembled properly and that the hose has not become pinched or detached from the cassette or the pump.
7. Periodically monitor the employee throughout the workday to verify sample integrity is maintained and cyclical activities and work practices are identified. When the employee leaves the work area for lunch or breaks in a clean, uncontaminated area, post calibrate the sampling train, remove the sampling media, and continue pump operation. Document the post calibration and stop time. When the employee returns, calibrate the sampling train, begin sampling, record the start time and calibration. Turning off the pump during lunch is not recommended unless equilibration is performed.
8. Take detailed notes concerning visible airborne contaminants, work practices, potential interferences, movements, and other conditions to assist in determining appropriate engineering controls.
9. Prepare blank(s) during the sample period, a minimum of two blanks or 10% for any given sampling period. The blanks should be opened but not used to take samples. They should be handled in the same manner as any sampling media used in sampling air contaminants, with the exception that no air is drawn through them.
10. Before removing the pump at the end of the sample period, post calibrate the sampling train.
11. Turn off the pump and record the ending time.
12. Remove the cassette from the pump and seal with caps.

4.2.2 Area Monitoring

4.2.2.1 Rationale

Asbestos area air monitoring samples will be collected during the asbestos abatement to determine the levels of airborne fibers inside and outside the work area.

4.2.2.2 Method

Sampling and analysis of airborne concentrations of asbestos fibers will be performed as specified herein. Area sampling during asbestos abatement work, NIOSH Method 7400 Phase Contrast Microscopy (PCM) with optional confirmation of results by NIOSH Method 7402 Transmission Electron Microscopy (TEM) will be used. All area sampling will be conducted at a sufficient flow rate and time to collect a sample volume necessary to establish the limit of detection of the method used at 0.01 f/cc. The sampling and analytical method used for final clearance air sampling will be the mandatory EPA TEM Method specified at 40 CFR 763. The sampling cassettes will be archived for the duration of the project plus 6 months.

4.2.2.3 Field Procedures

4.2.2.3.1 Equipment

Filter
0.8 micrometer pore size, 25-mm diameter mixed cellulose ester filter with a back-up pad and a fully conductive cassette with conductive extension cowl.

Pump
Medium to high volume area sampling pumps capable of maintaining a flow of 2 L/min to 16 L/min.

Rotameter
Precision rotameter (a secondary calibration device) capable of indicating flow rates from 2 L/min to 16 L/min.

4.2.2.3.2 Sample Collection

Area samples will be collect at the following locations: One in each clean room, one sample for each 50,000 cubic feet of air space (minimum of two samples) inside the work area, and at least two samples outside the asbestos containment near the work area using the following method.

1. Assemble the sampling train and operate the pump for 5 to 10 minutes to equilibrate the equipment. Do not connect the cassette during the equilibration period.
2. Attach the cassette to pump tubing. The inlet should always be in a downward vertical position to avoid gross contamination.
3. Calibrate the pump and record the starting time.

4. Observe the pump operation for a short time after starting to make sure it is operating correctly.
5. Record the information on the Air Sampling Form.
6. Check the sampling train regularly. More frequent checks may be necessary when heavy filter loading is possible. Verify the sampler is still assembled properly and that the hose has not become pinched or detached from the cassette or the pump.
7. Take detailed notes concerning visible airborne contaminants, work practices, potential interferences, movements, and other conditions to assist in determining appropriate engineering controls.
8. Prepare blank(s) during the sample period, a minimum of two blanks or 10% for any given sampling period. The blanks should be opened but not used to take samples. They should be handled in the same manner as any sampling media used in sampling air contaminants, with the exception that no air is drawn through them.
9. Before removing the pump at the end of the sample period, post calibrate the sampling train.
10. Turn off the pump and record the ending time.
11. Remove the cassette from the pump and seal with caps.

4.2.2.3.3 Sampling Design

Background Monitoring: Background samples may be collected 24 hours prior to the isolation of the work area. Background samples will comply with methods and locations as described in 12 NYCRR Part 56–17-2.

Asbestos Abatement Monitoring: During asbestos abatement, the air technician will collect air samples at locations described in 12 NYCRR Part 56-17.3.

Clearance Monitoring: The clearance sampling for airborne fiber concentrations will be in accordance with 40 CFR 763 and shall comply with methods and locations as described in 12 NYCRR Part 56-17.2.

4.3 RADIOLOGICAL

4.3.1 Personal Monitoring

4.3.1.1 Rationale

Personal air monitoring samples will be collected from 33% of the workers during the asbestos assessment and during the asbestos abatement to determine the levels of airborne radioactivity in the employees breathing zone.

4.3.1.2 Method

Sampling and analysis of airborne radioactivity concentrations will be performed as specified herein. Analysis of air filters for long-lived gross alpha activity is based on U.S. Department of Energy Environmental Measurements Laboratory Procedures Manual HASL-300 methodology.

4.3.1.3 Field Procedures

4.3.1.3.1 Equipment

Filter
0.8 micrometer pore size, 37-mm diameter mixed cellulose ester filter with a back-up pad and cartridge.

Pump
Low volume personal sampling pumps capable of maintaining a minimum flow of 4 L/min over an eight hour period.

Rotameter
Precision rotameter (a secondary calibration device) capable of indicating minimum flow rates of 4 L/min.

Radiation Detection Instruments
Scintillation detector (Ludlum Model 43-10-1 or equivalent) and compatible scaler (Ludlum Model 2000 or equivalent)

4.3.1.3.2 Sample Collection

Personal breathing zone air samples collected for airborne radioactivity determination will be analyzed on-site routinely for long-lived gross alpha activity, but may also require off-site isotopic analysis at the end of the project for radium, thorium, and uranium isotopes. This will only occur if workers are likely to have exceeded an internal radiation committed effective dose equivalent of 100 mrem, which is very unlikely given the nature and extent of radioactive contamination in Building 401. The following method will be used to collect the samples:

1. Connect an unused, loaded sample cartridge to the personal sampling pump.

2. Connect the precision rotameter to the cartridge inlet and turn the pump on. Adjust the sampling pump flow screw until the desired steady flow rate is obtained.

3. Record the sample start time (using the time or a zero as applicable), beginning flow rate, collection date, and wearer's name on the Air Sampling Form.

4. Attach the sampling pump to the wearer with the sample cartridge within 12 inches of the wearer's nose and mouth. The inlet opening of the cartridge should face down.

5. Connect the rotameter to the cartridge inlet and measure the flow rate, turn pump off and remove the cartridge from the pump.

6. Record the sample stop time or minutes ran (as applicable), ending flow rate, pump ID number, and rotameter ID number on the Air Sampling Form. Remove the cartridge from the sampling pump.

7. The sample should be stored for a minimum of 72 hours to allow short-lived radon-222 and radon-220 daughters to decay. More rapid analysis (i.e., "quick count") is possible and may be performed as determined necessary by the JE Health Physicist on a case-by-case basis.

8. The sample shall be analyzed following the appropriate decay period using the alpha scintillation detector and compatible scaler. Record analysis results, analysis times, posted detector background count rate, posted detector efficiency, and detector & scaler serial number.

9. Sample Calculations. The long-lived gross alpha activity concentration is calculated using the following equation:

$$C = \frac{\frac{SC}{SCt} - DR}{DE x V x 2.22E+06}$$

where:

C = long-lived gross alpha activity concentration (uCi/ml)
SC = sample count
SCt = sample count time (minutes)
DR = detector background count rate (cpm)
DE = detector efficiency (cpm/dpm)
V = air volume (ml)

The Critical Level Concentration (CLC) is the level of air filter analysis instrument response at which there is a 5% probability of incorrectly identifying an instrument background value as a "greater than background" result. The units for the CLC have been converted from number of instrument counts to airborne radioactivity concentration in uCi/ml. The CLC equation is derived from Equation (3-2) of NUREG-1507, *Minimum Detectable Concentrations with Typical Radiation survey Instruments for Various Contaminants and Field Conditions*. The CLC is calculated using the following equation:

$$CLC = \frac{1.645x\sqrt{\frac{DR}{SCt}+\frac{DR}{BCt}}}{DExVx2.22E\ +\ 06}$$

where:

CLC = critical level concentration (uCi/ml)
DR = detector background count rate (cpm)
SCt = sample count time (minutes)
BCt = background count time (minutes)
DE = detector efficiency (cpm/dpm)
V = volume (ml)

4.3.2 Area Monitoring

4.3.2.1 Rationale

Airborne radioactivity area air monitoring samples (one upwind and one downwind) will be collected outside Building 401 to verify engineering controls are effective at minimizing airborne radioactivity emissions. When possible, outdoor area air monitors will be placed in areas that are free from obstructions or conditions that could affect the air sampling results. The air monitors will normally be placed twice the distance from an obstruction or structure as the obstruction or structure is high (i.e. air monitor would be placed 10 feet away from a 5-foot tall obstruction). In addition, outdoor air monitors will be placed when possible in areas that do not have turbulent air conditions, such as nearby roads or active equipment.

Inside Building 401, personal air monitoring will be used initially versus general work area monitoring because of the likelihood of work groups being widely dispersed within the building. In such cases it can be difficult for general work area air monitoring to be representative of the air inhaled by the various work groups. However work area air monitoring may be performed when work groups are confined to a local area within the building. Also, personal air monitoring will be reduced or eliminated should initial air monitoring results during abatement confirm that airborne radioactivity levels are low (i.e. less than 5% DAC).

4.3.2.2 Method

Sampling and analysis of airborne radioactivity concentrations will be performed as specified herein. Analysis of air filters for long-lived gross alpha activity is based on U.S. Department of Energy Environmental Measurements Laboratory Procedures Manual HASL-300 methodology.

4.3.2.3 Field Procedures

4.3.2.3.1 Equipment

Filter
Five (5) micrometer pore size, 4 inch diameter minimum, glass fiber filter with a backing pad and sampling head.

Pump
Area sampling pumps capable of maintaining a minimum flow of 200 L/min over a 24 hour period.

Rotameter
Precision rotameter (a secondary calibration device) capable of indicating flow rates of at least 200 L/min.

Radiation Detection Instruments
Gas proportional or scintillation detector (Ludlum Model 43-68 ot 43-89 or equivalent) and compatible scaler.

4.3.2.3.2 Sample Collection

1. Without touching the filter, place an unused filter and backing pad on the sampling head. Do not invert the filter face: side facing out in the package must also face out on the sampling head.

2. Connect the sampling head to the precision rotameter, and connect the rotameter to the pump. Turn the pump on and adjust the flow screw until the desired steady flow rate is obtained. When ambient temperatures are below 32^{o} F, check the flow rate again after 10 minutes of pump operation.

3. Record the sample start time, beginning flow rate, and collection date on the Air Sampling Form.

4. At the end of the sampling period, connect the rotameter between the sampling head and the pump to measure the flow rate.

5. Shut off the pump. Without touching the filter, remove the filter and backing pad and place them in a protective envelope.

6. Record the sample stop time, ending flow rate, pump ID number, and rotameter ID number on the Air Sampling Form.

7. The sample should be stored for a minimum of 72 hours to allow short-lived radon-222 and radon-220 daughters to decay. More rapid analysis (i.e., “quick count”) is possible and may be performed as determined necessary by the JE Health Physicist on a case-by-case basis.

8. The sample shall be analyzed following the appropriate decay period using the gas proportional or scintillation detector and compatible scaler. Record analysis results, analysis times, posted detector background count rate, posted detector efficiency, and detector & scaler serial number.

9. Sample Calculations. The long-lived gross alpha activity concentration is calculated using the same equation specified in Step 9 of Section 4.3.1.3.2 above.

10. Samples may be sent off-site for isotopic analysis to achieve lower detection limits as needed.

4.3.3 Bulk Waste Monitoring

4.3.3.1 Rationale

Radiological monitoring for surface contamination will be performed by JE health physics technicians on all items (including bulk waste containers) being removed from radioactive material areas. Asbestos waste removed from radioactive material areas will not be allowed to leave the site, but may be transferred on site to another designated radioactive material storage area. In such cases surface contamination monitoring will be performed on the container exteriors to ensure removable radioactive contamination is not being tracked out of radioactive material areas.

4.3.3.2 Method

Screening of the waste using a alpha detector and/or GM detector similar. Refer to Appendix III of the Radiological Control Contingency Plan for a description of the screening method.

4.3.3.3 Field Procedures

Refer to Appendix III of the Radiological Control Contingency Plan for a description of the field screening procedures.

4.3.4 Waste Water Monitoring

4.3.4.1 Rationale

All decontamination water, shower water, and water collected from radioactive material areas will be filtered, collected, and monitored for gross alpha activity. Only waters which meet the requirements of 10 CFR 20.2003 will be released to the sanitary sewer system.

4.3.4.2 Method

Sampling of water collected from radioactive material areas will be performed as specified herein, and analysis is based on EPA 600/4-80-032, “Prescribed Procedures for Measurement of Radioactivity in Drinking Water”.

4.3.4.3 Field Procedures

4.3.4.3.1 Equipment

Container
Plastic sealable jar - minimum of 750 ml in size.

4.3.2.3.2 Sample Collection

1. Collect water in labeled sample container.

2. Add sufficient nitric acid to adjust ph<2. The container will then be sealed for shipping.

3. Place samples into an appropriate secondary containment designed for shipping (e.g., plastic cooler). Samples will not be shipped as radioactive material, since they will be below the Department of Transportation (DOT) definition of radioactive material (49 CFR 173.403).

5.0 FIELD OPERATIONS DOCUMENTATION

5.1 DAILY QUALITY CONTROL REPORTS (QCR)

Daily quality control will be performed in accordance with the Contractor's Quality Control Plan.

5.2 FIELD LOGBOOK AND/OR SAMPLE FIELD SHEETS

5.2.1 Asbestos Bulk Samples

A bound field logbook will be used to record the information concerning the asbestos bulk samples collected. At a minimum, the following information will be recorded:

- date of sample collection
- sample location
- sample number
- visual description of material being sampled
- quantity of material present
- condition of material
- sampler's name
- comments

The identified suspect ACM will be quantified by dimension. All volumes recorded will be reported in cubic feet. In the case of pipe insulation, the lengths of pipe containing the insulation, pipe diameters, and insulation thickness of diameter, and insulation condition shall be recorded.

5.2.2 Asbestos Air Samples

Asbestos air sample information will be recorded on air sampling field sheets. The sheets will include the following information:

- date of sample collection
- sample location
- sample number
- start time and stop time
- flow rate
- volume
- analytical procedure required
- sampler's name
- comments

See Appendix II for an example of an air sampling field sheet.

5.2.3 Radiological Records

The Radiation Control Contingency Plan contains the standard forms which will be used to document:

- airborne radioactivity sampling results (both area and personal monitoring)
- surface contamination monitoring results for equipment and material being brought out of radioactive material areas
- surface contamination monitoring results generated in support of pre- and post-construction radiological surveys, and
- surface contamination monitoring results generated in support of MARSSIM Class 1, 2, and 3 final status surveys.

5.3 PHOTOGRAPHIC RECORDS

Digital photographs shall be taken of the various areas of the building, asbestos materials, and sampling locations.

5.4 SAMPLE DOCUMENTATION

5.4.1 Sample Numbering and Labeling

A unique sample ID number will be assigned to each sample location. This ID number will be on the sample container, a plastic zip-lock sample bag for bulk samples, and will be placed on a label to be affixed to the air sampling cassette for air samples. The sample ID will consist of four distinct fields and will be as follows:

BN – Date – X###

where,

BN	=	Building Number (401)
Date	=	Date in MMDDYY format
X	=	Matrix Type "AB" for asbestos bulk sample "AP" for asbestos air personal sample "AA" for asbestos air area sample "RW" for radiological water "RA" for radiological air
###	=	Sample number

Example:

401 – 102001 – AB001: Bulk asbestos sample number one from building 401 collected on October 20, 2001.

For bulk asbestos samples the sample ID number and the sample location will be recorded on the sample area diagram, in the field logbook, and the Chain of Custody sheet. Upon completion of the field survey, the bulk sample bags will be placed in a second oversized zip-lock bag, along with the Chain of Custody sheet, placed in a FEDEX box, and sent overnight delivery to EMSL for analysis.

For air samples the sample ID number will be placed on a label and affixed to the cassette and the sample location and other information recorded on the air sampling field sheet. Samples for onsite reading will be taken to the onsite laboratory for analysis. Samples for offsite analysis will be documented on a Chain of Custody sheet. Upon completion of sampling the cassettes, a copy of the air sampling form, and the Chain of Custody form will be placed in an oversized zip-lock bag, placed in a FEDEX box, and sent overnight delivery to EMSL for analysis.

5.4.2 Chain-of-Custody Records

A Chain of Custody (COC) form will be used for all samples being shipped off-site to a laboratory. The COC will contain the following information:

- Sampler
- Sample number
- Date
- Sample type
- Analysis requested
- Signature of person releasing samples
- Signature of laboratory person receiving samples

See Appendix II for an example of a COC form.

5.5 FIELD ANALYTICAL RECORDS

Field analytical records will be maintained for onsite radiological analysis performed. Refer to the Radiation Control Contingency Plan for standard forms which will be used to document the following:

- airborne radioactivity sampling results
- surface contamination monitoring results

5.6 DOCUMENTATION PROCEDURES/DATA MANAGEMENT AND RETENTION

Data generated onsite or offsite by laboratories will be maintained onsite in the Jacobs construction trailer throughout the assessment and abatement of Building 401. After completion of the records a final closeout report will be generated to include the data generated during the

assessment and abatement. These records will be maintained for 10 years following completion and submittal of the closeout document.

6.0 SAMPLE PACKAGING AND SHIPPING REQUIREMENTS

6.1 PACKAGING

6.1.1 Asbestos

Asbestos samples will be packed and sealed into a hard cardboard box designed for shipping. Packing material for air samples (if required) will be of a material that is not electrostatic in nature (paper or cardboard, not styrofoam).

6.1.2 Radiological

Radiological water samples will be collected in a plastic sample container, which will be sealed after collection. Samples will be placed into an appropriate secondary containment designed for shipping (e.g., plastic cooler). Samples will not be shipped as radioactive material, since they will be below the Department of Transportation (DOT) definition of radioactive material (49 CFR 173.403).

Should radiological air samples need to be sent off-site for isotopic analysis, they will be managed as described above for asbestos samples.

6.2 SHIPPING

Shipping on all samples will be Fed-Ex Priority Overnight directly to the laboratory.

7.0 INVESTIGATION-DERIVED WASTE (IDW)

7.1 EQUIPMENT AND PERSONAL PROTECTIVE EQUIPMENT (PPE)

PPE generated outside of radioactive material areas that becomes contaminated with asbestos and that cannot be decontaminated and materials used to decontaminate equipment and personnel that is used during the asbestos assessment will be bagged up and left onsite in the area to be disposed by the abatement contractor during abatement activities.

7.2 LABORATORY SAMPLES

The laboratory will dispose of samples in accordance with their disposal plan and in accordance with federal, state, and local regulations.

8.0 NONCONFORMANCE/CORRECTIVE ACTIONS

The following is a list of potential discrepancies and problems, and possible corrective actions.

Discrepancy	Corrective Action
Improper Sampling Procedures	Resample following correct procedure
Problems/Inconsistencies in Laboratory Sample Receipt	Telephone coordination with laboratory to resolve problems, resampling if required.
Laboratory is unable to handle sample load in the required turnaround time.	Laboratories have offices throughout the nation capable of performing the required analysis. Jacobs will coordinate with the laboratory and arrange to send the samples to another office to meet analytical turnaround time.

Appendix I

Resumes

VIRGIL W. JANSEN, P.E.
Program Manager

EDUCATION
MS, Environmental Engineering – University of Illinois, 1972
BS, Civil Engineering – University of Illinois, 1971

LENGTH OF SERVICE
Sverdrup Hire Date: 1996
Other firms/agencies: 24 years (Entered the profession 1972)

REGISTRATIONS/CERTIFICATIONS
Registered Professional Engineer in Illinois (1975)
40-Hour HAZWOPER
Executive Enterprise Environmental Regulations Course
Lion Technology Hazardous Waste Management Workshop

BACKGROUND/EXPERTISE
Mr. Jansen has 26 years of experience in environmental engineering project management, and construction management of remediation work.

PROJECT EXPERIENCE

UST Program, Louisville District, USACE – IN. Senior Project Manager responsible for negotiation, cost estimating, management, and execution of 22 delivery orders at 10 sites in Indiana for this $12-million contract.

UST Program, Louisville District, USACE – Various Sites, KY. Project Manager responsible for all project management duties for this $6-million UST program, including oversight of site superintendent, scheduling, and cost control/cost accounting.

Navy Environmental Job Order Contract. Program Manager responsible for management of an indefinite quantity-indefinite delivery order contract for environmental remediation construction at Great Lakes Naval Training Center involving asbestos UST, wetlands restoration, and contaminated soil removal projects.

Philip Environmental Services Corporation – Columbia, IL. Construction Manager responsible for supervision of proposal and project managers in remediation contracting arena. Also, act as liaison between consulting and remediation divisions in the Columbia office. Project Management including proposal preparation, cost estimate review, subcontract negotiations, implementation of corporate special projects team. Major projects included: construction management of interim remedial measures project at former MPG facility; project management of site remediation of former open dump located din Louisville, KY; and project management of source removal action at former MGP facility.

Central States Environmental Services, Inc. – Centralia, IL. Engineering Manager responsible for technical management functions related to LUST and hazardous waste remediation projects, client and regulatory agency liaison, project cost estimation, contract negotiation and project management for an environmental remediation contractor. Major projects include: project management of LUST projects including investigation, tank removal and soil/water remediation, regulatory agency contact on 20+ sites throughout Illinois; and project management of hazardous waste remediation sites, including engineering evaluations, cost estimation, site specific work plan, health and safety plan, client liaison, and negotiation with regulatory agencies.

Barttelbort Engineering & Excavating, Inc. – Freeburg, IL. Vice President and Project Manager responsible for all management functions, including daily office management, estimating, and overall project planning and management, liaison with project owner and/or engineer for general construction corporation. Involved in subdivision development, residential construction, utility and treatment (water and sewer) facility construction, and storm sewers systems.

Haier Plumbing & Heating, Inc. – Okawville, IL. Project Manager responsible for limited management functions, day-to-day office management, estimating and overall construction project management. Also, acted as a liaison with project owner and/or engineer for a mechanical and municipal utility construction firm.

WORK HISTORY

Jacobs Sverdrup Constructors 1996 to Present

LEO F. MANN III
Environmental Engineer - Project Manager – Asbestos and Lead Services

EDUCATION
MBA – Lindenwood University, 1996
BS, Marketing and Management – University of Missouri-St. Louis, 1983

LENGTH OF SERVICE
Jacobs/Sverdrup Hire Date: 1993
Other firms/agencies: 6 years (Entered the profession in 1987)

REGISTRATIONS/CERTIFICATIONS
Certificate, Hazardous Waste Operations and Emergency Response
Certificate, EPA AHERA Asbestos Abatement Contractor/Supervisor
Certificate, EPA AHERA Asbestos Course for Inspector Training
Certificate, EPA AHERA Asbestos Project Designer Course
Certificate, EPA Lead Inspector and Risk Assessor
Certificate, NIOSH 582 Sampling and Evaluating Airborne Asbestos Dust
Certificate, Lead Abatement Contractor/Supervisor
Red Cross First Aid/CPR
OSHA 10-Hour Construction Safety Training
License, Asbestos Supervisor, Missouri
License, Asbestos Inspector, Missouri, Illinois, North Carolina, Kentucky
License, Asbestos Project Designer, Missouri, California
License, Certified Site Surveillance Technician, California
License, Lead Inspector, Missouri

PROFESSIONAL AFFILIATIONS/AWARDS
Environmental Information Association (EIA)

BACKGROUND/EXPERTISE
With more than fourteen years of experience, Mr. Mann performs, as well as manages, a staff that provides asbestos/lead/PCB in-buildings surveys and designs, UST removal design and oversight, phase I site assessments, formulates detailed construction estimates, provides contractor project management and project oversight, and performs asbestos/lead/PCB related industrial hygiene services.

PROJECT EXPERIENCE

Survey, Design, Abatement, Project Management (PM) – Project Oversight

Parkway School District ($500K) – St. Louis County, MO Project Principle for a two-year, multi-phase, environmental consulting services contract providing professional services in conjunction with the school districts renovation activities at any of the 31 campuses within the Parkway School District. Responsibilities include survey and design, project construction estimating, contract management, subcontractor oversight, UST removal, asbestos, lead paint,

PCB, and mercury-containing fluorescent light abatement services, daily and clearance air monitoring, and project closeout.

Survey, Design, Abatement, Project Management (PM) – Project Oversight

Morgan Hill Unified School District ($2 million) – San Jose, CA. Project Manager for a two-year, multi-phase, modernization project providing asbestos, lead paint, PCB, and mercury-containing fluorescent light abatement services in conjunction with $30 million renovation activities to the Live Oak High campus. Responsibilities include survey and design, project construction estimating, contract management, subcontractor oversight, daily and clearance air monitoring, and project closeout.

Survey, Design, Abatement, Project Management (PM) – Project Oversight

Santa Monica-Malibu Unified School District ($2 million) – Santa Monica, CA. Project Manager for a two-year, multi-phase, modernization project providing asbestos, lead paint, PCB, and mercury-containing fluorescent light abatement services in conjunction with $50 million renovation activities to 16 schools in the Santa Monica-Malibu Unified School District. Responsibilities include survey and design, project construction estimating, contract management, subcontractor oversight, daily and clearance air monitoring, and project closeout.

FAA Air Traffic Control Center – Honolulu, HW Project Manager providing asbestos/lead/PCB survey and abatement design in conjunction with the demolition of a 50,000 sf FAA facility located in Diamond Head crater.

Keebler Company – Elmhurst, IL. Project Manager providing asbestos survey and bakery oven assessments and developing a report for each of the newly acquired President Bakery facilities located in: Marietta, Oklahoma; Birmingham, Alabama; Augusta, Georgia; Lake Bluff, Illinois; Little Rock, Arkansas; Houston, Texas; Charlotte, North Carolina; Louisville, Kentucky; and Cleveland, Tennessee.

Keebler Company – Louisville, KY. Project Manager responsible for providing Keebler a "turn-key" service for the demolition and disposal of an asbestos-containing 200-ft-long baking line unit during a two-week plant shutdown.

Keebler Company – Augusta, GA. Project Manager responsible for providing Keebler a "turn-key" service for the demolition and disposal of three abandoned asbestos-containing baking line units. The work was performed over a weekend while working around the clock in order to meet the facilities startup schedule.

Keebler Company – Elmhurst, IL. Project Manager providing an Asbestos Management Plan for each Keebler facility located in Louisville, Kentucky; Charlotte, North Carolina; Birmingham, Alabama; Cleveland, Tennessee; Marietta, Oklahoma; Lake Bluff, Illinois; Little Rock, Arkansas; and Augusta, Georgia.

Okaloosa County School District – Okaloosa County, FL ($3 Million). Project Manager for a three-phase, five-year project providing asbestos, lead paint, and PCB abatement services in conjunction with $140 million renovation activities to 30 schools in the Okaloosa County School District. Responsibilities include: survey and design, project construction estimating, contract management, subcontractor oversight, daily and clearance air monitoring, and project closeout.

FAA Air Traffic Control Center – Oakland, CA. Site Surveillance Technician providing industrial hygiene services for asbestos and lead during abatement activities in the occupied air traffic control center.

Concorde Brands – St. Louis, MO. Project Manager performing asbestos-containing building material abatement design for the removal of friable and non-friable materials in the single-story office building damaged as a result of fire.

Pet Building – St. Louis, MO. Project Manager performing asbestos-containing building material, lead paint, and PCB inspection of the 15-story building. Provided asbestos abatement project design, air monitoring, and oversight for the 15th floor abatement of 4,000 square feet of friable material.

Survey, Design, Industrial, Hygiene Services

IDO HTRW Contract, U.S. Army Corps of Engineers – Nashville District (Ohio River Division), KY.

- *Fort Campbell Facility, Old Hospital Complex Demolition, Fort Campbell, KY* – Project manager for a three-phase project providing industrial hygiene services for monitoring asbestos abatement work at the Old Hospital Complex (OHC) Demolition. The project includes the abatement of asbestos-containing materials from and demolition of the 54 buildings that comprise OHC.
- *Fort Campbell Facility, Demonstration Project, Fort Campbell, KY* – Developed project design, supervised the field activities of the asbestos and demolition contractors, provided environmental and clearance air monitoring, formulated the project's final report, and consulted the Fort Campbell Environmental Office in their communications with Kentucky Department of Air Quality. Project involved demonstrating the feasibility of removing and disposing of drywall and joint compound as a non-ACM homogeneous material, with no significant fiber release.

Survey, Design

DFAS – San Bernardino, CA. Project Manager performing asbestos-containing building material and lead paint inspection, and project design of two three-story, 70,000-sq-ft office buildings.

Building 5500 – Edwards AFB, CA. Project Manager performing asbestos containing building material and lead paint inspection of the three-story hospital building.

29 Palms Marine Base – 29 Palms, CA. Project Manager performing asbestos-containing building material, lead paint, and PCB inspection of nine warehouse buildings.

Pentagon Navy Annex – Arlington, VA. Project Manager performing asbestos-containing building material surveys in an occupied office building.

NASA Goddard Space Flight Center – Greenbelt, MD. Project Manager performing asbestos-containing building material surveys in six occupied buildings totaling over 500,000 square feet.

Los Angeles World Airport – Los Angeles, CA. Project Manager performing asbestos/lead/PCB/mercury containing lamps survey and abatement design for two 80,000-sq-ft air freight building.

Abatement, Project Management

Brand Environmental Services, Inc. – St. Louis, MO. Project Manager and estimator covering a territory that includes Missouri, Illinois, Kansas, Iowa, and Nebraska. Responsibilities included: coordinating subcontractors and defining their scope of work; creating job scheduling using time line software; and managing commercial and industrial jobs ranging in price to $5 million.

May Company – St. Louis MO. Project Manager for all asbestos removal work for the May Company facilities located throughout Missouri, Kansas, and Illinois.

Lambert International Airport – St. Louis, MO. Project Manager/Estimator for a one-year asbestos removal project at the main terminal of Lambert International Airport.

Union Electric – St. Louis, MO. Project Manager/Estimator for asbestos removal and insulation projects located at various Missouri power plant sites.

Monsanto Company – Sauget, IL. Project Manager for a second shift asbestos removal crew that worked seven days per week in order to complete scheduled project during Monsanto Krumrich Plant process shutdown.

Survey, Abatement, Project Management

Monsanto Company – St. Louis, MO. Asbestos Project Manager performing asbestos in buildings survey at the Monsanto Queeny Plant.

Survey

State Farm Regional Headquarters – Columbia, MO. Asbestos Project Manager performing asbestos in-buildings survey in an occupied office facility covering 200,000 square feet.

OMNITRANS – San Bernardino, CA. Project Manager performing an asbestos survey of a 30,000-sq-ft bus maintenance and office facility scheduled for demolition.

TECHNICAL PAPERS/PUBLICATIONS

Presentations

EIA National Conference, 1995 – Presented the project design and results of the U.S. Army Corps of Engineers – Nashville District's Fort Campbell, Kentucky Demonstration Project

WORK HISTORY

Jacobs Engineering Federal Programs 1993 to Present

DAVID L. FLEMING

Health Physicist

Education: Coursework Completed for M.S. Environmental Engineering, Health Physics emphasis, Northwestern University, Evanston, Illinois
B.S., 1986, Physics, Bradley University, Peoria, Illinois

Mr. Fleming has over thirteen years of experience in environmental health physics. He has worked at the DOE Weldon Spring Site Remedial Action Project for twelve of these thirteen years. He is currently the ES&H Department Worker Protection Group Supervisor responsible for all programmatic aspects of WSSRAP radiation protection and industrial hygiene programs, including the site 10 CFR 835 PAAA Coordinator position. He is also the Jacobs corporate radiation safety officer, providing advice and guidance on radiation protection issues to Jacobs offices as needed.

Mr. Fleming has prepared environmental documents in support of the NEPA/CERCLA process, has developed project documents as required by DOE Orders and federal regulations, and has participated in numerous health physics field activities in support of WSSRAP project needs. Mr. Fleming had previously held the ES&H Field Operations Supervisor position for over three years. He was responsible for ES&H oversight and support for the WSSRAP radioactive/mixed waste disposal cell, and was active in all phases of site remediation including initial characterization, building demolition, asbestos abatement, contaminated soils removal, and disposal cell operations. He supervised a staff of up to 20 ES&H technicians during various phases of the project.

Mr. Fleming's WSSRAP experience in technical report writing includes:

- Development and revision of the WSSRAP Radiological Control Manual
- Development of the WSSRAP Radiation Protection Program required by 10 CFR 835
- The WSSRAP Internal Dosimetry Technical Basis Manual
- The Weldon Spring Quarry Radon Emissions Modeling Report
- The Weldon Spring Chemical Plant Buildings Radiological Characterization Report
- Soil contamination sections of both the Weldon Spring Site and the Weldon Spring Quarry Remedial Investigation reports
- Technical review of subcontract documents, NEPA/CERCLA related documents, health and safety plans, characterization reports, and standard operating procedures.

His operational health physics experience at Weldon Spring includes:

- Former Field Operations Group Manager. Managed a staff of 20 environmental safety and health technicians providing field support of remediation activities
- Preparation of radioactive material shipments in accordance with DOT requirements
- Radiation protection health and safety support
- Radiological data acquisition for soils, buildings, and equipment characterization, as well as environmental monitoring data acquisition
- Radiation Protection Training Instructor

- Currently providing technical guidance and support to ongoing remediation activities as required.

Mr. Fleming's experience also includes one year as Project Engineer at the DOE Argonne National Laboratory in Argonne, Illinois, where he was responsible for field oversight of a radioactive decontamination and decommissioning project. This work involved removal and disposal of radioactively contaminated water, mixed waste sediment, activated metal objects, and radioactively contaminated cast iron pipes and concrete.

Mr. Fleming's duties included developing and implementing sampling plans for characterization of the various waste streams, reviewing work plans, daily field oversight of subcontractor work activities, coordination with other Argonne National Laboratory departments to accomplish support tasks for the project, and ensuring compliance with work procedures and health and safety plan requirements.

Employment History:

Jacobs Engineering Group Inc. Health Physicist	1994 to date
Espo Engineering (Subcontractor to Argonne National Laboratory) Project Engineer	1993 - 1994
Jacobs Engineering Group Inc. Health Physicist	1988 - 1993

Appendix II

Forms

Jacobs Engineering Group, Inc.
13723 Riverport Drive
Maryland Heights, MO 63043
Tel. (314) 436-7600 Fax (314) 770-5110

AIR SAMPLE LOG

Project Name:	Date Sample___ Date Analyzed___	LEVEL OF RESPIRATORY PROTECTION
Address:	Comments:	1/2 Mask APR
City/State:		Full Face APR
Sampled by:		PAPR
Method:		SAR (Demand or Continuous Mode)
	Cassette Γ 25mm Γ 37 mm	SAR (Pressure Demand Mode)

Sample Number	Location	Type*	Time On	Time Off	Total Mins.	Flow Rate/LPM			Volume Liters	Detection Limit	Fibers/ Fields	Fibers/ mm^2	Fibers CC	LCL	UCL	8 Hr. TWA	30 Min. Excursion
						Begin	End	Avg.									

*BACK : Background AREA: Area PERS: Personal BAR: Barrier LO : Load Out DECON: Decontamination Unit
IWA: Inside Work Area OWA: Outside Work Area

Jacobs
13723 Riverport Drive
Maryland Heights, MO 63043
Tel. (314) 436-7600 Fax (314) 770-5110

CHAIN OF CUSTODY

PROJECT:

LOCATION:

COLLECTOR:

SAMPLE NUMBER	DESCRIPTION	SAMPLE TYPE	DATE	ASBESTOS	LEAD									REMARKS

RELINQUISHED BY:	DATE	TIME	RECEIVED BY:	DATE	TIME

RELINQUISHED BY:	DATE	TIME	NOTES:

RECEIVED FOR LABORATORY BY:	DATE	TIME	

QUALITY ASSURANCE PROJECT PLAN
FOR THE
ASBESTOS ABATEMENT OF BUILDING 401
NIAGARA FALLS STORAGE SITE
LEWISTON, NEW YORK

PREPARED FOR:

DEPARTMENT OF THE ARMY

CORPS OF ENGINEERS, BUFFALO DISTRICT
BUFFALO, NEW YORK
CONTRACT DACW49-00-D-0007

Prepared by:

Jacobs Engineering Group, Inc. - Federal Operations
13723 Riverport Drive
Maryland Heights, MO. 63043

October 2001

TABLE OF CONTENTS
QUALITY ASSURANCE PROJECT PLAN (QAPP)

Figures

Tables

Appendices

1.0 PROJECT LABORATORY ORGANIZATION AND RESPONSIBILITIES

1.1 Asbestos Laboratory

EMSL Analytical, Inc. (EMSL) will provide the primary analytical laboratory services for the asbestos assessment and abatement monitoring. EMSL is a full-service environmental analytical laboratory. The firm has fully equipped laboratories with the latest instrumentation and an advanced data management system. USACE validation in accordance with EM-200-1-1 is not applicable to asbestos analysis, however EMSL is accredited by the National Voluntary Laboratory Accreditation Program (NVLAP) for analysis of asbestos samples by Transmission Electron Microscopy (TEM) and Polarized Light Microscopy (PLM), by the American Industrial Hygiene Association (AIHA) for Phase Contrast Microscopy (PCM), and by the State of New York for asbestos analysis, refer to Appendix IX for copies of EMSL's certificates.

Acceptance of this plan shall constitute project specific approval by the USACE to utilize this facility in the capacity as specified herein.

EMSL's key laboratory personnel are discussed in section 2 of EMSL's Quality Assurance Plan (QAP), refer to Appendix I. The point of contact is John VanVoorhees. The laboratory address is below:

EMSL Analytical, Inc.
107 Haddon Avenue
Westmont, NJ 08108
Office: (800) 220-3675 Fax: (856) 858-7141

1.2 Radiological Laboratory

Severn Trent Laboratories, Inc. (STL) will provide the analytical laboratory services for the radiological water analysis. STL is a full-service environmental analytical laboratory that has been evaluated by the USACE Hazardous, Toxic and Radioactive Waste Mandatory Center of Expertise (USACE-HTRW-MCX) and is approved as a Hazardous Waste Testing Laboratory in support of their environmental investigations. The firm has fully equipped laboratories with the latest instrumentation and an advanced data management system.

Acceptance of this plan shall constitute project specific approval by the USACE to utilize this facility in the capacity as specified herein.

STL's key laboratory personnel are discussed in section 4.1 of STL's QAP, refer to Appendix IV. The point of contact is Richard H. Mannz. The laboratory address is below:

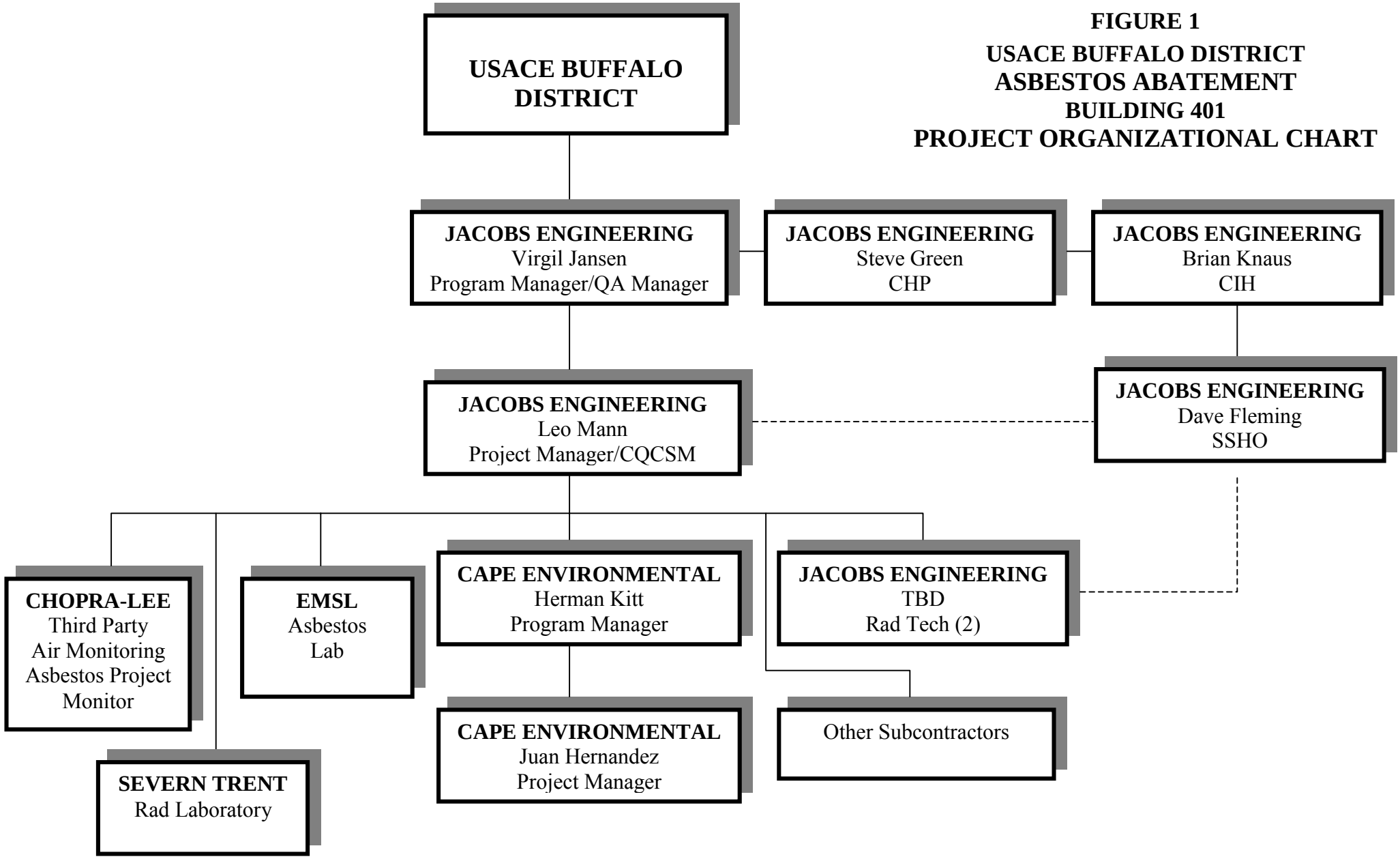
STL St. Louis
13715 Rider Trail North
Earth City, MO 63045
Office: (314) 298-8566 Fax: (314) 298-8757

1.3 Organizational Chart

Figure 1 is the overall organizational chart for the project. EMSL's laboratory organizational chart is presented in section 2 of EMSL's QAP, refer to Appendix I. STL's laboratory organizational chart is presented in section 4.1 of STL's QAP, refer to Appendix IV

FIGURE 1

USACE BUFFALO DISTRICT
ASBESTOS ABATEMENT
BUILDING 401
PROJECT ORGANIZATIONAL CHART



2.0 DATA ASSESSMENT ORGANIZATION AND RESPONSIBILITIES

Data assessment will be performed by the laboratories and by by Jacobs. Jacobs independent assessment activities include procedures for asbestos samples and radiation chemistry samples. The following describes the roles and responsibilities of project personnel that will be involved with data assessment.

2.1 LABORATORY DATA ASSESSMENT

Each laboratory is responsible for it's own internal data assessment. This will include at a minimum a review analyst and by a supervisor or data review specialist. Refer to section 7.2 for specifics concerning laboratory assessment.

2.2 JACOBS DATA ASSESSMENT

2.2.1 Jacobs Project Manager

The Jacobs Project Manager (PM) will be responsible for assuring overall data assessment is carried out. The PM will also specifically be responsible for the review and validation of asbestos laboratory results as they apply to the asbestos assessment portion of the project. Refer to section 7.3 for items that will be reviewed during the data assessment.

Leo Mann III will serve as the Project Manager.

2.2.2 QA Manager

The Jacobs QA Manager is responsible for assuring that the Jacobs QA program is implemented in all project activities including:

- QA protocols and procedures,
- audits to see that data assessment is properly performed
- documentation of quality objectives

Virgil Jansen will serve as the Project QA Manager.

2.2.3 Asbestos Data Assessment

2.2.3.1 Site Manager

In addition to the PM the Site Manager will be responsible for review and validation of asbestos laboratory results that will be developed during the abatement portion of the project. Refer to section 7.3 for items that will be reviewed during the data assessment.

Leo Mann III will serve as the Site Manager.

2.2.4 Radiation Chemistry

2.2.4.1 Site Safety Health Officer

The Jacobs Site Safety and Health Officer is responsible for the overall implementation of the Safety and Health plan during onsite activities. Due to the SSHO qualifications and the nature of radiation the SSHO is also responsible for data assessment specific to radiation. Refer to section 7.3 for items that will be reviewed during the data assessment.

David Fleming will serve as the SSHO.

3.0 DATA QUALITY OBJECTIVES

Data Quality Objectives (DQO) are defined as an integrated set of thought processes which define data quality requirements based on the intended use of the data. DQOs are necessary in obtaining sufficient data of known quality, both technically and legally defensible, for the intended data use.

The QAPP employs a systematic program of Quality Control procedures and checks designed to support and document the attainment of established DQOs. Since field sampling procedures, sample handling procedures and laboratory testing procedures are all potential sources of error for data, the QA program contains QC checks intended to monitor these aspects of data collection. The project DQOs are expressed as a series of requirements for the sampling procedures, sample handling procedures, analytical procedures, and analytical sensitivity; as well as precision, accuracy, representativeness, completeness, and comparability (PARCC) parameter goals for project QC check results.

3.1 BACKGROUND

3.1.1 Project Objectives

The objectives of this project are to collect sufficient information to determine if suspect asbestos containing materials (ACM) contain asbestos, to determine if the materials contain radioactive contamination, to remove and dispose of non-radioactive ACM, and to remove and set aside for disposal from other radioactively contaminated ACM. Specific project decision statements are as follows:

(1) determine ACM and radioactive contamination areas

(2) remove and dispose of non-radioactive ACM in accordance with EPA and OSHA

(3) remove and set aside for disposal by others radioactive ACM in accordance with EPA, OSHA, and NRC

The level of quality required of the collected data to be used for these intended purposes is such that it meets 12 NYCCR Code Rule 56 “Asbestos”. The method-specific DQOs for precision, accuracy, and sensitivity have been established for each measurement parameter based on prior knowledge of the specific measurement system used and method validation studies employing standards, blanks, instrument calibrations, control charts, and project specific requirements.

3.2 MEASUREMENT QUALITY OBJECTIVES FOR DATA MEASUREMENT

To meet the required data needs of the project, sampling will be conducted following EPA guidelines as set forth in 40 CFR 763 subpart E for asbestos and all suspect ACM surfaces will be scanned for radioactive material to determine if the levels exceed NRC Reg Guide 1.86 surface contamination guidelines for Ra-226 and Th-232. Specific sampling locations,

rationales, and number of samples of will be determined in the field. The minimum amount of asbestos samples for each matrix required to meet project DQOs are discussed in detail in Section 4 of the FSP. Table 3-1 provides a summary of the number of field samples required for each matrix and the required analytical testing.

TABLE 3-1 FIELD SAMPLES

Sample Type	Method	Estimated Samples	Comments
Bulk ACM	12 NYCRR Code Rule 56	98	
Radiological Survey	NRC Reg Guide 1.86 Surface Contamination Guideline for Ra-226 and Th-232		Continuous screening during assessment and abatement. Based on MARSSIM Table 5.3*
Personal Air – ACM	NIOSH 7400	330	
Area Air – ACM	NIOSH 7400	990	
Clearance Air ACM	AHERA 40 CFR 763 Subpart E	50	
Personal Air – Rad	Radiation Control Contingency Plan Appendix 5	425	
Area Air – Rad	Radiation Control Contingency Plan Appendix 5	132	
Waste Screening – Rad	Radiation Control Contingency Plan Appendix 3		Continuous screening during waste loadout of all containers.
Waste Water Rad	Gross Alpha per EPA 600/4-80-032	25	

*: Refer to Section 3.1.2.3 of the Sampling and Analysis Plan (SAP) for further details

Sampling procedures are presented in Section 4 of the FSP and sample handling procedures are presented in Section 5 and 6 of the FSP. Analytical procedures and sensitivity requirements are presented in Section 6 of this QAPP. Goals for analytical parameters are discussed in the following paragraphs.

3.2.1 Precision

Precision is a measure of the degree of reproducibility of an analytical value. Precision may be affected by the natural variation of the matrix or contamination within that matrix, as well as by errors made in field and/or laboratory handling procedures. For chemical parameters which do not allow homogenization prior to sample acquisition (e.g. asbestos samples), precision values must be viewed accordingly. Precision objectives for this project are presented on Table 3-2 for each measurement parameter.

TABLE 3-2 - PRECISION

Parameter	Precision	Frequency
Bulk ACM – EPA Bulk	Not Determined	N/A
Air Samples ACM - NIOSH 7400	45% of Relative Standard Deviation	10% Recount
Air Clearance Samples – AHERA Method	Not defined according to method.	N/A
Fixed Point Measurements for total alpha and beta surface contamination	±30% for measurements greater than 5 times detection limits	1 in 20
Rad Air Samples – Personal & Area (environmental)	±30% for measurements greater than 5 times detection limits	1 in 20
Waste Water – Gross Alpha per EPA 600/4-80-032	±40% for measurements greater than 5 times detection limits	1 in 20

3.2.2 Accuracy

Accuracy is a measure of bias in a measurement system (i.e. how closely an analytical result agrees with the "true" or actual value). Potential sources of error are the sampling process, field contamination, preservation, handling, sample matrix sample preparation and analysis techniques. Accuracy objectives are presented on Table 3-3 for each measurement parameter.

TABLE 3-3 ACCURACY

Parameter	Accuracy
Bulk ACM – EPA Bulk	Not Determined [1]
Air Samples ACM - NIOSH 7400	Evaluation of Method [1]
Air Clearance Samples – AHERA Method	Evaluation of Method [1]
Daily source checks on instruments for monitoring total & removable alpha and beta surface contamination –per Radiation Control Contingency Plan Appendix 4	±20% of the known check source activity
Daily source checks on instruments for monitoring long-lived gross alpha activity on air samples – per Radiation Control Contingency Plan Appendix 4	±20% of the known check source activity
Waste Water – Gross Alpha	±20% of the known spike gross alpha activity

1 – Accuracy for asbestos bulk and air samples is confirmed through the samplers following specific protocol in sampling, the laboratory handling of samples, calibration of equipment, training, and proper calculation. The methods do not define a specific accuracy criteria but refer you to follow the protocol to minimize error in the sampling and analysis.

3.2.3 Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represent actual site conditions. Representativeness is a qualitative parameter most concerned with the proper design of the sampling program or subsampling of a given sample. The representativeness criterion is satisfied by employing appropriate sampling strategies and techniques. The representativeness of the data will be evaluated by:

- comparing actual sampling procedures and chain of custody forms to those described in the work plan,
- identifying and qualifying nonrepresentative data in site characterization activities,
- examining blanks for cross contamination.

The objective of this work plan is to generate representative data. This shall be accomplished by utilizing trained personnel and employing standardized and approved sampling and analytical procedures. These procedures shall be explicitly followed, with any exceptions thoroughly documented.

3.2.4 Comparability

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared to another. For this project, these data sets may include data generated by laboratories in previous investigative phases or data generated by the same laboratory over a period of several years. The comparability objective of this project is to generate data comparable with other measurement data for similar samples and sample conditions. This goal will be accomplished by using standard techniques to collect and analyze samples, following these methods and procedures explicitly, documenting any exceptions, and reporting results in appropriate units. Any planned deviation from procedures will be approved in advance and well documented.

Comparability is assessed by evaluating the procedures for collecting the samples against the known sampling strategies and laboratory procedures.

3.2.5 Completeness

Completeness is defined as the percentage of measurements made which are judged to be valid compared to the total number of measurements planned. A value of 90% or higher is the goal. For values less than 90%, problems in the sampling or analytical procedures will be examined and possible solutions explored.

3.2.6 Sensitivity

Sensitivity is a general term which refers to the calibration sensitivity and the analytical sensitivity of a piece of equipment or analytical method. The calibration sensitivity is the slope of the calibration curve evaluated in the concentration range of interest. The analytical sensitivity is the ratio of the calibration sensitivity to the standard deviation of the analytical signal at a given analyte concentration.

3.2.6.1 Method Detection Limits

The method detection limit (MDL), which is based on the sensitivity of the analysis, is the smallest reported concentration in a sample within a specified level of confidence. MDL's for asbestos based samples are determined by each method and parameter.

Radiological MDLs for field instrumentation (i.e. alpha and beta gas proportional and scintillation detectors) are known as Critical Detection Levels (CDL). When applied to airborne radioactivity air sampling concentrations, they are known as Critical Level Concentrations (CLC). They are determined for each instrument using the formulas below.

The MDL for gross alpha in water analysis is 1 pCi. The required detectable concentration for release of waste water to sanitary sewers is 3E-7 uCi/ml (i.e. the maximum release concentration for Th-232 in Table 3 of Appendix B to 10 CFR 20). To be able to detect this, the sample aliquot analyzed by the laboratory must be at least 4 ml.

EPA Bulk Analysis

MDL = <1% asbestos containing

NIOSH 7400 MDL

MDL = 2.695/Volume

V – volume of sample collected.

AHERA TEM

MDL = Structures/Area Analyzed

Critical Levels (CL) are calculated as follows:

The instrument CDL is calculated:

$$CDL(dpm) = 1.645 \frac{\sqrt{\frac{bcr}{bct} + \frac{bcr}{sct}}}{de} \text{ x } (100 / da)$$

where bcr = background count rate
bct = background count time
sct = sample count time
de = detector efficiency
da = physical detector area in cm^2

The critical level concentration (CLC) is calculated using the following equation:

$$CLC = \frac{1.645x\sqrt{\frac{DR}{SCt}+\frac{DR}{BCt}}}{DExVx2.22E + 06}$$

where:

CLC = critical level concentration (uCi/ml)
DR = detector background count rate (cpm)
SCt = sample count time (minutes)
BCt = background count time (minutes)
DE = detector efficiency (cpm/dpm)
V = volume (ml)

Laboratory specific MDLs for each project parameter are summarized in Table 3-4:

TABLE 3-4 MDL'S

Parameter	MDL
Bulk ACM – EPA Bulk	<1%
Air Samples ACM - NIOSH 7400	= 2.695/Volume
Air Clearance Samples – AHERA Method	14.3 s/mm^2
Field instrumentation CDL	See equation above
Airborne Radioactivity CLC	See equation above
Waste Water – Gross Alpha	1 pCi

3.2.6.2 Practical Quantitation Limits

Practical quantitation limits (PQL) represent the sum of all of the uncertainties in the analytical procedure plus a safety factor. For asbestos sampling the PQLs are equal to the MDL's except for NIOSH 7400 where the PQL is approximately 14 times the PQL. In general, radiological laboratory PQLs shall be set at levels 3-5 times the MDL. The laboratory shall be required to analyze the low calibration standard for each analysis at or near the project PQL. Laboratory specific PQLs for gross alpha activity in water samples is 5 pCi/L. There are no PQLs for radiological field instrumentation measurements, but for practical purposes a PQL of 5 times the detection limit will be used.

3.2.6.3 Rationale for Sensitivity Objectives

Reported chemical concentrations from project samples will be screened against the values listed by 12 NYCCR Code Rule 56 "Asbestos", and measured surface contamination levels will be compared to the NRC 1.86 limits for Ra-226 and Th-232 to determine if constituents are present at levels of concern. Asbestos is determined present in bulk samples if the values exceed 1% asbestos by volume. Asbestos clearance samples are determined clean if the sample results are less than 70 s/mm^2.

Asbestos samples will not be collected from areas found to exceed NRC Reg. Guide 1.86 surface contamination limits for Ra-226 and Th-232. These limits are further detailed in Section 3.1.2.2 of this Sampling and Analysis Plan. Waste water is determined to be releasable to sanitary sewers if the sample results indicate < 3E-7 uCi/ml.

3.2.6.4 Laboratory Reporting Requirements and Sensitivity

In an effort to meet sensitivity goals, the laboratory shall report non-detect values to the MDL (with any necessary corrections for moisture and sample dilution). Concentrations of constituents detected above the MDL but below the PQL will be flagged by the laboratory as estimated.

4.0 SAMPLE RECIEPT, HANDLING, CUSTODY, AND HOLDING TIMES

In order to preserve the quality and integrity of samples from time of collection until time of analysis, sample preparation, preservation, storage and shipment procedures have been established. Additionally, to accurately track all samples collected, a logical sample numbering system will be developed. Procedures and methods for accomplishing these tasks are described in Section 5 "Field Operations Documentation" of the FSP.

The appropriate type and number of sample containers, method of preservation, and analytical holding times are specified for each class of project parameters. These requirements are summarized below.

TABLE 4–1 SAMPLE REQUIREMENTS

Parameter	Method	Required Containers	Required Preservative	Maximum Holding Times (measured from sample collection)	
				To Extraction	To Analysis
Bulk Asbestos	Polarized Light Microscopy	Plastic ziploc bag	None	None	None
Asbestos Air Personal/Perimeter	NIOSH 7400	0.8 um 25 mm Cassette	None	None	None
Asbestos Air Clearance	AHERA	0.4 um 25 mm Cassette	None	None	None
Surface Contamination Monitoring	Radiation Control Contingency Plan Appendix 3	None	None	None	None
Airborne Radioactivity Sample Monitoring	Radiation Control Contingency Plan Appendix 5	Plastic ziploc bag	None	None	None
Waste Water Gross Alpha	EPA 600/4-80-032	500 ml plastic bottle	HNO_3: pH<2	180 days	180 days

5.0 ANALYTICAL PROCEDURES

5.1 LABORATORY METHODS

The level of analytical support required to meet project DQOs is such that the resulting data meets 12 NYCRR Code Rule 56 "Asbestos".

Analytical parameters for this project will be determined using the methods summarized on Table 5-1. All analytical methods will be followed explicitly as stated. No exceptions or method modifications are planned. If exceptions or modifications are found to occur following project execution, the procedures used will be well documented in the field logbook and the final closeout document along with the reason for the deviation. Equivalent methods will only be substituted for the listed methodology if prior approval by the USACE is given.

TABLE 5-1 ANALYTICAL METHODS

Parameter	Method
Bulk Asbestos	Polarized Light Microscopy
Asbestos Air Personal/Perimeter	NIOSH 7400
Asbestos Air Clearance	AHERA
Surface Contamination Monitoring	See Radiation Control Contingency Plan Appendix 3
Airborne Radioactivity Sample Monitoring	See Radiation Control Contingency Plan Appendix 5
Waste Water Gross Alpha	EPA 600/4-80-032

5.2 CALIBRATION PROCEDURES AND FREQUENCY

This section of the QAPP discusses the calibration procedures that will be used by the subcontracted laboratory. Issues addressed in this section include defining the number of calibration standards to be used, the calibration range, and the procedures used to establish and verify the calibration of the laboratory instrumentation.

5.2.1 Analytical Support Areas

5.2.1.1 Standard/Reagent Preparation

A critical element in the generation of quality data is the purity/quality and traceability of the standard solutions and reagents used in the analytical operations. Preparation and maintenance of standards and reagents will be performed per the specified methods. The subcontract laboratory shall continuously monitor the quality of reagents and standard solutions through a series of well-documented procedures. Primary reference standards and standard solutions used by the subcontract laboratory shall be obtained from the National Institute of Standards and Technology, a USEPA supplier, or other reliable commercial sources to ensure the highest purity possible. All standards and standard solutions will be catalogued to identify the supplier, lot number, purity/concentration, receipt/preparation date, preparer's name, method of preparation, expiration date, and all other pertinent information. Both stock and working standard solutions shall be validated before use. Validation procedures can range from a check for

chromatographic purity to verification of the concentration of the standard using a standard prepared at a different time or obtained from a different source. Stock and working standards shall be checked regularly for signs of deterioration, such as discoloration, formation of precipitates, or change in concentration. Care shall be exercised in the proper storage and handling of standard solutions, and all containers are labeled to identify the chemical(s), concentration, solvent, expiration date, initials of preparer, and date of preparation. Reagents are to be examined for purity by subjecting an aliquot or subsample to the analytical method in which it will be used. The subcontract laboratory shall not use standards or reagents in which the expiration dates are exceeded; and shall maintain complete documentation for all standards and reagents used.

5.2.1.2 Refrigerators/Freezers

All refrigerators and freezers shall be monitored for proper temperature by measuring and recording internal temperatures on a daily basis. Thermometers used for these measurements shall be calibrated annually at a minimum. Temperatures shall be recorded on appropriate log sheets. Appropriate acceptance ranges (2°-6° C for refrigerators) shall be clearly posted on each unit in service and corrective measures established if necessary.

5.2.1.3 Water Supply System

The subcontract laboratory shall maintain an appropriate water supply system that is capable of furnishing American Society for Testing and Materials (ASTM) Type II 'polished' water to the various analytical areas. The quality of the water shall be documented on a regular basis. An ion-exchange treatment is recommended for inorganic areas, and UV cartridges or carbon absorption treatments are recommended for organic purposes.

5.2.2 LABORATORY INSTRUMENTS

Calibrations of instruments will be performed to ensure that each analytical system is operating correctly and functioning at the proper sensitivity to meet established quantitation limits. Frequency of calibration, calibration verification; and the number and concentration of calibration standards are specified by the specific analytical method(s). Laboratory calibration procedures and frequency will be as stated in the specific analytical method and the laboratory QAP for each analysis. Table 5-2 identifies the reference for each analysis calibration procedure
.

TABLE 5-2 CALIBRATION REFERENCE

Method	Reference:
Polarized Light Microscopy	Appendix II PLM SOP Section 6.1
NIOSH 7400	Appendix II PCM SOP Section 3
AHERA	Appendix II TEM SOP Section 9.2
EPA 600/4-80-032	Appendix IV Gross Alpha SOP Section 10

5.3 LABORATORY QC PROCEDURES

This section of the QAPP identifies the specific internal QC methods used by the analytical laboratory. Type and frequency of QC samples performed by the laboratory are dependent upon the specified analytical method and the project DQOs. Internal QC methods require performance on a sample batch basis and include analyses of method blanks and actual environmental samples as duplicates. Additional information concerning laboratory internal QC checks is included in the respective laboratory QAP.

The overall QA objective of this project is to implement QC procedures during laboratory analysis and reporting that will provide data to the degree of quality consistent with their intended use. The sample set, chemical analysis results, and interpretations shall be based on data that meet or exceed the QA objectives established for the project.

Internal QC checks are used to determine if analytical operations are in control, as well as determining the effect sample matrix may have on data being generated. The type and frequency of specific QC samples performed by the subcontract laboratory shall be consistent with the specified analytical method and requirements outlined in this QAPP. Each method's minimum laboratory QC are specified within the referenced methods. Acceptance criteria and/or target ranges for QC samples are specified within the project DQOs and, at a minimum, are equivalent to those specified within the referenced method. Data which vary from these target ranges shall result in the implementation of appropriate corrective measures, potential application of qualifiers, and/or an assessment of the impact these corrective measures have on the usability of the data in the decision-making process. Corrective action requirements are discussed in Section 6 of this QAPP.

5.3.1 Blanks and Interlaboratory Samples

QC samples for asbestos will be analyzed according to EMSL SOP for Phase Contrast Microscopy (PCM) and in accordance with NIOSH Method 7400. Accordingly 2 or 10%, whichever is greater, blanks will be submitted to the laboratory for analysis. In addition to the blanks the laboratory will reanalyze 10% of the air samples submitted to compare the values as to whether or not they fall within the standard deviation parameters as indicated in Section 3.

Blanks and interlaboratory samples for gross alpha in water analysis will be in accordance with Appendix IV Gross Alpha SOP Section 10.

5.3.2 Other Laboratory QC Samples

Additional appropriate QC requirements are detailed within the specific analytical methods and the laboratory's QAP, and will be analyzed and reported if required.

6.0 NONCONFORMANCE/CORRECTIVE ACTIONS

This section of the QAPP addresses corrective actions that must be implemented if the laboratory QA specifications are not met. Corrective action procedures will be implemented if problems are observed with incoming samples, sample holding times, instrument calibration procedures, specified practical quantitation limits, or internal QC sample results. Corrective actions may include resampling, reanalyzing samples, or auditing laboratory procedures. Procedures for identifying and documenting corrective actions and for reporting and follow-up of corrective actions are also specified.

When errors, deficiencies, or out-of-control situations exist, the subcontract laboratory's QA plan provides corrective actions which shall be implemented to resolve problems and restore proper functioning to the analytical system. Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation procedure for possible errors, checks the instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter will be referred to the laboratory supervisor; manager, and/or QA department for further investigation. Once resolved, full documentation of the corrective action procedure shall be filed with the project records, and the information summarized within the case narrative. At a minimum, full documentation of all actions taken will be recorded within a case narrative, and transmission of this information to USACE will be with the laboratory data package. If necessary, USACE will receive immediate verbal notification to the USACE Contracting Officer or District Chemist for input on corrective action requirements, deviations to protocol taken on future samples, final decisions of data usability, etc.

6.1 INCOMING SAMPLES

Problems noted during sample receipt shall be documented on an appropriate form (Receipt Form) by the sample custodian. If necessary, USACE shall be contacted immediately for problem resolution. All corrective actions taken shall be thoroughly documented. Potential corrective actions include reporting data with qualifiers or resampling and reanalysis.

6.2 SAMPLE HOLDING TIMES

If samples cannot or were not extracted/digested and/or analyzed within the appropriate method required holding times, USACE shall be notified immediately for problem resolution. All corrective actions shall be thoroughly documented. Potential corrective actions include reporting data with qualifiers or resampling and reanalysis.

6.3 INSTRUMENT CALIBRATION

Sample analysis shall not be allowed until all initial calibrations meet the appropriate requirements. All calibrations must meet method requirements or corrective action must be performed in accordance with method requirements. All continuing calibrations that do not meet method requirements shall result in a review of the calibration, rerun of the appropriate

calibration standard(s), and if necessary, reanalysis of all samples affected back to the previous acceptable calibration check.

6.4 PRACTICAL QUANTITATION LIMITS

Appropriate sample cleanup procedures shall be employed to attempt to achieve the practical quantitation limits as stated in Section 3 of this QAPP. If difficulties arise in achieving these limits due to a particular sample matrix, the subcontract laboratory should notify the JEG project manager, who will in turn notify the USACE Contracting Officer and District chemist of this problem for resolution. Any dilutions made shall be documented in a case narrative along with the revised practical quantitation limits for those analytes directly affected.

6.5 METHOD QC

All method QC, including blanks, laboratory control samples, and other method-specified QC samples shall meet the requirements as specified within the analytical method and within the project QAPP. Failure of method-required QC shall result in the review of all affected data, which may result in the resampling and reanalysis of samples. USACE shall be notified as soon as possible to discuss possible corrective actions should unusually difficult sample matrices be encountered.

6.6 CALCULATION ERRORS

Reports shall be reissued if calculation and/or reporting errors are noted with any given data package. The case narrative shall clearly state the reason(s) for reissuance of a report.

7.0 DATA REDUCTION, VALIDATION, AND REPORTING

This section of the QAPP discusses the data review process that is required to assure the validity of the data. This process includes a combination of individual data reduction, review, validation, and reporting procedures which are discussed in the following paragraphs.

All analytical data generated by the subcontract laboratory shall be reviewed by the laboratory to assure the validity of the reported data. This internal data review process shall consist of data generation, a minimum of two levels of documented review, and reporting. In each stage, the review process shall be documented using an appropriate checklist form that is signed and dated by the reviewer and the completed forms maintained in the laboratory project files.

7.1 LABORATORY DATA REDUCTION PROCEDURES

Data reduction procedures are summarized within the laboratory QAP along with the persons responsible for this task. These procedures address any statistical approaches used for reducing data, and include applicable units and any term definitions.

In general, data will be reduced by an analyst in one of the following ways:

- Manual computation of results directly on the laboratory bench sheet or on calculation pages attached to the data sheets.

- Input of raw data for computer processing.

- Direct acquisition and processing of raw data by a computer.

If data are manually processed by an analyst, all steps in the computation are provided including the equations used and the source of input parameters such as response factors, dilution factors, and calibration constants. If calculations are not performed directly on the data sheet, calculations are done on standard calculation paper and attached to the data sheets.

If data are input and processed using a computer, a copy of the input is kept and uniquely identified with the project number and other information as needed. The samples analyzed shall be evident and the input signed and dated by the analyst.

If data are directly acquired from instrumentation and processed, the analyst verifies that the following are correct: project and sample numbers, calibration constants and response factors, output parameters such as units, and numerical values used for detection limits (if a value is reported as less than). The analyst signs and dates the resulting output.

7.2 LABORATORY DATA REVIEW PROCEDURES

The laboratory's data review process is detailed within the respective laboratory QAP and is summarized in this section. The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of that data. Each step of the review process involves evaluation of data quality based on both the results of the QC data and the professional judgement of those conducting the review. This application of technical knowledge and experience to the evaluation of data is essential in ensuring that data of quality are generated consistently. All data generated and reduced shall follow well-documented in-house protocols, including two levels of technical review:

(1) Level 1 technical data review, performed by the analyst;

(2) Level 2 technical review, performed by a supervisor or data review specialist; and

Laboratory review of analytical data shall be consistent with analytical methods and applicable laboratory standard operating procedures. One hundred percent of laboratory generated data will be subjected to internal data review. If problems are identified during analysis, method modifications such as additional cleanup steps, sample volume changes, and analytical procedure revisions will be attempted and documented. If method modifications do not remedy the problem, alternative procedures will be proposed. The laboratory will assign qualifiers to the data to indicate impacts to data use. At a minimum, the following information will be evaluated by the laboratory, as applicable:

- Calibration (initial and continuing) and tuning check results.
- Analyte identification and quantification are correct.
- QC samples and method blanks are within control limits.
- Data summaries and reports for transcription and typographical errors.
- Holding times, sample preservation, and sample storage criteria have been met.
- Sample chain of custody documentation for completeness, accuracy, and to ensure sample integrity has been maintained.
- Sample preparation information for completeness and accuracy.
- Documentation (including the case narrative) is complete and correct.

Treatment of Outliers and Non-Conforming Data

Corrective action measures will be taken to resolve problems and restore proper function to any analytical system generating data which indicate that the system is not performing adequately. Corrective measures may be necessary when the following occurs:

- QC data are not within control for precision and accuracy.
- Blanks are found with contaminants above acceptable levels.
- Calibration data or instrument performance parameters are not within acceptance criteria.
- Undesirable trends are observed in QC data or calibration data.
- There are sudden changes in instrument sensitivity or performance.

- Deficiencies are identified during audits or from the results of Performance Evaluation samples.

Initiation of corrective action resulting from the evaluation of QC results will be the responsibility of the laboratory QA manager in consultation with the Project Manager. Corrective action may include, but is not limited to the following:

- Reanalysis of the samples.
- Documentation of interferences or matrix effects that result in poor analytical performance.
- Evaluating and changing sampling or analytical procedures.
- Resampling and reanalysis, if the completeness or usability of the data set does not meet the criteria for acceptability.

7.3 INDEPENDENT DATA VALIDATION

A review of the data quality will be performed by JEG personnel independent of the laboratory generating the data. Data evaluations will also be based on the QA/QC requirements of the referenced analytical procedures, QC objectives presented in this QAPP, and professional judgement of the evaluator.

At a minimum, specific data evaluations shall include evaluation of:

- contractual compliance
- QC samples and method blanks are within control limits.
- Data summaries and reports for transcription and typographical errors.
- Holding times, sample preservation, and sample storage criteria have been met.
- Sample chain of custody documentation for completeness, accuracy, and to ensure sample integrity has been maintained.
- Documentation (including the case narrative) is complete and correct.
- constituent reporting limits,
- review of data based on historical parameters
- JEG supplied information matching laboratory inputted data
- Comparability is assessed by evaluating the procedures for collecting the samples against the known sampling strategies and laboratory procedures

The data quality review shall include evaluation of 100% of these data. If this review reveals trends of data quality deficiencies or systematic laboratory problems, appropriate additional QC data will be obtained from the laboratory for review.

7.4 DATA REPORTING

The required data reporting format for use with this project is a data package that will be submitted to the USACE to determine ACM and radioactive levels in Building 401.

The following data is will be supplied from the subcontracted laboratory:

- Sample identification numbers cross-referenced with laboratory identification numbers.
- All QC batch numbers designating QC samples to associated field samples.
- Problems with any arriving samples noted on receipt forms and summarized in a case narrative.
- Problems with any chemical analyses summarized in a case narrative.
- Each analyte reported as an actual value or less than the specified MDL.
- Dilution factors, collection dates, extraction dates, and analysis dates.
- All original sample chain of custody forms.
- All analytical instrument calibration and other laboratory raw data shall be available upon request.

7.5 LABORATORY TURNAROUND TIME

Turnaround time shall be as necessary for the completion of the project and as specified in the Asbestos Assessment and Abatement Plan. Refer to Appendix III and VII for letters from the laboratories indicating they are capable of handling the sample loads.

APPENDIX I
EMSL's QUALITY ASSURANCE PLAN

EMSL

ANALYTICAL, INC.

Outline of the
LABORATORY
QUALITY ASSURANCE
PROGRAM
For:
PHASE CONTRAST MICROSCOPY
TRANSMISSION ELECTRON MICROSCOPY
POLARIZED LIGHT MICROSCOPY

Table of Contents

The Quality program at EMSL is built on a commitment to quality and continued improvement. This program is a primary part of our every day work : developed, utilized, and maintained by all the dedicated staff at EMSL.

1.0 Introduction:

This Program Outline provides a comprehensive overview of the Quality Assurance Program. It provides the reader with a summary of the Laboratory policies and procedures as they relate to the technical aspects of Corporate Quality objectives.

This program follows quality guidelines as documented by the American Industrial Hygiene Association (AIHA), the EPA's National Voluntary Laboratory Approval Program (NVLAP) and other applicable state and federal regulatory agencies.

This QA program is designed to ensure that the highest level of quality professional services and technical excellence is provided to our clients. This is accomplished by the implementation of program policies including:

- Development of company standard quality control programs
- Standardization of reporting formats
- Review of regional laboratory QC performance
- Providing technical training for all staff levels
- Achieving traceability of data
- Performance of quality audits
- Participation in applicable Accreditation Programs
- Participation in applicable third party proficiency testing programs

The objectives of these program polices ensure the quality, accuracy and integrity of our analytical data.

The Quality Assurance objectives, policies and procedures are formally documented in the Quality Assurance Manual – EMSLQAASB100.4. An outline and summary of this manual is presented on the following pages.

2.0 General and Administrative

2.1 Scope

The Objectives of this Manual are to ensure the following:

- Quality and accuracy of analytical results.
- Conformance with all analytical methodologies
- Conformance with Corporate mandated QA/QC requirements.
- Delivery of the highest quality of professional services and technical excellence to our clients.
- Ensure data integrity

To achieve these goals, this Manual directs implementation of the Quality Assurance program and describes responsibilities and duties of all personnel, and addresses all aspects of Quality Assurance for phase contrast microscopy (PCM), transmission electron microscopy (TEM) and polarized light microscopy (PLM) laboratory operations.

This Manual is to be kept accessible to all employees, and all employees are responsible for being familiar with, and adhering to its contents. Each employee is to sign the signature page acknowledging an understanding of the contents of this document. A copy of this signature page is submitted to the QA Department .

This Quality Assurance Program will be reviewed at least annually by the QA Manager. It will also be reviewed any time a problem arises that indicates a possible program flaw. In such an instance, the QA Manager will discuss the problem with Regional and Laboratory Management, Quality Control Supervisor and Analysts to ensure needed input from all levels within the Laboratory.

2.2 Implementation of Goals and Objectives

The program is designed to plan and institute Company policies and quality objectives throughout the branch laboratories. It is intended to provide support and issue policies including:

- Development of company standard quality control programs
- Issue standard reporting formats
- Review regional laboratory QC performance
- Provide training
- Perform periodic quality audits
- Oversee accreditation programs
- Provide regional coherency
- Control and maintain round robin programs

This program is also designed to provide a method, which achieves traceability of data to national standards. This is accomplished by setting requirements, which include:

- Use of Standard Reference Materials as certified and traceable to the National Institute of Standards and Technology for calibration:
- Laboratory participation in proficiency testing programs as available

from applicable governing agencies

The program is managed and maintained by the Corporate Quality Assurance Manager as described in section 2.0 Organization and Responsibility.

It is our intention to ensure that all goals and objectives of our Quality Program are met and maintained. Quality policies and procedures are integrated into our daily work, and are constantly reviewed by Regional and Laboratory Management and by the Quality Assurance (QA) Manager. To ensure integrity of sample results, these policies include:

- Clear job descriptions delineating responsibilities of each employee involved at all steps of laboratory procedures, data analysis and report generation.
- Completion of Quality Control (QC) samples.
- Proper documentation of analytical data.
- Good laboratory technique that ensures a contamination-free environment.
- Use of appropriate analytical technology including review of current literature to capture recent applicable developments.
- Review of reports to clients.
- Understanding and compliance with procedures which insure Client confidentiality

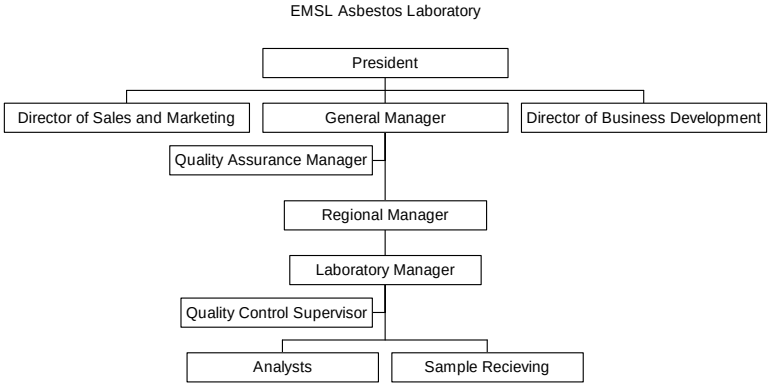
Any departure from the procedures and policies as stated in this document must under go a comprehensive review by the Quality Assurance Manager and Corporate Management prior to approval and effect. This will include-at a minimum:

- Reason for deviation from method
- Applicability of alternative method
- Availability of needed Resources (if applicable)
- Assurance that data is reported with appropriate references and disclaimers (if applicable)
- Record of alternative procedure or policy is maintained as part of the corporate files.

New Technical Service

Prior to the implementation of any new technical service, a comprehensive review is performed by Corporate Management. This review includes market applicability and availability of resources. The General Manager, Director of Business Development, or the Company President must grant formal approval

2.3 Organization and Responsibility



2.4 Ethics

One of the objectives of the Quality Assurance Program is to insure the staff of EMSL is provided training in the aspects of ethics as they pertain to corporate policy. The goals of this training program are:

- For each staff member to understand the responsibility to provide true and accurate information
- The understanding of the consequences of questionable conduct
- Provide direction to employees regarding ethics issues
- Provide support to employees regarding ethics issues
- Define right and wrong (as it is job related)
- The understanding of the impact of our actions

Training will be provided in the form of workshops, required readings and Corporate issued news letters. The Quality Assurance Department is responsible for insuring that this training is provided to the staff and that records are maintained documenting such training.

2.5 Standard Operating Procedures

Technically specific Operating Procedures are documented in the SOP manuals, located at each laboratory facility. These SOPs include step by step procedures for the preparation, analysis, and reporting of data. These documents are controlled by the QA Department and include:

EMSL.XXTEMSOP.200.x – Standard Operating Procedures for Transmission Electron Microscopy
EMSL.XXPLMSOP.200.x – Standard Operating Procedures for Polarized Light Microscopy
EMSL.XXPCMSOP.200.x - Standard Operating Procedures for Phase Contrast Microscopy
EMSL.QCPRGMSOP.200.x – Standard Operating Procedures for the Quality Control Program
EMSL.QAAUDITSOP.200.x – Standard Operating Procedures / Quality Assurance Audits

These SOPs cover methodology for analytical procedures, calibrations, contamination checks Quality Control frequency, procedures, and internal audit policies

Each analytical SOP (TEM, PLM, and PCM) is edited specifically for the laboratory operation. The Laboratory Manager is responsible for insuring the SOP's reflect the

actual laboratory procedures and are reviewed and updated annually.

2.6 Sample Tracking

Rigorous sample tracking is fundamental to a QA Program. The most thorough and complete analysis is useless if performed on the wrong sample.

Our sample-tracking program is designed, to the extent that it is possible, to meet all litigation requirements. It is also designed to have redundancy safeguards wherever possible.

In order to ensure the integrity of any sample, records of its custody must be maintained throughout the sample collection in the field, acceptance by the laboratory, and analysis.

A sample will not meet litigation requirements without a chain of custody that begins at the sample collection point. Since the client collects samples for analysis, the laboratory cannot be responsible for issuing a chain of custody at the time of sampling. However, the laboratory will advise all clients regarding sampling requirements (sampling materials, recommended sampling volumes, packaging, instructions for shipping, etc.) and chain-of-custody, and recommend that they use our form if they do not have their own.

The chain-of custody form will include:

- Analysis Required
- Date of Sampling
- Location of Sampling (if supplied)
- Sample volume (if supplied)
- Unique sample ID for each sample submitted
- Date submitted to laboratory
- Record of Custody

Prior to accepting samples, the Sample Receiving Coordinator inspects them to determine if they conform to laboratory acceptance criteria. If they do not, or if the clerk has any question as to the validity of the sample, the Laboratory Manager or an analyst trained to analyze such samples will determine whether the damage to integrity is sufficient to cause rejection. Rejections of samples are to be followed up by immediate notification of the client with an explanation and return of the questionable sample, if required by the client.

Samples are judged unacceptable under the following circumstances:

- Analysis requested outside laboratory's scope of accreditation
- Analysis requested outside laboratory capability (such as lack of equipment or staffing resources).
- Obviously damaged or compromised samples, i.e. opened air cassettes, cassettes with torn or ripped filters, water samples in leaking or faulty containers.
- Improper labeling
- Improper packaging
- Impossible deadlines
- Obvious faulty sampling technique
- Improper sample media

- Incompatible samples packaged together (i.e.- air samples with bulk samples)
- Inappropriate analytical methodology requested

Log in of samples is normally done by the Sample Receiving Coordinator, but may be done by any other employee familiar with the process. Information is entered for samples received into the Laboratory Information Management system (LIMS). LIMS is a computer laboratory management system which serves to track all samples from receipt through the analysis, reporting, and billing processes. Access to the LIMS system is restricted to approved personnel only. The Laboratory Manager is responsible for assigning computer rights to all applicable personnel and is accountable for ensuring that sound security measures are maintained.

The Sample Receiving Coordinator inspects the samples for integrity, verifies that all samples listed on the chain of custody are present, and logs them into the computer system. Any damages are noticed, and are reported to the laboratory manager.

All analyses must be carried out in accordance with the SOP(s) indicated. All SOPs used in this Laboratory will be found in the EMSL Laboratory Standard Operating Procedure Manuals.

2.7 Data Recording

Once analysis of a sample has been completed, the analyst signs the analytical worksheet and any other appropriate documentation. Chain of custodies and analytical worksheets are copied and placed in the laboratory master files. Originals are submitted to the Laboratory Manager for review and approval, before preparation of a client-ready report. All records are to be retained for a minimum of 7 years or as requested by the client.

Analytical data storage, processing, and reporting is facilitated through use of Laboratory Information Management System (LIMS) computer software. When samples are received by the laboratory personnel, sample information is entered into the LIMS system, which assigns the batch of samples a unique project identification number and generates analytical worksheets. The samples and worksheets are then forwarded to the analysts to be analyzed.

Once sample analysis has been completed, this result data is entered into the LIMS software. Analytical result data is entered either by approved data entry personnel, or by the analysts themselves. The LIMS software stores the analytical data, performs calculations where applicable, and generates the final report for the project. This final report is reviewed and approved before being forwarded to the appropriate client. (see also, Reporting Results)

2.8 Archival and Disposal of Samples

Once the analysis is complete and the analysis worksheet is signed, the analyst stores the sample in the appropriate storage box, as indicated in the SOP. All storage boxes are to be stored in a safe manner for the period indicated for that category of waste, in accordance with regulatory requirements. When a storage box is full, the month of which the samples where analyzed (or similar reference numbering system as appropriate for the operations, i.e. billing number) is marked on it. A new storage box replaces the old one which is then to be stored until time of disposal.

All bulk and air samples are held for a minimum of 3 months, unless a longer period is requested by the Client. All TEM grids are held for 3 years. Asbestos containing

samples are disposed of by a licensed contractor, and a copy of the waste manifest is obtained and kept on file. If requested, samples will be returned to the Client.

2.9 Quality of Materials

The high quality of materials used in this Laboratory shall be assured through specific purchasing and verification procedures and/or proper preparation techniques.

Selection of the appropriate grade of reagent(s) is designated in the reagent section of each analysis SOP and in addition may be specified by the Laboratory Manager in unusual circumstances. As a general practice, reagents will be of at least ACS reagent quality.

Reagents inclusive of SRM shall be purchased in accordance with the analytical needs of this Laboratory as determined by the Laboratory Manager. When received by the laboratory, these item's labels are dated and initialed with date received and expiration dates (if appropriate) as indicated /suggested by the manufacturer. Labels are also dated and initialed when opened and/or when reagent mixtures are prepared.

Verification will consist of confirming that the priority grade recorded on the reagent label conforms to the requirements of the SOP unless analysis difficulties indicate a possible problem or regulatory agency requirements specify otherwise. In the latter case, the appropriate analytical SOP will indicate the proper verification procedure.

2.10 Equipment/instrument maintenance

Maintenance schedules for equipment will be established by the Laboratory Manager. The Laboratory Manager shall also determine whether each microscope is maintained and repaired in-house or by an outside agency following EMSL administrative procedures. Servicing will also be performed when a need had been identified by calibration or other QC checks.

A maintenance file will be maintained for all equipment. In addition to a schedule of normal preventive maintenance, this file will contain a record of servicing.

2.11 Contamination Management

This Section describes reagent control, contamination management, and use of controlled procedures for this Laboratory. Proper observance of laboratory procedures is necessary to guarantee accuracy of results and the safety of Laboratory staff members.

Contamination both of samples and of the environment (including reagents used in analysis) must be avoided to provide the highest quality, legally defensible data to our clients. In order to achieve this goal, Laboratory staff must adhere to various preventative measures and use the testing procedures for contamination detection as established by the QA Manager.

If analysis of the blank samples indicate the possibility of contamination, the area and tools are cleaned and another slide prepared and analyzed. If the second slide shows contamination, applicable reagents are checked (acetone, triacetin, dispersion oils, etc.). A new box of slides is used to prepare a third slide. If analysis of the third slide shows contamination, a complete investigation is conducted to determine the contamination source.

If contamination is detected in any situation, the source of contamination must be traced and the problem resolved to prevent reoccurrence. All procedures taken to resolve a

contamination circumstance shall be documented properly and completely in the laboratory files.

2.12 Document preparation and control

In order to prepare and distribute documents in an organized fashion, procedures for initiation, preparation, review, approval and issuance of controlled copies will be followed. This program is a coordinated effort involving both technical review and custodial control. Analysts are to use only controlled, i.e., approved documents for all calibrations, analyses, final reports, and other activities performed in this laboratory.

2.13 Reporting results

Analytical data storage, processing, and reporting is facilitated through use of Laboratory Information Management System (LIMS) computer software. The Corporate MIS staff and the Laboratory Manager control the security of the LIMS software. Each LIMS user is assigned rights and privileges specific to the tasks, which are the responsibility of the user to perform. Access to the LIMS software is password protected on a user-by-user basis to ensure security. The MIS staff is responsible for insuring access to the LIMS is controlled, assignments are held secure and based on Laboratory Management approval.

All calculations and reporting performed by the LIMS software is implemented by the Corporate MIS staff, as instructed by EMSL's Quality Assurance (QA) department. No change is made to the LIMS software that affects how analytical data is stored, processed, or reported, without approval from the QA department. This coordination between the QA department and MIS department allows the LIMS software to be continually altered as necessary to comply with regulatory requirements. Compliance with regulatory agencies and/or accrediting bodies is implemented in this manner.

All final client reports are to be reviewed, approved, and signed by the Laboratory Manager prior to being sent to the client. They are also subject to review by the QA Manager.

Results are cleared for reporting by quality control data review and confirming analysis. Quality Control statistics shall be reviewed on a regular basis as determined by the QA Manager in accordance with regulatory agency requirements. Specific Quality Control procedures are detailed in the 'Performance Criteria' sections for each of the Method Modules found in this document. In general, 10% of analyses are reanalyzed using various QC procedures as appropriate for the methodologies. Samples are chosen randomly. The analyst records their reanalysis results on the data sheet in addition to notes whether any serious discrepancy exists. The Laboratory Manager periodically reviews the data sheets and the reanalysis data. If the difference between analyses is within control limits for QC analysis, the results will be cleared for reporting. As long as those statistics are deemed acceptable, client reports will continue to be processed.

If the difference between analyses exceeds control limits the Laboratory Manager and the analyst will review the sample data and resolve the differences. A detailed corrective action report recording all activity is submitted to the QA Manager. (See Procedures for Dealing with Deficiencies below)

In addition to QC review, analytical data is reported with confidence based on

compliance with this QA program. The traceability of the data reported is insured through the procedures and policies as documented in this manual, including:

- Delineation of Responsibility
- Compliance with Analytical Standard Operating Procedures
- Following Calibration Protocols
- Fulfillment of the Required Amount of Quality Control Analysis
- Satisfaction of Training Requirements

2.14 Records Retention

The following records shall be maintained for 3 years:

- Copy of Chain of Custody Documents
- Original Analytical Data Recording Worksheets
- All other records relating to the preparation of the client report

2.15 Procedures for dealing with deficiencies

Any complaint by a client will be treated as a non-conformance, and treated with the same corrective action follow-up as a discrepancy seen in following internal Quality Control procedures.

If a client makes a complaint about a test result, the sample in question will be reanalyzed by a second Analyst. If the second result agrees with the original the Laboratory Manager shall advise the client in writing that a quality control check has confirmed the original analysis.

In all cases where a deficiency is discovered, the QA Manager will initiate a corrective action review to determine the root cause of the problem and action to take to prevent reoccurrence. A report will be issued to the Laboratory Manager, who is responsible for the corrective action implementation.

The corrective action will consist of a review of all steps leading up to the non-conformance. This will include review of QC data, sample tracking, data transcription, instrument calibration, training documentation, and discussion with personnel.

Following the review, the QA Manager will prepare a report detailing the cause of the error and corrective action to take to prevent re-occurrence. The QA Manager will also follow up on the corrective action to ensure its implementation

2.16 Analytical Performance Criteria

Performance criteria will be determined three ways:

1) Results from intra-lab and round robin testing will be plotted to see if they fall within warning and action limits.

2) The administering agencies for proficiency testing will determine performance criteria.

3) Achievement of internal on-site Quality audits by the Regional or QA Manager. These audits will verify compliance with all QA and QC policies as documented in this manual. The Quality Audit process is detailed below.

Quality Control is performed continuously throughout the course of laboratory sample analysis regardless of laboratory productivity and is made part of the normal course of

laboratory sample analysis. Frequency and volume of QC analysis is based on regulatory requirements and Good Laboratory Practice. These requirements are listed for each analysis type in Appendix A of this manual.

These methods will be used according to the scope of the laboratories accreditation status and quality control requirements for each type of analysis. Performance criteria will be maintained for both individual analysts and for the entire laboratory. The standards for acceptance criteria are documented in the EMSL Quality Control Standard Operating Procedure Manual, *EMSLQCPGRMSOP.200.x.*

2.17 Quality Audits

Quality Audits will be performed for each laboratory location on an annual basis or more frequently as deemed necessary by Corporate, Regional or Laboratory Management. Audit procedures and policies are issued by the Quality Assurance Department and include:

- Review of compliance with the Quality system
- Compliance with Quality Control analysis
- Identification of any problem areas and suggestions for resolution

The Quality Assurance Department develops the guidelines and overall manner by which a Quality Audit is performed. These policies are detailed in the Standard Operating Procedure for Quality Audits, *QAASBAUDITSOP.200.0.*

2.18 Management Reviews

Management reviews are designed to provide the Corporate Management of EMSL with an overview of the performance of the QA activities of the laboratory operations. It addresses the Quality topics documented in ISO 17025 for each laboratory location and includes:

- quality control activities
- outcome of recent internal audits
- assessments by outside agencies
- corrective actions
- major changes in personnel, work load, service areas
- client dealings

2.19 Proficiency Testing Programs

Laboratories participating in proficiency testing programs will insure the analysis is performed using the same analytical methodology and staff as under normal, client sample conditions. At no time is there inter-laboratory exchange of samples.

Records of proficiency testing analysis are to be completed and maintained in a separate laboratory PT file. This data is also maintained for each participating analyst in his or her personal training file.

3.0 Analytical Quality Control Programs

The Quality Control program as established and managed by the QA Manager ensures that this Laboratory produces quality data. This process ensures, at a minimum, that our data is legally defensible and that all personnel perform their responsibilities properly . The Laboratory Manager will determine how QC testing is implemented operationally (e.g., after the analyses of every ten samples or at the end of each day, etc.) QC analysis is performed on a minimum of 10 % of sample volume. QC testing occurs on a regular basis and is not scheduled around the amount of workload.

In addition , the QA Manager will inspect the results of all QC testing on a regular basis and provide the necessary support and directives to the Laboratory Manager to ensure the QC program is properly executed.

3.1 Quality Control - General

Our laboratories internal QC program includes at a minimum, 10% quality control on all samples received for analysis. These are summarized below in each analytical section and include:

- Analysis of standard reference materials
- Intra analyst QC
- Inter analyst QC
- Analysis of blank samples
- Participation in inter laboratory programs
- Participation in proficiency testing programs

This QC data is graphed on control charts designed specifically for each analysis type. The description of these control charts are detailed in the Standard Operating Procedure EMSLQCPROGSOP.200.

The Laboratory Manager (or Managers designee, i.e. QC Supervisor) of each laboratory is responsible for implementing the day-to-day QC testing and ensuring the correct types of testing occurs at the appropriate frequencies. The Laboratory Manager is also responsible for ensuring complete records of QC testing are maintained.

3.2 Training - General

New analysts with no prior formal training must complete the EMSL training program in asbestos analysis in order to perform such analysis independently. The Lab Manager will draw on the candidate's previous training, if any. The candidate will receive sufficient in-house training to demonstrate proficiency and understanding in all related topics to the Lab Manager's satisfaction.

Practical Factors: When the candidate has received sufficient training to analyze samples, he/she will work in the laboratory along side an experienced analyst. The candidate will not sign any reports. All samples will be checked by an experienced senior analyst, who will officially report the results for review and signature, by the Laboratory Manager.

Proficiency Analysis: The candidate will be deemed proficient when quantitation within l laboratory norms, as established by the QC schedule are met.

Additionally, the trainee must perform analysis on past proficiency samples and succeed in generating data within the acceptable range as established by the agency(ies) statistical analysis. (see training SOP for additional detail)

Records are kept of the candidate's progress by the analyst-training log. When all areas are signed off, the candidate may perform independent analysis.

3.3 Phase Contrast Microscopy (PCM)

Method following: NIOSH 7400

The Quality Control program for phase contrast microscopy includes intra-analyst sample testing, participation in inter-lab programs, and statistical evaluations and calibrations.

Calibration

Calibration procedures must be followed prior to the analysis of air samples to insure that results of analysis reflect true and accurate data. The following summarizes the type and frequency of calibration for the analysis of fibers in air by PCM. Details of these procedures are found in the PCM SOP, *EMSL.XXPCMSOP.200.x.*

1) Microscope calibration
   - Phase Ring Alignment
   - Contamination control
   - HSE/NPL Test Slide
   - Measurement of Walton Beckette Graticule

2) Analysts calibration
   - Standard reference slide (past Proficiency test slide)

3) Operational Calibration
   - Air monitoring
   - Hood calibration

Details on the calibration procedures for PCM can be found in the Standard Operating Procedures Manual.

Quality Control Analysis

Each analysis performed in this Laboratory has its own Quality Control requirements that have been determined by the QA Manager and are delineated in that SOP. These requirements include but are not limited to the following:

Intra-Analyst
Reference
Proficiency Testing
Round Robin Testing
Laboratory Blank Analysis

The Laboratory Manager (or Managers designee, i.e. QC Supervisor) of each laboratory is responsible for implementing the day-to-day QC testing and ensuring the correct types of testing occurs at the appropriate frequencies. The Laboratory Manager is also responsible for ensuring complete records of QC testing are maintained.

3.4 Transmission Electron Microscopy (TEM)

Method following: AHERA - 40 CFR, Part 763, Subpart E, EPA Level I, II, III - EPA Contract # 68-02-3266, ASTM D 5755-95

The QA/QC program for the analysis of asbestos via TEM insures compliance with standard regulatory guidelines and follows Good Laboratory Practice (GLP). The program includes:

- Achievement of Verified Status
- Classification of Structures
- Calibrated Measurements at .5 micron
- Calibrations
  - -alignments
  - -magnification
  - -camera constant
  - -plasma asher
  - -detector resolution
  - -grid opening measurements
  - -analytical balance
  - -muffle furnace
- Fiber Id and Sizing
- SAED Indexing
- Ambient air monitoring

Quality Control Analysis

Each analysis performed in this Laboratory has its own Quality Control requirements that have been determined by the QA Manager and are delineated in that SOP. These requirements include but are not limited to the following:

Intra-Analyst
Intra-Analyst repreparation
Inter-Analyst
Reference Standards
Verified Analysis
Proficiency Testing
Round Robin Testing
Laboratory Blank Analysis

3.5 Polarized Light Microscopy
Method following: EPA-600/R-93/116, EPA-600/M4-82-020
Quality control procedures in the PLM laboratory follow guidelines as documented by the NVLAP accreditation program.

Calibration procedures must be followed prior to the analysis of samples to insure that results of analysis reflect true and accurate data. The following summarizes the type and frequency of calibration for the analysis of asbestos in bulk materials by PLM. Details on the performance of these functions are found in the PLM SOP.

1) Microscope calibration
   Center Stage or objective, & condenser
   Align polars
   Crosshair alignment fixed in polarizer's privileged direction

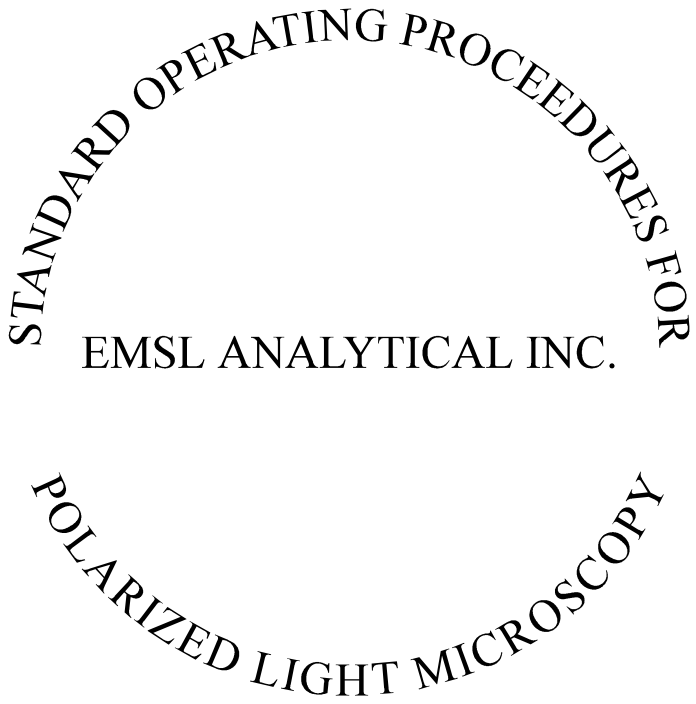
2) Analysts calibration
   Standard reference sample
   Contamination check with fiberglass sample
   Check Standard Amosite mount for proper dispersion colors, refractive index
3) Operational
   Calibrate Analytical Balance
   Air monitoring
   Refractive mounting oil calibration
   Calibrate muffle furnace temperature
   Hood calibration

Details on the calibration procedures for PLM can be found in the Standard Operating Procedures Manual.

Quality Control Analysis
Each analysis performed in this Laboratory has its own Quality Control requirements that have been determined by the QA Manager and are delineated in that SOP. These requirements include but are not limited to the following:

Intra-Lab Testing
Inter-Analyst
Intra-Analyst
Reference Standards
Proficiency Testing
Round Robin Testing
Laboratory Blank Analysis

APPENDIX II
EMSL's PLM, PCM, AND TEM STANDARD OPERATING PROCEDURES



Standard Operating Procedures
Asbestos Analysis
PLM Analysis of Bulk Samples

Revision Date: July, 2000
Original Date: June, 1995

Issue Date: August 1, 2000

EMSL Analytical Inc., Quality Assurance Dept.

EMSL ANALYTICAL

Standard Operating Procedures for the Analysis of Asbestos by Polarized Light Microscopy

TABLE OF CONTENTS

I. LOG-IN REPORTING AND SAMPLE HANDLING

SECTION

SECTION

II. DETERMINATION OF ASBESTOS IN BULK SAMPLES BY POLARIZED LIGHT MICROSCOPY (PLM) WITH DISPERSION STAINING

SECTION

SECTION

TABLES and FIGURES

APPENDICES

A. MICROSCOPE CARE AND MAINTENANCE

1.0 General Discussion
2.0 Equipment
3.0 Reagents
4.0 Background & Definitions
5.0 Method
6.0 Quality Control
7.0 References

B. ALIGNING THE OPTICAL SYSTEM – POLARIZED LIGHT MICROSCOPY (PLM)

1.0 General Discussion
2.0 Equipment
3.0 Background & Definitions
4.0 Method
5.0 Quality Control
6.0 References

C. CALIBRATION OF REFLECTIVE INDEX LIQUIDS

1.0 Overview
2.0 Materials & Equipment
3.0 Methodology
4.0 Procedure
5.0 Quality Control
6.0 Calculations
7.0 References

D. ASBESTOS AND NONASBESTOS MINERALS (DESCRIPTIVE INFORMATION)

SIGNATURE PAGE

I

LOG-IN, REPORTING AND SAMPLE HANDLING

1.0 INTRODUCTION

Standard Operating Procedures (SOPs) documented in this manual are intended for information and use of all laboratory personnel employed by EMSL, Inc. at all laboratories that perform asbestos analysis of bulk material by Polarized Light Microscopy. Currently, PLM analysis is performed at all EMSL laboratories. The Corporate Quality Assurance Department is responsible for the control and oversight of any revision in these Standard Operating Procedures. All laboratory personnel must adhere to procedures documented in this manual. Any major deviations must be approved y the Quality Assurance Department. Standard Operating Procedures for all other analysis are presented in other SOP's.

2.0 SAMPLE RECEIPT/LOG IN PROCEDURES

Care should be used to avoid cross contamination of bulk samples and air samples when handling incoming samples.

Acceptance of Bulk Samples

Incoming samples are inspected by the Sample Receiving Coordinator to determine if they conform to laboratory acceptance criteria. Samples are judged unacceptable under the following circumstances:

- Bulk samples must not be packaged with air samples
- Samples must not be submitted in obviously damaged or compromised packaging
- Analysis requested must not be outside laboratories capability or accreditation
- Sufficient amount of sample must be provided.
- The sample identification must correspond to those listed on the client's Chain of Custody form
- Requested turnaround must be achievable
- Proper sampling technique must have been performed
- Appropriate analytical methodology must be cited

Samples, which do not meet the above requirements, are rejected. Deficiencies are reported to the client and noted on the Chain of Custody in the comments section of the form. A deficiency/corrective action report is completed and filed.

Samples are accepted upon signing the chain of custody and logged into the Laboratory Information Management System (LIMS), as described in the following section.

2.1 Verification of Chain of Custody for Bulk Samples

All samples received must be submitted with a Chain of Custody Form. An EMSL Chain of Custody form is available for clients who do not have one. The Chain of Custody form contains information regarding samples, which is essential for proper analysis, sample tracking, and record keeping. All facilities must verify the information contained on this form prior to the log-in procedure, beginning with sample sets with the quickest turnaround time.

The Chain-of-Custody Form must be checked for the following:

- The sample numbers on the chain of custody form must match the numbers on each sample exactly.
- Each sample received must be listed on the Chain-of-Custody form.
- The turnaround time should be clearly stated. In the case of a regular client, these may be known in advance. If the turnaround time is not known or is incorrect, the client must be notified immediately.
- Contact information for reporting of results or for addressing any questions or problems, is included on the Chain-of-Custody form.
- The person relinquishing the samples must sign where appropriate.
- If the client wishes to make changes i.e., turnaround time or type of analysis, the client must provide such information in writing. If possible, the client will send the written statement by facsimile.

2.2 Assigning Laboratory Numbers and Sample Log-In

After this information has been verified, each sample to be analyzed may now be entered into the computerized log in system and assigned a unique laboratory number. Instructions on Computer Data Entry can be found in the LIMS Computer Manual.

2.3 The Following Information Is Recorded When Samples Are Logged In:

- Client Name and Address
- Job Number or Name of Project (or both if available)
- Quantity of Samples Ordered
- Type of Analysis
- Turnaround Time
- Sample ID
- Phone Number
- Results to:
- Fax #
- Purchase Order # (if available)
- Name of individual logging samples

The computer then assigns a laboratory number to each sample.

Voided samples

If at any time during acceptance, log in, or analysis procedures a sample or sample set is voided for any reason, the laboratory must document such an event. This includes a notation on the Chain of Custody and record on the Deficiencies and Corrective Action Report (EMSLdfcorr1.98).

Samples may be voided by the client or the Laboratory Manager. Examples of causes may be:

- Work order cancelled by the client
- Analysis requested outside laboratory capability
- Obviously damaged or compromised samples, i.e. opened air cassettes, cassettes with torn or ripped filters, water samples in leaking or faulty containers.
- Obvious faulty sampling technique
- Improper sample media
- Incompatible samples packaged together (i.e.- air samples with bulk samples)
- Inappropriate analytical methodology requested

The client must be immediately notified by the laboratory manager or designee when the sample is voided.

2.4 Required Documents

After samples are assigned a unique laboratory number and have been logged in, additional data and special forms are generated for each sample set.

Forms are generated either by entering data into the computerized log in data system (see the LIMS Computer Manual) or manually, depending on the specific form.

Billing Worksheets, PLM Analysis Worksheets, and Tracking Labels are generated for each set of samples.

2.5 Billing Worksheet

The Billing Worksheet is produced by computer after sample information has been entered and samples have been assigned a unique laboratory number. Instructions for producing a computerized Billing Worksheet are in the Computer Manual.

The Billing Worksheet contains the following information:

- Computer Billing #
- Client Name and address
- Project ID
- Turnaround Time
- Type of Analysis
- Quantity
- Date
- Laboratory #'s
- Sample ID and location
- Purchase Order #
- Accounting code
- Phone #, Fax #
- Logged in by and time
- Prepared by and Date
- Analyzed by and Date
- Data entered in by and Date
- Screened by and Date
- Mailed out and Date
- Comments
- Laboratory location

2.6 PLM Analysis Worksheets

Analysis Worksheets are generated by the computer at the time of log in. Specific instructions for preparing a computerized PLM Analysis worksheets are found in the LIMS manual.

The analysis worksheet contains the following information:

- Client information (address, phone, etc.
- Time logged in
- Date/time Due
- Project Identification (if provided)
- Turnaround time
- Billing number
- Client sample identification number
- Location from which the sample was collected
- Appearance of sample
- Treatment
- % and type of asbestos
- % type of non-asbestos
- optical properties of non-asbestos
- Optical properties of asbestos
- Analysts signature, date

3.0 BULK SAMPLE PREPARATION – OVERVIEW

Bulk samples are prepared under a HEPA filtered hood in an area separate from air samples. Technicians are instructed in basic sample preparation techniques, instrument calibration and safety procedures. After receipt and verification of all paperwork as specified in Sample Receipt, Section 2.0, the following steps are taken:

Open the sample container under the HEPA filtered hood and place entire contents on a clean sheet of paper. In the event that the sample size is too large to fit onto the paper, make a composite sample with several representative portions of the sample and place them on the sheet. Observe the sample under the stereomicroscope; examining for homogeneity, color, and obvious fibers. Determine the appropriate category for sample preparation technique, which should be employed, i.e. crushing, teasing, dissolving or other mechanism and estimate as much as possible the percentage of all material present (see Section II). Note on the PLM Analysis Worksheet the sample color, method of treatment used, and degree of homogeneity.

Preparation Techniques

Teasing

Take two pairs of cleaned forceps and hold the sample down with one of the forceps. Use the other pair of forceps to pull and tease the sample material apart. If curly fibers are present, place a drop of 1.55 RI liquid on a pre-cleaned glass slide. Grasp the sample material with the forceps and place it into the liquid on the slide and cover with a coverslip. Flatten material between the coverslip and slide by gently rubbing a pencil eraser over the slipcover. If straight fibers are observed, indicating the potential presence of amphiboles, mount the sample in 1.68 RI liquid and follow the same procedure as above. If both curly and straight fibers are observed, prepare multiple sample preparations as discussed above. If no fibers are observed, mount in 1.55 RI liquid.

Crushing

Use forceps or other appropriate instrument that has been cleaned to isolate a small, representative piece and scrape or crush the material. If curly fibers are present take a drop of 1.55 RI liquid and place on a pre-cleaned glass slide. Grasp the sample material with the forceps and place into the liquid on the slide and cover it with a coverslip. Flatten the material between coverslip and slide by rubbing a clean pencil eraser over the coverslip. If straight fibers are observed, mount the sample in 1.68 RI liquid and follow the same procedures as above.

If both curly and straight fibers are observed, prepare multiple sample preparations as discussed above. If no fibers are observed mount in 1.55 RI liquid.

Dissolving

Isolate a small representative sample using cleaned scalpel and forceps. Place the isolated sample on a pre-cleaned slide and dissolve with the appropriate solvent (i.e., acetone, chloroform, tetahydrofuran, toluene, and amyl acetate). Allow the solvent to evaporate before addition of RI liquid (air evaporation, or hot plate evaporation).

Note: Perform this preparation technique under a fume hood suitable for organic compounds. See EMSLCHP100.0

If curly fibers are present mount the sample in 1.55 RI liquid and cover with a coverslip. Flatten the material between coverslip and slide by rubbing a clean pencil eraser over the coverslip. If straight fibers are observed, mount the sample in 1.68 RI liquid and follow the same procedure as above. If both curly and straight fibers are observed prepare multiple sample preparations as discussed above.

Carefully fold the sheet of paper used during stereoscopic review and dispose of in designated as asbestos waste receptacle.

4.0 ANALYSIS OF BULK SAMPLES – OVERVIEW

PLM Analysis Procedure

After a sample has been prepared, observe under the polarized light microscope. All fibrous materials are identified. If amphiboles are suspected, preparations are made consecutively in 1.68 and 1.605 liquids to determine the refractive index of the fiber. Observations of optical properties of suspect asbestos fibers are made and recorded to include:

- morphology
- pleochroism (if any)
- retardation
- thickness (as applicable to determine birefringence)
- refractive index parallel and perpendicular to polarizer
- sign of elongation
- angle of extinction
- color of fiber

Optical characteristics of suspect non-asbestos fibers are also recorded. These may include:

- Isotropic
- Undulating
- High Birefringe
- Scaly surface
- Double sign of elongation

An area percent of asbestos is determined by calibrated visual estimation or by point count criteria. Record all information obtained during analysis the PLM Analysis Worksheet.

5.0 DATA REPORTING

Procedures for Reporting Data

Following sample analysis, the analyst initials the billing worksheet and signs the analyst worksheet containing the sample results. The analyst then submits the results for data processing. Following data entry and report generation, the report is submitted for review and signature by the laboratory manager or designee. Procedures for report clearance includes:

- Overall compliance with the Quality Assurance Program
- Check of daily calibrations including standard analysis
- Review of intra analyst QC data associated with sample set
- Comparison of original data with typed report
- Check of completeness of bench forms

Approved signatories are assigned by the Regional Laboratory Managers or the Quality Assurance Manager.

- The billing worksheet is signed and dated by appropriate personnel.

Report filing

Files are maintained of the original bench worksheets, billing worksheet, copy of the chain of custody and any other applicable associated paperwork. All documents are filed by billing number.

6.0 DATA ENTRY AND THE FINAL REPORT

Data produced during analysis is entered into the computer. Specialized computer programs corresponding to different types of analyses have been written. Instructions for entering data are kept in the LIMS Computer Manual.

All reports must be signed by an approved signatory and will contain the following disclaimers:

> "The above test report relates only to the items tested. This report may not be reproduced, except in full, without written approval by EMSL. The above test must not be used by the client to claim product endorsement by NVLAP nor any agency of the United States Government".
>
> "Laboratory is not responsible for the accuracy of results when requested to physically separate and analyze layered samples"

Verification of Data

After the report is printed, the data entry operator reviews all reports as follows:

- Correct client information appears on the report.
- Sample numbers are correct
- Checks for any obvious errors
- Sample components sum to 100%

Assembling Reports

Place the pages of the report, the original bench worksheets and all other project related documents together for final review by the laboratory Approved Signatory.

Signing the Final Report

Prior to signing, the printed final report with all its components is reviewed in detail by an approved signatory. The report is reviewed for:

- Overall compliance with the Quality Assurance Program
- Typographical and transposition errors, which may occur between the client chain-of-custody form, analysis worksheets and final report
- All required signatures or initials are present in the Analysis Worksheets.
- Review of microscope calibration records and intra analyst QC data
- Review of original data for technical accuracy and completeness
- Check of daily calibrations including standard analysis

When the report review is complete, the approved signatory signs the report in the space required and places the documents in the ‘to be mailed’ bin. Reports containing errors or lacking information are held back for corrections by the data entry staff.

Final reports are mailed by the clerical staff under the direction of the Laboratory Manager.

7.0 FILING SYSTEM

After final report is sent to the client, the billing worksheet, original bench worksheet and a copy of the chain of custody is filed by billing number. Final reports are maintained electronically by the LIM's system. Copies of chains of custody are stored at the corporate office in computer files.
All records are stored for 7yrs.

8.0 SAMPLE STORAGE AND DISPOSAL

All samples are placed in ziplock bags and kept for one month (unless otherwise requested by the client). Samples containing <1% asbestos are discarded into the trash while those containing ≥1% asbestos are discarded through a licensed hazardous waste removal company. A copy of the waste manifest is stored in the laboratory files.

9.0 PROCEDURE FOR DEALING WITH CLIENT COMPLAINTS OR PERFORMANCE DEFICIENCIES

If a client calls with a complaint, the call is referred to the Laboratory Manager. An investigation into the cause of the problem is initiated. The Laboratory Manager will review all applicable documentation related to the sample including bench notes, chain of custody and Quality Control data. The Laboratory Manager may need to discuss the analysis with the responsible analyst. When the discrepancy is resolved, the client is notified and a corrective action report is completed and placed in the file. In the event a resolution is not accomplished, the Corporate QA Manager is called upon to aid in satisfying the issue.

A client complaint or performance deficiency may be defined as:

- Reporting errors such as typographical errors, transcription errors
- Missed turnaround time requirements
- Analytical or technical errors
- Sample numbering
- Results reported to wrong client contact
- Miscalculated results

Steps for resolution may include:

- Additional training-This may include one-on-one training, refresher workshops, etc.
- Revisions to report review procedures
- Adding to laboratory resources (staff, equipment, etc.)

If the problem has not been resolved, and involves a result discrepancy, it is EMSL's policy to suggest the sample(s) be analyzed by another laboratory. If a discrepancy is still found, the samples will be sent to a third, referee laboratory. The laboratory should be selected and agreed upon by EMSL and the client.

For each event, a deficiency corrective action form (EMSLdfcorr1.98) must be completed and maintained in the laboratory files.

II

DETERMINATION OF ASBESTOS IN BULK SAMPLES BY POLARIZED LIGHT MICROSCOPY (PLM) WITH DISPERSION STAINING

1.0 OVERVIEW

1. This method describes the procedures for the determination of the presence or absence of asbestos in bulk samples of building material. Samples are initially examined under low magnification using a stereo microscope, contained in a hood equipped with a HEPA filter. Initial observations should note gross material appearance (homogeneity, fibrous/non-fibrous) and physical characteristics (color, texture, friable/non-friable).

2. Analysis by polarized light microscopy (PLM) is used for the positive identification of suspect fibers. Positive identification of asbestos requires the determination of several optical properties peculiar to the six types of asbestos: chrysotile asbestos, grunerite asbestos (amosite), riebeckite asbestos (crocidolite), anthophyllite asbestos, tremolite asbestos and actinolite asbestos.

3. Quantitative estimates of the asbestos content, and other major constituents, of the sample are made based on a combination of the estimates from both the gross and the PLM examinations.

4. Interference's from other inorganic and organic fibrous constituents, cleavage fragments of natural minerals, binders, coatings, and man-made fibers may be encountered. Moisture may interfere with the determination of some optical properties. Therefore, wet samples should be dried prior to analysis.

5. The sample matrix may cause a variety of interference's under PLM observation. Special matrix reduction techniques may be necessary to reduce these interference's.

2.0 EQUIPMENT

1. A low power binocular microscope (preferable stereomicroscope), with a magnification range of approximately 10-45X, and an auxiliary light source.

2. A compound microscope set-up for polarized light microscopy, to include a polarizer, analyzer, port for a wave retardation plate, a 360^{o} graduated rotating stage, substage condenser, lamp and lamp iris.

    - Objective Lenses: 10X, 20-25X, 40-45X, and dispersion staining objective.
    - Ocular Lens: 10X minimum
    - Eyepiece reticule: Cross hair
    - Compensator plate: 550 millimicron retardation (first-order red or gypsum)

3. The type of material being examined will dictate the various apparatus needed for sample preparation. At a minimum, the following will be required:

    - Negative pressure hood equipped with a HEPA filter at the exhaust
    - Microscope slides: ~75 mm x 25 mm, 1 mm thickness
    - Coverslip: No. 1, 22 mm^2
    - Tweezers, tungsten probes, dissecting needles, scalpels, glazing pliers, forceps.
    - Glass plates, petri dishes or disposable containers(e.g. weighing boats $5"^2$)
    - Mortar and pestle (agate or porcelain)

4. Auxiliary equipment may include a Wylie mill, centrifuge, filtration apparatus, and low temperature ashers, assorted beakers, and miscellaneous glassware, a vacuum cleaner equipped with a HEPA filter.

3.0 REAGENTS

1. Refractive index liquids

    - N_D = 1.550, 1.605, 1.630, 1.680, 1.700

2.

    - Dilute acetic acid (CH_3COOH): ACS reagent grade
    - Dilute hydrochloric acid (HCI): ACS reagent grade
    - Acetone (CH_3COOH_3): ACS Reagent grade
    - Chloroform ($CHCI_3$): ACS Reagent grade

3. Asbestos reference standards, and standards for various minerals and man-made materials typically encountered in bulk materials containing asbestos. Use NIST Certified SRM 1866a/Common Commercial Asbestos, SR1867/Uncommon Commercial Asbestos.

4.0 BACKGROUND AND DEFINITIONS

The name asbestos, a Greek word mistakenly thought to mean incombustible, was given to fibrous minerals hundreds of years before the science of mineralogy evolved. The Greek word actually means unquenchable, inextinguishable (not incombustible) according to the etymology of the Oxford English Dictionary.

The definition of asbestiform minerals includes three aspects: morphology, structure, and chemistry. Morphologically, asbestiform mineral varieties separate into flexible fibers or flexible bundles of fibers. Flexible fibers bend readily and only break across the fibers into distinct pieces with some difficulty. Structurally, the asbestiform minerals are limited to the serpentine and amphibole mineral groups. Chemically, these minerals are all hydroxylated silicates. The term “hydroxylated” is preferred over “hydrated” because these minerals contain OH ions rather than water or crystallization. The serpentines contain approximately 13-weight percent water; and the amphiboles, approximately 2.5 weight percent water.

There is no “group” of asbestos minerals. “Asbestos” is a general term applied to certain minerals (which are themselves classified under crystal-structure-based groups) when these minerals crystallize as the asbestiform variety. Table 2 lists some common silicate minerals and their asbestiform varieties, together with their relationships and formulas in Tables 3 & 4.

Only very small quantities of the amphibole and serpentine minerals under particular geological circumstances occur as an asbestiform variety of the mineral. The asbestiform varieties occur in veins or small veinlets within rock containing or composed of the common (nonasbestiform) variety of the same mineral.

In some rare instances, the mineralogical occurrences contain sufficient quantities of usable asbestiform minerals to be economically mined for commercial asbestos. The soft, silky fibers of asbestos (sometimes called mineral silk) are so flexible that they can be spun into threads from which cloth can be woven. The resulting material is fireproof, is a good thermal and electrical insulator, and has moderate to good resistance to acids. It has been used from Roman times, and is most familiar in daily use in brake lining for automobiles and as the “asbestos” siding used in residential construction.

The six asbestos minerals are defined under two mineral groups:

1. The serpentine group and
2. The amphibole group.

Serpentine Asbestos

Chrysotile is the only commercial asbestos mineral belonging to the serpentine group. Moderate amounts of aluminum may substitute for silicon and moderate amounts of iron may substitute for magnesium. Small amounts of manganous oxide (Mn)), calcium oxide (CaO), potassium monoxide (K_2O) and sodium monoxide (Na_2O) are also reported in the chemical analyses.

The crystal structure of chrysotile asbestos consists of double layers. Each layer consists of a linked SiO_4 tetrahedral coordinated to a second layer of linked MgO_2 $(OH)_4$ octahedral through a sharing of oxygen atoms; the composite double layer rolls up (like a window shade) to form long hollow tubes. The diameters of the individual tubes are on the order of 35 mm, and the length-to-diameter ratio can vary from 10:1 to well over 10,000:1.

Chrysotile is characterized by a combination of (1) a distinctive shape, (2) a chemical composition close to $Mg_3Si_2O_5$ $(OH)_4$, and (3) characteristic X-ray and electron diffraction pattern.

Amphibole Asbestos

Five of the six commercial asbestos minerals belong to the amphibole mineral group. These are grunerite asbestos (usually but improperly referred to by the acronym amosite); riebeckite asbestos (usually referred to by the variety name crocidolite); anthophyllite asbestos, tremolite asbestos; and actinolite asbestos. A considerable amount of substitution of other elements for Fe^{2+}, Fe^{3+}, silicon, sodium, calcium, and magnesium can take place in these minerals.

The Crystal structures of the amphibole minerals, including the asbestiform varieties, are composed of strips or ribbons of linked polyhedra, which join to form the three-dimensional crystal. The individual stripes are composed of three elements: These are two double chains of linked (Si, Al)O_4 tetrahedral and a strip of linked MgO_6, FeO_6 or AlO_6 octahedral.

4.1 Properties of Asbestos

Asbestos is a fibrous mineral of unique properties. It is used in a multitude of different applications because it can confer superior properties on products, including the following:

- stability in resistance to heat, moisture and microorganisms;
- insulation against noise, heat and electricity
- resistance to wear and to deformation under load or impact
- improved smoothness, hardness and opacity
- resistance to chemical attack, leaching and decay.

4.2 Asbestos Related Terms

In the following discussion, asbestiform refers only to asbestos. The other term, "fibrous", "mineral fiber", "fibril" and "fibril structure" applies to both asbestiform and non-asbestiform varieties.

Asbestos: A collective mineralogical term encompassing the asbestiform varieties of various minerals; an industrial product obtained by mining and processing primarily asbestiform minerals.

The quality of asbestos depends on the mineralogy of the asbestiform variety, the degree of asbestiform development of the fibers, the ratio of asbestiform fibers to acicular crystals of other impurities, and the length and flexibility of the fibers. The major asbestiform varieties of minerals used for asbestos are chrysotile, tremolite-actinolite asbestos, cummingtonite-grunerite asbestos, anthophyllite asbestos, and crocidolite. Asbestos may be marketed by its mineral name such as Amosite or Montasite. Some asbestos products contain non-asbestiform minerals (for example, asbestos-cement and asbestos-magnesia); consequently, the mineralogical and the industrial definitions of asbestos do not always coincide.

Fibrous: The occurrence of a mineral in bundles of fibers, resembling organic fibers in texture, from which the fibers can usually be separated (for example, satin-spar, and chrysotile).

The term "fibrous" has been used during the last 200 years to describe all kinds of minerals that crystallized in habits resembling organic fibers, including asbestos minerals. However, the related term "asbestiform" was never used for fibrous mineral habits other than asbestos. Accordingly, "fibrous" is the more general term, and asbestiform is a specific type of fiber.

Mineral Fiber: The smallest elongated crystalline unit which can be separated from a bundle or appears to have grown individually in that shape, and which exhibits a resemblance to organic fibers. (Examples: fiber bundles, chrysotile and crocodile, individual fibers, epsomite and Millerite).

The term “fiber is not limited to asbestos. However, it is distinct from “acicular” because it requires the resemblance to organic fibers.

Fibril: A single fiber, which cannot be separated into smaller components without losing its fibrous properties or appearances.

Most fibers are single structural entities, such as Millerite and nickel sulfide, and some may be called fibrils. However, some fibers are composed of two or more fibrils that are less readily separable from each other than fibers are from bundles (for example, chrysotile and crocidolite).

Fibril Structure: A systematically deformed and/or defective crystal structure of a fibril. A defect structure would involve various type of dislocation. The fibril structure may be exhibited by a single crystal, a group of single crystals, or at twinned single crystal.

The scroll-like fibril structure of chrysotile, the twinned single crystal fibrils of chrysotile, and the incompletely resolved fibril structure of an amphibole are all examples illustrated in the literature.

Some acicular single crystals may have the appearance of fibers and fibrils, yet there is nothing unusual about their crystal structures. Other acicular single crystals may have significant structural deviation sin addition to appearance which result in the display of certain properties usually found in fibers such as high tensile strength along the fiber axis. Thus, fibril structure is not limited to asbestiform structures, but may occur in a minor form in non-asbestiform structures.

Asbestiform: A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.

“Asbestiform” and “asbestos” are essentially synonymous in current usage. Some special properties of asbestiform varieties, including optical extinction and surface charge, are either not fully understood or are not uniformly applicable to all asbestiform fibers; consequently, they cannot be considered fundamental characteristics at this time.

4.3 Commercial Asbestos Minerals

Chrysotile: found in white, wavy, silky, lustrous fiber bundles. The fibers are usually much longer than they are wide. Chrysotile is often found in woven materials because of its flexibility.

Amosite: found in tan-brown, straight, brittle, rigid, inflexible fiber bundles.

Crocidolite: found in blue-blue-gray, straight, rigid fiber bundles. It is probably the most toxic form of asbestos we know.

Anthophyllite: usually colorless to pale brown. It may be found as singly crystals or fiber bundles. Fibrous anthophyllite is generally long and thin.

Actinolite and tremolite: difficult to identify, appear as acicular (bladed) and prismatic (more massive) cleavage fragments.

4.4 Polarized Light Microscopy Terminology

Crossed polarized (polarizer and Analyzer crossed): A fiber is isotropic (has only one refractive index) if it appears black (dark on a dark background) as the stage is rotated. It is extinct at all angles. Such a fiber cannot be an asbestos fiber.

A fiber is anisotropic (has more than one refractive index) if it shows up, as the stage is rotated, alternately light on a dark background

Sign of Elongation: A first order red plate is a section of quartz. It produces a 530nm retardation between the fast ray (X' along the long edge of the plate) and the slow ray (z' along the short edge of the plate). At crossed polars, if the fiber turns yellow in a NW-SE direction (parallel to the red plate port), it displays a positive sign of elongation. If the fiber turns blue when oriented in a NW-SE direction, it displays a negative sign of elongation. Crocidolite is the only asbestos mineral with a negative sign of elongation.

Dispersion Staining: λo is the wavelength at which solid and liquid match in refractive index. Dispersion staining requires "stops" in a special objective. The annular stop allows colors through. The central stop allows complementary (white light -λo) colors to pass through. Reference tables exist which show the complementary annular and central stop colors for different asbestos minerals in different immersion liquids. If fiber and liquid RI's are too far apart, then no dispersion staining colors will result.

Pleochroism: Pleochroism is one of the least reliable asbestos identification characteristics. Pleochroism refers to the tendency of a fiber to change color tint when rotated on the stage in plane polarized light. Most asbestos minerals are nonpleochroic. That is, they do not appear to change color tint as the stage is rotated in plane polarized light. Filler-binder materials contained in the insulation sample, however, may coat the asbestos fiber bundles and create a false pleochroic response. The most strongly pleochroic asbestos mineral is crocidolite, which usual appears to change from a blue to a blue-gray as the stage is rotated.

5.0 ANALYTICAL METHODOLOGY

Note : Exposure to airborne asbestos fibers is a health hazard. Bulk samples submitted for analysis are usually friable and may release fibers during handling or matrix reduction steps. All sample and slide preparations should be carried out in a ventilated hood or glove box with continuos airflow (negative pressure) and a HEPA filtered exhaust. Handling of samples without these precautions may result in exposure to the analyst and contamination of samples and the work environment, by airborne fibers. The cleanliness of the air in the work area is also ensured by testing the air quarterly with TEM analysis.

5.1 Sample Preparation

Gross examination of bulk samples is performed under low magnification (10-45X) to identify homogeneity, layering color, texture, friability and the presence or absence of fibrous constituents.

The sample is carefully removed from the sampling container and placed in an examination disk. Sample integrity is maintained at this point in order to note any layering, and if possible, orientation of the top and bottom surfaces. When discrete layers are identified, each is treated as a separate material, identifying and quantifying fibers in each layer. Each layer is analyzed and reported separately.

All fibrous materials are isolated (subsamples) and prepared for examination by polarized light microscopy. Isolation of these materials results in the loss of sample integrity since the sample must be “picked” through using forceps, probes, and needles. If the sample is not readily friable, a mortar and pestle can be used to crush the sample, or smooth jawed glazing pliers used to break the sample.

The type of sample matrix must be considered when determining sample preparation methodology. In samples such as floor tiles, roofing felts, tars, mastics and chalking, the fibrous materials of interest are often bound in a non-friable, organic substance, which makes observation of asbestos fibers difficult. Special techniques are used to reduce or remove these interference’s such as ashing and solvent dissolution. These techniques are detailed below.

5.2 Sub-Sample Preparation

Representative sub-samples of suspect fibrous material must be obtained from a variety of matrix materials. In most cases, forceps and probes are sufficient to isolate fibrous materials for analysis by PLM.

Sub-samples are immersed in an appropriate refractive index liquid on a microscope slide, teased apart, covered with a cover glass, and observed with the polarized light microscope. A refractive index liquid is chosen based on the fiber's morphology as observed under the stereomicroscope.

The selection of appropriate procedures for identifying and collecting sub-samples is dependent on the sample matrix. The following are presented as sample preparation steps for typical bulk sample materials.

5.2.1 Spray-on Fireproofing and Acoustic Material

In general these materials contain some combination of cellulose, vermiculite, perlite, clay, binder, and possibly asbestos. They are very friable, and by nature of their preparation, are of a heterogeneous, mixed appearance. These materials are easily probed to isolate suspected fibrous materials.

The presence of cellulose, vermiculite and binder present their own unique problems

Cellulose may have approximately the same index of refraction as chrysotile-asbestos. For this reason, it is frequently confused with chrysotile. However, cellulose fibers frequently pinch and swell along their length exhibit internal cellular structure and lack splayed ends: they are not composed of bundles of smaller fibers.

Vermiculite may be confused with chrysotile. It has similar index of refraction and while it is not fibrous, its extinction characteristics under crossed polars may give the impression that the particles are composed of masses of matted fibers. The problem is compounded by the fact that chrysotile and vermiculite are a common mixture in sprayed-on coatings.

Sprayed-on binder materials may coat fibers and affect color or obscure optical characteristics. Fine particle of other materials may also adhere to fibers.

5.2.2 Cementitious Materials (plaster/transite)

These materials are usually non-friable, or not easily friable, and of a heterogeneous, mixed appearance, containing combinations of perlite, vermiculite, calcium carbonate, gypsum, hair, quartz, and possible wollastonite and/or asbestos.

Probing is not easy and a mortar and pestle may be necessary to facilitate size reduction of the sample.

This procedure is not recommended for samples, which contain amphibole minerals or vermiculite. Grinding of amphiboles may result in the separation of fiber bundles or the production of cleavage fragments, which have aspect ratios greater than 3:1 and will be classified as asbestos fibers. Grinding of vermiculite may also produce fragments with aspect ratios greater than 3:1.

Transite board may be broken into pieces to expose “fresh” surfaces where fibers may be more easily identified.

Cleavage fragments of many natural minerals including amphiboles, talc, gypsum, wollastonite and vermiculite may appear as elongated anisotropic particles. The aspect ratio of these particles may be as great as 20:1. Therefore, aspect ratio alone is not sufficient for the identification of asbestos. Other properties of the asbestiform habit such as curved fibers, fiber bundles exhibiting splayed ends, and fibers with aspect ratios in excess of 20:1 must be observed in order to be sure asbestiform material is present in the sample. Therefore, once asbestos is known to be present, other properties (such as index of refraction an aspect ratio) can be used to identify asbestos and determine which particles will be counted in making a quantitative estimate of the amount of asbestos in the sample.

5.2.3 Thermal Insulating Material

These materials are usually very friable, white, and chalky in texture. Foam glass is an exception and may be covered by an outer layer (1/32 - 1/8”) of asbestos-containing material. Careful attention must be given to identifying and investigating this layer. Suspected fibrous constituents can be easily isolated with probes and forceps. Problems associated with cellulose and vermiculite have been noted under the discussion about spray-on materials.

Certain minerals may be found in the construction materials, which are fibrous, or asbestiform, but which are not asbestos containing. These minerals include but are not limited to fibrous talc, fibrous brucite (nemalite), zeolites and dawsonite.

Fibrous glass including both mineral wool and fiberglass is very common in these materials. Its isotropic character makes it readily distinguishable from asbestos.

5.2.4 Ceiling Tiles

These materials are typically friable and have a fibrous matrix.

Three predominate mixtures of the constituents:

1) cellulose, fibrous glass, perlite, filler/binder
2) cellulose and filler/binder
3) fibrous glass, filler/binder

When asbestos is present, it is usually either chrysotile or amosite. These materials are easily probed to isolate suspected fibrous materials. Particular attention should be given to the outer binder/paint layer of the three- (3) types mentioned. Type 3 is most likely to be asbestos containing.

5.2.5 Floor Coverings (Vinyl and Asphalt tiles, Linoleum)

Two types of materials are of concern in this category, block tile and sheet goods. They are non-friable (unless severely damaged by water, heat and weathering) and composed of a combination of tar, vinyl, quartz, calcium carbonate, and occasionally cellulose or fibrous glass. The mastic used to apply the product to the sub-floor is also a suspect for containing asbestos.

Isolation of suspect fibrous materials is not easily accomplished with probes and forceps. A “fresh” broken surface must be exposed to facilitate the location of any fibers. The use of the gravimetric reduction preparation methods is currently the best available for this sample type. See section 5.3

Caution must be exercised in handling these samples so that the mastic is not contaminated with debris and dust. Probe the mastic for potential fiber bundles. Clean as much of the mastic as possible off the fibers using a tungsten probe. A solvent such as acetone, chloroform, or the refractive index liquid can be used to wash off any remaining mastic.

Note: Dissolved mastic will change the refractive index of the RI liquid in which a fiber is mounted. If discoloration of the liquid is noted it may be “wicked” out from under the coverslip with a piece of paper towel. Fresh RI liquid can then be drawn, by capillary action, under the coverslip.

If fibrous material is not immediately apparent in the mastic, a smear of a small amount of mastic on a slide should be examined via PLM.

After the mastic is thoroughly examined, break or cut the tile to expose a “fresh” edge.

View these edges under the stereoscope to identify and isolate fibers or fiber bundles for PLM analysis.

The felt backing of vinyl sheet goods must be examined thoroughly. This material is easily probed for fibrous entities.

5.2.6 Asphalt Roofing Material

Roofing materials (roofing felts and asphalt shingles) are typically non-friable and have a heterogeneous, mixed matrix. Heavy probes must be used to tear through the material to isolate any fibrous materials. The use of the gravimetric reduction preparation methods is currently the best available for this sample type. See section 5.3 .

Solvent washing (acetone or RI liquid) may also be required to clean the fibers or bundles sufficiently for analysis. Fresh RI liquid must be used, after the cleaning, for PLM analysis.

5.2.7 Miscellaneous Materials (wall coverings, window and stage curtains)

Man-made fibers such as carbon, aluminum oxide, polyamides (nylon), polyester (Dacron) and polyolefins (polyethylene), and rayon are occasionally encountered in building materials.

The manufacturing process, and thus the physical form of these particles, can help to identify them. Typically, they are continuous, colorless, transparent cylinders with a round cross section. Two exceptions would be triacetate (Arnel and rayon fibers which demonstrate lengthwise striations and a multi-lobed cross section. Acrylic fibers show a cross section varying from kidney bean to dumbbell in shape. Synthetic fibers typically demonstrate high birefringence.

5.2.8 Gravimetric Reduction

Using the combination of high temperature and solvent dissolution, a samples matrix may be removed (or reduced), facilitating the microscopic examination of the fiber of interest. Eliminating the interference's of tar, vinyl, calcium carbonate, etc. gives the analyst a clear view of the fibrous materials, allowing for the measurement of key optical properties.

Procedure:
Organic Reduction

- Weigh approximately .5-1.5 grams of the sample into a tarred crucible. Record weight.
- Ash sample at 480 degrees Celsius for a minimum of 6 hours
- Record weight of ashed sample
- Calculate the amount of organic constituent lost in %.

Inorganic Reduction

"Wash" remaining ash with .5 ml distilled water and add concentrated HCL. After 15 mins., dilute with additional distilled water. Pour into filtration apparatus with .4 micron polycarbonate filter. Apply vacuum. Dry filter in tarred plastic petri dish. Weigh filter and calculate % mineral lost.

5.3 Asbestos Identification

Positive identification of asbestos requires the determination of the following optical properties:

a. morphology
b. color and pleochroism
c. refractive indexes
d. birefringence
e. extinction characteristics
f. sign of elongation

Table 5 lists the optical properties for a variety of fibrous constituents encountered in the analysis of building and insulation products. Table 7 presents a flow chart for the qualitative analysis of some of these materials. Central stop dispersion staining colors are listed in Table 6. It must be remembered that natural geological variations of asbestiform mineral deposits will produce exceptions to the data in Tables 5 and 6, and differences from laboratory standards.

The prepared slide is scanned identifying asbestos fibers using the optical properties of morphology, refractive indices, color pleochroism, birefringence, extinction characteristics, sign of elongation and dispersion staining characteristics.

5.3.1 Pleochroism

This is a property exhibited by some colored anisotropic substances. When viewed by polarized light pleochroic crystals change color as they are rotated. Examine the fiber of interest in plane polarized light (i.e. polarized in, analyzer out), and observe any color changes which result as it is rotated through 360^{o}.

5.3.2 Isotropic/ Anisotropic

With the polarizer and analyzer crossed (i.e., dark field) rotate either the slide or the stage and observe the fiber of interest. An isotropic particle will remain dark (essentially invisible against the dark background). Conversely, anisotropic particles will present an image, which appears to fade in and out of the background (at 90^{o} intervals) as it is rotated.

5.3.3. Angle of Extinction

As mentioned in Section 5.3.2., any anisotropic crystal extinguishes four times, between crossed polars, during a complete rotation.

This extinction occurs when the directions of vibration of the slow and fast rays of the fiber coincide with those of the polarizer and analyzer. Extinction may be one of three types:

1) Parallel or straight, when the fiber extinguishes parallel to the vibration direction the analyzer or polarizer (Figure I).
2) Symmetrical, when in the extinction position the vibration direction of the analyzer and polarizer are parallel to the diagnosis of a rhombic cross-section through a crystal (Figure I).
3) Oblique of inclined, when the fiber extinguishes at an oblique angle to the vibration directions of the analyzer and polarizer. This angle is known as the extinction angle, which is usually determined in terms of the slow vibration direction of the crystal.

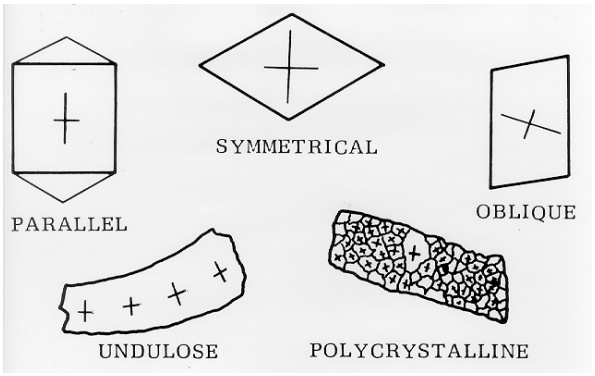


FIGURE I. Types of Extinction

5.3.4 Birefringence

Birefringence (the difference between two indices of a particle on a given view) can be estimated from the interference colors observed when polarizers are crossed. As the stage (or slide) is rotated, isotropic particles (e.g., fibrous glass) will remain dark against the dark background. Particles with weak birefringence (e.g., quartz) will exhibit first order grays, whites, or yellows. As birefringence increases, higher order interference colors (reds, blues, greens, etc.) may be observed. As a rule, highly birefringent minerals appear brighter when rotated under crossed polarizers than do particles with weaker birefringence.

TABLE 1 - CATEGORIES OF BIREFRINGENCE STRENGTH WITH EXAMPLES

BIREFRINGENCE	INTERFERENCE COLOR IN SECTIONS 0.03 MM THICK	EXAMPLES, AND BIREFRINGENCE OF EXAMPLE
Weak: 0.0.010	First order gray, white or yellow	Apatite: 0.0003
Moderate: 0.010-0.025	First order red to second order green	Cancrinite: 0.0023-0.029
Strong: 0.025-0.100	Upper second order into fifth order	Zircon: 0.062
Very Strong: 0.100-0.200	High order-sixth and higher	Calcite: 0.172
Extreme: 0.200 and up	Very high order	Rutile: 0.285

5.3.5 Sign of Elongation

Using a first-order red 1 plate and crossed polars determine the sign of elongation by positioning the fiber at an angle of 45^{o} to the analyzer and/or polarizer. When the slow ray of the red plate is parallel to the elongation of fiber, and the interference color of the fiber is yellow, the mineral has a negative sign of elongation. Vice versa, if the interference color of the fiber is blue, the mineral has positive sign of elongation. In other words, the arrangement of colors:

(in negative crystals)
yellow NW-SW elongation
blue SE-NW elongation

(in positive crystals)
yellow SE-NW elongation
blue NE-SW elongation

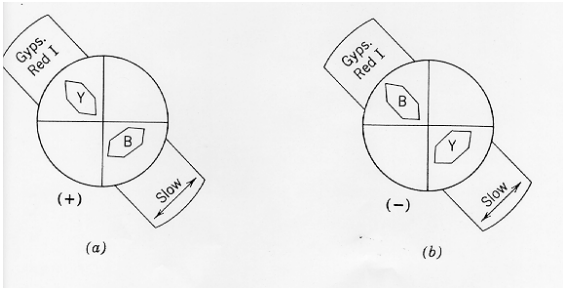


FIGURE II. Determination of Sign of Elongation

(a) positive elongation (b) negative elongation

5.3.6 Dispersion Staining Colors

Dispersion staining is a technique for particle identification based on the difference between the dispersion of refractive index for a particle and the liquid medium in which the particle is immersed. In order to produce dispersion staining colors, the particle, and immersion liquid must have dispersion curves that intersect sharply in the visible light region. A special objective, containing annular and central stops in the back focal plane is required.

After isolating fibers of interest (sub-samples), follow the analysis flow chart (Table 7)

Note: Differences from standard characteristics may be observed due to natural variations in the conditions under which the minerals were formed and/or subjected to.

In the 1.55 HD refractive index oil, chrysotile will be readily identifiable from mineral wool or fiberglass by crossing the polars and using the 550-millimicron retardation plate to observe the colors of chrysotile. Both of the glass species are isotropic and will not show any colors. Many varieties of cellulose are close to 1.55 in index, but will not show chrysotile central dispersion staining colors. Characteristic magenta and blue colors identify chrysotile.

a. If the fibers in the sample have a higher index of refraction than 1.55, have a negative sign of elongation, and appear blue by transmitted light, crocidolite is suspected. Prepare another slide with 1,700 refractive index oil. The color of crocidolite will be much bluer with an annular stop. The central stop dispersion staining colors are sometimes difficult to impossible to see because of the opacity of the dark blue fibers. If the fibers with the higher index than 1.55 are not blue, prepare a slide using 1.670 refractive index oil. Amosite has a positive sign of elongation and in the oil has central stop dispersion staining colors of yellow and magenta-blue.

b. If the refractive index of the fibers is between 1.550 and 1.670 mount another preparation in 1.605 or 1.620 HD. The refractive indices for anthophyllite, tremolite, and actinolite vary naturally within the species. Anthophyllite can be distinguished from the other two by its parallel extinction. Actinolite has a light to dark green color with some pleochroism in transmitted light. The dispersion staining colors will have to be checked (i.e., actinolite DS colors in 1.63 RI oil are blue-magenta). A common interference mineral in this refractive index range is wollastonite. It also has a typical cleavage fragment morphology similar to the three asbestos minerals. Wollastonite has both a positive and a negative sign of elongation, parallel extinction and central stop dispersion stain colors in 1.605 HD of pale yellow and pale yellow to magenta. If further confirmation of wollastonite versus anthophyllite is

needed, wash a small portion of the sample in a drop of concentrated hydrochloric acid on a slide. Place the slide, with a coverslip in place, on a warm hot plate until dry. By capillary action, place 1.62 refractive index oil under the slipcover and then examine the slide. Wollastonite fibers will have a "cross-hatched" appearance across the length of the fibers and will not show central stop dispersion colors. Anthophyllite and tremolite will still show dispersion colors.

5.4 Quantification

If a sample is properly prepare (i.e., components are recognizable) it is possible to estimate its composition with considerable accuracy. It is desirable, though not always realistic, that the components be of the same size and specific gravity. Simple mixtures (2-3 components) can generally be analyzed to an accuracy of + 10%. Greater accuracy is attainable with practice. Comparison to a reference set of standard slides or photomicrographs can facilitate a more accurate analysis.

Always keep in mind that many conditions must be fulfilled in order to carry out a more or less accurate quantitative analysis. Three of the most important are:

1. The sample to be examined must be representative of the material to be analyzed. This will eliminate most of the errors arising from sampling.
2. The components of the sample must be distinguishable in appearance and/or properties. In some cases, additional treatment may be necessary to accentuate them.
3. Analysts ability to quantitative correctly as established with calibration and training.

After the asbestos species have been identified, scan the entire area under the coverslip of the slide with the 1.55 HD refractive index oil and determine an estimated volume percentage of each asbestos mineral. The slides with the other index oils can be used to help confirm these estimates, if needed.

1. Since complete homogeneity of the sample is often difficult to obtain, care must be exercised when estimating a single percentage.
2. If the estimate is in question, several microscopists can examine the slide and conclude amongst each other.
3. Estimate of the volume percentages of the other fibers in the sample can also be determined and reported as required

Make quantitative estimate of the asbestos content of the sample from the appropriate combination of the estimates from both the gross and microscopic examinations. Use either Calibrated Visual Estimation or Point Count technique.

Note: The point counting method only produces accurate quantitative data when the material on the slide has a uniform thickness, which is difficult to obtain. The point counting technique is recommended by the EPA and various State agencies to determine the amount of asbestos in bulk samples.

After performing qualitative analysis on each prepared slide, quantitative analysis must be performed by area estimation and recorded on the PLM worksheet.

Heterogeneous (Layered) Samples

For heterogeneous samples, a slide must be prepared for each distinct layer. A heterogeneous sample is a sample consisting of one or more analyses, which can be easily distinguished into layers and are not uniformly mixed. Each individual layer of a heterogeneous sample may have more than one material present.

From the prepared slide, examine a random field/graticle area at 100X under PLM. To determine the area percent of the materials visible, note the dispersion colors, the morphology and/or the sign of elongation for each different material. Estimate and record the percentage of each individual analyte to include asbestos fibers, non-asbestos fibers, and nonfibrous materials.

For each random field, the total area percentage must add up to 100%. A minimum of five random fields must be read from each slide preparation.

To determine the individual quantity of each analyte present in a homogeneous sample, find the average of the recorded area percentages for each. These percentages will be representative of the entire sample.

To determine the individual quantity of each analyte present in a heterogeneous sample the analyst must first determine the percentage of each distinct layer present in the sample as a whole. Examine the entire sample under the stereoscope to determine the layer percentages and record these percentages in decimal units. Find the average of the recorded area percentage for each individual analyte in each distinct layer. For each individual analyte in a layer, multiply the layer percentage (decimal) by the average area percent (whole number). When complete, add the percentages together to achieve a representative quantity of each analyte present for the entire sample. Use the PLM worksheet/layers form to enter data.

Calibrated Visual Estimation

The technique used for visual estimation is calibrated against the point count technique. To calibrate an analyst, he/she must quantitate a number of samples using the point count technique and reanalyze the same samples with visual estimation. The number of samples analyzed must be at a minimum of 20, in various ranges (see Monthly QC report). The data is compared using variance statistics as discussed below. This comparison of technique is performed until proficiency is established for all ranges.

Point Count

In addition to Calibrated Visual Estimation, the point count technique is a tool available to the analysts for the determination of asbestos concentration. Commonly used for petrographic applications, it is a widely accepted technique for the estimating asbestos concentrations based on a volume* percent of the binding matrix material. It can be used on a variety of material types, including friable matrices as well the residue ash of a non-friable organically bound material (NOB)

**Note: unless the densities of each of the materials being measured are known, concentrations based on weight percent are not achievable*.

EPA Point-count method

This technique is outlined in EPA-600/M4-82-020, ' Interim Method for the Determination of Asbestos in Bulk Insulation Samples' and EPA/600/R-93/116, 'Method for the Determination of Asbestos in Bulk Building Materials'

Procedure:

1. Prepare 8 slide mounts using the appropriate refractive index. Care should be taken to achieve a uniform distribution of materials throughout each slide prep.
2. Using the single discrete point of a cross hair reticule, or Chalkey Point Array (a reticule with 25 "dots",) count points superimposed on either an asbestos or non asbestos matrix material for a total of 50 non empty points per slide preparation.

Reporting-
% asbestos is calculated:

% asbestos = (a/n) 100%
a = number of asbestos points
n = number of non asbestos points (total of 400-minimum)

If a=0, report ND
If $0 < a \leq 3$, report <1% asbestos

All values should be reported to the nearest %.

New York State, 198.1 (unofficially referred to as the "Stratified Point Count")

The State of New York has adopted the point count technique as the only acceptable quantitation method for friable building materials. NYS has incorporated a variety of modifications to the EPA's method

Procedure:

1. Prepare 4 slide mounts
2. For each slide preparation count all superimposed points until either one asbestos point is counted, or 50 nonempty points are counted.
3. If 4 asbestos points have been counted after all 4 preps have been analyzed, analysis can be halted.
4. If less than 4 points have been counted, 4 additional slides must be prepared and analyzed (at the rate of 50 nonempty points/prep) until: a) at least 4 asbestos points have been counted, or b) at least 400 nonempty points from the 8 slide preps have been counted.

Scanning Option: If the initial stereoscopic scan hints at the sample being negative, the analyst may opt to use the scanning option. All 4 mandatory slide preparations must be scanned at 100X mag. If no asbestos is detected the sample is reported as ND. The percentages of all other non-asbestos fibers may be determined by visual estimation. However, if asbestos is detected during this scan, the stratified point count must be initiated. The analyst begins the point count at the slide in which the asbestos was observed. Slides from that particular sample which were already scanned and contained no asbestos can be considered to contain 50 – non-asbestos points each.

Reporting

% asbestos = (a/n) 100%

a = number of asbestos points
n = number of non asbestos points (total of 400-minimum)

a = number of asbestos points
n = number of non asbestos points (total of 400-minimum)

Results are rounded off to 2 digits.

If no asbestos was detected, report ND
If the sample contained 0 (zero) asbestos points out of 400 (or more) non-empty points but did contain asbestos (observed during scanning) report <1%.

Note: this method does not refer to the "0 < a $\leq$ 3, report <1% asbestos" reporting protocol as indicated in the EPA method – see above.

Application:

The point count methods are used in a variety of applications. These include:

- samples analyzed under New York State regulatory guidelines
- as an additional quantitation tool to verify visual estimation values
- as a referee method for boarder line concentrations, <10%
- compliance with the NESHAP's ruling for sample concentrations <10%
- at the clients request
- for improved detection limits

Detection limit

Under normal conditions, the practical detection limit for this method is one (1) percent. Detection limits can vary with sample type, amount of sample analyzed or method of quantitation used. For example, the 1,000 point count method can report values down to 0.1%.

These detection limits are based on the limits as referenced in the documented methodology (EPA-600/M4-82-020, EPA/600/R-93/116). These limits have been widely accepted, recognized, and understood by the analytical community. A true specific method detection limit (MDL) study has not been performed by EMSL laboratories.

Results are reported for samples containing asbestos below the detection limits as “less than “ one percent (<1%). With training in the use of known standards at various ranges of concentrations at, near and less than 1%, the analysts can accurately determine if the concentration of asbestos is <1% in the sample. Data collected from the comparison of point count data with visual estimation at levels below 1% is also a useful tool for the analysts. These procedures for ‘the calibration of the analyst” is detailed in the EMSL training policies and procedures.

Any sample determined to contain no asbestos is to be reported as “none detected”.

A note on trace: It is EMSL policy not to use the term ‘trace’ when reporting sample results, as this terminology is ill defined and ambiguous.

6.0 QUALITY CONTROL

6.1 Instrument Calibration/Maintenance

Follow the manufacturer's instructions for illumination and condenser alignment and other microscope adjustments. Specific guidelines for calibration, alignment, and maintenance can be found as Appendices in this manual.

Maintenance is a daily activity and is the responsibility of each analyst. Preventive maintenance is emphasized. Repairs beyond the capabilities of the analyst must be referred to an outside vendor.

Alignment/calibration must be checked weekly or more frequently, especially if the microscope is transported. See Calibration Frequency Reference Guide in Appendix section of this manual for a detailed schedule of calibration frequencies.

6.2 Personnel Training and Qualifications

Technicians hired for PLM analysis, which have not had previous experience or formal training, require the greatest amount of training and attention. They are introduced to basic information prepared by our more experienced analyst and are simultaneously led through the basics in the prep room and at the microscope. The trainee will work very closely with the mentor, helping and learning but not analyzing alone. Following this initial training period, the trainee analyzes samples, which are also run by the mentor. Through this activity, the progress of the trainee can be gauged without endangering the quality of data being reported by the lab. If the progress is good and the mentor feels confident about his/her student, an attempt is made to allow the candidate to run the QC samples, both the re-analysis and re-preps re-analysis. The trainee will be deemed proficient when quantitation and qualification within laboratory norms as established by our QC program is demonstrated on 100 consecutive samples. Additionally, the trainee must perform analysis on five rounds of past proficiency samples and succeed in generating data with the acceptable range as established by the agency(ies) statistical analysis.

Using NIST Standard Materials, the analysts must also be able to demonstrate ability to measure optical properties of asbestos fibers. The analyst must be able to determine the properties of all six asbestos types.

During this stage, the training analyst is trained and tested in textbook theory on microscopy and optical mineralogy. The analyst continues to receive extra attention and coaching as he/she analyzes on a microscope physically situated near that one operated by the mentor. Furthermore, whenever possible, the novice analyst is encouraged to participate in continuing education workshops and in-house programs related to PLM analysis. An analyst who has received the above training and attention and who consistently performs well will be considered qualified. Qualification status is determined by the Laboratory Manager or Quality Assurance Manager.

6.3 Intra-Analyst Reanalysis/Quality Assurance

Intra-analysts analysis is performed on 2%, or 1/50 of the samples analyzed daily (at a minimum). These samples are randomly chosen by the laboratory analyst. The same sample is prepared and analyzed a second time by the same individual. This data is then entered into the monthly Quality Control program.

6.4 Inter-Analyst Quality Assurance

Inter-analysts analysis (by at least one other analyst) must be performed on 7%, 1/15 of all samples analyzed. Samples are chosen randomly. Each analyst will have no knowledge of the other analyst's (original) results prior to his/her evaluation. This data is entered into the monthly Quality Control program.

Non friable organically bound (NOB) samples-reprep
Complete repreparations of NOB samples shall be performed on 5% or 1 in 20 for interanalyst QA. This includes all steps for gravimetric reduction .

6.5 Inter-Laboratory Quality Assurance

Inter-Laboratory analyses are samples that are reanalyzed by another laboratory. This process evaluates the precision of the participating laboratories preparation and analysis procedure.

Each EMSL laboratory will exchange samples with another laboratory on 1 in 500 samples and at a frequency of 4 times a year. The Regional Manager is responsible for maintaining and managing the program. The data is submitted to the Quality Assurance Department in the Monthly Quality Control Report.

6.6 Blanks

Blank analysis is performed using NIST traceable materials (1866a). Results are recorded daily to insure the absence of asbestos contamination in the work area. If contamination is found, its source must be immediately identified and eliminated. Target sources include:

a. Tools
b. Slides
c. Work areas
d. Preparation agents (acids, solvents)
e. Mounting oils

Non friable organically bound materials
At least one non- ACM NOB must be prepared and analyzed with every 20 samples. This includes the complete full preparation regime.

6.7 Daily Reference Slide Analysis

Samples are prepared from past proficiency testing samples with known concentrations and type of asbestos or NIST traceable standards. These samples are analyzed daily and recorded on applicable worksheets. In addition to monitoring the accuracy of the analyst quantitation techniques, reference slide analysis is used to determine ability to measure optical properties. Measurements are compared with the known standards. The measurements recorded include:

- Refractive Index
- Birefringence
- Pleochroism & color
- Anisotropic/isotropic
- Sign of elongation
- Angle of extinction

The Laboratory Manager reviews the data for accuracy and records proficiency in the analysts files.

This data is entered into the monthly quality control report. These results establish analyst accuracy as well as precision.

6.8 Proficiency Testing

All analysts in the NIST accredited PLM laboratories participate in the NIST's National Voluntary Laboratory Accreditation Program for Bulk Sample Analysis. Every active analyst must perform an individual analysis on the proficiency test sample, and report the data to the laboratory manager for review.

7.0 STATISTICAL ANALYSIS

Intra, Inter Analysts

The Quality Control calculations used for the Intra/Inter-analyst analysis based on a simple comparison of the relative difference of two values. These measures of variance are recorded and plotted over time to determine any trends or problems with the analysis.

Variance is calculated using:

For Intra-Analysts

$$R = |(A-B)|/((A+B)/2)|$$

Where: R = the measure of variance for the analysis

A = the value of the first analysis in %

B = the value of the second analysis in %

The Pass/Fail criteria for the QC analyses are as follows:

$R \leq 1$ - PASS

R > 1 - FAIL

Incorrect Asbestos ID - FAIL

Asbestos missed during analysis (false negative) - FAIL

Asbestos incorrectly identified to be present in a negative sample (false positive) - FAIL

For any failure of the above criteria the cause of the failure is identified and corrected. A deficiency /corrective action report is completed and place in the file.

For Inter-Analysts

$$R = (A-B)/((A+B)/2)$$

Where: R = the measure of variance for the analysis

A = the value of the first analysis in %

B = the value of the second analysis in %

The Pass/Fail criteria for the QC analyses are as follows:

$-1 \leq R \leq 1$ - PASS

$R > 1$ and $R < -1$ - FAIL

Incorrect Asbestos ID - FAIL

Asbestos missed during analysis (false negative) - FAIL

Asbestos incorrectly identified to be present in a negative sample (false positive) -FAIL

For any failure of the specified criteria the cause of the failure is identified and corrected. A deficiency /corrective action report is completed and place in the file.

For Standard Reference Analysis

Accuracy of analysis of standard, known samples, is determined using percent recovery calculations. Results are quantified and charted to determine analyst, as well as laboratory precision and accuracy, using the following formula for Percent Recovery.

$$\%R = (A/F) \times 100$$

Where: %R = percent recovery

A = the analytical result

F = the formulated standard weight

Quality Control Data Management

The Management of QC data is reported, tracked, and analyzed using the EMSL Quality Control program. Using custom EXCEL spreadsheets, this program is designed to provide accuracy and precision information for all qualified analysts.

8.0 REFERENCES

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9. EMSL.QAASB 101.0. Revision 1

TABLE 2 – ASBESTOS MINERALS, CHARACTERIZED BY THEIR MINERALOGY

Group	Designation of Mineral	Type of Asbestos	Chemical Abstracts Number[2]
Serpentine Amphibole	Serpentine	chrysotile	12001-29-5
	Riebeckite (glaucophane)	crocidolite(blue asbestos)	12001-28-4
	Grunerite (cummingtonite-grunerite)	grunerite asbestos (amosite)	12172-73-5
	Anthophyllite (gedrite)	anthophyllite asbestos[b]	77536-67-5
	Tremolite (ferroactinolite)	tremolite asbestos[b]	77536-68-6
	Tremolite - actinolite	actinolite asbestos	77536-66-4

TABLE 3 – CHEMICAL FORMULAS FOR TYPICAL ASBESTOS STRUCTURES

Mineral	*Chemical Formula*[c]
Chrysotile	$Mg_3\ (si_2O_5)(OH)_4$
Amosite	$(Mg, Fe)_6(Si_8O_{22})(OH)_2$
Crocidolite	$Na_2Fe^{2+3}\ Fe^{3+2}(Si_8O_{22})OH)_2$
Anthophyllite	$Mg_7(Si_8O_{22})(OH)_2$
Cummingtonite	$(Mg, Fe)7(Si_8O_{22})(OH)_2$
Tremolite	$Ca_2Mg_5(Si_8O_{22})(OH)_2$
Ferroactinolite	$Ca_2Mg_5(Si_8O_{22})(OH)_2$
Actinolite	$Ca_2(Mg,Fe^{2+})_5(Si_8O_{22})(OH)_2$
Glaucophane	$Na,\ Mg_3,\ Al_2(Si_8O_{22})(OH)_2$

TABLE 4 - ASBESTOS AND ANALOG FORMS

Asbestos	*Non-Asbestos Analog*
Chrysotile	Angigorite-lizardite
Crocidolite	Riebeckite
Amosite	Cummingtonite-grunerite
Anthophyllite	Anthophyllite
Tremolite Asbestos	Tremolite
Actinolite Asbestos	Actinolite

a) Taken from ASTM Practice E849-82
b) These varieties have no special designations.
c) Taken from Dee, Howie and Zusman, Rock Forming Minerals, Vol. 3, Longmans, London, 1967.

TABLE 5 - OPTICAL PROPERTIES OF ASBESTOS FIBERS

Mineral	Morphology,Color[1]	Refractive Indices[2] α		Birefringence	Extinction	Sign of Elongation
Chrysotile (asbestiform) serpentine	Wavy fibers. Fiber bodies have splayed ends and "kinks". Aspect ratio typically > 10:1, Colorless[4], plechroic.	1.49-1.560	1.517-1.562[3] (normally 1.556)	.002-.014	\\ to fiber length	+ (length slow)
Amosite (asbestiform grunerite)	Straight, rigid fibers. Aspect ratio typically > 10:1. Colorless to brown, nonpleochroic or weakly so. Opaque inclusions may be present	1.635-1.696	1.655-1.729[3] (normally 1.696-1.710)	.010-.033	\\ to fiber length	+ (length slow)
Crocidolite (asbestiform riebeckite)	Straight, rigid fibers. Tick fibers and bundles common, blue to purple-blue in color. Pleochroic. Birefringence is generally masked by blue color	1.654-1.701	1.668-1.717[5] (normally close to 1.700)	.014-.016	\\ to fiber length	- (length fast)
Anthophyllite	Straight, single fibers, some larger composite fibers. Anthophyllite cleavage fragments may be present with aspect ratios < 10:1[6] . Colorless to light brown	1.596-1.652	1.615 - 1.676[3]	.019-.024	\\ to fiber length	+ (length slow)
Tremolite-actinolite-asbestos	Tremolite-asbestos may be present as single or composite fibers. Tremolite cleavage fragments may be present as single crystals with aspect ratios< 10:1[6]. Colorless to pale green	1.599-1.668	1.622-1.688[3]	.023-.020	oblique extinction, 10-20^{o} for fragments. Composite fibers show \\ extinction	+ (length slow)

1. From reference 7, colors cited are seen by observation with plane polarized light
2. From references 7 and 4
3. \\ to fiber length
4. Fibers subjected to heating may be brownish
5. Fibers defined as having aspect ratio > 3:1

TABLE 6 – CENTRAL STOP DISPERSION STAINING COLORS[a]

MINERAL	RI LIQUID	I	\\
Chrysotile	1.550^{HD}	blue	Blue-magenta
"Amosite"	1.680	Blue-magenta to pale blue	Golden-yellow
	1.550^{HD}	Yellow to White	Yellow to White
Crocidolite[b]	1.700	Red-magenta	Blue-magenta
	1.550^{HD}	Yellow to White	Yellow to White
Anthophyllite-asbestos	1.605HD	Blue	Gold to gold-magenta
Tremolite-asbestos	1.605HDC	Pale-blue	Yellow
Actinolite-asbestos	1.605HD	Gold-magenta to blue	Gold
	1.630HDC	Magenta	Golden-yellow

a) From reference 3. Colors may vary slightly
b) Blue absorption color
c) Oblique extinction view

TABLE 7:

POLARIZED LIGHT MICROSCOPY QUALITATIVE ANALYSIS: For each type of material identified by examination at low magnification. Mount specially dispersed sample in 1.550 RI liquid and view at 100x in plane polarized light and crossed polars.

Fibers Present

Fibers Absent

Analyze two additional preps at 100x

Fibers Present

Fibers Absent

Fibers are isotropic (disappear at all angles of stage rotation with crossed polars).

Examination Complete

Possible fibers include:

Fiberglass 1-20 nm diameter
RI typically <1.53

Mineral wool 0-200nm diameter
RI typical <1.53
non-uniform morphology

Fibers are anisotropic

1. Determine extinction
2. Determine sign of elongation

SOE is Positive

SOE is Negative

n= 1.55
morphology = wavy (Chrysotile)
(Crocidolite)
morphology = ribbon (Cellulose)

n= 1.68
morphology = strait (Amosite)

n= 1.605
morphology = strait

DS Colors = yellow - blue (Tremolite)
DS Colors = yellow - yellow (Actinolite)

n= 1.68
morphology = strait
pleochroism = blue

A.

MICROSCOPE CARE AND MAINTENANCE

1.0 GENERAL DISCUSSION

With careful treatment, most microscopes remain practically maintenance free for many years. General maintenance is confined to protection from dust, chemically aggressive substances and fumes, excessive temperatures (>45^{o}C) and direct insulation. Specific precautions and instructions can be found in the manufacturer's operating manual(s).

2.0 EQUIPMENT

1. Sable hair brush (clean and dry)
2. Expanded polystyrene
3. Cotton swabs
4. Dust cover
5. Rubber bulb blower or canned compressed air
6. Chamois

3.0 REAGENTS

1. Ether or non-chlorinated solvents such as xylene
2. Distilled water

4.0 BACKGROUND & DEFINITIONS

See enclosures (FIGURES III, IV, IV, and TABLE 8)

5.0 METHOD

1. The optical parts of the microscope must be kept absolutely clean. All external surfaces and mechanical parts must also be cleaned periodically. Caution must be exercised during the use of solvents on any part of the microscope.

   Note: Alcohol and chlorinated solvents must never be used, which might destroy the cement between lens elements and the coatings on the exterior surfaces.

2. Dust on optical elements will degrade the image quality. Therefore, when not in use, the microscope must be covered with a dust cover. Special care should be taken to ensure that the tubes of the microscope are always closed with either an eyepiece or a dust plug.

3. When dust particles on the eyepiece will only give rise to patches in the image - which not every observer finds disturbing - a dirty objective front lens may hopelessly reduce the sharpness of the image, or at least its contrast. The eyepiece is close to the specimen, possibly to the immersion oil, but particularly to the hand operating the nosepiece. Due to these facts, the front lens of the objective is in special danger of becoming soil Even the lightest fingerprint may have grave consequences. Before starting important work, it is advisable - particularly if the microscope is not used by one observer alone. To unscrew every objective and check it carefully with the aid of a magnifier. Dirt is easily recognized if the objective is held so that the image of a light source is reflected from its plane surface. In the case of objectives that have a concave front lens, a different approach is indicated (to examine the surfaces of the front lens from the screw thread side). The remaining lens's elements can also be examined easily and any faults (cracks, "starting" of the cement) detected without difficulty.

4. The optical components of the polarized light microscope (PLM), arranged between the polars, are to be rotated from heavy mechanical stress in order to maintain the high degree of the optical isotropy of these components. This is to include things such as shock, fall, impact, tension, and pressure. For the same reason, the objectives of the microscope may be only slightly tightened against the contact face; stronger torsion would result in birefringence due to stress.

The cleaning of the objectives is confined to cleaning of the front and rear lenses with a dry brush and a rubber blower. Do not disassemble the objectives! Alcohol must not be used for cleaning purposes.

The knurled ring of the nosepiece should be turned, assuring that no objective is left in the light path during storage or transport of the microscope.

5. Should structures be found in the image which are suspected of being extraneous to the specimen, the fault may be traced as follows:

   If the trouble can be eliminated by slight adjustment of the condenser, the cause must be sought in the bulb of the lamp, the lamp condenser, or the filter in front of it. However, if adjustment of the condenser does not produce results, the next step is to turn the focusing adjustment, which should eliminate all faults due to soiling of the condenser front lenses or the specimen. If this does not lead anywhere, slightly turn first the objective and then the eyepiece and you will immediately notice in which case the foreign body follows the rotation.

   Dust particles are most clearly seen when the aperture diaphragm has been fully closed, because in this case the depth of focus is at its greatest.

6. In almost all cases, it will be sufficient to clean the outer lens faces with the aid of a grease-free brush. If necessary, wash first or with a frequently washed absolutely dust free linen rag (or chamois) and distilled water, (produced easily by breathing upon the surface to be cleaned).

   If an organic solvent cannot be dispensed with, it is advisable to use very little ether or xylene instead of water, but never alcohol, which might destroy the cement between lens elements.

   Ether is usually preferred because it evaporates most quickly and any harmful effect is thus less likely. Finally, residues are always removed with water as described above.

   Should compressed air be available for cleaning, be sure to use a filter of cotton wool.

7. If the air in the workroom continuously has a relative humidity of more than 60%, certain precautions should be taken to avoid fungus growth on the optical elements. Do not keep microscopes under plastic covers. Do not store them in cabinets, but ensure good ventilation. If necessary, ventilate with the aid of a fan. In a particularly humid climate it is advisable to keep optical parts in perfectly air proof containers provided with a disinfectant or in which, for instance, a lamp and a fan circulate air of 40-50^{o}C (100-120^{o}F). The grease applied for corrosion protection on mechanical parts without surface coatings (operational surfaces) have to be renewed from time to time, the old grease to be removed by a solvent first.

8. In case of electrical trouble, check first to see if the projection lamp is burnt out, or whether the fuse remains good.

   If the electrical trouble cannot be eliminated by replacement of the fuse or lamp, contact a repair representative.

9. Repairs and maintenance, other than discussed in 5.1 thru 5.8 should not be attempted by any laboratory staff. Appropriate arrangements can be made with a local vendor who repairs and services microscopes.

6.0 QUALITY CONTROL

1. An Instrument Record Card is maintained for each microscope. The information on this card is to include: model number, instrument type, serial number, I.D. number, location of instrument, checking/calibration frequency and responsibility, service representative and telephone number and a chronological record of all problems, repairs and calibrations.

2. An annual preventive maintenance visit is scheduled with a local service representative. This visit is beyond any service calls performed during the year.

7.0 REFERENCES

7.1 Reichert Scientific Instrument, “Reference Manual: Stereostar Zoom Stereoscopic Microscope”,
No. 560-101.1/86.

7.2 Jenoptick, Jena GMBH&DDR, “Instruction Manual: LABOVAL-2 Polarizing Microscope”, No. 30-g526-2.

7.3 F.K. Mollring, Microscopy from the Beginning, Carl Zeiss, Oberkochen, West Germany, 1968

7.4 W.J. Patzelt, Polarized-light microscopy, Ernst Leitz Wetzler GMBH, 1974.

7.5 Nikon Biological Microscopes, “Phase Contrast equipment Instructions:, Optiphot, Labophot Phase Contrast Microscope, (84.8A) H&E-10S and (85.6B) H&E-3S.

TABLE 8: SUMMARY OF DAILY MAINTENANCE

External surfaces of objectives

Eyepieces, condensers:	Dust: remove with soft, dry sable brush. Finger marks: remove immediately with a damp piece of linen or chamois leather; if necessary, use Light petrol. Resistant dirt: remove with damp piece of fine linen or chamois leather. Clean the lens first with a highly volatile solvent and allow all the solvent to evaporate. Additional cleaning with expanded polystyrene has been found very reliable with dirt difficult to remove. The type of white, granular expanded polystyrene will known as packing material for instruments in particularly suitable. Break a piece off, press it against the dry lens with a protection grain of the fresh fracture surface, and rotate it as coaxially as possible with the lens axis. This removes, even from the recessed rims of the lens mount. Even the most minute residues of immersion oil, skin grease and solvents, which otherwise spread across the surface of the lens and partly counteract the reflection-reducing action of the coating layers. Any adherent grains of expanded polystyrene can be simply blown away or dislodged with an absolutely clean slate brush specially reserved for this purpose. Cleaning is also possible with cotton wool wrapped around a wooden stick.
Oil immersion objectives:	Clean immediately after use: dab off with a piece of blotting paper or a small piece of linen. Remove the residual oil with a piece of linen soaked in light petrol. Final cleaning: If necessary, with a petrol-soaked piece of linen. Never use methylated spirits or alcohol.

Internal surfaces of eyepieces

Condensers:	Dust: Blow it away softly or clean with a sable brush.

FIGURE III: STEREOSCOPIC ZOOM MICROSCOPE

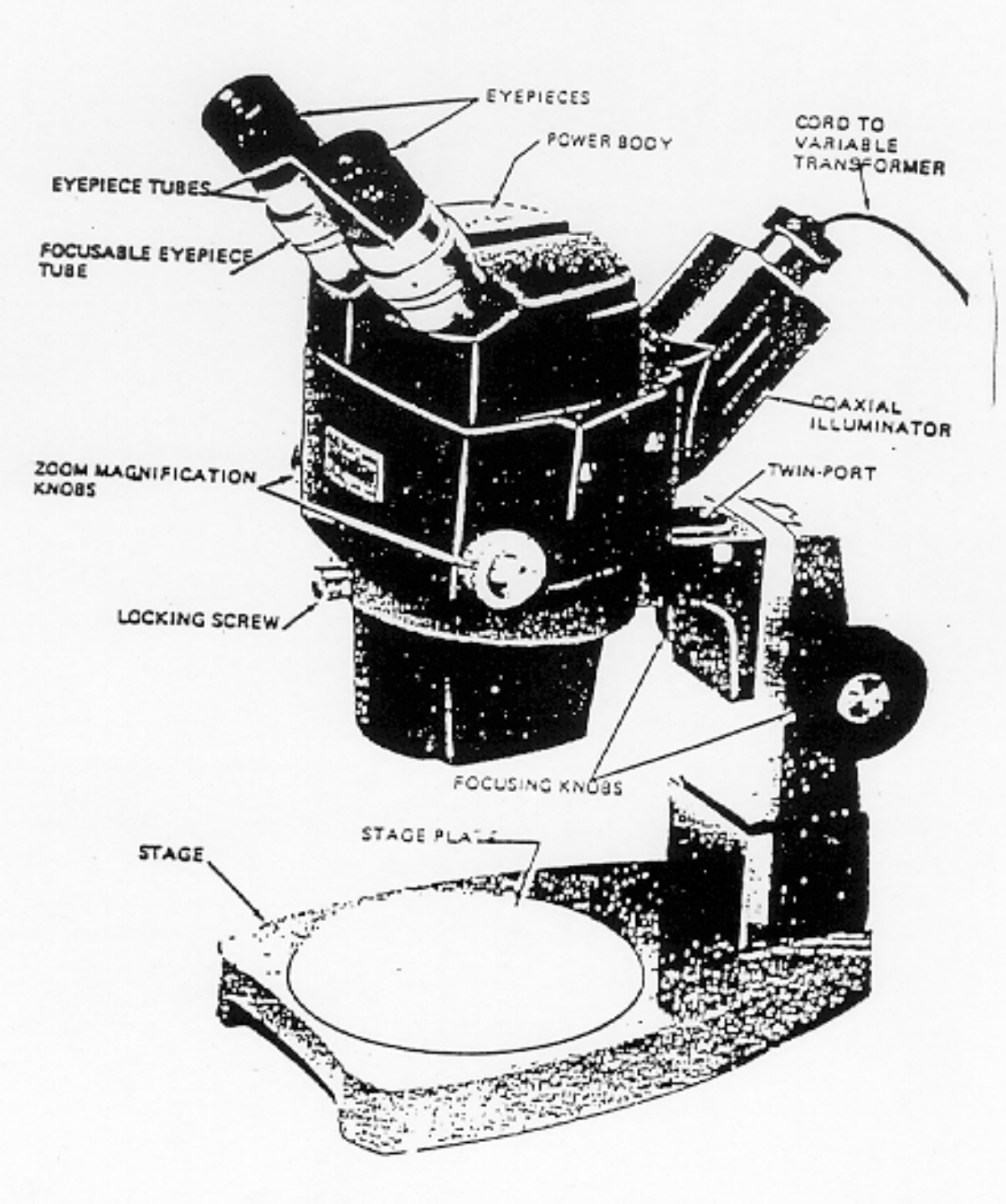
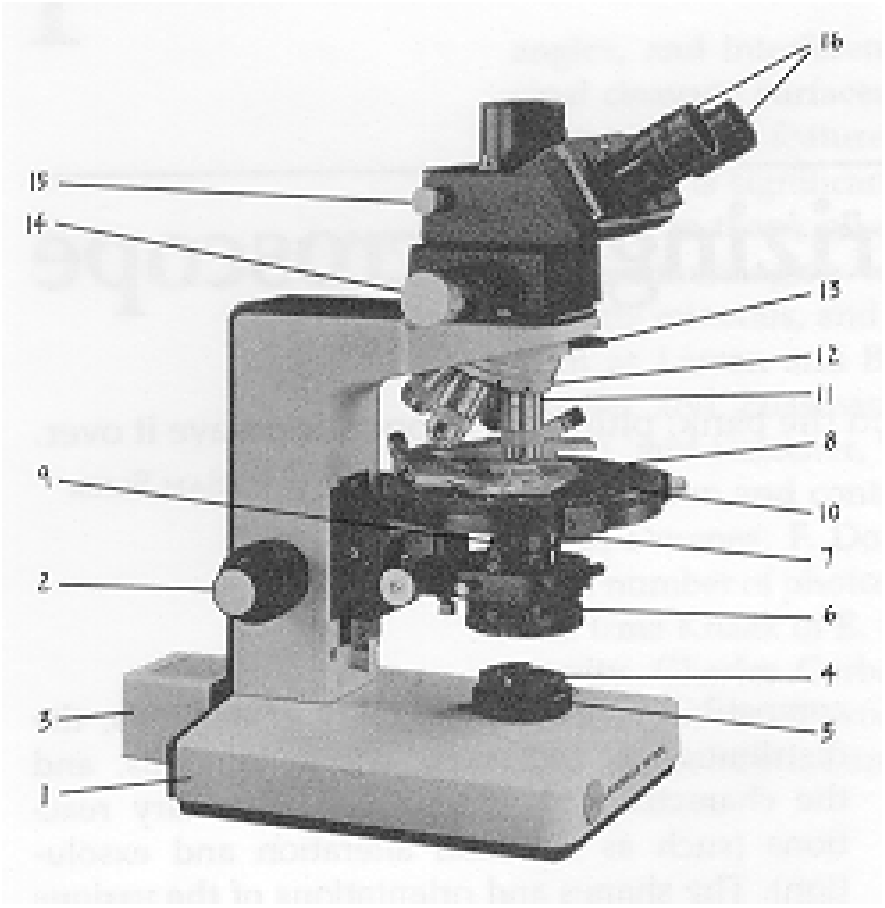
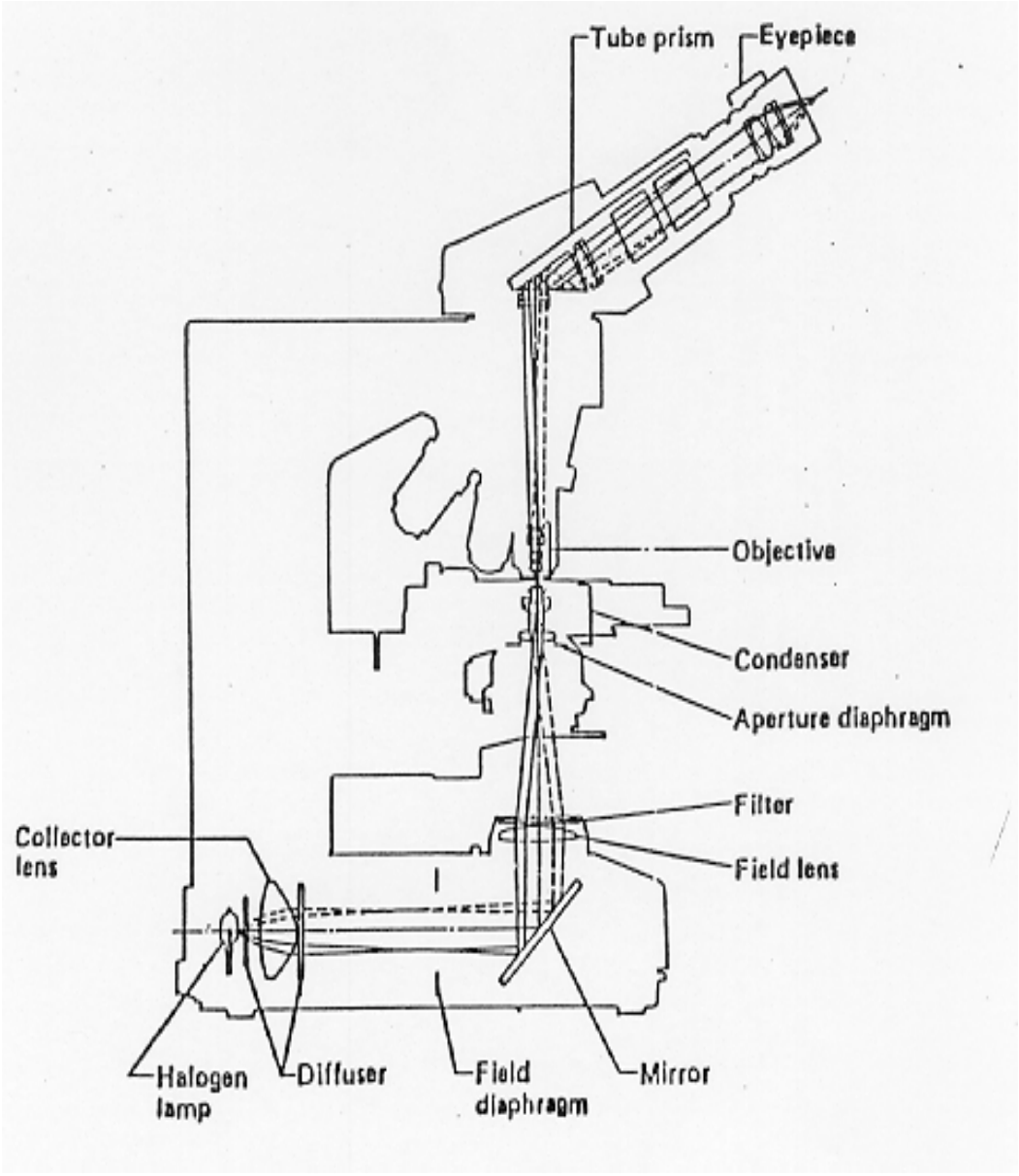


FIGURE IV: Polarizing Light Microscope



1. Base
2. Course and Fine Focus
3. Illumination Source
4. Daylight Filter
5. Field Iris Adjustment Control
6. Rotatable Polarizer
7. Substage Iris Diaphragm
8. Rotatable Stage
9. 1.0 - 1.5x Condenser Lens
10. Slide Holder
11. Objective Lenses
12. Revolving Nose Piece for Objectives
13. Analyzer
14. Bertrand Lens and Polerizer
15. Camera Shutter Knob
16. Oculars

FIGURE V: POLARIZING LIGHT MICROSCOPE



B.

ALIGNING THE OPTICAL SYSTEM

POLARIZED LIGHTMICROSCOPY (PLM)

1.0 GENERAL DISCUSSION

This procedure is to be used for aligning the optical system of the microscope for image formation of optimum quality, based on an illumination of the object with transmitted light.

2.0 EQUIPMENT

A compound microscope set up for polarized light microscopy to include a polarizer, analyzer, port for a wave retardation plate, a 360^{o} graduated rotating stage, substage condenser, lamp and lamp iris.

Objective Lenses: 10X, 20-25X, 40-50X and a dispersion-staining objective.
Ocular lenses: 10X minimum.
Eyepiece Reticule: Cross hair.
Compensator Plate: 550-millimicron retardation (first-order red or gypsum).

3.0 BACKGROUND & DEFINITIONS

3.1 Polarizing Light Microscope (PLM)

This is an instrument for qualitative and quantitative work in either transmitted or reflected light. The majority of PLMs are used to study the optical properties of substances (e.g., geology, mineralogy, metallurgy, fiber research) in linearly polarized light.

The substage polarizer converts the unpolarized light of the light microscope into linearly polarized light. Between the objective and the eyepiece, there is a second polarizer, called the analyzer. Both are in calibrated mounts rotatable to 180^{o} or 360^{o}, and they can be swung out of the path of light. All optical elements between the polarizers must be strain-free. The objectives are individually centered. The tubes permit the use of an Amici-Bertrand lens to observe interference figures in the exit pupil of the objectives (conoscopic path of rays). The conoscopic image (e.g., together with a gypsum or quartz plate red first-order) renders information on the type of crystal examined (e.g. uni-axial or bi-axial, positive or negative). Most investigations require the polarizers crossed (directions of oscillation of polarizer analyzer oriented at 90^{o} to each other), The crosshair of the oculars is congruent with these directions. The stage is of high precision and rotatable by 360^{o}.

3.2 Light Source

The light source is generally an incandescent lamp with a concentrated filament for low voltage, in a pre-centered, pre-focused socket. The collator forms a magnified image of the light source in the plane of the aperture stop of the condenser, thus allowing for a light source of small dimension and low heat dissipation. The light intensity required for highest magnifications and binocular observations is much higher than required at lower magnifications. reductions of the light intensity can be made by interposing a neutral density filter. A less preferable alternate would be lowering the voltage to the lamp, with a resultant change of its “color temperature”. This is not particularly objectionable for visual observations when a blue (daylight) filter is interposed in the light path, or for black and white photography, but must be considered when using color film. For polarized light microscopy a 100-watt incandescent lamp is preferred.

3.3 Condenser

The condenser, located in the substage, concentrates light on the specimen. The substage is generally permanently attached to the microscope. It should be equipped with a focusing device, for the condenser to focus the image of the field stop in the plane of the object. In addition, it should have a centering mount for the condenser, to center its optical axis with that of the objective (when the image of the field stop is concentric with the periphery of the field of view).

The numerical aperture of the condenser must be variable allowing for adjustment to that of the objective in order to obtain optimum resolution and contrast. The condenser images the field diaphragm into the specimen plane.

Selection of the components of the condenser system for illumination of the object from lowest to highest magnifications varies for different manufacturers. Optimum illumination conditions are explained in the manufacturer’s instrument booklet.

3.4 Objectives

The microscope objective is the most important component of the optical system as it essentially determines the image quality. the majority of microscopes use two stage magnifications (objective plus ocular). The objectives required are divided into different categories according to their optical design and type of correction (e.g., achromats, fluorite, apochromats).

3.5 Oculars

The ocular at the upper end of the microscope tube enlarges the immediate image formed by the objective. Together with an attachment camera, it can also form the final image on a film plane. All oculars are derived from the either Huygens or Ramsden type.

Due to the low numerical aperture of oculars, only astigmatism, field curvature, distortion and the chromatic difference to magnification need to be corrected. The commonly used type of oculars is the compensating eyepiece, whose chromatic difference of magnification is equal but opposite to that of the objective. For use with flatfield objectives, differently corrected oculars are used in order to fully utilize the performance of the flatfield objectives. Many eyepieces have a high eye point to permit the microscopist to wear his corrective glasses.

Special eyepieces are available for measurements (filar eyepieces, micrometer eyepieces, interference eyepieces, image-splitting eyepieces, and so on) and for teaching purposes (pointer eyepieces and demonstration eyepieces). The magnifications range from 5X to 25X, of which 8X, 10X, 12.5X and 15X eyepieces are mainly used.

4.0 METHOD

These instructions are specific for a binocular polarizing light microscope (PLM) equipped with the McCrone dispersion staining (DS) objective.

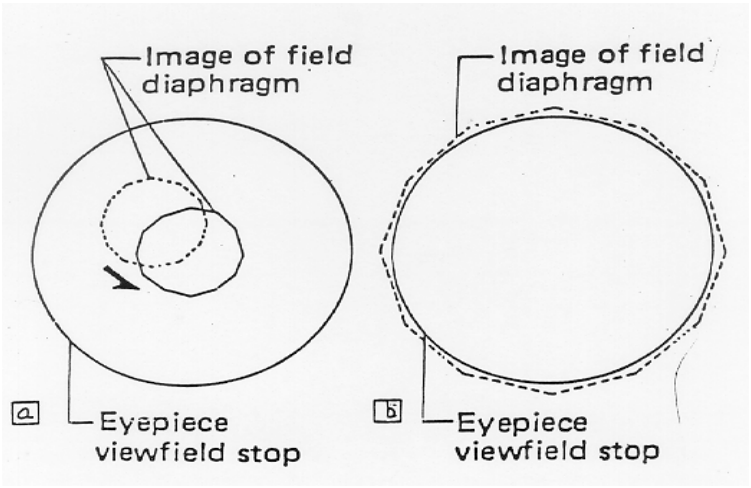
4.1 Alignment of the Illumination/Condenser System

Turn on the main switch and observe the light in the ground glass plate. If no light is observed, assure that the scope is plugged in, power is on and that the fuse and the bulb are in good condition. The illumination control should be increased to maximum. Assure uniform illumination in the object plane of the lower power objective.

Now an objective of medium magnification (10X) should be used so that when the field diaphragm is fully open, the entire field of view is illuminated. Focus the 10X objective and the 10X eyepiece on a specimen. The aperture diaphragm should be open and the field diaphragm closed just until its image is visible in the object field of the microscope (Figure VI). The condenser should be moved up and down until the image of the field diaphragm is as sharp as possible. The image will not be as sharp as that of the object, and color fringes may be visible at its edge (condenser too high the color is yellowish, too low and the color is bluish). When the field images is in best focus, center the image by means of the square socket wrenches.

With the image of the specimen and the centered field diaphragm in focus, the field diaphragm should be opened only so much that its image disappears at the edge of the field of view, but no further (Figure VI). When the diameter of the illuminated area is larger than the field of view, there is no increase in the intensity within this field, but the detrimental effect of glare increase.

FIGURE VI: OBJECTIVE DIAGRAMS



If the objective, required for a specific analysis, differs from that with which the alignment was made, it should now be placed in the light path and the alignment checked.

Note: Never swing an object into place by handling the objective directly. Always use the knurled ring of the nosepiece to rotate an objective into place. Unnecessary stress on the objective can create strain on the objective lenses, resulting in loss of image quality.

Insert the daylight filter into the filter holder and adjust lamp intensity according to individual needs.

The condenser should always remain in the positions as adjusted when objectives are changed, except for correction of concentration when necessary. Any displacement of the condenser in the direction of the optical axis (e.g., for varying the light intensity within the field of view), must be avoided. Many users of microscopes adhere to this incorrect procedure. Variations of the light intensity should be made by interposing a neutral density filter, or sometimes by varying the voltage of the light source (within certain limits and when variations in color balance are not important). When all the adjustments as described have been completed, and the aperture stop is still fully open, the image will appear with low contrast. The aperture stop should now be closed very gradually while the observer critically evaluates the image quality to detect slight improvements. You will notice that at a somewhat reduced numerical aperture, a fairly abrupt change in the optical quality of the image occurs. The contrast between the smallest resolved object detail increases slightly, the colors of stained objects appear slightly more “saturated”; there is a slight increase in the depth of field. At that position of the aperture stop, the image quality is at an optimum, and the microscope performs to the limit of its capacity with the selected optical components. This procedure is performed daily (see Calibration and Contamination Worksheet).

4.2 Centering the Objectives

Place a centering plate onto the specimen stage and focus it in the eyepiece with the aid of 10/0.25 achromatic objective. For this purpose, lift the stage carrier to the upper stop by actuating the combined control. Turning the control within the range of the two stops causes fine adjustment, turning the control beyond the stops causes coarse adjustment of the specimen. Bring the center of the centering cross and that of the eyepiece crosshair into coincidence, either by hand or with the aid of the objective traverser. Correct any deviations from this position occurring when the state is rotated, half by shifting specimen and half by turning the centering screws by means of the socket wrenches. Repeat this procedure until both crosshair centers remain in coincidence when the stage is rotated. According to the stage's center of rotation found in this way, adjust other objectives by means of centering screws so that the centers of the stage centering cross and eyepiece crosshair are in coincidence.

5.0 QUALITY CONTROL

1. An Instrument Record Card is maintained for each microscope. Information on this card is to include: model number, instrument type, serial number, EMSL I.D. number, location of instrument, checking/calibration frequency and responsibility, service representative and telephone number and a chronological record of all problems repairs and calibrations.

2. An annual preventive maintenance visit is scheduled with a local representative. This visit is above and beyond any service calls performed during the year.

6.0 REFERENCES

6.1 Zieler, HW., The Optical Performance of the Light Microscope, Parts 1 and 2 Microscope Publications, Ltd., 1974

6.2 Considine, D.M. (Editor), Van Nostrand's Scientific Encyclopedia, Volume II, Van Nostrand Reinhold Co., 1983

6.3 Jenoptick Jena GmbH DDR, "Instruction Manual" LABOVAL-2 Polarizing Microscope", No. 30-G526-2

C.

CALIBRATION OF REFRACTIVE INDEX LIQUIDS

1.0 OVERVIEW

Refractive index is one of the primary characteristics for the identification of asbestos. In order to accurately identify the RI of the particle it is important to know if the refractive index oil is within an acceptable range. (0.004) Two methods for the calibration of RI oils are documented here. The first is using an Abbe refractometer. The second is with Cargille glass standards and a method written by Dr. Shu-Chun Su.

The Refractometer:

1. Follow the manufacturers specifications for calibrating the instrument at 25^{o}c using the water bath to stabilize the temperature.
2. After the instrument is calibrated, clean the prisms and stage.
3. Place 1 to 2 drops of RI oil onto the stage and allow to sit for one minute to stabilize the temperature of the oil.
4. Following the manufacturers instructions read and record the RI of the oil. It should be $\pm$ 0.004 the stated RI.

The Optical Glass Method: by Shu-Chun Su

2.0 MATERIALS & EQUIPMENT

1. Calibrated optical glass standards
2. Polarized Light Microscope with Dispersion Staining Objective
3. Thermometer with 1 degree divisions

3.0 METHODOLOGY

There are four steps in performing this calibration using optical glass standards and dispersion staining.

1. Temperature

Find out what is the temperature, *t*, of the RI liquid to be calibrated. Generally, it is assumed that the temperature of the liquid is in equilibrium with the room temperature. If this is the case, the room temperature can be measured to represent the liquid temperature.

2. Determine λ_o

Compare the liquid RI with that of an optical glass with accurately and precisely known RI's at various wavelengths to determine at which wavelength their RI values are equal. This wavelength is called the *matching wavelength*, λ_o, which can be derived from the dispersion staining color exhibited by the glass particles (See table 6).

3. Determine n^L_D

Calculate n^L_D the RI value of the liquid at the wavelength of Fraunhofer spectral D line or 589nm and temperature *t* by using the following equation:

$$n^L_D = n^S_D - (\Delta^L - \Delta^S) \bullet k_D$$

Where: n^L_D = the refractive index of the liquid at 589nm and t^oC

n^S_D = the RI value of the optical glass at 589nm, which is listed in the optical constant table supplied with every set of glasses

Δ^L = The dispersion coefficient, $(n_F - n_c)$, of the liquid, which is printed on the bottle label

Δ^S = The dispersion coefficient, $(n_F - n_c)$, of the liquid, which is listed in the optical constant table supplied with every set of glasses

k_D = a coefficient determined by the matching wavelength λ_o as listed in Table 6 (Reference 3)

4. Determine n_D^{25deg}

If the room temperature is not 25°C, apply temperature correction to n^t_D to find n_D^{25deg}, the RI value of the liquid at 25°C and 589nm. The equation used for temperature correction is;

$$n_D^{25} = n^L_D + (25-t) \bullet dn/dt,$$

Where: n_D^{25} = the RI of the liquid at 25°C and 589 nm
n^L_D = the RI value of the liquid at 589 nm and *t*°C
t = the temperature in centigrade at which the calibration is performed
dn/dt = the temperature coefficient of the liquid, which is printed on the bottle label and is always a negative value for RI liquids.

No temperature correction is necessary for the glass if the temperature is within the range of 25° ± 10°C, because the temperature coefficient of Cargille glasses are so small that the resultant variation of RI will not exceed ± 0.0001.

4.0 PROCEDURE

1. Measure and record the room temperature with an accuracy of ± 2°C.
2. Select an optical glass standard whose RI is closest to that of the liquid to be calibrated, for example 1.55 glass for 1.550 liquid, 1.60 glass for 1.605 liquid.
3. Mount the glass in the liquid and observe the *predominant* dispersion staining (DS) color in central stop mode.
4. Convert the observed CS-DS color into the corresponding matching wavelength by referring to a DS color chart.
5. Convert λ_o and *t* into the corresponding n_D^{25} by referring to an appropriate conversion table:

Table 9: Refractive Index Oil Conversion Table

Cargille RI liquid		Cargille Optical Glass Standard		Conversion
Nominal or labeled n_D^{25}	Series	Nominal or labeled RI	Lot No.	Table No.
1.550	E	1.55	B C	3 4
1.605	E	1.60	B	5
1.680	B	1.68	B C	6 7
1.700	B	1.70	B D	8 9

6. Compare the resulted n_D^{25} with the nominal or labeled value on the bottle of the RI liquid. If the absolute difference between the two values is less than or equal to 0.0044, this liquid can be used for bulk sample analysis. If the difference is greater than 0.004 then the liquid should be discarded.
7. Record the calibration result.

EXAMPLE:

If the Cargille 1.55 glass of Lot C is used to calibrate a Cargille 1.550 (Series E) RI Liquid at the room temperature of 21^o and the predominant CS-DS color observed is bluish-purple, the corresponding λ_o is then 570nm. λ_o = 570 and *t* =21 yield 1.548, which is the average of 1.5475 and 1.5485.

The calibration result shows that the RI of this bottle of 1.550 liquid at 589 nm and 25^o is *actually* 1.548. Because the difference is 0.002, using $\pm$0.004 criterion, this RI liquid is considered acceptable for use in bulk sample analysis.

Charts are located in the appendix.

5.0 QUALITY CONTROL

1. A Refractive Index Liquid calibration card (Figure VII) is maintained for each liquid. Information on this card is to include: manufacturer, catalog number, date received, received by whom, date RI verified, verified by whom, calibration responsibility and frequency, calibration instruction number, supplier, address and telephone number.

   The reverse side is to include: reference refractive index (n) and temperatures (T), measured n and room temperature (T_R), the corrected n, and the temperature coefficient.

2. A label containing the manufacturer's name, the initial calibration date, expiration date, and initials of the person performing the calibration will be attached to each bottle of RI liquid. The expiration date is 6 months from the time the oil was initially opened.

3. A RI liquid exhibiting a change in the refractive index of greater than = 0.005 must be replaced.

4. All RI liquids used daily (HD series 1.550, 1.605, 1.630, 1.680, 1.700) will be calibrated monthly.

5. The PLM QA/QC Manager at the main lab will receive copies of the data on a quarterly basis.

6.0 CALCULATIONS

As an example of the use of Equation 1, assume a mineral has been matched in index to a particular RI liquid (n_D = 1.530, T = 20°C, dn/dt = 3.00×10^{-4}), and the room temperature (TR) at the time of the match is 25° C. The actual refractive index of the liquid (and therefore the mineral) at the time of the match is:

$$n_{corr} = n_D + (T_R - T)\ dn\ dt$$
$$n_{corr} = 1.530 + (25 - 20)\ (-0.0003)$$
$$n_{corr} = 1.5285$$

The index reported for the mineral is then rounded off to 1.528. Reporting a refractive index of 1.5285 implies a greater accuracy than is attainable by this method ($\pm$ 0.002).

7.0 REFERENCES

1. El-Hinnawi, Essam E., Methods in Chemical and Mineral Microscopy, Elsevier Publishing Co., 1966

2. Nesse, William D., Introduction to Optical Mineralogy, Oxform University Press,1986

3. Bloss, F. Donald, An Introduction to the Methods of Optical Crysallography. Holt, Rinehart Winston, 1972

4. Schaeffer, Harold F., Microscopy for Chemists, Dover Publications, Inc.1953

5. Air pollution Training Institute, “Course 420 - Air Pollution Microscopy Laboratory Manual”, June 1979

FIGURE VII - REFRACTIVE INDEX LIQUID CALIBRATION CARD

Manufacturer: _____ Date Recv'd: _____

Catalog #: _____ Recv'd by: _____

Batch #: _____ Date R.I. verified: _____

Assigned to: _____ Verified by: _____

Calibration Responsibility: _____

Calibration Instruction #: _____

Supplier: _____

Telephone #: _____ Address: _____

_____

Comments: _____

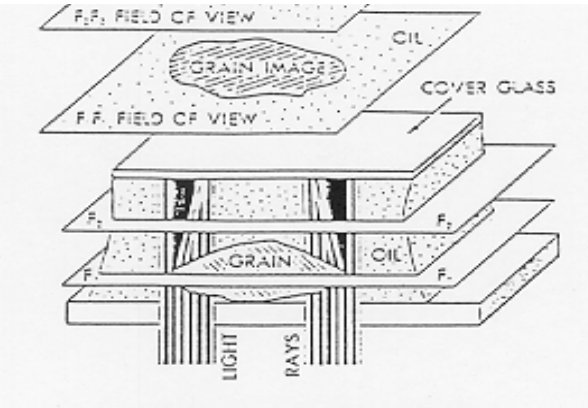
Reference			Measured		Corrected	Calibration		Accept/
n_D	T	dn/dt	n	T	n	Date	Initials	Reject

TABLE 10: THE PROBABLE ERROR IN REFRACTIVE INDEX DETERMINATION

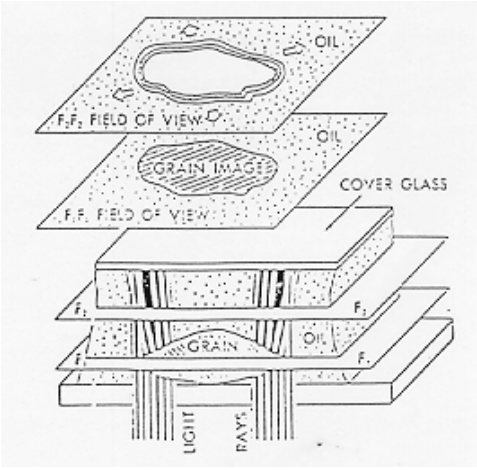
METHOD USED	PROBABLE ERROR (±)
Central illumination	0.002 - 0.003
Becke line colors	0.002
Oblique illumination	
Single diaphragm	0.002 - 0.003
Double diaphragm	0.0001 - 0.0002
Dark-field immersion method	0.001 - 0.002
Focal screening method	
Apertural screening	0.001 - 0.002
Unilateral screening	0.0001 - 0.002
Central screening	0.001
Variation methods	
Thermal variation	0.001
Dispersion	0.001
Double variation	0.0004
Glass method	0.0002 - 0.0003

FIGURE VIII: BECKE LINE

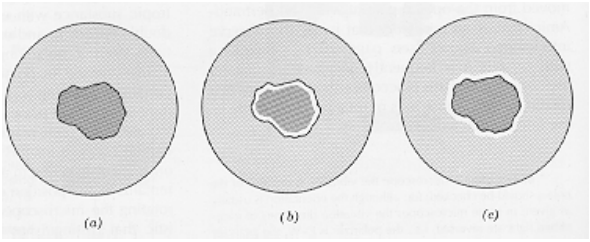
A. GRAIN INDEX IS GREATER THEN OIL'S



B. GRAIN INDEX IS LESS THAN OIL'S

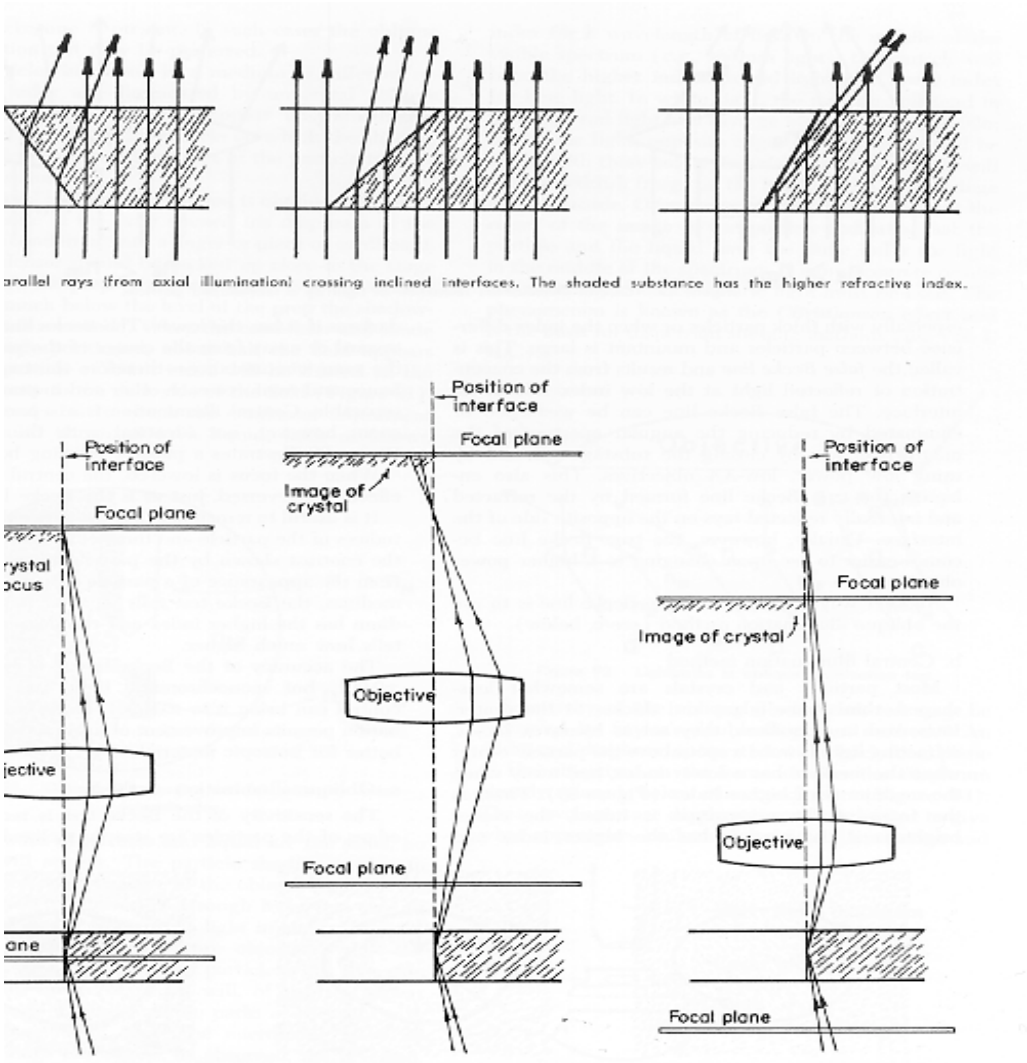


The disposition of movement of Becke Lines, field of view, F_1F_1 is that observed if the microscope is focused upon plan F_1F_1 to produce the sharpest image; here the Becke Lines are not too obvious. However, if observed while the microscope is racked upward toward a focus on plan F_2F_2, the Becke Lines become increasingly apparent, the brighter lines moving (as indicated by the hollow arrows) toward the medium having the greater refractive index.



a) oil = particle: b) oil < particle: c) oil > particle

FIGURE IX: BECKE LINE MOVEMENT



iagram (after Wahlstrom) illustrating lateral movement of the Becke line as focus is changed. Two rays reflected from the s of a vertical interface are shown to represent the Becke line. (Other rays contributing to the Becke line are omitted for simplicity.)

D.

ASBESTOS & NONASBESTOS MINERAL DESCRIPTIVES

Mineral Information

Asbestos:	Chrysotile	*Non-Asbestos:*	Augite
	Anthophyllite		Diopside
	Actinolite		Hedenbergite
	Tremolite		Enstatite
	Amosite		Hyperstene
	Crocidolite		Halloysite
			Kaolinite
			Palygorskite
			Talc
			Wollastonite

ASBESTOS MINERALS

ANTIGORITE, LIZARDITE, & CHRYSOTILE

Class: Phyllosilicate
Group: Serpentine

Antigorite	$Mg_3Si_2O_5(OH)_4$
Chrysotile	$Mg_3Si_2O_5(OH)_4$

Color: various shades of green; brownish, gray, white, or yellow: translucent to opaque.
Luster: waxy or greasy; fibrous varieties silky; massive varieties earthy.
Transparency: translucent to opaque
Habit: Occurs mainly as fibrous chrysotile or as lamellar or platy antigorite.
Antigorite & Lizardite: Massive, fine grained, lamellar
Chrysotile: Fibrous
Hardness / Specific Gravity: variable 2.5 -5.0 / 2.5 - 2.6
Cleavage / Fracture: Basal, perfect / conical, splintery

Antigorite

Optics: (+)
Pleochroism = none
Refractive Indices: 1.55-1.56
Birefringence: low (0.007)
Extinction Angle: 0, 90°

Crystallography: Monoclinic; 2/m (Orthorhombic Antigorite & Chrysotile are rare)
a=5.30; b=9.20; c=7.46; *B*=91°24' a:b:c=0.567:1:0.811
Z= 2. d's: 7.30(10), 3.63(8), 2.52(2), 2.42(2), 2.19(1)
Composition: MgO 43, SiO_2 44.1, H_2O 12.9
Ni & Fe may sub. for Mg and Al for Si
Cleavage: {001} basal, perfect. Corrugated, finite layers parallel to {001}

Chrysotile

Optics: (+)
Pleochroism: none
Refractive Indices: x= 1.55-1.56 y= 1.545-1.556
Birefringence: Moderate (0.011)
Extinction angle: 0- 90°

Crystallography: monoclinic; 2/ m (Orthorhombic Antigorite & Chrysotile are rare)
a=5.34 b=9.25 c=14.65 β=93°16' a:b:c=0.577:1:1.584
Z= 4.
Composition: MgO 43, SiO_2 44.1, H_2O 12.9%
Ni & Fe may sub. for Mg and Al for Si
K - Factor Ratios: Na=0.00, Mg=0.70, Si=1.0, Ca=0.00, Fe<0.02
Cleavage: none. A mismatch between the t and o layers causes the structure to scroll forming cylindrical tubes

Distinguishing features:
color, luster, smooth, greasy feel.

Optically:

(chrysotile asbestos) wavy bundles under polarized light and scanning electron microscope; hollow, tubular fibrils in transmission electron microscope. Antigorite; plate aggregates under PLM & SEM; Plate aggregates with perfect cleavage under TEM.

Occurrence:

Serpentines are secondary minerals formed from other deposits such as olivine, orthopyroxene, amphibole, and magnesium silicates. They are found in both igneous and metamorphic rock formations.

ANTHOPHYLLITE

Class: Inosilicate
Group: Amphibole

Anthophyllite (Mg, Fe)7 Si8 O22 (OH)2

Color: white, gray green, & brown. (brown predominates in cummingtonite series.)
Luster: vitreous, silky for fibrous varieties.
Transparency: Translucent. Transmits light on thin edges.
Twinning: Common.
Habit:: Orthorhombic; 2/m 2/m 2/m. Distinct crystals are rare. Commonly lamellar, fibrous, radiating, slender & prismatic or aggregates of fibrous crystals.
Hardness / Specific Gravity: 5.0 - 6.0 / 2.8 - 3.4
Cleavage / Fracture: {210} prismatic, perfect.

Optics: (+)
Pleochroism : none
α=1.60-1.69, β=1.61-1.71, γ=1.62-1.72. Indices will increase with Fe.
2V=70-100^{o} , X=a, Y=b. absorption Z > Y and X.
Birefringence: moderate (0.02)
Pleochroism: none

Crystallography:
Pnma; a=18.56; b=18.08; c=5.28; β= 90^{o}0' a:b:c=1.027:1:0.292
Z= 4. d's: 8.26(6), 3.65(4), 3.24(6), 3.05(10), 2.84(4)
(210)^(2-10) = 55^{o}

Composition:
Forms a solid solution series from Mg7Si8O22(OH)2 to Fe2Mg5Si8O22(OH)2
At higher Fe concentrations, cummingtonite results.

Distinguishing Features:
Light brown color. Needle like or fibrous, often radiating habit. Indistinguishable from cummingtonite-grunerite series with out microanalysis. Lack of reaction to HCl. Thin plate like fibers under TEM. Anthophyllite can form in the same environment as talc and during elemental analysis may contain similar Mg to Si ratios. In this scenario, the diffraction pattern is the key to distinguishing between the two structures. Talc has a hexagonal pattern while anthophyllite primarily yields a series of parallel spots clearly marking the (h,k,0) and the (h,k,l) rows. In addition, the c axis may contain alternate or (missing) spots as in the [010], [140] & [320] zone axis. Fusible at 5 to a magnetic black enamel.

CUMMINGTONITE
GRUNERITE (Amosite Asbestos)

Class: Inosilicate
Group: Amphibole

Cummingtonite	(Mg, Fe)7 Si8 O22 (OH)2
Grunerite	Fe7 Si8 O22 (OH)2

Color: White-gray. Varying shades of lt. brown are common in cummingtonite series.
Transparency: Translucent. Transmits light on thin edges.
Luster: Vitreous. Silky in fibrous form.
Twinning: Common.
Habit:: Monoclinic; (Cummingtonite 2/m; Grunerite C2/m). Distinct crystals are rare. Commonly Lamellar, fibrous, radiating, slender & prismatic or aggregates of fibrous crystals.
Hardness / Specific Gravity: 5.0 - 6.0 / 2.8 - 3.6
Cleavage / Fracture: {110} prismatic, perfect.

Optics: (-) grunerite; (+) cummingtonite; (heated grunerite as amosite)
α= 1.65-1.69 (-1.74); β= 1.67-1.71 (-1.95); γ= 1.69-1.73
2V large; Y = b; Z ^ c= 13-20^{o}; r<v for cummingtonite; r>v for grunerite.
Pleochroism: none (May appear yellow to red)
Birefringence: Moderate 0.03 (up to 0.19)

Crystallography: Monoclinic
a= 9.59, b= 18.44, c= 5.34, β= 102^{o}0'. Unit cell length decreases with increase in Mg.
a:b:c= 0.520:1:0.289
Z= 2. d's; 9.21(5), 8.33(10), 3.07(8), 2.76(9), 2.51(6)

Composition:
A solid solution series exists between cummingtonite and grunerite starting from approximately Fe2 Mg5 Si8 O22 (OH)2 to Fe7 Si8 O22 (OH)2. Members with an atomic percentage of Mg > Fe is referred to as cummingtonite. 30 atomic percent is used as a division between members. Al2O may range up to 0.4 weight percent and CaO as high as 0.9 percent.

Distinguishing Features:
Light brown color. Needle like or fibrous, often radiating habit. Indistinguishable from anthophyllite with the naked eye. Lack of reaction to HCl. Fe up to 80% that of Si. Thin plate like fibers under TEM with distinctive, easily obtained diffraction patterns.

GLAUCOPHANE
RIEBECKITE (Crocidolite Asbestos)

Class: Phyllosilicate
Group: Amphibole

Glaucophane Na2 Mg3 Al2 Si8 O22 (OH)2
Riebeckite Na2 $Fe3^{2+}$ $Fe2^{3+}$ Si8 O22 (OH)2

Color: Glaucophane is blue-grey, to lavender-blue. Riebeckite is dark blue to blue-black. Darker with increased Fe content.
Transparency: Translucent
Luster: Vitreous, silky in fibrous varieties.
Habit: Monoclinic; 2/m Slender acicular crystals, aggregated, or in fibrous form.(Crocidolite Asbestos)
Hardness / Specific Gravity: 5.5 - 6.0 / 3.0 - 3.4
Cleavage / Fracture: {110} prismatic, good / Uneven.

Optics: (-)
Pleochroism = blue to blue-grey. X<Y<Z
Refractive Indices: α=1.61-1.70, β=1.62-1.71, γ=1.63-1.72
Birefringence: moderate (0.045)
Extinction Angle: 0, 90^{o}
Glaucophane, (Riebeckite)

Crystallography:
C2/m; a=9.58 (9.769); b=17.80 (18.048); c=5.30 (5.335);
a=90^{o}, β=103^{o}48' (103^{o}59'); a:b:c = 0.538:1:0.298
Z= 2. d's: 8.42(10), 4.52(5), 3.43(6), 3.09(8), 2.72(10).
2V = 40 - 90^{o}; Y = b, Z^c = 8^{o}; X < Y < Z.

Composition:

The composition changes with the substitution of Fe^{2+} for Mg and Fe^{3+} for AL. Care should be taken during chemical analysis as the elemental make up of Riebeckite may closely resemble that of Grunerite. The distinguishing feature that is most prominent is the occurrence of Na. Also known as crocidolite in its fibrous form.

Occurrence:

Glaucophane is found in metamorphic rocks such as marble and schists. Reibeckite most commonly occurs in igneous rocks. Tiger-eye is an ornamental stone where quartz has taken the place of crocidolite while preserving a fibrous texture.

ACTINOLITE
TREMOLITE

Class: Inosilicate
Group: Amphibole

Actinolite Ca2 (Mg, Fe)5 Si8 O22 (OH)2
Tremolite Ca2 Mg5 Si8 O22 (OH)2

Color: white to gray (tremolite), light to dark green (actinolite)
Luster: vitreous, often with silky sheen.
Transparency: transparent to translucent
Twinning: common
Habit:: usually aggregates of fibrous crystals. Individual crystals are rare.
Hardness / Specific Gravity: 5-6 / 3.0 - 3.3
Cleavage / Fracture: {110} perfect

Optics:

	SHAPE	PLEOCHROMISM	INDICES	BIREFRINGENCE	EXTINCTION
TREMOLITE	fibrous	none	1.60 - 1.62	mod. 0.02	0 - 5
	non-fibrous	none	1.60 - 1.62	mod. 0.02	0 - 20
ACTINOLITE	fibrous	none	1.63 - 1.65	mod. 0.02	0 - 5
	non-fibrous	none	1.63 - 1.65	mod. 0.02	0 - 16

Crystallography: Monoclinic; 2/m
Actinolite, (Tremolite)
C2/m; a=9.86 (9.84); b=18.11 (18.02); c=5.34 (5.27);
a=90^{o} β=105^{o} (104^{o}95'); a:b:c= 0.545:1:0.293
Z= 2. d's: 8.38(10), 3.27(8), 3.12(10), 2.81(5), 2.71(9)
Orientation: 2V = 80^{o}; Y = b, Z^c = 15^{o}

Composition:
There is a solid solution series from tremolite to ferroactinolite.
(Ca2Fe5Mg5Si8O22(OH)2.)

Distinguishing features:
Radiating tremolite may resemble wollastonite however can be distinguished by a lack of reaction with HCl. Actinolite is lighter in color than the majority of hornblende's.

Optically
Thin strait fibers under PLM & SEM analysis with a slender prismatic habit being evident. TEM analysis reveals platy bundles with good cleavage giving the edges a choppy look. The chemical composition of the structure is a good indication of its nature.

Occurrence:

Tremolite commonly occurs in metamorphosed dolomitic limestone's and may, in rare instances, form in some serpentines. Radiating tremolite may resemble wollastonite, however it shows no reaction to HCl that is a distinguishing feature of wollastonite. A felted aggregate of tremolite fibers is also known as *mountain leather* or *mountain cork*.

Actinolite usually forms in green schist's produced from low to medium grade metamorphism. It also coexists with quartz and epidote. A tough, compact variety that supplies much of the material for *jade* is called *nephrite* (*jadeite*).

NONASBESTOS MINERALS

AUGITE
DIOPSIDE
HEDENBERGITE

Class: Inosilicate
Group: Pyroxene (Clinopyroxene)

Augite (Ca,Na)(Mg,Fe,Al)(Si,Al)2O6
Diopside CaMgSi2O6
Hedenbergite CaFeSi2O6

Color: White to pale green & black. Darkness increasing with Fe towards the black of augite.
Luster: vitreous
Transparency: transparent, translucent to opaque
Twinning: common
Habit: crystals usually square or eight-sided cross section; also massive, granular, lamellar
Hardness / Specific Gravity: 5.5 - 6.5 / 3.2 - 3.6
Cleavage / Fracture: {110}, at 87^{o} & 93^{o} imperfect. Frequently parting on {001} and less commonly on {100}. Fracture is uneven.

Optics: (+)
Pleochroism : varies in darker members only.
(X=pale green, Y=Yellow-green, Z=dark green)
Refractive Indices: α=1.66-1.73, β=1.67-1.73, γ=1.69-1.75
2V=55-65^{o} , Y=b, Z^c=39-48^{o}; r>v.

Crystallography: Monoclinic; .2/m
C2/c; a=9.73; b=8.91; c=5.25; $\beta=105^{o}50'$; a:b:c=1.092:1:0.589
Augite a=9.755; b=8.928; c=5.204; $\beta=106^{o}11'$
Diopside a=9.761; b=8.926; c=5.258; $\beta=105^{o}79'$
Hedenburgite a=9.827; b=8.994; c=5.261; $\beta=105^{o}52'$
Z= 4. d's: 3.23(8), 2.98(10), 2.94(7), 2.53(4), 1.748(4).
m(110)^m'(1-10)=$92^{o}50'$, c(001)^(p(111)=$33^{o}50'$,
s'(-111)^s'(-1-11)=$59^{o}11'$, c(001)^a(100)=$74^{o}10'$.

Composition: A solid solution series exists between all members of this group toward acmite-augite. A solid solution series exists between diopside and hedenbergite with Mg and Fe^{2+} substitution. Most of the members of this group have a 1 - 3% Al_2O_3 content. Augite has substitution in Mg - Fe^{2+} and Al substitutes for Mg, Fe^{2+} & Si. Na and Ti may also be present.

Distinguishing Features:

Two cleavages almost at right angles (87 & 93^{o}). Augite is usually a darker green than diopside. Insoluble in acids. Fusible at 4 to a green glass.

Occurrence:

Diopside and hedenbergite are native to metamorphic rock formations. Diopside occurs in impure limestone and occasionally in basaltic igneous rocks.

ENSTATITE
HYPERSTHENE

Class: Inosilicate
Group: Pyroxene

Enstatite MgSiO3
Hyperstene (Mg, Fe)SiO3

Color: Pale green to dark brownish green, yellowish, or greenish-white.
Luster: vitreous, pearly on cleavage surfaces. Translucent to opaque.
Transparency: Translucent to opaque
Habit: Crystals are prismatic; usually grains or massive; fibrous , lamellar
Hardness / Specific Gravity: 5.0 - 6.0 / 3.2 - 4.0
Cleavage / Fracture: Prismatic, good {210}, {100} & {001} are less common. Fracture is uneven.

Optics: enstatite (+), Hyperstene (-)
Pleochroism : varies
Refractive Indices: α=1.650-1.715, β=1.653-1.728, γ=1.658-1.731
2V=35-50^{o} , X=b, Z=c. Indices increase with Fe.

Crystallography: Orthorhombic; 2/m 2/m 2/m
Pbca; a=18.22; b=8.81; c=5.21; β= 90.00 a:b:c=2.068:1:0.590
Z= 8. d's: 3.17(10), 2.94(4), 2.87(9), 2.53(4), 2.49(5)
(210)^(2-10) = 91^{o}44'

Composition:
Fe:Mg ratios rarely exceed 1:1. Pure enstatite contains no Fe. Mg may be substituted for by Fe $^{2+}$ up to 90%.

Distinguishing features:
Two cleavages may appear to intersect at or near right angles. Varieties that are high in Fe content may appear almost black, similar to augite in appearance. Enstatite-Hyperstene is commonly recognized by its cleavage and luster. Thin, nearly infusible, edge may be rounded. Fibers are common under high magnification. It is commonly located in metamorphic rock and may coexist with amphiboles.

Occurrence:
Igneous rock such as peroxenite. They may give way to amphiboles especially anthophyllite in metamorphic rocks.

HALLOYSITE

Class: Phyllosilicate
Group: Clay Mineral

Halloysite Al2 Si_2 O_5 $(OH)_4$ & Al_2 Si_2 O_5 $(OH)_4 \bullet 2H_2O$ *dehydrates to the first composition with the loss of inner layer water molecules.*

Color: white
*Luste*r: dull, earthy
Habit: massive, fibrous with tubular morphology
Hardness / Specific Gravity: 1.0 - 2.0 / 2.0 - 2.2

Optics: (+)
Pleochroism: none
Refractive Indices: α= 1.539, β= 1.589, γ= 1.589
Birefringence: moderate
Extinction Angle: 0, 90^o

Crystallography: Monoclinic
c= 5.242

Distinguishing features:
The high presents of Al. This Aluminum Silicate may appear in fibrous form with a tubular morphology.

Occurrence:
See kaolinite

KAOLINITE

Class: Phyllosilicate
Group: Clay Mineral

Kaolinite $Al_2 Si_2 O_5 (OH)_4$

Color: white; may be colored by impurities.
Luster: dull, earthy
Habit: Usually white earthy masses may be colored by surrounding material. Crystals are usually hexagonal plates.
Hardness / Specific Gravity: 2 - 2.5 / 2.6 - 2.7
Cleavage / Fracture: {001} perfect, rhombic or hexagonal plates

Optics: (+)
pleochroism: none
Refractive Indices: α=1.539, β=1.589, γ=1.589
Birefringence: moderate (0.045)
Extinction Angle: 0, 90^o

Crystallography: Triclinic; -1
P-1; a=5.27; b=9.12; c=18.85; $\beta=100^o0'$ a:b:c=0.578:1:2.067
Z= 4. d's: 9.34(10), 4.66(9), 3.12(10), 2.48(7), 1.87(4)
Orientation: $2V=6\text{-}30^o$, Z=b, X$\perp${001}; r>v

Composition:
MgO 31.7, SiO_2 63.5, H_2O 4.8
Al or Ti may substitute for Si. Fe may substitute for Mg. Cleavage: {001} perfect. Thin flexible folia. Care should be taken during TEM analysis. The chemical analysis may yield a breakdown similar to chrysotile asbestos and the platy aggregates can easily lie upon one another in a way as to appear tubular in morphology. The distinguishing feature is the hexagonal diffraction pattern quite unlike that of chrysotile. Usually found in clay like masses.

Distinguishing features:
infusible, insoluble. When moistened with cobalt nitrate and ignited it takes on a bluish color.

Occurrence:
The chief constituent of kaolin or clay. It is always a secondary mineral formed by the hydrothermal alteration of aluminum silicates, particularly feldspar.

PALYGORSKITE

Class: Phyllosilicate
Group: Clay Mineral

Palygorskite $(Mg,Al)_5 (Si,Al)_8 O_{20} (OH)_2 \bullet 8H_2O$

Color: white, gray
Luster: dull, earthy
Habit: massive, fibrous with tubular morphology
Hardness / Specific Gravity: 1.0 - 2.0 / 2.0 - 2.2

Optics: (+)
Pleochroism: none
Refractive Indices: α= 1.539, β= 1.589, γ= 1.589
Birefringence: moderate
Extinction Angle: 0, 90^o

Crystallography: Orthorhombic
Pn: *a*= 12.725; b= 17.872; c= 5.242 β= 92.23^o

Distinguishing features:
The high presents of Al. This Magnesium Aluminum Silicate may appear in fibrous form with a tubular morphology.

Occurrence:
See kaolinite

TALC

Class: Phyllosilicate
Group: Clay Mineral

Talc $Mg_3 Si_4 O_{10} (OH)_2$

Color: white, gray or pale green; often stained reddish and translucent.
Luster: dull, pearly on cleavage surfaces.
Transparency: translucent
Habit: Usually granular or foliated masses; crystals and fibers are rare.
Hardness / Specific Gravity: 1 (will make a mark on cloth) / 2.7 - 2.8
Cleavage / Fracture: basal, perfect {001}

Optics: (+)
Pleochroism: none
Refractive Indices: α=1.539, β=1.589, γ=1.589
Birefringence: moderate (0.045)
Extinction Angle: 0, 90^o

Crystallography: Monoclinic; 2/m
C2/c; a=5.27; b=9.12; c=18.85; $\beta=100^o0'$ a:b:c=0.578:1:2.067
Z= 4. d's: 9.34(10), 4.66(9), 3.12(10), 2.48(7), 1.87(4)
Orientation: $2V=6\text{-}30^o$, Z=b, $X \perp \{001\}$; r>v
Composition: MgO 31.7, SiO_2 63.5, H_2O 4.8
Al or Ti may substitute for Si. Fe may substitute for Mg.
Cleavage: {001} perfect. Thin flexible folia.

Distinguishing features:
Greenish white color, extremely soft, soapy feel. Flexible but not elastic.

Optically:
Usually tabular with hexagonal or rhombic plate outline. Foliated. Sometimes massive, talc may appear as fine thin ribbons under PLM. TEM usually shows platy or rhombic outline with near perfect cleavage. Diffraction analysis yields distinct hexagonal patterns, which remain constant during tilting.

Care should be taken during TEM analysis. The chemical analysis may yield a breakdown similar to chrysotile asbestos and the platy aggregates can easily lie upon one another in a way as to appear tubular. The distinguishing feature is the hexagonal diffraction pattern quite unlike that of chrysotile.

Occurrence:
Talc is formed by the alteration of magnesium silicates such as pyroxenes, amphiboles and olivine. Usually found in metamorphic rock. Known as *soapstone* in its massive form.

WOLLASTONITE

Class: Inosilicate
Group: Pyroxenoid

Wollastonite Ca Si O3

Color: colorless or white to gray
Luster: vitreous; pearly on cleavage surfaces; silky when fibrous
Transparency: subtransparent to translucent
Habit: Triclinic; (-1)
Hardness / Specific Gravity: 4.5-5.0 / 2.8-3.0
Cleavage / Fracture: {100} & {001} perfect, {-101} good giving splintery fragments.

Optics: (-)
α=1.620, β=1.632, γ=1.634
2V=40^{o}, Y near b, X^c=32^{o}

Crystallography:
P-1; a=7.94; b=7.32; c=7.07; α=90^{o}2', β=95^{o}22', γ=103^{o}26'; a:b:c=1.084:1:0.966
Z=6. d's: 3.83(8), 3.52(8), 3.31(8), 2.97(10), 2.47(6)

Composition:
CaO 48.3%, SiO_2 51.7% for pure $CaSiO_3$. Some Fe, Mn, & Mg may replace Ca. Pseudowollastonite is stable above 1120^{o}C, has a larger unit cell (Z=24) as compared to (Z=6) of wollastonite.

Distinguishing Features:
Fusible at 4 to a white almost glassy globule. Soluble in HCl. Distinguishable by two perfect cleavages of about 84^{o}.

Sample Reports and Worksheets

Signature Page

In signing this, I acknowledge having read and understood the previous pages of this document.

APPROVED BY:

LABORATORY Manager (print)	LABORATORY Manager (sign)	DATE

READ AND UNDERSTOOD BY:

Print Name	Signature	Date
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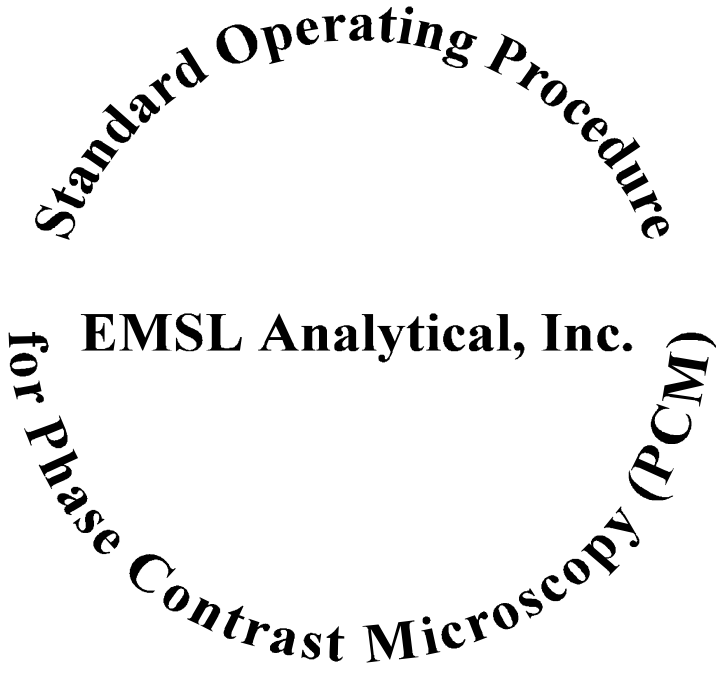

Standard Operating Procedures
Asbestos Analysis
Phase Contrast Microscopy (PCM)

Revision Date: May 1999
Original Date: June 1995

EMSL Analytical Inc., Quality Assurance Dept.

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Table of Contents

1.0 SAMPLE RECEIVING

1. Upon receipt of samples, check that the sample information on the Chain of Custody matches the information on the samples and any other paperwork. Any discrepancies must be dealt with before proceeding. If the samples do not have a COC, then provide a blank one, having the client fill out the necessary information.

   Information includes but is not limited to:

   - Client name, address, telephone number, fax #, contact person, etc.
   - State of origin
   - Number of samples sent and their ID
   - Type of analysis requested
   - Sample volumes or areas, where applicable
   - Turn around time
   - Signature, dates and time of the person relinquishing the samples.
   - Signature, date and time of an EMSL employee receiving the sample.

2. Acceptability of the condition of the samples is acknowledged upon signing the chain of custody.

   Insure samples comply with the following criteria:

   - Correct methodology requested
   - Properly labeled with unique identification information
   - Samples are not packaged using expanded Styrofoam type pieces
   - Samples are submitted separately from bulk asbestos samples
   - Correct sampling media is used

3. Log samples into LIMS system with all appropriate data:
   a. Computer assigns unique, sequential billing/job numbers, and unique sequential individual sample numbers.
   b. Sample batches are labeled with billing number, due date and time.
   c. Worksheets and billing sheets are printed and placed with sample batch

4. Samples with chain of custody, work sheet, billing worksheet, and all other associated paperwork are taken to the air sample preparation room.

2.0 SAMPLE PREPARATION

MCE FILTERS

1. Place the samples in an order corresponding to the clients COC.

2. On a clean 1X3-microscope slide oriented in a horizontal position, using a permanent marker, write the sample numbers on the slide.

3. Cut a wedge from the filter of each sample and place it on the slide above the appropriate sample number.

4. Collapse the filters using the acetone vapor generator. Inject acetone into the vapor generator with a syringe.

5. Place a drop of triacetin on the filter and cover with a clean cover slip.

6. With permanent marker (such as a ‘Sharpie’), outline the collapsed filter on the underside of the slide.

3.0 MICROSCOPE CALIBRATION

1. Turn on the microscope.

2. Slowly increase the power to the lamp.

3. Adjust the light source for even illumination across the entire sample.

4. Insert the telescope ocular into the head of the microscope in place of the 10x ocular.

5. Focus the image of rings in the ocular.

6. If the rings do not form concentric circles adjust them using the adjustment screws on the condenser.

7. Record the date and alignment in the microscope logbook.

8. Make sure the field iris is in focus, centered on the sample and open only enough to fully illuminate the field of view.

9. **(Weekly)** Check the phase-shift detection limit using the phase-contrast slide.
   - Center the HSE/NPL phase-contrast slide to focus under the PCM.
   - The lines in blocks 1 through3 must be fully visible showing row 4 & 5 as partially visible and the least distinct.
   - Rows 6 and 7 must be invisible. Record results in logbook.

8. **(Monthly)** Check measurement of the Walton Beckett graticle. Using a stage micrometer, determine the diameter of the graticle circle. Acceptable values are 100 microns +/- 2 microns. Calculate area of graticule using πR^2 where:

   $\pi = 3.14$
   R =radius (mm)

   The calculated value should be 0.00785 mm^2 for a graticle measuring 100 µm.

9. If image quality is poor, clean the microscope optics or inform the supervisor.

4.0 SAMPLE ANALYSIS

1. Fill out the appropriate information on the PCM bench sheet provided.

2. Place the slide containing the first sample onto the stage and center it under the objective lens. *Make sure to use the 40x objective.*

3. Focus the sample taking care not to allow the objective lens to touch the slide.

4. Starting at the upper left corner of the sample, traverse randomly down the filter in increments large enough to avoid possible overlapping of the fields of analysis.

5. If more than 50% of the filter for each field of view is covered with particulate, analysis must be discontinued and the sample declared overloaded.

6. Once stopped on a field of analysis slowly over and under focus to see any fibers that might be imbedded in the filter medium.

7. Working magnification is 400x.

8. Fiber criteria
    - Aspect ratio of 3:1
    - Must be $\geq$5micron in length

9. Count those fibers, which are within the boundaries of the circular graticule.

10. For fibers crossing the boundaries of the graticule
    - Count as a half fiber any fiber with only one end lying within the
    - graticule field.
    - Do not count any fiber, which crosses the boundary more than once.
    - Count bundles of fibers as one fiber unless observing both ends of a fiber can identify individual fibers.

11. Sizing of fibers
    - Count if $\geq$5 micron in length with 3:1 aspect ratio
    - $\geq$5 micron in length with 3:1 aspect ratio protruding from a matrix material

12. Stopping rules
    - Count 100 graticule fields or 100 fibers whichever comes first.
    - Count a minimum of 20 fields regardless.
    - Complete count of final field; do not terminate count mid-field.

13. Blanks
    - Prep and analyze all blanks
    - Subtract the average of the blanks from the sample result for the associated batch

14. Required filters
    - 0.45 – 1.2 micron pore size, 25mm mixed cellulose ester (MCE)

15. Pass / Fail limit
    - Depends on job requirements

5.0 CALCULATIONS

Results are reported in units of fibers/cc if sampling information is provided. In the event sample volume information is not submitted, report results in concentrations of fibers/mm^2.

To calculate fiber density in fibers/mm^2 :

- Total fibers counted minus mean field blank count = fibers
- Fibers / # fields counted x area of graticule = f/mm^2

To calculate fibers/cc:

- Fibers/filter = f/mm^2 x effective field area of sampling cassette (385mm for 25mm)

- $$f/cc = \frac{\text{fibers/filter}}{\text{sample volume (l) X 1,000}}.$$

Similarly

- Fibers/mm^2 $(E) = (F/n_f - B/n_b) / A_f$
- Fibers/cc $(C) = (E)(A_c) / V \times 10^3$

Where:

E	= fiber density in fibers/mm^2
F/n_f	= average fiber count per graticule field
B/n_b	= mean field blank count per graticule field
A_f	= graticule field area (approx. 0.00785 mm^2)
A_c	= effective collection area of filter (approx. 385 mm^2 for 25 mm cassette)
V	= volume in liters

EXAMPLE

Fibers = 35
Mean blank value = 0
Fields = 53
Volume = 1200 liters
Cassette size = 25mm (effective field area of 385mm)

f/mm^2 : 35.0 fibers/(53 fields x .00786) = 84.0 f/mm^2

Fibers /cc $= \frac{84.0 \times 385}{(1200 \times 1000)} = 0.027$ f/cc

6.0 REPORTING RESULTS

Data is reported in the final report and includes:

- Identification of laboratory location
- Page number
- Name and address of client
- Unique ID of test report
- Name and address of client
- Test method (NIOSH 7400 Issue 2, Fourth Edition August 15, 1994)
- Sample ID#, location, sample date, volume, fibers counted, fields counted, and limit of detection.
- Concentrations f/mm^2 and f/cc

Disclaimers cited in each report are as follows:
"The laboratory is not responsible for data reported in fibers/cc, which is dependent on volume collected by non-laboratory personnel."

"This report relates only to the samples reported above. This report may not be reproduced, except in full, without written approval by EMSL"

Limit of Detection
The recognized method detection limit for NIOSH 7400 is 5.5 fibers counted in 100 fields. Concentrations below 5.5 fibers are reported as less than (<) 5.5 fibers in 100 fields, or a fiber density of <7 fibers/mm^2.

7.0 QUALITY CONTROL

Procedures

1. After daily microscope calibration and before beginning analysis, analyze one reference slide.
    - Record results
    - If results are acceptable, proceed with analysis of samples.

2. Analyze all field blanks that have come with the sample set.
    - If the blanks contain >7 fibers in 100 fields then report possible contamination to the client.

3. Analyze a blind recount for every ten samples analyzed, record results in QC logbook.

4. All analysts participate in a Round Robin and PAT program.

5. New analyst proficiency is determined through evaluation of their skills using a training log.

QC Data Management

Accuracy

The analysis of reference slides (old proficiency test samples with known concentrations) are analyzed daily by each analyst. This analysis is performed prior to analysis of client samples. This data is checked against acceptable limits as determined by the issuing agency.

Precision

Daily reference sample data for each analyst is collected and tracked in the EMSL Monthly QC report. For each set of 20 data points, a coefficient of variation (CV) is determined for each individual analyst in fiber ranges of:

5-20 fibers/100fields
21-50 fibers/100 fields
51-100 fibers/100 fields

As these standard samples are analyzed, current data replaces the oldest data point.

Sample reanalysis is performed as intra analyst (same analysts) QC at the rate of 10% of sample volume. This data is tracked and managed in the EMSL Monthly QC report. The pass/fail criteria follows NIOSH 7400 requirements as follows:

Pass - If the absolute value of the difference between original and QC analysis (in f/mm2) is less than or equal to the value calculated by the constant (2.77) multiplied by the average of the square root of the original and QC fiber counts times the calculated CV value divided by 2.

$$\left| \sqrt{originalf\ \ / mm^2} - \sqrt{qcf\ \ / mm^2} \right| \leq 2.77 \left(\frac{\sqrt{f\ / mm^2} + \sqrt{f\ / mm^2}}{2} \right) \frac{CV}{2}$$

Fail - If the absolute value of the difference between original and QC analysis (in f/mm2) is greater than the value calculated by the constant (2.77) multiplied by the average of the square root of the original and QC fiber counts times the calculated CV value divided by 2.

$$\left| \sqrt{originalf\ \ / mm^2} - \sqrt{qcf\ \ / mm^2} \right| > 2.77 \left(\frac{\sqrt{f\ / mm^2} + \sqrt{f\ / mm^2}}{2} \right) \frac{CV}{2}$$

References:

NIOSH Manual of Analytical Methods (NMAM) 7400 Issue 2:August 15, 1994. Fourth Edition

American Industrial Hygiene Association: *Quality Assurance Manual for Industrial Hygiene Chemistry*. Fairfax, VA. American Industrial Hygiene Association 1995

Signature Page

In signing this, I acknowledge having read and understood the previous pages of this document.

APPROVED BY:

LABORATORY Manager(print)	LABORATORY Manager (sign)	DATE

READ AND UNDERSTOOD BY:

Print Name	Signature	Date
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Standard Operating Procedures
Analysis of Asbestos
Transmission Electron Microscopy &
Energy Dispersive X-Ray Micro Analysis

Revision Date: **June 1999**
Original Date: **June 1995**

EMSL Analytical Inc., Quality Assurance Dept.

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EMSL ANALYTICAL
Standard Operating Procedures
Analysis of Asbestos
Transmission Electron Microscopy &
Energy Dispersive X-Ray Micro-analysis

TABLE OF CONTENTS

SECTION

SECTION

SECTION

1.0 SAMPLE RECEIVING

Upon receipt of samples, check that the sample information on the Chain of Custody (COC) matches the information on the samples and other paperwork. Any discrepancies must be resolved before proceeding. If the samples do not have a COC then one is completed at time of log in. Have the client fill out the necessary information completely.

INFORMATION REQUIRED:

- Client name, address, telephone number, contact person, etc.
- Project number and state where samples were taken
- Number of samples sent and there ID
- Type of analysis requested
- Sample volumes or areas, where applicable
- Turn around time needed
- Date and time of delivery
- A signature, date and time of the person relinquishing the samples.
- A signature, date and time of an EMSL employee receiving the sample.

Check to see if the samples match the COC and if the cassettes are open, damaged, contaminated, or double bagged.

If the samples are damaged or if the COC does not match, notify the client.

Clock in the samples and place your initials next to the time received.

If any of the following information is not supplied, contact the client.

- Client name, address, telephone number, contact person, fax number
- Project number/ name, state where samples where taken
- Number of samples sent and sample ID's
- Type of analysis requested
- Sample volumes or areas if applicable
- Turn around time-"RUSH" is not acceptable
- A signature, date, and time of the person relinquishing the samples
- All samples MUST be accounted for with the proper sample ID's
- All samples MUST be sealed, properly bagged and undamaged

Record the following information for water samples: the volume of sample received in milliliters (mls.)

Note: All water samples are to be filtered within 48 hr. of collection. Samples are stored at 4° C.

2.0 EQUIPMENT START UP

2.1 JEOL 100-CX II/HITACHI

1. Turn on the chiller.
2. To turn on the TEM, turn the key to the "START" position and release.

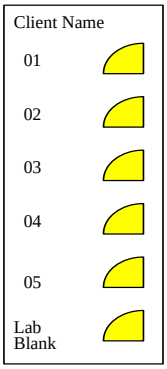
2.2 OTHER EQUIPMENT

1. Turn on the acetone vapor generator.
2. Turn on the Denton carbon coater. All valves should be closed.
    a. Turn on the cooling water.
    b. Turn on the main power
    c. Turn on mechanical pump
    d. Turn on the diffusion pump heater.
    e. Open the backing valve
    f. Turn the High Vacuum Gauge to the on position.
3. Turn on the condensation washer. (Optional)
    a. Turn on the cooling water.
    b. Turn on the heater.

3.0 SAMPLE PREPARATION

3.1 MCE FILTERS

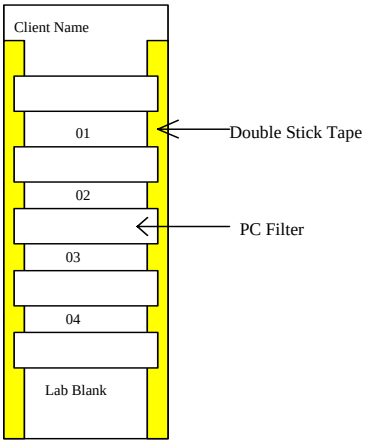
1. Place the samples in an order corresponding to the clients COC.
2. Add a Laboratory Blank to the end of the series.
3. On a clean 1X3-microscope slide, scribe the billing number and the sample numbers with a diamond scribe.
4. Cut a wedge from the filter of each sample and place it on the slide above its ID number



5. Collapse the filters using the acetone vaporizer and fresh acetone.
6. For AHERA, samples go to ASHING THE SAMPLE.
   All other analysis goes to CARBON COATING THE SAMPLES.

3.2 POLYCARBONATE FILTERS

1. Place the samples in an order corresponding to the clients COC.
2. Add a Laboratory Blank to the end of the series.
3. On a clean 1X3-microscope slide write the clients name and the sample numbers with a colored Sharpie permanent marker and cover with clear tape. This color will be used to designate that set of samples through analysis.
4. Using double stick tape, run a thin section down each side of the slides.
5. Cut rectangular sections of the polycarbonate filter and place on the slide so that each end adheres to the tape.
6. Go to CARBON COATING THE SAMPLES.



3.3 ASHING THE SAMPLES (MCE Filters Only)

BIORAD LOW TEMPERATURE ASHER

1. Turn on the oxygen and check that the only buttons lit are the POWER and AUTO/MAN.
2. Vent the chamber to load samples. [Depress the VENT button]
3. Load samples and turn off VENT.
4. Hold the chamber door closed and depress the PUMP button.
5. When the chamber pressure is between 4 and 10-1 mbar depress the STANDBY button.
6. Wait for the chamber pressure to reach 50-1 mbar.
7. Depress the GAS 1 button. DO NOT ADJUST GAS FLOW.
8. Depress the RF button. The REFLECTED POWER is to be 5 watts.
9. The FORWARD POWER should be 50 watts.
10. **The REFLECTED POWER should never exceed 8 watts and the FORWARD POWER is not to exceed 100 watts or resisters blow**
11. Start your timing device.
12. Ash for the calibrated time.

TO REMOVE SAMPLES:

a. Turn off the RF button.
b. Turn off the STAND BY, and GAS 1 buttons.
c. Turn off the PUMP.
d. Depress the VENT.

TO SHUT DOWN THE SYSTEM:

a. Hold the chamber door closed and PUMP the chamber for a few seconds.
b. Turn off the PUMP.
c. The only lights on should be the POWER and the AUTO/MAN buttons
d. Turn off the POWER button.

3.4 CARBON COATING THE SAMPLES

DENTON CARBON COATER

TO START THE SYSTEM
(MAKE SURE THAT ALL VALVES ARE CLOSED AND SWITCHES ARE OFF)

1. Turn on main power.
2. Turn on cooling water.
3. Turn on mechanical pump.
4. Turn on diffusion pump.
5. Open backing valve.
6. Turn on thermocouple gauge, set to TC2 position, and turn on the high vacuum gauge.
7. Wait 15 minutes for diffusion pump to warm.

TO COAT A SAMPLE

1. Vent the chamber and lift off the bell jar.
2. Replace the used carbon rod with a new sharpened rod.
3. Place the sample in the coater affixing it to the metal plate with double stick tape.
4. Replace bell jar after checking for any debris around the rubber gasket.
5. Close the backing valve and the chamber vent.
6. Open the roughing valve.
7. Wait for the chamber pressure to reach below 50 mtorr.
8. Close the roughing valve.
9. Open the backing valve, then the main valve.
10. Set the high vacuum gauge range to the 10^{-4} range. IF THE RED LIGHT DOES NOT REMAIN ON WAIT TWO MINUTES AND TRY AGAIN.
11. Once the gauge drops below $1.0x10^{-4}$ switch the range to 10^{-5} and wait for the needle to reach $3X\ 10^{-5}$.
12. Turn on rotary power.
13. Turn on fill/glow power.
14. Slowly increase power until the carbon is just sparking, and continue until the carbon tip has been evaporated.
15. Turn off the fill/glow power.
16. Turn off rotary power.
17. Close the main valve.
18. Open the chamber vent and lift off the bell jar after venting is complete.

TO SHUT DOWN THE SYSTEM

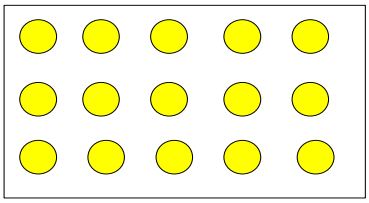
1. Replace the bell jar.
2. Turn off the diffusion pump.
3. Close the backing valve and the chamber vent.
4. Open the roughing valve and pump the chamber to below 100 mtorr.
5. Close the roughing valve and open the backing valve.
6. Wait 15 minutes for the diffusion pump to cool.
7. Close all vents and turn off all gauges.
8. Turn off the mechanical pump.
9. Turn off the system power.
10. Open the mechanical pump valve to vent pump.

NOTE: WHEN SYSTEM IS OFF ALL VALVES, VENTS AND SWITCHES SHOULD BE TURNED OFF.

3.5 DISSOLVING THE FILTER

3.5a THE JAFFE WICK

1. Place a piece of cut Kimwipe onto the metal mesh screen in the petri dish making sure that the level of the solvent rises to touch the underside of the paper. For MCE filters use acetone and to dissolve PC filters use chloroform. (Optional)DMF and DMSO may replace acetone and in many cases will yield a better preparation.
2. Place three grids per sample in order on top of the Kimwipe making sure that the dull side of the grid is up.
3. Cut the collapsed coated sample filters in a grid pattern and carefully, using clean forceps, peal up one square at a time and place it onto one of the copper grids, carbon side up. (*Carbon side down for NIOSH 7402)*

Setup as shown 

4. Replace the lid and label.
5. Allow to stand for at least 30 minutes for MCE and 60 minutes for PC. If time allows, leave the samples in the solution for one to two hours.
6. Pull from the solvent bath and allow drying for a minute before storing into a grid box.

3.5b THE CONDENSATION WASHER: (Optional)

1. Place a small piece of tissue paper onto a small piece of screen.
2. Place the screen into a petri dish with acetone for MCE or chloroform for PC. Similar to the Jaffe Wick.
3. Put your grids onto the damp tissue paper.
4. Carefully place your sample onto the grid.
5. After the solvent has been brought to its boiling point, remove the cold finger from the condensation washer.
   Make sure the condensation washer is filled with acetone for MCE and chloroform for PC filters.
6. Carefully lift the tissue paper and screen off the wick and place onto the cold finger.
7. Replace the cold finger into the condensation chamber.
8. Wait at least five minutes then remove the cold finger.
9. Remove the screen and replace the cold finger.
10. Allow drying.
11. Mount samples on clip.

3.6 GRID STORAGE

1. Grids are attached to an asymmetric copper clip with pre-cut sections of carbon double-stick tape. The clips are rectangular with a rectangular elongate opening running along the long axis of the clip. One end has a semi-circular notch on one end.
2. Six grids fit on each clip and are arranged in a standard sequence. Counting away from the notch, the first and second grids are from sample one, the third and fourth from the second sample, and the fifth and sixth from the third sample. On the second clip are samples four in the first and second grid positions, five in the third and fourth positions and the lab blank in positions five and six. In the event of additional samples, continue this sequence, placing the blank at the appropriate end position. The notched end of the clip is oriented closest to the tip of the specimen arm,
3. Clips are lettered A through U inclusive, and placed in a specially designed holding box.
4. Grid boxes are uniquely and sequentially numbered
5. Included inside the box is a numbered inventory sheet with client name and billing number for tracking.
6. All unanalyzed grids (inclusive of the third prepped grid) are placed into standard numbered grid boxes, recorded on the grid box log sheet, and stored for three years.

4.0 SPECIALIZED SAMPLE PREPARATION

4.1 WATER SAMPLES-Method 100.2

1. Place the samples into the order noted on the COC.
2. Label a petri dish for each sample or for each dilution used per sample.
3. Setup the filtration apparatus, vertical walled fritted glass or disposable plastic units, with 0.22µm MCE filters and run a 100ml blank prior to sample filtering.
4. Thoroughly mix the samples by placing in a low temperature sonicator for 15 minutes and vigorously shake before removing the aliquot to filter.
5. Filter a series of aliquots and place the filter in the respective labeled petri dishes.
   For potable water filter 50ml, & 100ml.
   For other water filter 5ml, 10ml, & 25ml.
   Choosing the proper volume to filter comes with practice. If the aliquot is <50ml bring the sample to a volume of >50 using particle free water.
6. Allow the sample filters to dry. A heat lamp may be used to shorten the drying time of MCE filters only. *Do not subject the PC filters to heat.*
7. SEE THE PROCEDURES UNDER AIR SAMPLE PREP, FOLLOWING THIS ORDER:
   FOR MCE FILTERS
   a. Collapse & plasma ash the filter.
   b. Carbon coat the samples.
   c. Allow samples to soak in the Jaffe Wick.
   d. Dry grids and store.

   FOR PC FILTERS
   a. Cut and tape the filters to a clean glass slide.
   b. Carbon coat the samples.
   c. Allow samples to soak in the Jaffe Wick.
   d. Dry grids and store.
8. Quality Control
   a. Prepare a Laboratory Blank with each sample set.
   b. If the set contains three or more samples, perform a Duplicate Prep on the first sample of each set.
   c. Sample Container Contamination Check
      -one bottle in each batch or one bottle in 24.
      -use a pre-washed bottle with 800 mls. of fiber free water
   d. Record refrigerator temperature daily with NIST traceable thermometer. Temperature is to be maintained at 4°C ± 2°C.

4.2 WIPES & MICRO-VAC SAMPLES

1. Place the samples into the order noted on the COC.
2. Label a petri dish for each sample or for each dilution used per sample.
3. Setup the filtration apparatus, fritted glass, or disposable plastic units.
4. Carefully open the cassette or bag containing the sample.
5. Using distilled or particle free water, fill the cassette or a portion of the bag, and empty into a clean beaker or disposable-measuring cup.
6. Remove the filter or the wipe and place into the beaker.
7. Rinse the cassette or bag again and bring the wash in the beaker to 100ml volume.
8. Place the sample wash into a sonicator and sonicate for two minutes.
9. Remove the sample wash from the sonicator and remove the filter or wipe carefully wringing out any water back into the beaker.
10. Bring the wash up to 100ml volume with particle free water.
11. Filter a series of milliliters and place in the respected petri dishes. *Choosing the proper volume to filter comes with practice. When in doubt ask your associates.*
12. Allow the sample filters to dry. A heat lamp may be used to shorten the drying time of MCE filters only. *Do not subject the PC filters to heat.*
13. SEE THE PROCEDURES UNDER AIR SAMPLE PREP. FOLLOWING THIS ORDER:

    FOR MCE FILTERS

    a. Collapse the filter with the acetone vaporizer.
    b. Carbon coat the samples.
    c. Allow samples to soak in the Jaffe Wick.
    e. Dry grids and store.

    FOR PC FILTERS

    a. Cut and tape the filters to a clean glass slide.
    b. Carbon coat the samples.
    c. Allow samples to soak in the Jaffe Wick.
    d. Dry grids and store.
14. Quality Control

    Prep a Laboratory Blank with each sample set.

4.3 BULK SAMPLES (Qualitative Drop Mount)

1. Remove a portion of the sample and place into a glass scintillation vial.
2. Fill the sample vial with a reagent suitable for dissolving the sample matrix.
   - Chloroform, toluene, or di-chloromethane for floor tiles
   - Chloroform or toluene for tar
   - Water for gypsum board
   - Chloroform or acetone for wall or ceiling plasters
   - Some plasters or mortars, depending on the matrix material, may first be subjected to two to three drops of HCL to dissolve the matrix material fill the container with water.
4. Cap and vigorously shake the container until the sample is dissolved.
5. If the suspension appears too dense, dilute with more of the same reagent used to dissolve the sample. This comes with practice. Ask an experienced analyst or your supervisor if you have any questions.
6. Using a micro pipette dispense a drop of the suspension onto a carbon coated blank TEM grid placed onto a clean glass slide.
7. Allow the drop to dry before storing. Drying time varies depending on the reagent used.
8. Quality Control

   The Drop Mount is predominantly used as a QC check for bulk samples.

4.4 BULK SAMPLES (Chatfield & Non-Friable Organically Bound - NOB)

1. Place the samples into the order noted on the COC.
2. Set up a clean, pre-weighed crucible for each sample.
3. Set up a clean pre-weighed petri dish with a filter for each sample.
4. Separate floor tile from mastic if possible without cross contamination.
5. If the client requests, separate other easily separable layers. Otherwise homogenize the layers and take a representative sample.
6. Remove 100-500mg. of sample and weigh in the pre-weighed crucible.
7. Place into a pre-heated muffle furnace at 480° until the weight of the residue has stabilized.
8. Remove from the furnace and allow cooling to room temperature.
9. Re-weigh and log the weight on the NOB preparation form.
10. Grind the residue in the crucible with 0.5ml of distilled particle free water and 2-5mls of HCL.
11. Filter the sample through the pre-weighed 0.4µm filter and rinse the crucible filtering the rinse water until no sample remains in the crucible.
12. Allow the filter to dry in the petri dish.
13. Re-weigh and log the results.
14. Transfer the filter to a vial and add ethanol to cover the sample.
15. Ultrasonicate the sample for one minute.
16. Using a micropipette, remove a portion of the sample while it is sonicating. If necessary vigorously shake the sample as the heavy particles will settle and cause high asbestos readings.
17. Place a drop onto a carbon coated TEM grid and allow to dry.
18. Store.
19. Quality Control
    - Prep a Laboratory Blank with each sample set.
    - For (NOB) Perform a Duplicate Prep on one of the samples contained in each set.

4.5 BULK SAMPLES (Conventional)

1. Place the samples into the order noted on the COC.
2. Set up a clean, weighted crucible for each sample.
3. Separate floor tile from mastic if possible without cross contamination.
4. If the client requests, separate other easily separable layers. Otherwise homogenize the layers and take a representative sample.
5. Remove 100-500mg. of sample and weigh in the pre-weighed crucible.
6. Place into a pre-heated muffle furnace at 480° until the weight of the residue has stabilized.
7. Remove from the furnace and allow cooling to room temperature.
8. Add approximately 10ml of water and mix the sample.
9. Ultrasonicate the sample for one minute.
10. Using a pipette remove a portion of the sample while it is sonicating. If necessary vigorously shake the sample as the heavy particles will settle and cause high asbestos readings.
11. Filter the aliquot through a 0.45 μm filter and allow to dry. (A dilution series should be used in the event of incorrect loading.)
12. Go to Air Sample Prep.
13. Quality Control

    Prepare a Laboratory Blank with each sample set.

5.0 SAMPLE ANALYSIS

DEFINITIONS:

Working Magnification: The magnification at which analysis should be performed.

Fiber Criteria: The attributes, size and shape, that a structure must display in order to be counted.

Sizing of Fibers: How the size of a structure needs to be recorded on the worksheet.
For AHERA, a check is placed in a column according to a structure being < or ≥ 5μm length. For EPA Level II, the actual length and width will need to be recorded.

Required EDX: The frequency at which structures need to be analyzed by EDX. For AHERA, each structure, which will cause the sample to exceed 70str/mm^2 will need EDX analysis. In the case of AHERA samples, this usually equates to the first four structures.

Required Diffraction Patterns: The frequency at which structures need to be analyzed by SAED.

Stopping Rules: The criteria required before analysis can be suspended.

Required Analytical Sensitivity**:** The criteria required for the number of grid openings to be analyzed. AHERA requires an A.S. of 0.005str/cc so a sample with 1200 liters being analyzed on 0.0129 mm^2 grid opening will require 5 openings for analysis.

Laboratory Blanks**:** A blank filter supplied by the lab, which is prepped along side of the samples to test for contamination. This section states if a laboratory blank is required and if the blank will need to be analyzed at the time of sample analysis.

Required Filters: The type of filter that the sample is to be taken on or filtered through.

Pass / Fail Limit: The level or concentration at which a sample or set of samples is past acceptable limits.

Quality Control: The steps to follow for QC. (S= Intra Analyst; D= Inter Analyst; SR=Intra Analyst Reprep, DRI=Inter Analyst Reprep, Inter Laboratory; V= Verified, B=Blanks).

PROCEDURES

1. Remove the first sample grid from the box and insert it into the TEM.
2. Bring the TEM to a magnification of 300 to 500x and inspect the grids to determine if at least 50% if the grid openings are intact. If two of the three grids are not 50% intact then the samples will have to be reprepped.
3. Two grids are analyzed per sample. Separation of grid openings for each grid is recorded on the bench sheet with a one or two indicating the grid number and a line separating the two.

5.1 SAMPLE PREPARATION ACCEPTANCE

1. More than 50% of the grid must be covered by the replica.
2. Grids must have at least 50% intact grid openings
3. Grids must not have more than 10% opaque area due to incomplete filter dissolution.
4. Total Grid openings must have <50% overlapping of folded replica film
5. At least 20 grid openings with <5% holes and <5% opaque area due to incomplete filter dissolution.
6. Grid openings analyzed must not have rips or overlapping folds

5.2 AHERA PROTOCOL

1. At a magnification of 300 to 500x, orient an intact grid opening on the middle of the screen.
2. Increase magnification to 19,000x taking care to remain in the chosen grid opening.
3. Log the grid opening identification on the sample worksheet.
4. Move to the upper left corner of the grid square and begin traversing the grid using only one directional control. Once the opposite grid bar has been reached, move over one screen width and traverse to the original grid bar. Take care not to count any structure twice or to miss any area of the grid opening.
5. If no fibrous structures have been located repeat steps one through four until the stopping procedures have been reached. Do not analyze adjacent grid openings.
6. If a fibrous structure has been located, check its morphology against the morphologies for each asbestos type. If morphology is consistent with asbestiform fibers, proceed to EDX analysis.
7. Obtain an EDX of the sample by following the procedures outlined in ENERGY DISPERSIVE X-RAY ANALYSIS and compare it to the spectra for the asbestos types. Record the information on the sample worksheet. If the chemistry is consistent with one of the asbestiform minerals, proceed to Diffraction analysis.
8. Obtain a diffraction pattern for the sample by following the procedures outlined in DIFFRACTION ANALYSIS. Record diffraction information on the sample worksheet.

9. If the morphology, diffraction, and EDX information match that of an asbestos type, then record the structure as asbestos.
10. For asbestos structures, note whether it is a fiber, bundle, cluster, or matrix.
    - *Fiber:* a single fibril not connected to any non-fibrous debris.
    - *Bundle:* any group of three or more fibers lying parallel and with less than a single fiber diameter between them.
    - *Cluster:* a group of three or more fibers or bundles randomly oriented with three or more intersections.
    - *Matrix:* a bundle or fiber with one end free and the other end covered by or imbedded in a matrix material.
11. Working magnification is 19,000x.
12. Fiber criteria
    - Aspect ratio of 5:1
    - Must be $\geq$0.5µm length
13. Sizing of fibers
    - Record <5µm length and $\geq$5µm length
14. Required EDX (for each type of asbestos)
    - All fibers that cause the sample to exceed 70str/mm^2
15. Required diffraction patterns (for each type of asbestos)
    - All fibers that cause the sample to exceed 70str/mm^2
    - One micrograph per 5 samples that contain asbestos
16. Stopping rules
    - Count the number of grids required to reach a detection limit of <0.005
    - Analyst may stop if 50 fibers in a minimum of 4 grid openings is reached
17. Required detection limit
    - 0.005 fibers/cc
18. Laboratory blanks
    - One blank per sample set
19. Required filters
    - 0.45µm MCE
20. Pass / Fail limit
    - Average of <70str/mm^2
    - Pass the “Z” test (Optional)
21. Quality Control
    - Analyze QC during analysis for S's.
    - Leave D's for analysis by another analyst.
    - Analyze QC during analysis for Laboratory Blanks.

22. Required QC
    - One Laboratory Blank prepared per sample set.
    - Analyze Lab Blank for every 25 samples prepped.
    - If a sample set averages greater than 70 str/mm^2, analyze the Lab Blank.
    - Two percent Inter Analyst, same grid openings.
    - Two percent Intra Analyst, same grid openings.
    - Five percent Verified.
    - One percent Intra Analyst, reprepped.
    - One percent Inter Analyst, sent for Inter Lab analysis and reprepped.
    - One blank read for every 25 samples prepped, or 10 %, whichever is greater
23. Sample Requirements
    - Requires a volume of 1200 to 1800 liters in most instances
    - Requires Inside, Outside, and Blank samples

- If these requirements have not been met, a disclaimer must appear on the report.

5.3 EPA LEVEL II PROTOCOL

1. At a magnification of 300 to 500x, orient an intact grid opening on the middle of the screen.
2. Increase magnification to 15,000 to 19,000x taking care to remain in the chosen grid opening.
3. Log the grid opening number on the sample worksheet.
4. Move to the upper left corner of the grid square and begin traversing the grid using only one directional control. Once the opposite grid bar has been reached move over one screen width and traverse to the original grid bar. Take care not to count any structure twice or to miss any area of the grid opening.
5. If no fibrous structures have been located, repeat steps one through four until the stopping procedures have been reached. Do not analyze adjacent grid openings.
6. If a fibrous structure has been located, check its morphology against the morphologies for each asbestos type. If morphology is consistent with asbestiform fibers, proceed to EDX analysis.
7. Obtain an EDX of the sample by following the procedures outlined in ENERGY DISPERSIVE X-RAY ANALYSIS and compare it to the spectra for the asbestos types. Record the information on the sample worksheet. If the chemistry is consistent with one of the asbestiform minerals, proceed to Diffraction analysis.
8. Obtain a diffraction pattern for the sample by following the procedures outlined in DIFFRACTION ANALYSIS. Record diffraction information on the sample worksheet.
9. If the morphology, diffraction, and EDX information match that of an asbestos type, then record the structure as asbestos.
10. For asbestos structures-note whether it is a fiber, bundle, cluster, or matrix.
    - *Fiber:* a single fibril not connected to any non-fibrous debris.
    - *Bundle:* any group of three or more fibers lying parallel and with less than a single fibers diameter between them.
    - *Cluster:* a group of three or more fibers or bundles randomly oriented with three or more intersections.
    - *Matrix:* a bundle or fiber with one end free and the other end covered by or imbedded in a matrix material.
11. Working magnification is 15,000 to 19,000x.
12. Fiber criteria
    - Aspect ratio of 3:1
13. Sizing of fibers
    - Record length and width of fibers and bundles
    - Record length and width at 90° for clusters
    - Add the length and width of all protruding fibers and bundles for matrices

14. Required EDX
    - The first five structures and every tenth thereafter
15. Required diffraction patterns
    - The first five structures and every tenth thereafter
16. Stopping rules
    - Count ten grid openings
    - May stop upon the completion of the GO with the 100th structure
17. Required detection limit
    - None
18. Laboratory blanks
    - One blank per sample set
19. Required filters
    - 0.45µm MCE
    - 0.4µm PC
20. Pass / Fail limit
    - None
21. Quality Control
    - Analyze QC during analysis for S's.
    - Leave D's in the bin for analysis by another analyst.
    - Analyze QC during analysis for Laboratory Blanks.
22. Required QC
    - Prep one Laboratory Blank prepared per sample set.

5.4 NIOSH 7402 PROTOCOL

1. At a magnification of 300 to 500x, orient an intact grid opening on the middle of the screen.
2. Increase magnification 15,000 to 19,000x taking care to remain inside the chosen grid opening.
3. Log the grid opening number on the sample worksheet.
4. Move to the upper left corner of the grid square and begin traversing the grid using only one directional control. Once the opposite grid bar has been reached move over one screen width and traverse to the original grid bar. Take care not to count any structure twice or to miss any area of the grid opening.
5. If no fibrous structures have been located, repeat steps one through four until the stopping procedures have been reached.
6. If a fibrous structure has been located, check its morphology against the morphologies for each asbestos type. If morphology is consistent with asbestiform fibers, proceed to EDX analysis.
7. Obtain an EDX of the sample by following the procedures outlined in ENERGY DISPERSIVE X-RAY ANALYSIS and compare it to the spectra for the asbestos types. Record the information on the sample worksheet. If the chemistry is consistent with one of the asbestiform minerals, proceed to Diffraction analysis.
8. Obtain a diffraction pattern for the sample by following the procedures outlined in DIFFRACTION ANALYSIS. Record diffraction information on the sample worksheet.
9. If the morphology, diffraction, and EDX information match that of an asbestos type, then record the structure as asbestos.
10. For asbestos structures note whether it is a fiber, bundle, cluster, or matrix.
    - *Fiber:* a single fibril not connected to any non-fibrous debris.
    - *Bundle:* any group of three or more fibers lying parallel and with less than a single fiber diameter between them.
    - *Cluster:* a group of three or more fibers or bundles randomly oriented with three or more intersections.
    - *Matrix:* a bundle or fiber with one end free and the other end covered by or imbedded in a matrix material.
11. Working magnification is 15,000 to 19,000x.
12. Fiber criteria
    - Aspect ratio of 3:1 *regardless of length*
    - Fibers $\leq$0.25µm diameter or $\leq$5.0µm length label as undetectable by PCM
    - Report fibers $\geq$0.25µm diameter & $\geq$5.0µm length only *****see note***

13. Sizing of fibers
    - Record length and width of fibers and bundles
    - Record length and width at 90° for clusters
    - Add the length and width of all protruding fibers and bundles for matrices
    - Separate by (<5µm length X <0.25µm diameter) and ≥5µm
14. Required EDX
    - All fibers
15. Required diffraction patterns
    - All fibers
16. Stopping rules:
    - <5 fibers/ GO count a minimum of 40 grid openings
    - 5 to 25 fibers/ GO count either 40 or 100 fibers which ever comes first
    - >25 fibers/ GO count 6 grid openings or 100 fibers which ever comes first
17. Required detection limit
    - None
18. Laboratory blanks
    - One blank per sample set
19. Required filters
    - 0.8µm MCE
20. Pass / Fail limit
    - None
21. Quality Control
    - Analyze QC during analysis for S's.
    - Leave D's in the bin for analysis by another analyst.
    - Analyze QC during analysis for Laboratory Blanks.
22. Required QC
    - Prep one Laboratory Blank prepared per sample set.
23. Sample Requirements
    - Requires a volume >400

* If these requirements have not been met, a disclaimer must appear on the report.

Note: Count all fibers with a ratio of 3:1.
Report only the fibers that are ≥0.25µm diameter & ≥5.0µm length.
The report must have a disclaimer that states only the large fibers were used for calculations**.**

5.5 WATER 100.2

1. At a magnification of 300 to 500x, orient an intact grid opening on the middle of the screen.
2. Increase magnification to 10,000x taking care to remain in the chosen grid opening.
3. Log the grid opening number on the sample worksheet.
4. Move to the upper left corner of the grid square and begin traversing the grid using only one directional control. Once the opposite grid bar has been reached move over one screen width and traverse to the original grid bar. Take care not to count any structure twice or to miss any area of the grid opening.
4. If no fibrous structures have been located, repeat steps one through four until the stopping procedures have been reached.
5. If a fibrous structure has been located, check its morphology against the morphologies for each asbestos type. If morphology is consistent with asbestiform fibers, proceed to EDX analysis.
6. Obtain an EDX of the sample by following the procedures outlined in ENERGY DISPERSIVE X-RAY ANALYSIS and compare it to the spectra for the asbestos types. Record the information on the sample worksheet. If the chemistry is consistent with one of the asbestiform minerals, proceed to Diffraction analysis.
7. Obtain a diffraction pattern for the sample by following the procedures outlined in DIFFRACTION ANALYSIS. Record diffraction information on the sample worksheet.
8. If the morphology, diffraction, and EDX information match that of an asbestos type, then record the structure as asbestos.
9. For asbestos structures note whether it is a fiber, bundle, cluster, or matrix.
    - *Fiber:* a single fibril not connected to any non-fibrous debris.
    - *Bundle:* any group of three or more fibers lying parallel and with less than a single fiber diameter between them.
    - *Cluster:* a group of three or more fibers or bundles randomly oriented with three or more intersections.
    - *Matrix:* a bundle or fiber with one end free and the other end covered by or imbedded in a matrix material.
10. Working magnification is 10,000x.
11. Fiber criteria
    - Aspect ratio of 3:1
    - Must be $\geq$10μm length
    - Fibers intersecting top or left grid bars are recorded as twice the observed length.
    - Fibers intersecting the bottom or right grid bars are not counted.

12. Sizing of fibers
    - Separate into groups of <10 & ≥10μm (only ≥10μm are used for calculations).
13. Required EDX
    - All structures
14. Required diffraction patterns
    - All structures
15. Stopping rules
    - Completion of the grid opening in which an analytical sensitivity of 0.2 MFL is obtained.
    - May stop upon the completion of the GO with the 100th structure
16. Required detection limit
    - 0.2 million fibers per liter
17. Laboratory blanks
    - One blank per sample set
    - Prep blanks with 100ml particle free water and analyze to a D.L. of 0.05
18. Sample Container Contamination Check
    - One bottle in each batch or one bottle in 24.
    - Use pre-washed bottle with 800 mls. of fiber free water
19. Required filters
    - 0.22 MCE
20. Pass / Fail limit
    - 7 million fibers per liter
21. Quality Control
    - Analyze Laboratory Blanks accompanying sample sets.
    - Analyze Replicate preps accompanying sample sets.
    - Perform contamination checks on sample containers
    - Prepare and analyze one Laboratory Blank per sample set.
    - For sets with three or more samples, prepare and analyze a duplicate of one of the samples in the sample set.

5.6 WIPE & MICRO-VAC

1. At a magnification of 300 to 500x, orient an intact grid opening on the middle of the screen.
2. Increase magnification to 10,000 to 19,000x taking care to remain in the chosen grid opening.
3. Log the grid opening number on the sample worksheet.
4. Move to the upper left corner of the grid square and begin traversing the grid using only one directional control. Once opposite grid bar has been reached move over one screen width and traverse to the original grid bar. Take care not to count any structures twice or to miss any area of the grid opening.
5. If no fibrous structures have been located, repeat steps one through four until the stopping procedures have been reached.
6. If a fibrous structure has been located, check its morphology against the morphologies for each asbestos type. If morphology is consistent with asbestiform fibers, proceed to EDX analysis.
7. Obtain an EDX of the sample by following the procedures outlined in ENERGY DISPERSIVE X-RAY ANALYSIS and compare it to the spectra for the asbestos types. Record the information on the sample worksheet. If the chemistry is consistent with one of the asbestiform minerals, proceed to Diffraction analysis.
8. Obtain a diffraction pattern for the sample by following the procedures outlined in DIFFRACTION ANALYSIS. Record diffraction information on the sample worksheet.
9. If the morphology, diffraction, and EDX information match that of an asbestos type, then record the structure as asbestos.
10. For asbestos structures note whether it is a fiber, bundle, cluster, or matrix.
    - *Fiber:* a single fibril not connected to any non-fibrous debris.
    - *Bundle:* any group of three or more fibers lying parallel and with less than a single fibers diameter between them.
    - *Cluster:* a group of three or more fibers or bundles randomly oriented with three or more intersections.
    - *Matrix:* a bundle or fiber with one end free and the other end covered by or imbedded in a matrix material.
11. Working magnification is 10,000 to 19,000x.
12. Fiber criteria
    - Aspect ratio of 5:1
    - Must be $\geq$0.5µm length
13. Sizing of fibers
    - Record length and width of fibers and bundles
    - Record length and width at 90° for clusters
    - Add the length and width of all protruding fibers and bundles for matrices

14. Required EDX
    - First 5 structures of each asbestos type
    - 1 in 10 there after
15. Required diffraction patterns
    - First 5 structures of each asbestos type
    - 1 in 10 there after
16. Stopping rules
    - Count 10 grid openings
    - May stop if 50 structures is reached
17. Required detection limit
    - None
18. Laboratory blanks
    - One blank per sample set
19. Required filters
    - None
20. Pass / Fail limit
    - None
21. Quality Control
    - Analyze Laboratory Blanks accompanying sample sets.
22. Required QC
    - Prepare and analyze one Laboratory Blank per sample set.
    - For sets with three or more samples, prepare and analyze a duplicate of one of the samples in the sample set.

5.7 BULK (Qualitative Drop Mount)

1. At a magnification of 300 to 500x, orient an intact grid opening on the middle of the screen.
2. Increase magnification to 10,000 to 19,000x taking care to remain in the chosen grid opening.
3. Log the grid opening number on the sample worksheet.
4. Scan across the grid opening looking for the presence of asbestos.
5. If no fibrous structures have been located repeat steps one through four until the stopping procedures have been reached.
6. If a fibrous structure has been located, check its morphology against the morphologies for each asbestos type. If morphology is consistent with asbestiform fibers, proceed to EDX analysis.
7. Obtain an EDX of the sample by following the procedures outlined in ENERGY DISPERSIVE X-RAY ANALYSIS and compare it to the spectra for the asbestos types. Record the information on the sample worksheet. If the chemistry is consistent with one of the asbestiform minerals, proceed to Diffraction analysis.
8. Obtain a diffraction pattern for the sample by following the procedures outlined in DIFFRACTION ANALYSIS. Record diffraction information on the sample worksheet.
9. If the morphology, diffraction, and EDX information match that of an asbestos type, then record the structure as asbestos.
10. For asbestos structures note the dominant fiber type, whether it is a fiber, bundle, cluster, or matrix.
    - *Fiber:* a single fibril not connected to any non-fibrous debris.
    - *Bundle:* any group of three or more fibers lying parallel and with less than a single fibers diameter between them.
    - *Cluster:* a group of three or more fibers or bundles randomly oriented with three or more intersections.
    - *Matrix:* a bundle or fiber with one end free and the other end covered by or imbedded in a matrix material.
11. Working magnification is 5,000 to 19,000x.
12. Fiber criteria
    - Aspect ratio of 3:1
13. Sizing of fibers
    - Note the average size
14. Required EDX
    - As needed to confirm the existence of asbestos
15. Required diffraction patterns
    - As needed to confirm the existence of asbestos

16. Stopping rules
    - At least 10 grid openings
    - Stop at the 5th confirmed fiber
17. Required detection limit
    - None
18. Laboratory blanks
    - One blank per sample set
19. Required filters
    - None
20. Pass / Fail limit
    - None
21. Required QC
    - None

5.8 BULK (Conventional)

1. At a magnification of 300 to 500x, orient an intact grid opening on the middle of the screen.
2. Increase magnification to 15,000 to 19,000x taking care to remain in the chosen grid opening.
3. Log the grid opening number on the sample worksheet.
4. Move to the upper left corner of the grid square and begin traversing the grid using only one directional control. Once opposite grid bar has been reached move over one screen width and traverse to the original grid bar. Take care not to count any structures twice or to miss any area of the grid opening.
8. If no fibrous structures have been located, repeat steps one through four until the stopping procedures have been reached.
6. If a fibrous structure has been located, check its morphology against the morphologies for each asbestos type. If morphology is consistent with asbestiform fibers, proceed to EDX analysis.
7. Obtain an EDX of the sample by following the procedures outlined in ENERGY DISPERSIVE X-RAY ANALYSIS and compare it to the spectra for the asbestos types. Record the information on the sample worksheet. If the chemistry is consistent with one of the asbestiform minerals, proceed to Diffraction analysis.
8. Obtain a diffraction pattern for the sample by following the procedures outlined in DIFFRACTION ANALYSIS. Record diffraction information on the sample worksheet.
9. If the morphology, diffraction, and EDX information match that of an asbestos type, then record the structure as asbestos.
10. For asbestos structures note whether it is a fiber, bundle, cluster, or matrix.
    - *Fiber:* a single fibril not connected to any non-fibrous debris.
    - *Bundle:* any group of three or more fibers lying parallel and with less than a single fibers diameter between them.
    - *Cluster:* a group of three or more fibers or bundles randomly oriented with three or more intersections.
    - *Matrix:* a bundle or fiber with one end free and the other end covered by or imbedded in a matrix material.
11. Working magnification is 15,000 to 20,000x.
12. Fiber criteria
    - 3:1 aspect ratio
13. Sizing of fibers
    - Record length and width of fibers and bundles
    - Record length and width at 90° for clusters
    - Add the length and width of all protruding fibers and bundles

14. Required EDX
    - The first five structures and every tenth there after
15. Required diffraction patterns
    - The first five structures and every tenth there after
16. Stopping rules
    - Count ten grid openings
    - May stop if 50 structures is reached
17. Required detection limit
    - None
18. Laboratory blanks
    - One blank per sample set
19. Required filters
    - 0.45µm MCE
    - 0.4µm PC
20. Pass / Fail limit
    - 1%
21. Quality Control
    - Analyze Laboratory Blanks accompanying sample sets.
22. Required QC
    - Prepare and analyze one Laboratory Blank per sample set.
    - For sets with three or more samples, prepare and analyze a duplicate of one of the samples in the sample set.

Micrograms = Fibers and Bundles = (π/4) x Length x Diameter x Density x 10^{-6}
Clusters and Matrices = ((Width x Length x Thickness**) x Density) x 10^{-6}

Measurements are in micrometer (µm).
Densities = 3.0 for Amphiboles & 2.6 for Chrysotile
** = *Thickness is estimated from the width of the largest protruding bundle.*

Convert the total mass of asbestos into grams and determine the percent asbestos by...

% Asbestos = Mass of asbestos
(gms.....
.....
.....
.....
.....
.....) / (Initial mass of sample (gms.) X 0.01)

5.9 BULK (Chatfield & NOB)

1. At a magnification of 300 to 500x, orient an intact grid opening on the middle of the screen.
2. Increase magnification to 3,000 or 19,000x taking care to remain in the chose grid opening.
3. Randomly scan the sample choosing fields with a moderate loading of debris.
4. Estimate the percent asbestos vs. the percent debris on each field. Ignore any fields that do not contain debris. Cover several areas of the filter in case of a non-homogeneous sample. To better estimate the true percent, tilt the sample in order to visualize a three-dimensional picture as a two-dimensional image may be misleading causing false high asbestos percents.
5. Calculate the average asbestos concentration from all of the fields analyzed and report the range that the result corresponds with.
6. If a fibrous structure has been located, check its morphology against the morphologies for each asbestos type. If morphology is consistent with asbestiform fibers, proceed to EDX analysis.
7. Obtain an EDX of the sample by following the procedures outlined in ENERGY DISPERSIVE X-RAY ANALYSIS and compare it to the spectra for the asbestos types. Record the information on the sample worksheet. If the chemistry is consistent with one of the asbestiform minerals, proceed to Diffraction analysis.
8. Obtain a diffraction pattern for the sample by following the procedures outlined in DIFFRACTION ANALYSIS. Record diffraction information on the sample worksheet.
9. If the morphology, diffraction, and EDX information match that of an asbestos type, then record the structure as asbestos.
10. If a fibrous structure has been located, check its morphology against the morphologies for each asbestos type.
11. Obtain a diffraction pattern for the sample by following the procedures outlined in DIFFRACTION ANALYSIS for the scope that you are utilizing. Record diffraction information on the sample worksheet.
12. Obtain an EDX of the sample by following the procedures outlined in ENERGY DISPERSIVE X-RAY ANALYSIS and compare it to the spectra for the asbestos types. Record the information on the sample worksheet.
13. If the morphology, diffraction, and EDX information match that of an asbestos type then count the structure as asbestos.
14. For asbestos structures, there is no need to note fiber, bundle, matrix, or cluster.
15. Working magnification is 3,000 and 10,000 to 19,000x.

16. Fiber criteria
    - Estimate range percent of asbestos
17. Sizing of fibers
    - Not recorded
18. Required EDX
    - The first five structures and every tenth there after
19. Required diffraction patterns
    - The first five structures and every tenth there after
20. Stopping rules
    - Count ten grid openings
    - May stop if 50 structures is reached
21. Required detection limit
    - None
22. Laboratory blanks
    - One blank per sample set
23. Required filters
    - 0.4um PC
24. Pass / Fail limit
    - 1%
25. Quality Control
    - Analyze Laboratory Blanks accompanying sample sets.
    - Analyze any Replicate Prep. with sample sets.
    - Leave DR's in tray for analysis by another analyst.
26. Required QC
    - Prepare and analyze one Laboratory Blank per sample set.
    - Prepare and analyze a SR of one of the samples in the sample set.
    - Prepare and analyze a DR one in every twenty samples.

6.0 ENERGY DISPERSIVE X-RAY MICRO ANALYSIS (EDX)

6.1 KEVEX SYSTEMS

1. Center the structure in question in the center of the screen.
2. Remove the objective and selected area apertures.
3. Reduce to spot size 3.
4. Tilt the sample 30-40^{o} toward you.
5. Condense the beam to cover only the structure in question.
6. Depress the [CLEAR] key twice while holding down the [SHIFT] key. This clears the old spectrum from the screen and memory.
7. Depress the [ACQUIRE] key.
8. Adjusting the spectrum
    a. If the counts are below 100, try the following...
        - move the spot to a different area of the structure
        - spread the beam
        - increase the spot size one or two steps
    b. If the counts are above 5,000 try...
        - move the spot to a different area of the structure
        - reduce the spot size one to two steps
        - decrease the bias
    c. If the dead time is above 70% try...
        - reduce the spot size one or two steps
        - decrease the bias
    d. If you are unable to obtain a spectrum...
        - check the dead time
        - check the counts per second
        - you may be oriented too close to a grid bar or the holder
        - the sample may be organic

    **If you can not correct the problem call your supervisor for assistance.*
9. After obtaining a spectrum depress the [ACQUIRE] key again then the [MLK] key and a cursor appears on the screen. This cursor can be moved to correspond to each peak to verify which element that peak represents. To move the cursor, rotate the round knob at the top of the keyboard.
10. If the spectrum shows peaks corresponding to any of the six asbestos types, depress the [CNTR][EDS] keys at the same time to evoke the ratio program.

11. After the ratio program displays the peak ratios for the elements present compare them to the following list.

	Na	Mg	Si	Ca	Fe
CHRYSOTILE	0.0	0.6-0.95	1.0	0.0	0.0-0.05
ANTHOPHYLLITE	0.0	0.3-0.5	1.0	0.0	0.0-0.1
AMOSITE	0.0	0.02-0.11	1.0	0.0	0.7-0.95
CROCIDOLITE	0.1-0.5	0.0-0.02	1.0	0.0	0.55-0.7
ACTINOLITE	0.0	0.2-0.3	1.0	0.25-0.35	0.1-0.2
TREMOLITE	0.0	0.2-0.4	1.0	0.25-0.35	0.0-0.1

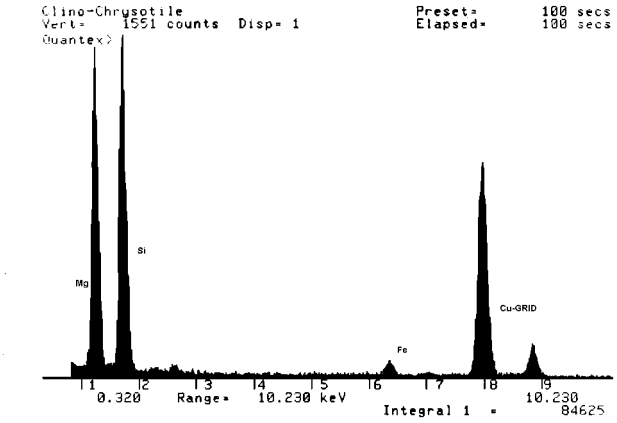
This list may vary for each instrument. Obtain a list for each instrument using NIST asbestos standards.
Ratios calibrated after background removal

Note: EDXA units vary so check these numbers using known asbestos standards. Most units will fall within these ranges.

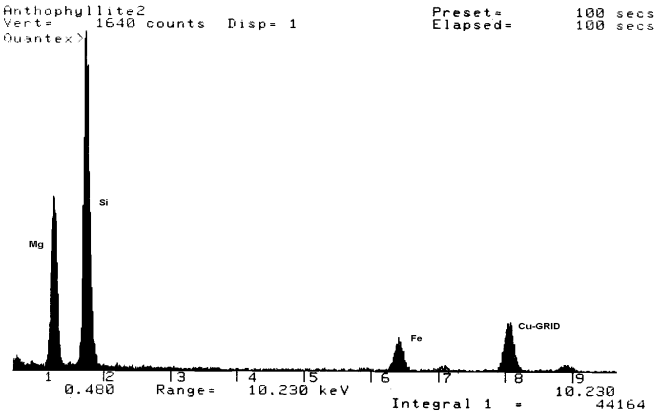
12. If the structure falls within any of these guide lines, it may be asbestos.
    - Minerals form under varying conditions the ratios may vary, however, the majority of each asbestos type will fall within the parameters listed.
13. Write the elements found in the EDX column on the sample worksheet.
14. Type "RE / OLD"[RETURN] to regain the original spectrum.
15. Depress [CLR][TEXT] and, using the arrow keys, type in the ID for each peak and the clients name, project #, sample #, and the type of asbestos found.
16. When finished depress [TEXT] and then [CNTR]P to print the spectrum for the client. (Optional)

6.2 Typical Spectra for the Six Asbestos Types

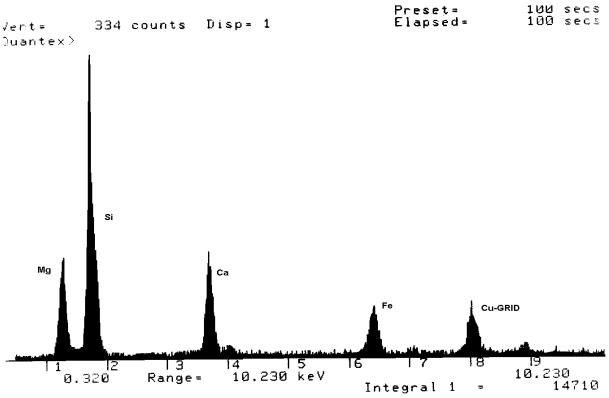
Chrysotile



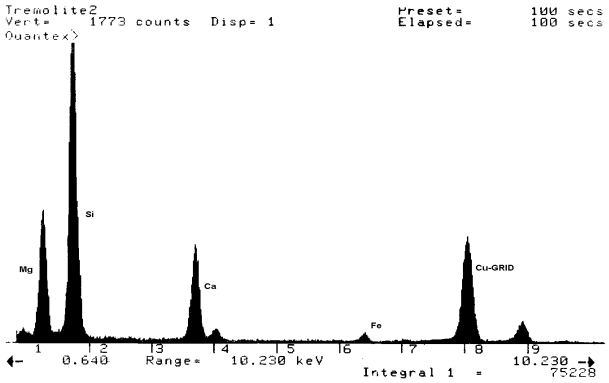
Anthophyllite



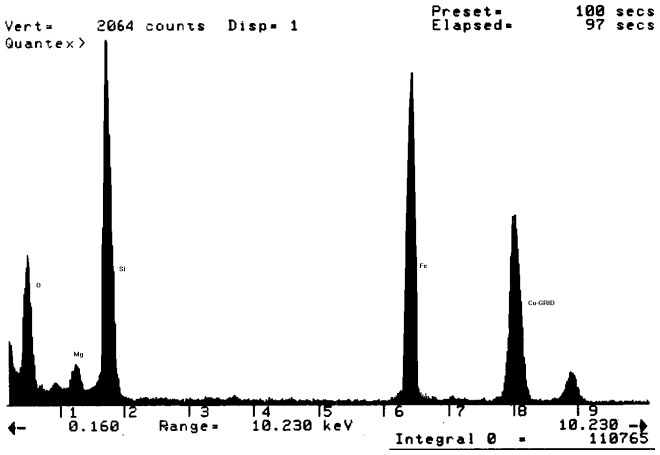
Actinolite



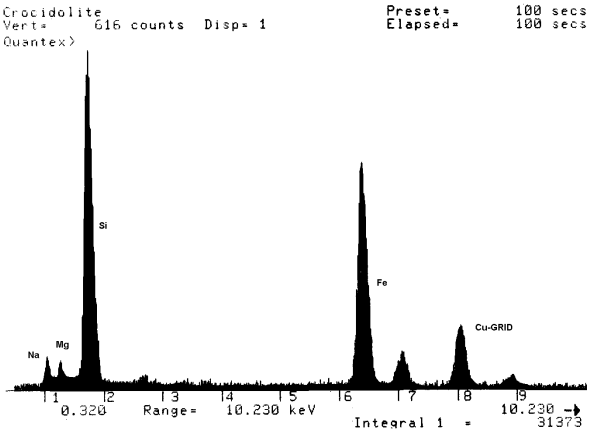
Tremolite



Amosite



Crocidolite



7.0 SELECTED AREA ELECTRON DIFFRACTION

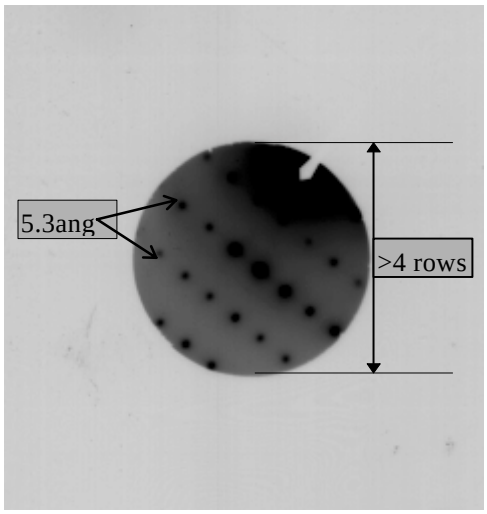
7.1 JEOL 100 CX II

1. Orient the sample in the center of the screen.
2. Remove the objective aperture.
3. Reduce the spot size one to two steps.
4. Tilt the sample using the rotating goniometer. If the sample slides across the screen then adjust it back to the center using the "Z" correction screw located on the side of the goniometer. Tilt back and forth across the 0^{o} mark and adjust until the sample remains stable in the center of the screen.
5. Insert & focus the selected area aperture choosing an aperture size that is slightly larger than the sample.
6. Trip the image wobbler and stabilize the image using the focus knobs.
7. Turn off the wobbler and depress the [DIFF] button.
8. Tilt up the screen using the square green button next to the column and insert the stereoscope for close examination of the pattern.
9. Insert the first objective aperture and adjust it to measure the number of rows of spots that are on the screen. Each microscope is different with respects to this measurement. To check yourself, insert a known asbestos standard into the scope and obtain a diffraction pattern. Insert the aperture and see how many rows are encompassed by the area of the aperture. This may be used as a quick measurement for the 5.3angstrom row spacing that are indicative of asbestos.

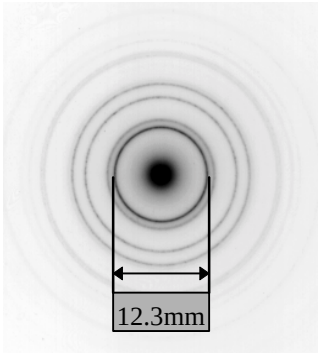
7.2 Measuring Patterns

Once a diffraction pattern has been obtained, insert the first objective aperture to help measure the row spacing.

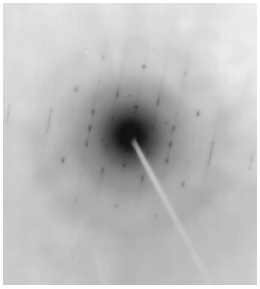
Each microscope is different. In order to find out how many row spacing are within the aperture calibrate using the gold standard.



Measuring a pattern from a micrograph requires a gold pattern as a standard.

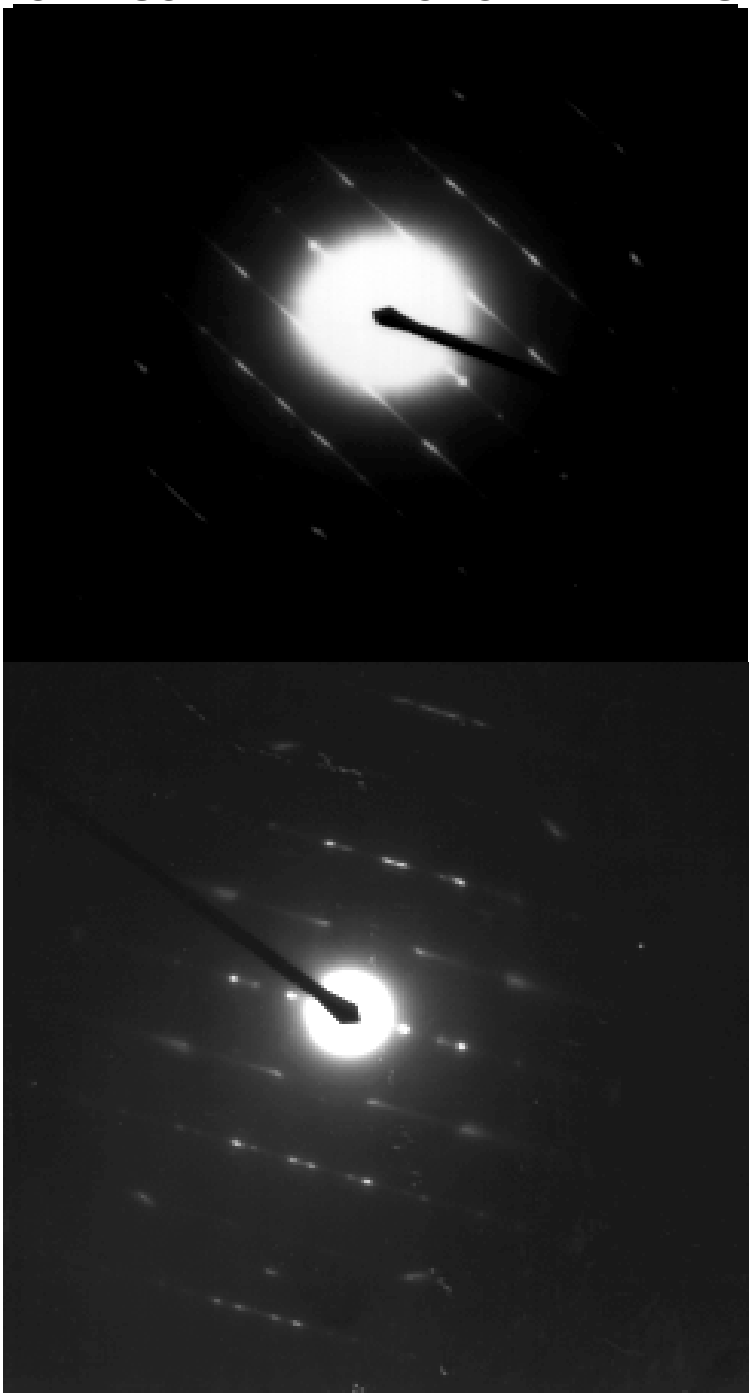


Measure the diameter (in mm) of the inner gold ring then divide by two to obtain the radius. Multiply the radius by 2.355 to get the Camera Constant. In this case the CC≅14.5. Next, measure the row spacing of the sample pattern using several rows and taking the average. The average row spacing of this picture is ≅2.7mm. Divide 2.7 into the camera constant and you get 5.37ang. row spacing. This is the row spacing for this diffraction pattern and it relates to the 5.3ang. row spacing of chrysotile asbestos.

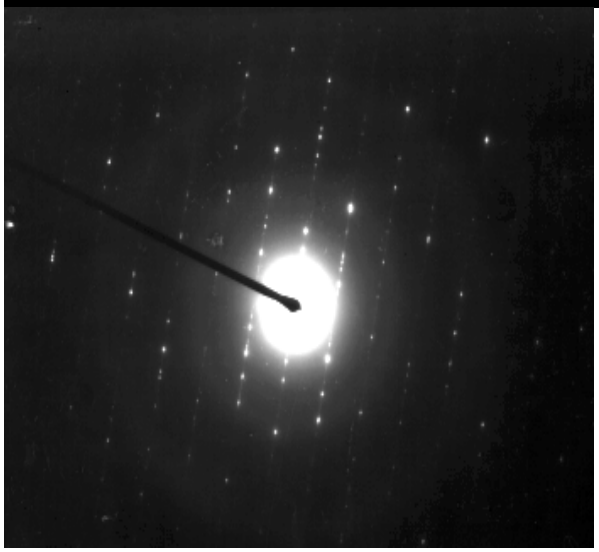


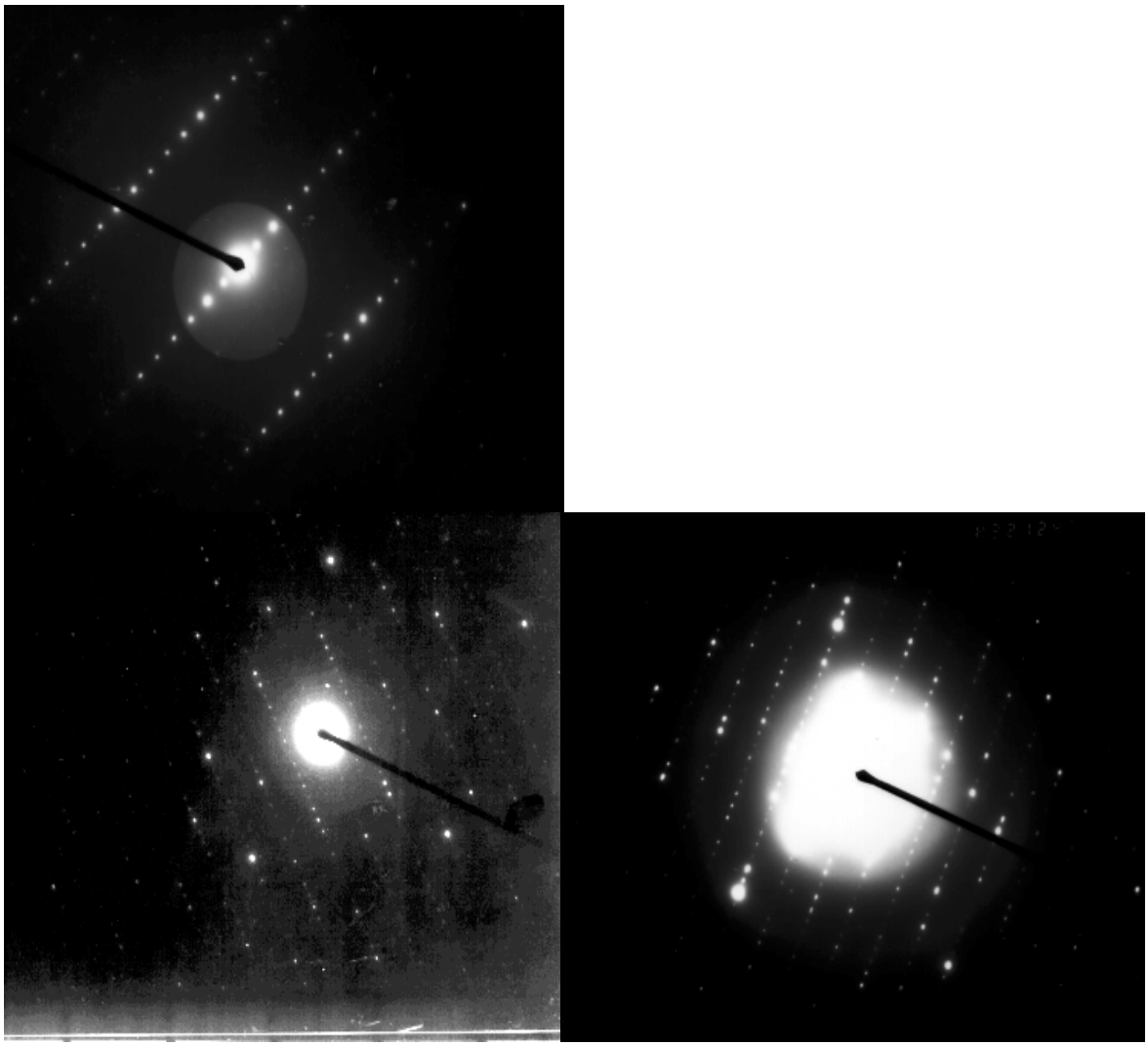
7.3 SAMPLE PATTERNS

CHRYSOTILE DIFFRACTION PATTERNS



7.4 COMMON AMPHIBOLE PATTERNS





8.0 EQUATIONS

All Equations are to be hand calculated to check the computer. The Approved Signatory must also recalculate the results prior to signing the final report.

NIOSH (3:1 Aspect Ratio)
AHERA (5:1 Aspect Ratio)
EPA Level II (3:1 Aspect Ratio)
AIR SAMPLES
EFA = Effective filter area of a 25mm cassette = 385mm^2
GOA = Grid opening area (0.00635)
N = Number of fibers (If N=0 then default to 1 structure)
V = Volume (1200 liters)
AA= Area analyzed (0.06985)
NO = Number of openings analyzed (11)
n = Number of Samples

If the concentration is "0" run all calculations using "1" structure and report as < this result.

Area Analyzed = (GOA x NO) [0.06985mm^2 = (0.00635 x 11)]

Structures /mm^2 = (N / AA) [14.3/mm^2 = (1 / 0.06985)]

Structures /cc = $\frac{385 \times N}{(V \times AA \times 1000)}$ $\frac{385 \times 1}{[0.0046/cc = (1200 \times 0.06985 \times 1000)]}$.

# of openings to read for 0.005^s/cc sensitivity NO = (EFA x 10^{-3}) / (GOA x V x 0.005)

Round all decimals UP to the next higher whole number 10.1 (11) = (385) / (1200 x 0.00635 x 10^3 x 0.005)

Geometric mean

$$\left[\sqrt[n]{N1 \times N2 \times N3 \times N4 \times N5} \right] \quad \left[2.6 = \sqrt[5]{(1 \times 2 \times 3 \times 4 \times 5)} \right]$$

Z" TEST

$\underline{Yi}$ = the mean of the natural logarithms of the insides (str/cc)
Yo = the mean of the natural logarithms of the outsides (str/cc)

$$\bar{Y} = \frac{\ln N_1 + \ln N_2 + \ln N_3 + \ln N_4 + \ln N_5}{n}$$

Assume the insides to be...	the outsides to be...
.06	.0062
.041	.0031
.063	.0032
.046	.0030
.036	.0034

"Z" Test = $\frac{\bar{Yi} - \bar{Yo}}{0.8\,(1/ni + 1/no)^{0.5}}$. $5.11 = \frac{(-3.04) - (-5.62)}{0.8\,(0.4)^{1/2}}$

The Site passes if Z < 1.65

EPA Level II analysis (3:1 ratio)

Micrograms = Fibers and Bundles = (π/4) x Length x Diameter x Density x 10^{-6}
Clusters and Matrices = ((Width x Length x Thickness**) x Density) x 10^{-6}

Measurements are in micrometer (μm).
Densities = 3.0 for Amphiboles & 2.6 for Chrysotile
** = *Thickness is estimated from the width of the largest protruding bundle.*

WATER (100.2) (<10um & ≥10um)
1385mm^2 = Effective filter area
V (ml) = milliliters of water filtered

$$\text{Million Fibers/ Liter} = \frac{1385 \times N}{(V(ml.) \times AA \times 1{,}000)} \times \frac{[\,0.23 \text{ million/l} = 1385 \times 1\,.]}{(100ml \times 0.06985 \times 1{,}000)}$$

VACUUM / DUST SAMPLES (3:1 ratio)

Dilution Factor = ml filtered / ml of solution [0.1 = 10ml / 100ml]

Million Fibers / area sampled =

$$\frac{N \times EFA}{(DF \times AA)} \times 0.000001$$

BULK (Gravimetric)
M = Initial mass of sample (0.02gms)
100ml = Volume of sample suspension
v = Volume of sample suspension filtered (10ml)
1385mm^2 = Effective filter area
m = Total mass of asbestos based on EPA II mass calculations (2496x10^{-12})
AA = Area analyzed (0.06985mm^2)

$$\text{wt\% Asbestos} = 100 \times \frac{((m/AA) \times 1385 \times (100/v))}{M}$$

$$\text{2.5\% Asbestos} = 100 \times \frac{((2496 \times 10^{-12} / 0.06985 mm^2) \times 1385 \times (100 / 10ml))}{0.02gms}$$

(BULK Conventional)

Micrograms = Fibers and Bundles = (π/4) x Length x Diameter x Density x 10^{-6}
Clusters and Matrices = ((Width x Length x Thickness**) x Density) x 10^{-6}

Measurements are in micrometer (μm).
Densities = 3.0 for Amphiboles & 2.6 for Chrysotile
** = *Thickness is estimated from the width of the largest protruding bundle.*

% Asbestos = Mass of asbestos (gms.) / (Initial mass of sample (gms.) X 0.01)

9.0 QUALITY ASSURANCE / QUALITY CONTROL

9.1 PLASMA ASHER CALIBRATION:

1. Make 2 preparations by collapsing 1 whole 25 mm MCE filter onto clean, pre-weighed glass slides.
2. Allow a minute for any residual acetone to evaporate from the filter & slide.
3. Re-weigh and record the weights of the slides and collapsed filters.
4. Ash the slides and filters for ten minutes.
5. Remove the slides and record the weight of each.
6. In the TEMCAL spreadsheet of the EMSL QC program enter the following data for each of the two slides:
    - Date of calibration
    - Total minutes ashed (this will usually be ten unless you have an extremely aggressive or anemic asher)
    - Initial slide weight
    - Weight of slide and collapsed filter
    - Weight of slide and collapsed and ashed filter
7. The program will then calculate the time needed to ash approximately 5% of the filter
8. The results will appear on the report page of the spreadsheet which is printed each month as part of the Monthly Quality Control Report

9.2 TEM Alignment

9.2.1 JEOL 100 CX II

1. Depress the Accelerating Voltage [HT] button and allow the voltage to stabilize at 100 keV.
2. Slowly increase the Filament Emissions to the stop.
3. Increase the magnification to 19,000x and condense the beam to the center of the screen.

4. Daily Alignments
   a. Condenser Alignment-
      Open the panel to the lower left and flip the white toggle switch labeled [WOBBLER] to the "X" position. If the image on the screen does not overlap then adjust the image using the Condenser & Corrector "X" knobs. Flip the white toggle switch labeled [WOBBLER] to the "Y" position. If the image on the screen does not overlap each other, adjust the image using the Condenser & Corrector "Y" knobs.
   b. Gun Tilt-
      Reduce the spot size to the #2 or #3 spot and condense the beam. If the beam is not in the center of the screen, adjust it using the Alignment TILT knobs located next to the column. Increase the spot size to 1 and adjust the beam using the Gun Alignment TRANS knobs located to the bottom right. Repeat this step until the spot stays centered through the series of spot sizes.

Note: If the movement in the spot is becoming greater then reverse the Alignment TILT / small spot size to Alignment TILT / large spot size and Gun Alignment TRANS / small spot size.

   c. Electron Gun Alignment-
      Return to the #1 spot size and condense the beam. Slowly reduce the Filament Emissions until a donut or semi-donut shape appears on the screen. If the image is not a donut shape, use the Gun Alignment TILT to correct it. Re-center the beam using the Gun Alignment TRANS knobs. Increase the Filament Emissions to the stop.

   d. Astigmation-
      Condense the beam at 19,000 times and use the CONDENS STIGMATOR knobs to adjust the beam to a circular shape.

   e. Condenser Aperture-
      Bring the TEM to approx. 10,000X and bring the beam to crossover. If the beam is sliding across the screen as it is being condensed then use the X and Y adjustment knobs on the upper most aperture on the right side of the TEM column. Simply adjust the knobs while sending the beam through over and under cross over until the spot no longer slides across the screen.

9.2.2 Hitachi

ZOOM Mode:

1. Insert an Al coated Holey Grid into the TEM.
2. Increase the beam voltage fully and at a 1.0 μm spot size and in SCAN mode select a grid opening without many replicas.
3. Move to ZOOM mode and increase the magnification to 20,000X.
4. Adjust the BIAS control (left panel) until the beam current VU meter displays a reading of 10 microamps of emission.
5. Center the beam spot with the BRIGHTNESS CENTERING control, and then bring the beam to crossover. Unsaturate the filament by rotating the FILAMENT control knob slightly counter-clockwise to view the undersaturated filament.
6. The filament should appear as a doughnut shape, centered within the beam spot. Adjust the GUN TILT X & Y controls until the undersaturated filament image is centered within the beam spot and resaturate the filament.
7. Move the beam spot back and forth through crossover and mechanically align the condenser aperture by adjusting the X & Y fine adjustment control stems on the Condenser Aperture control so that the beam spot remains concentrically positioned during this process.
8. Change the spot size to 0.4 μm and bring the beam spot to the crossover position. Adjust the COND STIGM X & Y control knob (leftmost knob on the lower right control panel) to make the beam spot as round as possible.
9. Bring the beam to the crossover position.
10. Center the beam spot on the screen with the BRIGHTNESS CENTERING controls.
11. Change the spot size to 1.0 or 2.0 μm (which ever will be used for analysis) and bring the beam to crossover.
12. Adjust the GUN HORIZ X & Y controls to center the beam spot on the screen.
13. Repeat the preceding steps 9 thru 12 until further adjustments are unnecessary to keep the beam centered after changing between the small and large spot size.
14. Move to SCAN mode and select a grid opening with an intact HOLEY replica.
15. Move back to ZOOM mode.
16. Bring the stage into eucentric position by bringing a feature of the replica to the center of the phosphor screen and depressing the **TILT** pedal in either direction. If the particle remains in the center of the screen, the stage is eucentric, and alignment may continue with step 21 after the stage has been brought back to 0° tilt. If the particle moves, follow steps 17 - 20 before continuing with alignment.
17. Using the **TILT** peddle continue tilting the stage in one direction until the selected particle moves to the edge of the phosphor screen or until the tilt reaches either **+10°** or **-10°.**
18. Using the **Z-axis** control on the sample holder, move the selected object back into the center of the screen.

19. Tilt the stage back into the 0° position with the **TILT** pedal.
20. If the selected object is still at the center of the screen, proceed to the next step, otherwise move the selected object back to the center of the screen using the translators. Repeat steps 16 thru 20.
21. Locate a round hole on a Holey grid, position it in the center of the phosphor screen and focus the hole using the WOBBLER button in conjunction with the OVER and UNDER FOCUS buttons.
22. Depress the HV MODUL button to observe an undulating movement of the hole.
23. Reduce the amount of movement by adjusting the BEAM TILT X & Y knob (second knob from the left on the lower right control panel) and then center the beam with the BRIGHTNESS CENTERING controls.
24. Bring the beam to crossover and recenter the beam with the BRIGHTNESS CENTERING controls then expand the beam again. Repeat steps 23 & 24 until no further adjustments are needed.
25. Turn off the HV MODUL button, center the beam with the BRIGHTNESS CENTERING controls.
26. Insert objective aperture #4 and center the aperture in the beam by adjusting the X & Y controls on the objective aperture stem.
27. Observe a hole and through-focus on it until the black over-focused ring of the Fresnel-fringe becomes visible.
29. Adjust the OBJ STIGM X & Y controls until the objective astigmatism is diminished as seen in the black-ring of the Fresnel-fringe becoming a uniform thickness. This function should be performed at a magnification higher than used for image recording.
30. Remove the objective aperture.

<u>SA Mode:</u>

1. Move to SA Mode and bring the Magnification to 20,000X.
2. Center the beam using the BRIGHTNESS CENTERING knob.
3. Focus the image by depressing the WOBBLER button and simultaneously adjusting the FOCUS buttons, until all movement have been eliminated.
4. Insert SA Aperture #4 and center it on the screen by adjusting the X & Y controls on the SA aperture stem.
5. Focus the SA Aperture opening using the FOCUS buttons on the left keypad below the F.PROBE and DIFF Mode buttons.
6. Move to Diffraction Mode and cycle through the different diffraction Camera Lengths by using the MAG UP and DOWN buttons to verify that all camera lengths are visible on the phosphor screen.

7. If the diffraction image at 0.9 on-screen camera length is not in the inner circle on the screen move it back to the center using the two small black unmarked knobs directly behind the column. They are at the same height level as the CRT screen and are mounted on a small exposed circuit board. Caution must be exercised during this operation not to disturb the X and Y translators or the diffraction pattern will move.
8. Return to SA mode and remove the SA #4 Aperture.

FINE PROBE Mode:

1. Move to ZOOM mode and focus the image by depressing the WOBBLER button and simultaneously adjusting the FOCUS buttons until all movement have been eliminated.
2. Locate a round hole on the Holey replica and position it in the center of the phosphor screen.
3. Move to FINE PROBE Mode.
4. Set the spot size to 100nm.
5. Bring the beam to crossover and round the beam using the COND STIGM X & Y knob (the middle knob on the lower right control panel).
6. Center the beam using the BEAM HORIZ X & Y knob (second knob from the right on the lower right control panel).
7. Expand the beam slightly and depress the HV MODUL button to observe an undulating movement of the hole.
8. Reduce the amount of movement by adjusting the BEAM TILT X & Y knob (the rightmost knob on the lower right control panel) and then center the beam with the BEAM HORIZ X & Y knob (second knob from the right on the lower right control panel).
9. Bring the beam to crossover and recenter the beam with the BEAM HORIZ X & Y knob (second knob from the right on the lower right control panel) then expand the beam again. Repeat steps 8 & 9 until no further adjustments are needed.
10. Turn off the HV MODUL button, center the beam with the BRIGHTNESS CENTERING controls.
11. Move to ZOOM Made.
12. Record any unusual observations in the Transmission Electron Microscope Maintenance LogBook. (Exhibit I) and complete the Daily TEM Calibration form (Exhibit II).

9.2.3 Eucentric Plane "Z"

Setting the "Z" is crucial to accurate measurements. Upon first inserting the sample holder, traversing multiple openings and before all diffraction measurements the "Z" should be checked. To check the "Z", rotate the specimen holder 10 degrees and observe the image for movement. If the image "slides" across the screen, adjust the "Z" with the setscrew located on the SEG until there is no movement. *If more information is required, see the manufacturers manual.*

9.3 Magnification

Magnification must be measured for each scope for those magnifications used during analysis (typically 10,000X and 19,000X). Measurements are obtained using a 2160 line per mm magnification grating.

The Magnifications used during analysis must be calculated at least monthly. The magnification must be determined on film and on screen.

On Film

1. Insert grid that has 2160 lines/mm grating.
2. Obtain eucentricity and critical focus at the magnification to be measured.
3. Adjust the illumination for photography
4. Record the image on film.
5. Record (at least) the negative number, date, initials and stated magnification into your photo log book
6. Once the pictures are developed and dry you can take measurements off the negative (preferably on a light box). To do this measure the distance across as many squares or lines as possible (avoid measuring all the way to the edges or any rows that show obvious distortion).
7. In the Magnification section of the TEMCAL spreadsheet enter the date, negative number, total lines measured and total mm measured from step 6 above.

The spreadsheet will calculate the actual magnification. The results are reported in the report section as well as graphically (showing percentage from mean and acceptance criteria at 2s<5% of mean) in the graphs section. Both sections are produced each month for the Monthly QC Report.

On Screen

1. Insert grid that has 2160 lines/mm grating.
2. Obtain eucentricity and critical focus at the magnification to be measured.
3. With your on screen measuring aids measure the row spacing of the 2160 grating.
4. In the Magnification section of the TEMCAL spreadsheet enter the date, number of total lines measured and total mm measured.

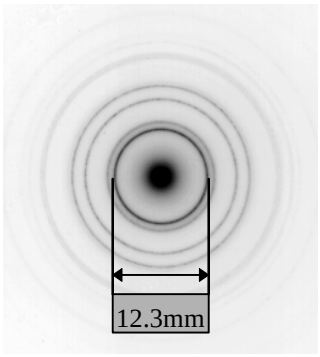
The spreadsheet will calculate the actual magnification. The results are reported in the report section as well as graphically (showing percentage from mean and acceptance criteria at 2s < 5% of mean) in the graphs section. Both sections are produced each month for the Monthly QC Report.

9.4 Camera Constant (CC)

The Camera Constant at the camera length(s) used during analysis must be calculated at least weekly. The camera constant must be determined on film and on screen.

On Film

1. Insert grid that has evaporated gold film on it
2. Obtain eucentricity and critical focus
3. Obtain diffraction pattern consistent with your normal asbestos analysis protocol
4. The typical camera length used for photography is approx. 55cm.
5. Focus the pattern and adjust the illumination for photography
6. Insert beam stop over the center spot
7. Record the diffraction pattern (series of concentric rings) on film.
8. Record (at least) the negative number, date, initials, and camera length into your photo log book
9. Once the pictures are developed and dry you can measure the diameters of the rings (preferably on a light box) as follows:
   - Measure the diameter of the first (innermost) ring at 0, 60, and 120 degrees.
   - Measure the diameter of the third ring at 0, 60, and 120 degrees.

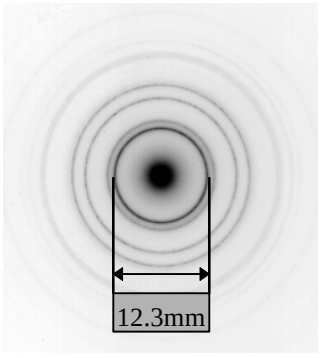


10. In the camera constant section of the TEMCAL spreadsheet enter the date, negative number, camera length stated on scope and the measurements obtained in step 9 above.

The spreadsheet will calculate the actual camera length and camera constant. The results are reported in the report section as well as graphically (showing percentage from mean) in the graphs section. Both sections are produced each month for the Monthly QC Report.

On Screen

1. Insert grid that has evaporated gold film on it
2. Obtain eucentricity and critical focus
3. Obtain diffraction pattern consistent with your normal asbestos analysis protocol
4. The typical camera length used for observation on screen is approx. 22cm.
5. Focus the pattern and adjust the illumination
6. Using the on screen measuring aids, measure the diameters of the first and third rings.



7. In the “on Screen” portion of the camera constant section of the TEMCAL spreadsheet enter the date, camera length stated on scope and the measurements obtained in step 6 above.

The spreadsheet will calculate the actual camera length and camera constant. The results are reported in the report section as well as graphically (showing percentage from mean) in the graphs section. Both sections are produced each month for the Monthly QC Report.

9.5 Chrysotile Beam Dose

Using NIST traceable 1866a chrysotile:

1. Obtain diffraction pattern of a single chrysotile fibril greater than or equal to 1 micron in length and 0.05 microns in width.
2. After 15 seconds observe and record whether the diffraction pattern is still visible.
3. Repeat steps one and two for ten fibrils.
4. In the beam dose section of the TEMCAL spreadsheet record:
    - Date
    - The number of patterns obtained (10)
    - The number of patterns still visible after 15 seconds

The program will calculate the percentage of patterns that are visible for at least 15 seconds. The report page will report this percentage and will be included in the monthly QC Report. Record machine settings used (aperture, spot size, bias, etc.) and post on machine. Use these settings for routine analysis.

9.6 Spot Size

The TEM spot size is measured by:

1. With no sample in the scope, reduce the beam to crossover at the spot size setting used during analysis.
2. Correct any astigmatism with the condenser stigmator.
3. Take a photograph at the shortest exposure time to obtain a photo of a distinct spot without “blooming”. Another way to minimize the blooming effect on the film caused by overexposure is to sweep the spot across the screen while the shutter is open. The result will be a line across the negative. The width of the line is the spot diameter.
4. Record (at minimum) date, negative number, magnification, and spot size in the photo log book.
5. After the negative has been developed and dried, measure the spot size diameter directly off the negative with a mm ruler.

6. In the TEMCAL spreadsheet record:

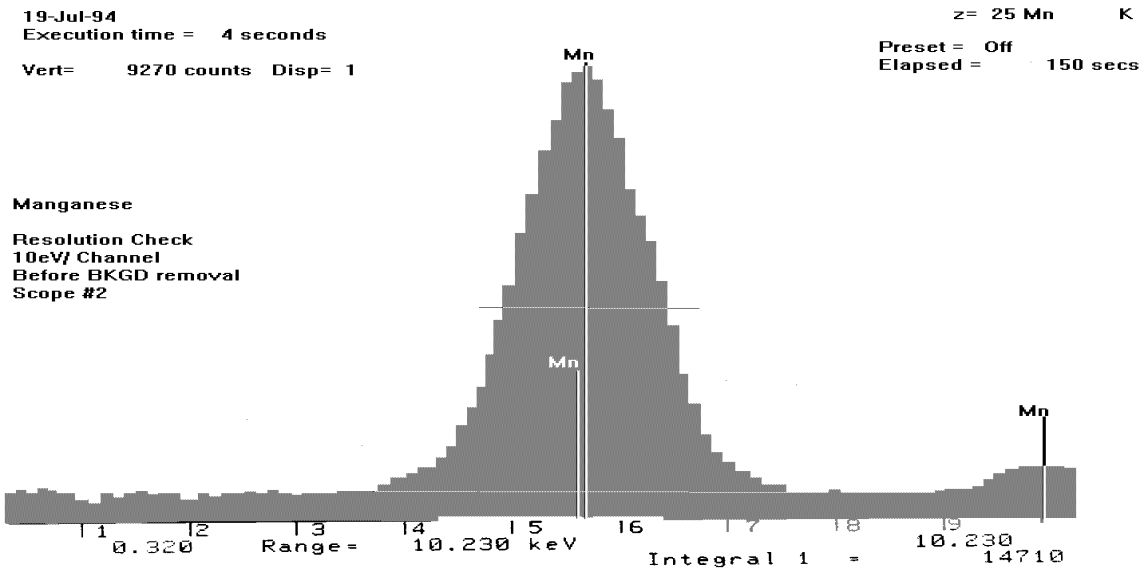
- Date
- Negative number
- The actual magnification from the most recent magnification calibration
- The diameter of the spot in mm measured on the negative (measures the spot along two perpendicular directions and takes an average).

The TEMCAL spreadsheet will calculate the actual diameter of the spot and report that actual diameter as well as whether that diameter passes the QC criteria of less than or equal to 250 nanometers.

9.7 Mn Resolution

The detector resolution is measured using a manganese grid. This must be performed quarterly.

1. Insert Mn grid into scope and set magnification to that used for routine analysis (circa 20,000X)
2. At spot size used for routine analysis, collect a Mn spectrum with approximately 10,000 counts vertical.
3. Spread the spectrum adequately to facilitate recording counts for each channel.
4. In the Resolution section of the TEMCAL spreadsheet record:
    - Date
    - Scope Number
    - KeV/channel
    - A representative low (left of the peak) and high (right of the peak) background count
    - From left to right enter the counts for each channel into the spreadsheet



The spreadsheet will perform the calculations to calculate the resolution at full width half max. The results are shown in the report section of the spreadsheet and are produced as part of the monthly QC report.

If the true resolution is >175 then the detector is in need of servicing. A common culprit is ice in the detector. This can usually be resolved by draining the detector and allowing it to dry to room temperature before slowly refilling.

9.8 Sodium Detection:

Sodium Detection is performed using NIST 1866a crocidolite standard on a quarterly basis.

1. Insert Crocidolite sample into scope
2. Set magnification to that used during analysis (circa 20,000X)
3. With spot size used during analysis, obtain an EDX spectrum for the crocidolite standard. Collect for 200-300 live time seconds in order to get sufficient counts.
4. Record the total counts in the sodium peak
5. Perform background subtraction and record the total counts in the background subtracted peak
6. In the TEMCAL spreadsheet record:
    - Date
    - Analyst's initials
    - Gross Counts from step 4 above
    - Net counts from step five above

The spreadsheet will calculate whether the sodium peak is statistically significant. This will be documented in the report section of the spreadsheet and is part of the Monthly QC Report.

If the Na is not present or significant then the detector may need servicing. A common culprit is ice in the detector. This can usually be resolved by draining the detector and allowing it to dry to room temperature before slowly refilling.

9.9 Al, Cu Calibration

Daily Check

Al, Cu peak calibration check is performed on a daily basis

1. Insert Al/Cu calibration grid into scope
2. At magnification and spot size used during analysis, obtain a spectrum.
3. Using the cursor, record the locations of the center channel of the Al and Cu peaks.
4. Record these 2 values on the Daily TEM calibration sheet
5. The Aluminum peak should be at 1.48 eV +/- 0.02
6. The Copper peak should be at 8.04 eV +/- 0.02

If the peaks do not fall within the specified range, then a full calibration will need to be performed.

Full Calibration

A full auto calibration should be run weekly or anytime the daily check indicates values outside the acceptable range

1. With the Al, Cu grid in the scope, proper mag, and spot size hit the PROGRAM key and choose item 4, Self Test.
2. From the next menu, select 4, EDC Calibration.
3. Type 1, [ENTER]
4. Allow the KEVEX to run through its calibration process and if the statement "Calibration Successful" appears then prints the results and the spectrum. Depress the PROGRAM key to exit the calibration mode.

9.10 Grid Opening Measurement System

Using copper 200 mesch locator grids, grid openings are measured using a phase contrast microscope fitted with a Walton Beckett graticule. The graticule is calibrated monthly using a stage micrometer.

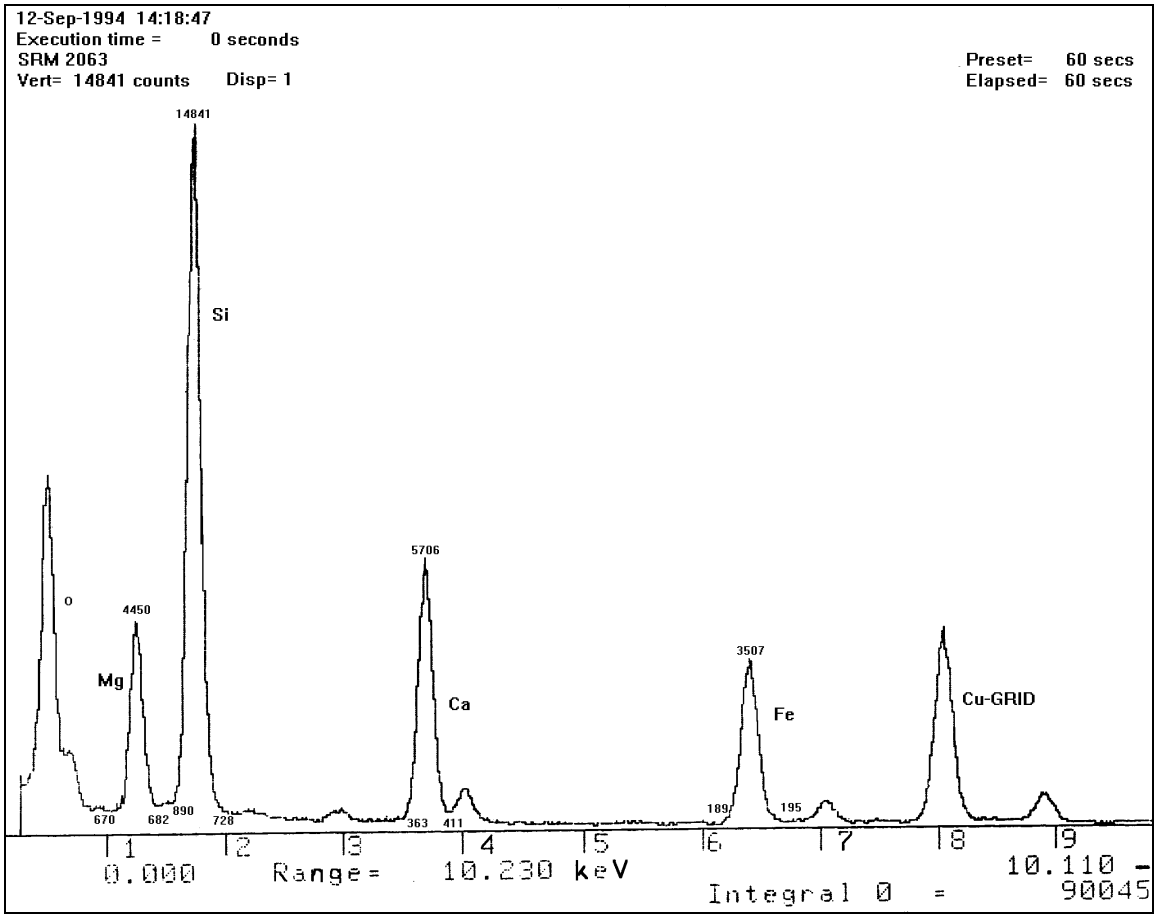
On 2% of each grid lot, measure both length (x-axis) and width (y-axis) on 20 grid openings per grid. Enter this data in the EMSL QC program and calculate the total grid opening area. The variation of the grid openings (defined as 2 X the standard deviation) must not be >5% of the mean.

9.11 K-Factor Calibration

Determination of K-Factors for Mg, Si, Ca and Fe using SRM 2063 or 2063a

1. Insert b2063 into scope an set mag and spot size to that used during routine analysis
2. Collect a spectrum allowing the Si peak to reach 10,000 counts.
3. Run an appropriate background subtraction routine
4. Enter the total counts for Mg, Si, Ca, and Fe peaks into the K-Factor section of the TEMCAL spreadsheet.
5. Repeat steps 1-4 for a total of six runs.

The spreadsheet will calculate the K-Factors for all of the elements as well as for Mg:Fe. The results are documented on the report page of the spreadsheet and are included in the Monthly QC report.



Determination of K-Factors for Na and Al using Albite

K-factors for Na and Al are obtained using an albite standard purchased from a commercial vendor with known composition based on Microprobe analysis.

1. Insert b2063 into scope an set mag and spot size to that used during routine analysis
2. Collect a spectrum for approx. 300 seconds to obtain a large number of Na counts.
3. Run an appropriate background subtraction routine.
4. Enter the total counts for Na and Al peaks into the K-Factor section of the TEMCAL spreadsheet.
5. Repeat steps 1-4 for a total of six runs.

The spreadsheet will calculate the K-Factors for all Na and Al. The results are documented on the report page of the spreadsheet and are included in the Monthly QC report.

10.0 TEM QC PROGRAM

Scope

The following section describes the type and frequency of quality control analysis performed by all EMSL Laboratories. The frequency and type of Quality Control analyses are dictated by the analytical procedures as defined in this document.

Air Analysis

Intra-Analyst QC

At least 2% (1/50) samples analyzed) of analyses are reanalyzed by the same analyst counting the same grid openings. The measure of variance is calculated using the formula

$$R = |(A-B)|/((A+B)/2)|$$

where: R = the measure of variance for the analysis

A = the value of the first analysis in structures

B = the value of the second analysis in structures

Measures of variance (R) are recorded and plotted over time to determine trends and problems in analyses.

The Pass/Fail criteria for the QC analyses are as follows:

< 5 structures = +/- 1 structure

5-20 structures = +/- 2 structures

<20 structures = +/- 3 structures

A failure based on the above criteria will result in a verified analysis in order to determine the cause of the problem. A cumulative record of False Positives, False Negatives, and True Positives based on the verified analysis are kept for each analyst.

Inter-Analyst QC

At least 4% (1/25) of analyses are reanalyzed by a different analyst counting the same grid openings. (Single analyst labs must perform 1/11.) The measure of variance is calculated using the formula

$$R = (A-B)/((A+B)/2)$$

where: R = the measure of variance for the analysis

A = the value of the first analysis in structures

B = the value of the second analysis in structures

Measures of variance (R) are recorded and plotted over time to determine trends and problems in analyses.

The Pass/Fail criteria for the QC analyses are as follows:

< 5 structures = +/- 1 structure

5-20 structures = +/- 2 structures

> 20 structures = +/- 3 structures

A failure based on the above criteria will result in a verified analysis in order to determine the cause of the problem. A cumulative record of False Positives, False Negatives, and True Positives based on the verified analysis are kept for each analyst. Single analysts laboratories are to perform interlaboratory analysis at a rate of 8%.

Verified Analysis

At least 1/100-grid openings analyzed are reanalyzed by at least one other analyst using a verified analysis technique. Structures within a grid opening are either sketched or plotted and their locations are verified. Twenty (20)% of the samples used for verified analysis must contain between 6-40 structures/grid (approximately 1,000 – 5,000 asbestos structures per mm^2), with the exception of verified analysis used to resolve discrepancies.

Inter-Laboratory Analysis

The Laboratories are currently part of a round robin testing program, which includes other area labs. Samples for each type of analysis performed in this Laboratory are exchanged on at least a semi-annual basis at the rate of five samples / quarter. Results are compared and charted.

Standard Reference Material Analysis

SRM 1876b must be analyzed by verified analysis once each year per analyst. Analysts in training are required to perform a greater amount of verified analysis as seen fit by the laboratory manager and as dictated by the analysts QC results.

Laboratory Blanks

A Laboratory blank is an unused filter that has been left open during the collapsing of a sample set. The blank is then collapsed and follows the samples through the remainder of the preparation procedures.

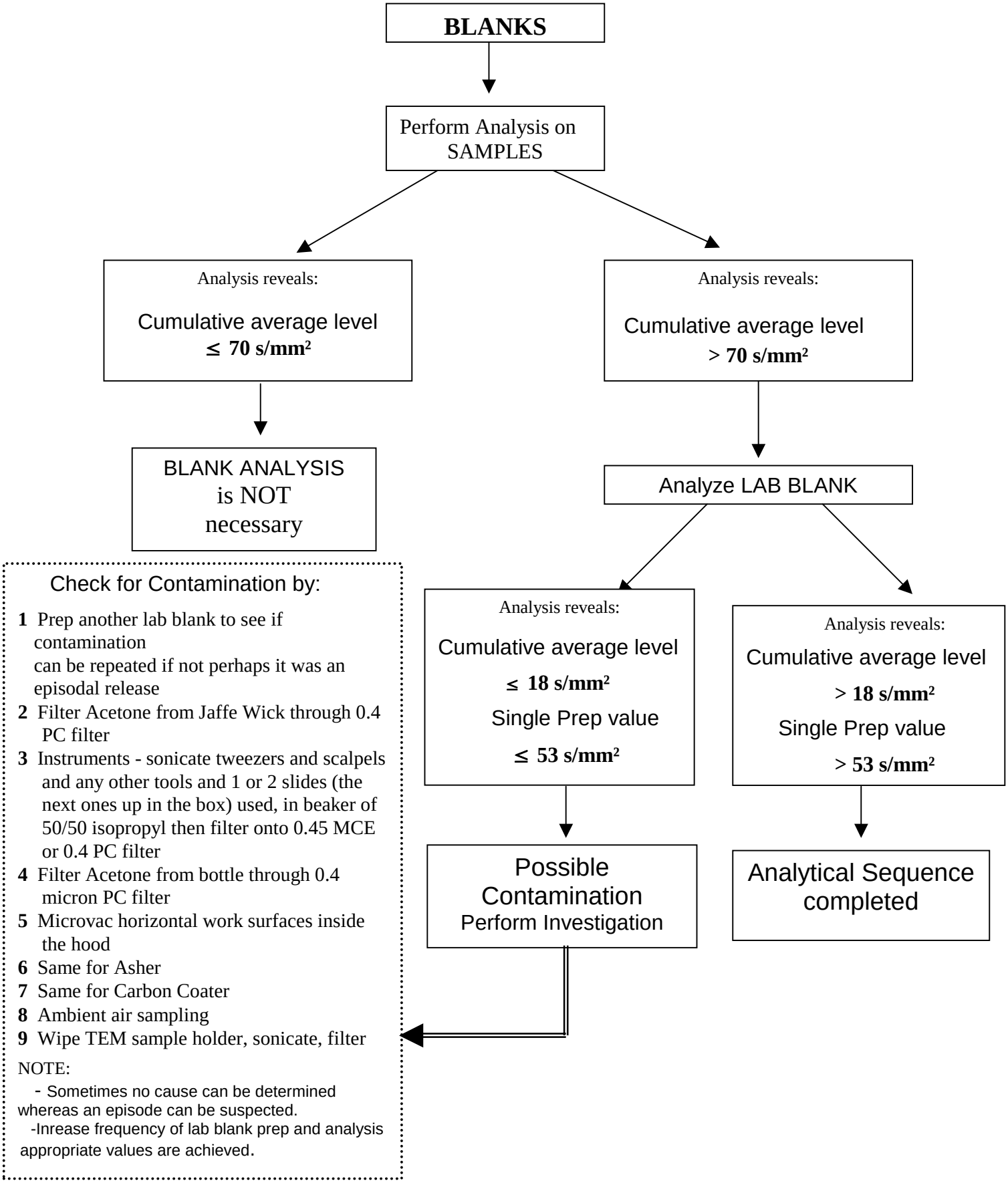
A Laboratory Blank is to be run with each set of samples, or ten percent of the daily total, whichever is greater.

One lab blanks is analyzed for every twenty-five samples prepped. The lab blank will be analyzed for any set of samples that averages greater than 70 structures / mm^2. (See chart on pg. 59)

All Field Blanks submitted with each set of samples for AHERA analysis must be prepped with the set.

A running average is maintained. The average must be less than 18 structures/mm^2, with no single value exceeding 53 structures/mm^2. Should either the average or the single value limits be exceeded, sample preparation stops until the source of contamination is found. Common sources of contamination are tools, reagents, and general housekeeping. When the source of contamination has been identified and eliminated, sample prep may continue. Record incident in equipment log.

CONTAMINATION FLOW CHART
for
AHERA TEM Laboratory Field Blanks


BLANKS
Perform Analysis on SAMPLES
Analysis reveals:
Cumulative average level
≤ 70 s/mm²
Analysis reveals:
Cumulative average level
> 70 s/mm²
BLANK ANALYSIS is NOT necessary
Analyze LAB BLANK
Analysis reveals:
Cumulative average level
≤ 18 s/mm²
Single Prep value
≤ 53 s/mm²
Analysis reveals:
Cumulative average level
> 18 s/mm²
Single Prep value
> 53 s/mm²
Possible Contamination
Perform Investigation
Analytical Sequence completed
Check for Contamination by:
1 Prep another lab blank to see if contamination can be repeated if not perhaps it was an episodal release
2 Filter Acetone from Jaffe Wick through 0.4 PC filter
3 Instruments - sonicate tweezers and scalpels and any other tools and 1 or 2 slides (the next ones up in the box) used, in beaker of 50/50 isopropyl then filter onto 0.45 MCE or 0.4 PC filter
4 Filter Acetone from bottle through 0.4 micron PC filter
5 Microvac horizontal work surfaces inside the hood
6 Same for Asher
7 Same for Carbon Coater
8 Ambient air sampling
9 Wipe TEM sample holder, sonicate, filter
NOTE:
- Sometimes no cause can be determined whereas an episode can be suspected.
-Inrease frequency of lab blank prep and analysis appropriate values are achieved.

Signature Page

In signing this, I acknowledge having read and understood the previous pages of this document.

APPROVED BY:

LABORATORY Manager(print)	LABORATORY Manager (sign)	DATE

READ AND UNDERSTOOD BY:

Print Name	Signature	Date
1)		
2)		
3)		
4)		
5)		
6)		
7)		
8)		

APPENDICIES

Appendix A: Mineral Information

Asbestos:
- Chrysotile
- Anthophyllite
- Actinolite
- Tremolite
- Amosite
- Crocidolite

Non-Asbestos:
- Augite
- Diopside
- Hedenbergite
- Enstatite
- Hyperstene
- Halloysite
- Kaolinite
- Palygorskite
- Talc
- Sepiolite
- Wollastonite
- Vermiculite

ASBESTOS MINERALS

Magnesium Silicates	Chrysotile Anthophyllite
Calcium rich Silicates	Actinolite Tremolite
Iron rich Silicates	Amosite Crocidolite

ANTIGORITE

LIZARDITE
CHRYSOTILE

Class: Phyllosilicate
Group: Serpentine

Antigorite	$Mg_3Si_2O_5(OH)_4$
Chrysotile	$Mg_3Si_2O_5(OH)_4$

Antigorite, Lizardite

Monoclinic; 2/m (Orthorhombic Antigorite & Chrysotile are rare)
a=5.30; b=9.20; c=7.46; *B*=91º24' a:b:c=0.567:1:0.811
Z= 2. d's: 7.30(10), 3.63(8), 2.52(2), 2.42(2), 2.19(1)
Composition: MgO 43, SiO_2 44.1, and H2O 12.9
Ni & Fe may sub. for Mg and Al for Si
Cleavage: {001} basal, perfect. Corrugated, finite layers parallel to {001}

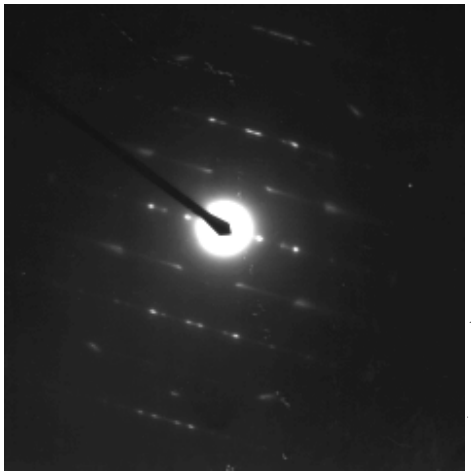
Chrysotile

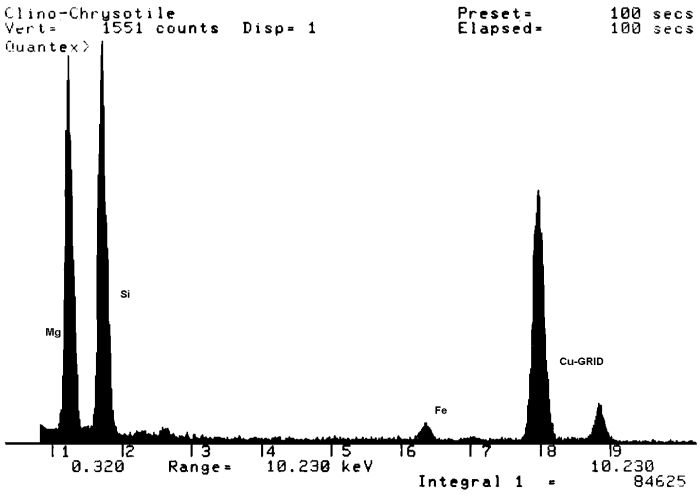
Monoclinic; 2/ m (Orthorhombic Antigorite & Chrysotile are rare)
a=5.34 b=9.25 c=14.65 β=93º16' a:b:c=0.577:1:1.584
Z= 4.
Composition: MgO 43, SiO_2 44.1, and H2O 12.9%
Ni & Fe may sub. for Mg and Al for Si
K - Factor Ratios: Na=0.00, Mg=0.70, Si=1.0, Ca=0.00, Fe<0.02
Cleavage: none. A mismatch between the t and o layers causes the structure to scroll forming cylindrical tubes

Distinguishing features: Chrysotile - hollow, tubular fibrils in transmission electron microscope. Distinctive diffraction pattern. *see Selected Area Electron Diffraction; Sample Patterns* Antigorite; Plate aggregates with perfect cleavage under TEM.

Antigorite & Lizardite; plate-like morphology with EDX similar to chrysotile. Antigorite and lizardite diffraction patterns may be hexagonal to rectangular but chrysotile has the distinct pattern depicted below.

CHRYSOTILE





ANTHOPHYLLITE

Class: Inosilicate
Group: Amphibole

Anthophyllite $(Mg, Fe)_7 Si_8 O_{22} (OH)_2$

Orthorhombic; 2/m 2/m 2/m.

Pmna; a=18.56; b=18.08; c=5.28; β= 102°0' a:b:c=1.027:1:0.292
Z= 4. d's: 8.26(6), 3.65(4), 3.24(6), 3.05(10), 2.84(4)
(210)^(2-10) = 55°

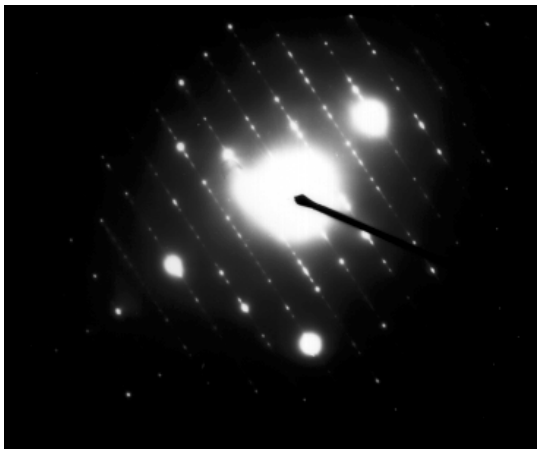
Three Strongest Diffraction Lines:

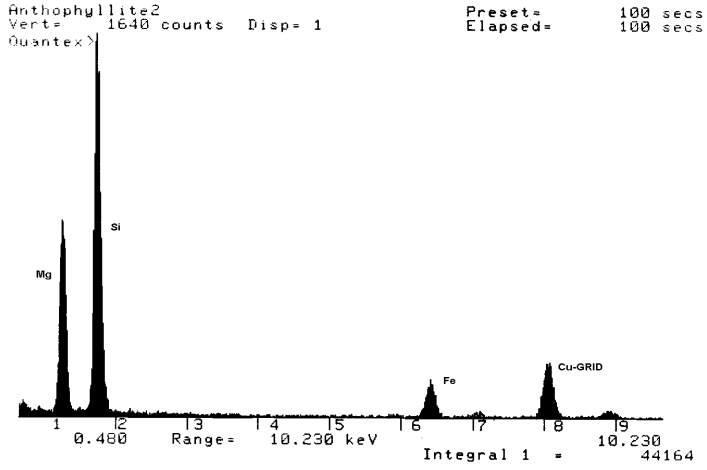
3.05	(100)
3.24	(60)
8.26	(55)

Composition: Forms a solid solution series from $Mg_7Si_8O_{22}(OH)_2$ to $Fe_2Mg_5Si_8O_{22}(OH)_2$
At higher Fe concentrations cummingtonite results.

Distinguishing Features: Thin plate like fibers under TEM. Anthophyllite can form in the same environment as talc and during elemental analysis may contain similar Mg to Si ratios. In this scenario, the diffraction pattern is the key to distinguishing between the two structures. Talc has a hexagonal pattern while anthophyllite primarily yields a series of parallel spots clearly marking the (h,k,0) and the (h,k,l) rows. In addition, the c axis may contain alternate or (missing) spots as in the [010], [140] & [320] zone axis.

ANTHOPHYLLITE





CUMMINGTONITE
GRUNERITE (Amosite Asbestos)

Class: Inosilicate
Group: Amphibole

Cummingtonite $(Mg, Fe)_7 Si_8 O_{22} (OH)_2$
Grunerite $Fe_7 Si_8 O_{22} (OH)_2$

Monoclinic; (Cummingtonite 2/m; Grunerite C2/m).

a= 9.59, b= 18.44, c= 5.34, β= 102°0'. Unit cell length decreases with increase in Mg.
a:b:c= 0.520:1:0.289
Z= 2. d's; 9.21(5), 8.33(10), 3.07(8), 2.76(9), 2.51(6)

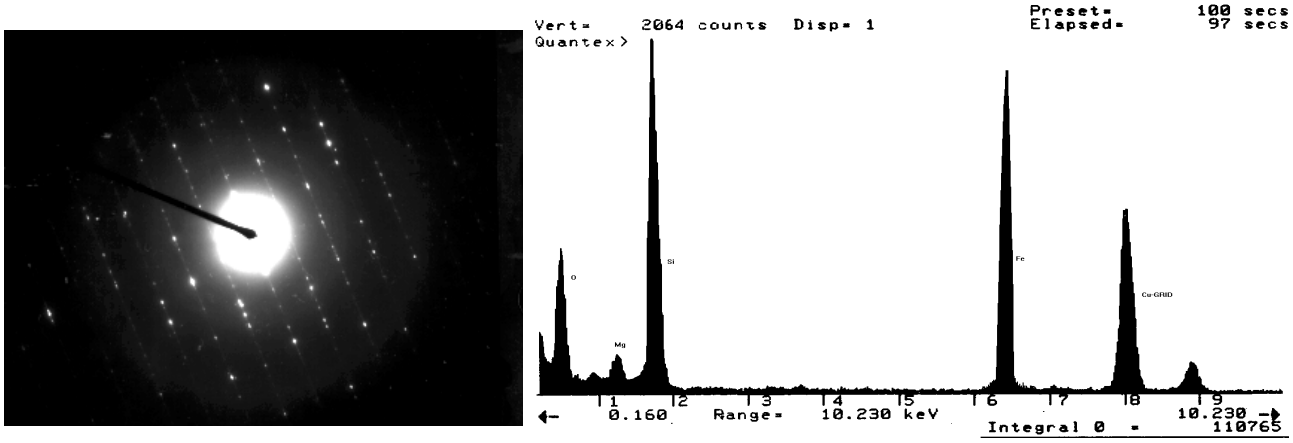
Three Strongest Diffraction Lines for Grunerite:

8.33	(100)
2.77	(90)
3.07	(80)

Composition: A solid solution series exists between cummingtonite and grunerite starting from approximately $Fe_2 Mg_5 Si_8 O_{22} (OH)_2$ to $Fe_7 Si_8 O_{22} (OH)_2$. Members with an atomic percentage of Mg > Fe are refereed to as cummingtonite. Thirty (30) atomic percent is used as a division between members. Al_2O may range up to 0.4 weight percent and CaO as high as 0.9 percent.

Distinguishing Features: Fe up to 80% that of Si. Thin plate like fibers under TEM with distinctive, easily obtained diffraction patterns.

AMOSITE:



GLAUCOPHANE
RIEBECKITE (Crocidolite Asbestos)

Class: Phyllosilicate
Group: Amphibole

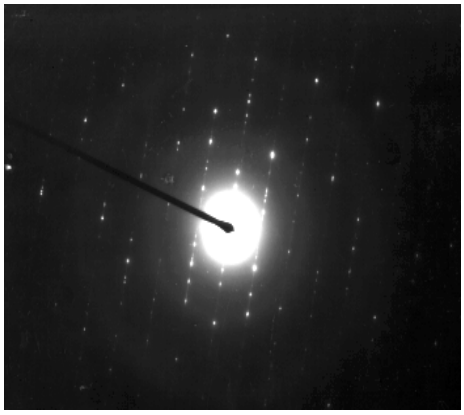
Glaucophane $Na_2 Mg_3 Al_2 Si_8 O_{22} (OH)_2$
Riebeckite $Na_2 Fe_3^{2+} Fe_2^{3+} Si_8 O_{22} (OH)_2$

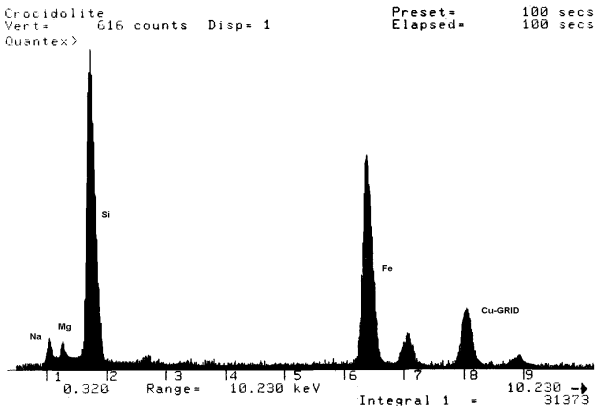
Monoclinic; 2/m
C2/m; a=9.58 (9.769); b=17.80 (18.048); c=5.30 (5.335);
a=90°, β=103°48' (103°59'), *y*=90°; a:b:c = 0.538:1:0.298
Z= 2. d's: 8.42(10), 4.52(5), 3.43(6), 3.09(8), 2.72(10).
2V = 40 - 90°; Y = b, Z^c = 8°; X < Y < Z.

Composition: The composition changes with the substitution of Fe^{2+} for Mg and Fe^{3+} for AL.

Care should be taken during chemical analysis as the elemental make up of Riebeckite may closely resemble that of Amosite. The distinguishing feature that is most prominent is the occurrence of Na. (Also known as crocidolite in its fibrous form.)

CROCIDOLITE





ACTINOLITE
TREMOLITE

Class: Inosilicate
Group: Amphibole

Actinolite $Ca_2 (Mg, Fe)_5 Si_8 O_{22} (OH)_2$
Tremolite $Ca_2 Mg_5 Si_8 O_{22} (OH)_2$

Twinning: common

Monoclinic; 2/m

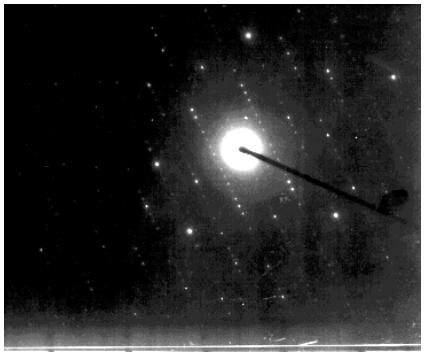
Actinolite, (Tremolite)
C2/m; a=9.86 (9.84); b=18.11 (18.02); c=5.34 (5.27);
a=90° β=105° (104°95') *y*=90° a:b:c= 0.545:1:0.293
Z= 2. d's: 8.38(10), 3.27(8), 3.12(10), 2.81(5), 2.71(9)
Orientation: 2V = 80°; Y = b, Z^c = 15°

Three Strongest Diffraction Lines for Actinolite & Tremolite:

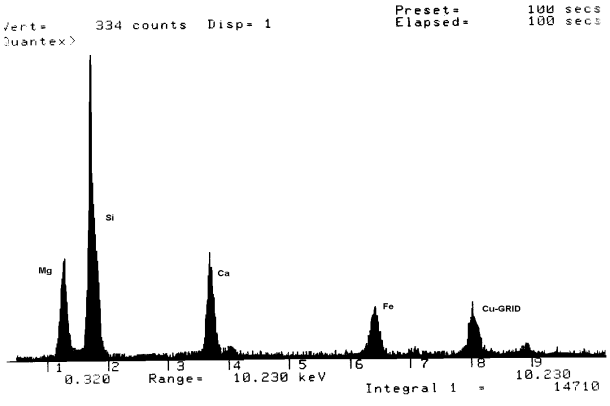
8.38 (100)
3.12 (100)
2.71 (90)

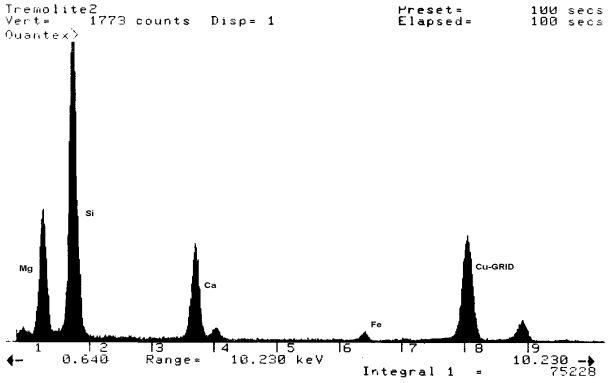
Composition: There is a solid solution series from tremolite to ferroactinolite.
($Ca_2Fe_5Mg_5Si_8O_{22}(OH)_2$.)

Distinguishing features: TEM analysis reveals platy bundles with good cleavage giving the edges a choppy look. The chemical composition of the structure is a good indication of its nature.



ACTINOLITE TREMOLITE





NON-ASBESTOS MINERALS

Mineral	Morphology	SAED	EDX
Antigorite	Plate-like	Hexagonal pattern	Similar to chrysotile
Lizardite	Plate-like	Rectangular pattern	Similar to chrysotile
Atapulgite	Tubular like chrysotile	Very faint	Al peak
Halloysite	Tubular like chrysotile	Streaked	Al, no Mg
Palygorskite	Tubular like chrysotile	Streaked	Al peak
Sepiolite	Tubular like chrysotile	Very faint, streaked	Mg:Si ≈ 0.5:1.0
Vermiculite	Plate-like	Hexagonal	Mg:Si ≈ 0.5:1.0
Talc	Plate-like	Hexagonal	Mg:Si ≈ 0.5:1.0
Wollastonite	Fibrous	Streaked	Ca, Si only
Augite	Plate-like may be acicular	Hexagonal or Rectangular pattern	Al peak
Enstatite	Acicular like amphibole	Hexagonal or Rectangular pattern	Mg:Si ≈ 0.5:1.0 + Fe

AUGITE
DIOPSIDE
HEDENBERGITE

Class: Inosilicate
Group: Pyroxene (Clinopyroxene)

Augite $(Ca, Na)(Mg, Fe, Al)(Si, Al)_2O_6$
Diopside $CaMgSi_2O_6$
Hedenbergite $CaFeSi_2O_6$

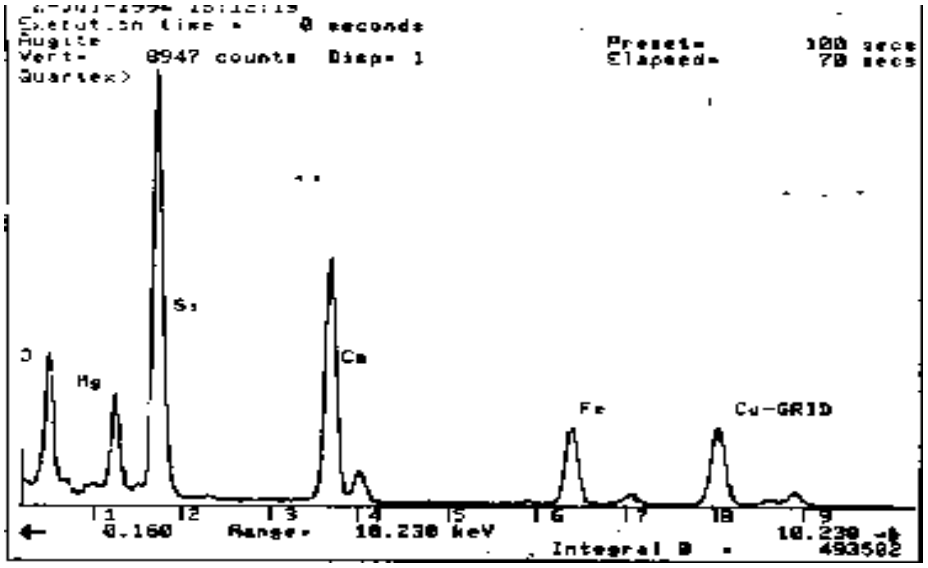
Augite:

Morphology:	Tabular, Plate-like, and non-fibrous
SAED:	Rectangular
EDX:	Presence of Al

Twinning: common

Monoclinic; 2/m
C2/c; a=9.73; b=8.91; c=5.25; β=105°50'; a:b:c=1.092:1:0.589
Augite a=9.755; b=8.928; c=5.204; β=106°11'
Diopside a=9.761; b=8.926; c=5.258; β=105°79'
Hedenburgite a=9.827; b=8.994; c=5.261; β=105°52'
Z= 4. d's: 3.23(8), 2.98(10), 2.94(7), 2.53(4), 1.748(4).
m(110)^m'(1-10)=92°50', c(001)^(p(111)=33°50',
s'(-111)^s'(-1-11)=59°11', c(001)^a(100)=74°10'.

Composition: A solid solution series exists between all members of this group toward acmite-augite. A solid solution series exists between diopside and hedenbergite with Mg and Fe^{2+} substitution. Most of the members of this group have a 1 - 3% Al_2O_3 content. Augite has substitution in Mg - Fe^{2+} and Al substitutes for Mg, Fe^{2+} & Si. Na and Ti may also be present.



ENSTATITE
HYPERSTHENE

Class: Inosilicate
Group: Pyroxene

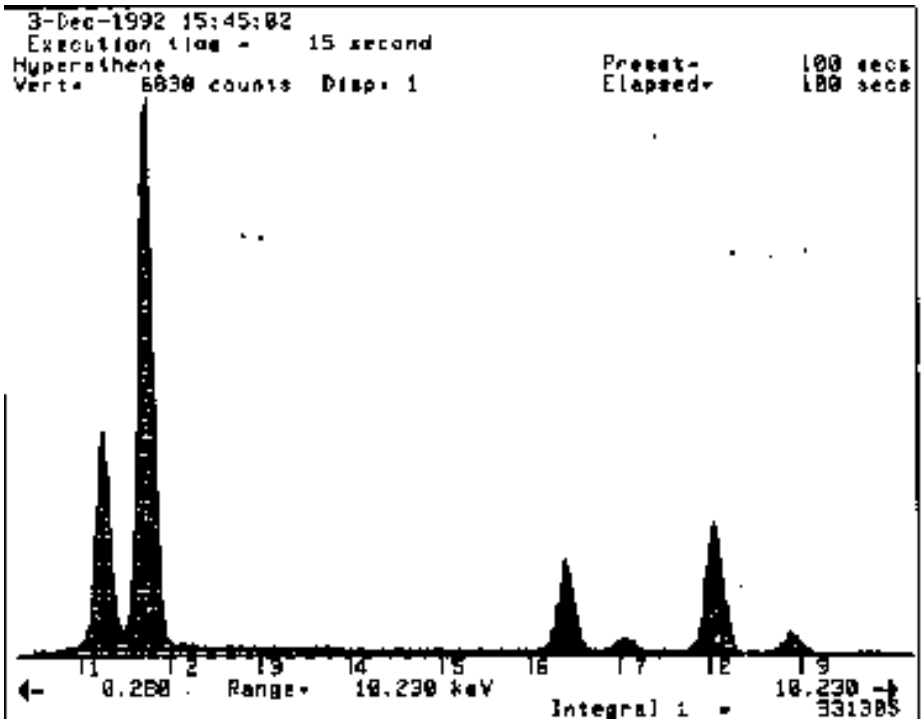
Enstatite $MgSiO_3$
Hypersthene $(Mg, Fe)SiO_3$

Enstatite:

Morphology: Acicular, fibrous, and similar to amphiboles
SAED: Rectangular
EDX: Presence of Fe and Mg:Si ≈ 0.4:1.0

Orthorhombic; 2/m 2/m 2/m
Pbca; a=18.22; b=8.81; c=5.21; β= 90.00 a:b:c=2.068:1:0.590
Z= 8. d's: 3.17(10), 2.94(4), 2.87(9), 2.53(4), 2.49(5)
(210)^(2-10) = 91°44'

Composition: Fe:Mg ratios rarely exceed 1:1. Pure enstatite contains no Fe. Mg may be substituted for by Fe^{2+} up to 90%. Enstatite may resemble chrysotile however the decreased Mg:Si ratio and the higher Fe content are indications as to its true nature.



GYPSUM

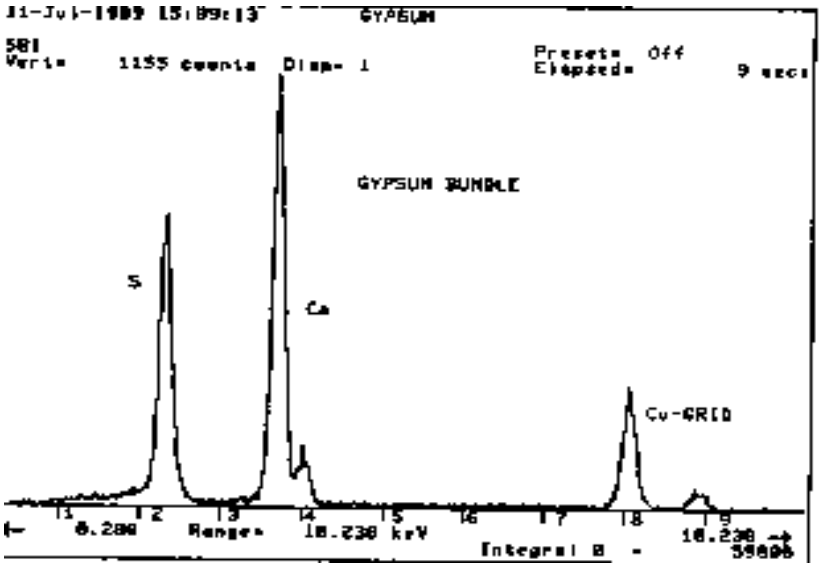
Class: Sulfates
Group: Hydrous/ Basic Sulfates

Gypsum: $CaSo_4 * 2H_2O$

Morphology: Plate-like
SAED: Pattern shows streaking
EDX: S & Ca only

Monoclinic
A2/n; a= 5.68, b= 15.18, c= 6.29; β= 113°50'
a:b:c= 0.374:1:0.414

Distinguishing features: The mineral consists of S & Ca.



HALLOYSITE

Class: Phyllosilicate
Group: Clay Mineral

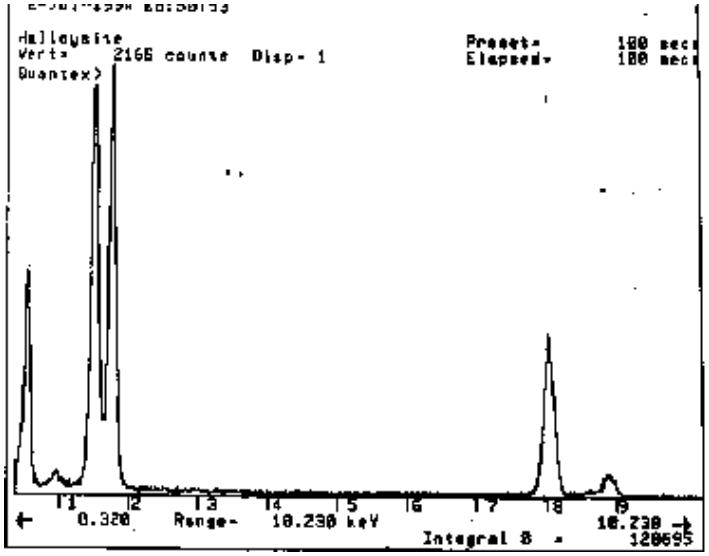
Halloysite $Al_2 Si_2 O_5 (OH)_4$ & $Al_2 Si_2 O_5 (OH)_4 \bullet 2H_2O$ *dehydrates to the first composition with the loss of inner layer water molecules.*

Morphology: Tubular, fibrous similar to chrysotile
SAED: Pattern shows streaking
EDX: Presence of Al and no Mg

Monoclinic
c= *5*.242

Distinguishing features: The high presents of Al will distinguish this clay mineral from asbestos. This Aluminum Silicate may appear in fibrous form with a tubular morphology.

Occurrence: *see also kaolinite*



KAOLINITE

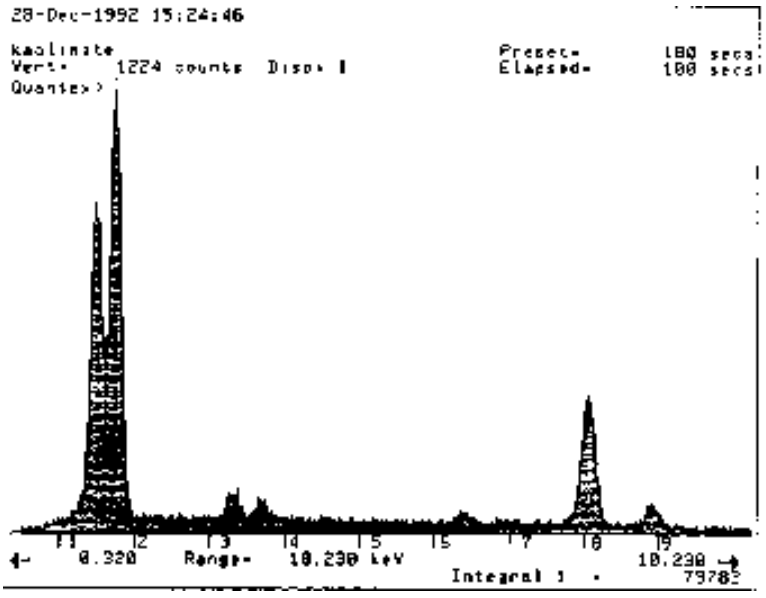
Class: Phyllosilicate
Group: Clay Mineral

Kaolinite $Al_2 Si_2 O_5 (OH)_4$

Triclinic; -1

P-1; a=5.27; b=9.12; c=18.85; β=100º0' a:b:c=0.578:1:2.067
Z= 4. d's: 9.34(10), 4.66(9), 3.12(10), 2.48(7), 1.87(4)
Orientation: 2V=6-30º, Z=b, X⊥{001}; r>v

Composition: MgO 31.7, SiO_2 63.5, H_2O 4.8
Al or Ti may substitute for Si. Fe may substitute for Mg. Care should be taken during TEM analysis. The platy aggregates can easily lie upon one another in a way as to appear tubular in morphology. The distinguishing feature is the hexagonal diffraction pattern quite unlike that of chrysotile. Usually found in clay like masses.



PALYGORSKITE

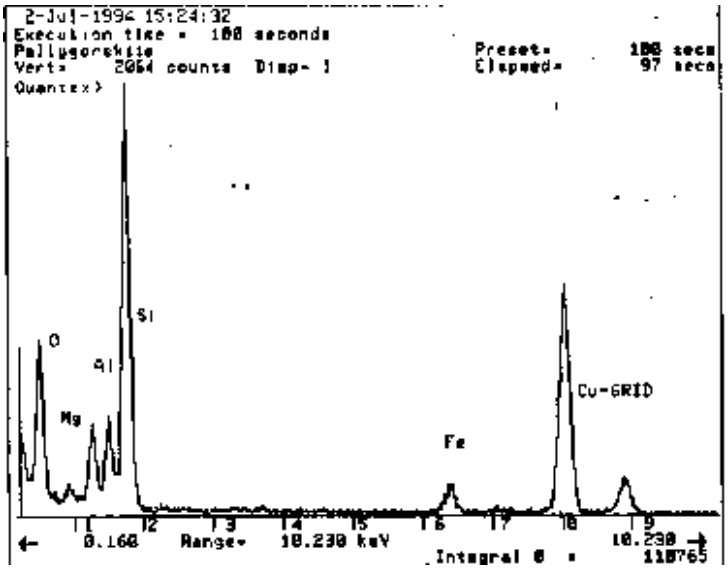
Class: Phyllosilicate
Group: Clay Mineral

Palygorskite $MgAlSi_4(OH)\bullet 4H_2O$
Morphology: Tubular, similar to chrysotile
SAED: Pattern shows streaking
EDX: Presence of Al

Monoclinic
Pn: *a*= 17.864; *b*= 12.681; *c*= *5*.127 β= 92.23°

Distinguishing features: The high presents of Al will distinguish this mineral from asbestos. This Magnesium Aluminum Silicate may appear in fibrous form with a tubular morphology.

Occurrence: *see also kaolinite*



SEPIOLITE (Meerschaum)

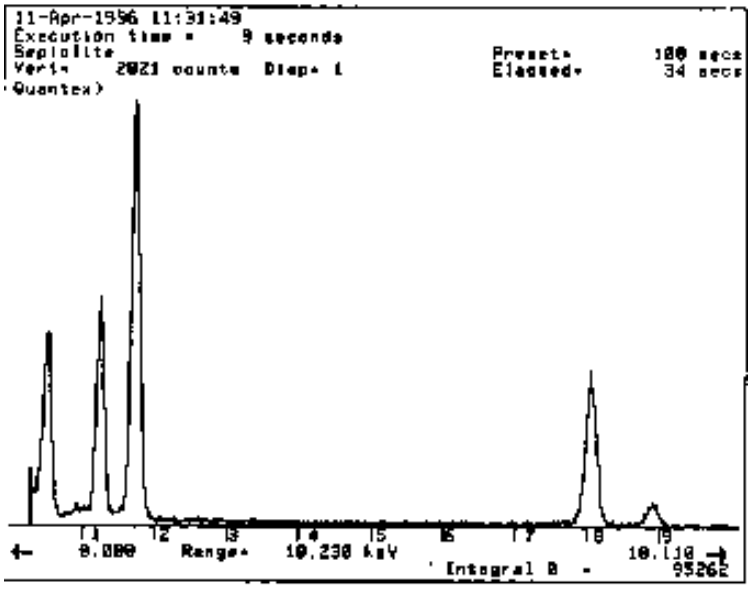
Class: Phyllosilicate
Group: Clay Mineral

Sepiolite $Mg_4Si_6O_{15}(OH)_2 \bullet 6H_2O$

Morphology: Tubular, similar to chrysotile
SAED: Pattern shows streaking, very faint
EDX: Mg:Si ≈ 0.5:1.0

Orthorhombic
Pn: *a*= 13.43; *b*= 26.88; *c*= *5.281*

Distinguishing features: Indistinct outline and poor diffraction patterns from instability in the electron beam. Mg:Si ≈ 0.5:1.0



TALC

Class: Phyllosilicate
Group: Clay Mineral

Talc $Mg_3 Si_4 O_{10} (OH)_2$

Morphology: Lamellar, plate-like, non-fibrous
SAED: Hexagonal spot pattern
EDX: Mg:Si ≈ 0.5:1.0

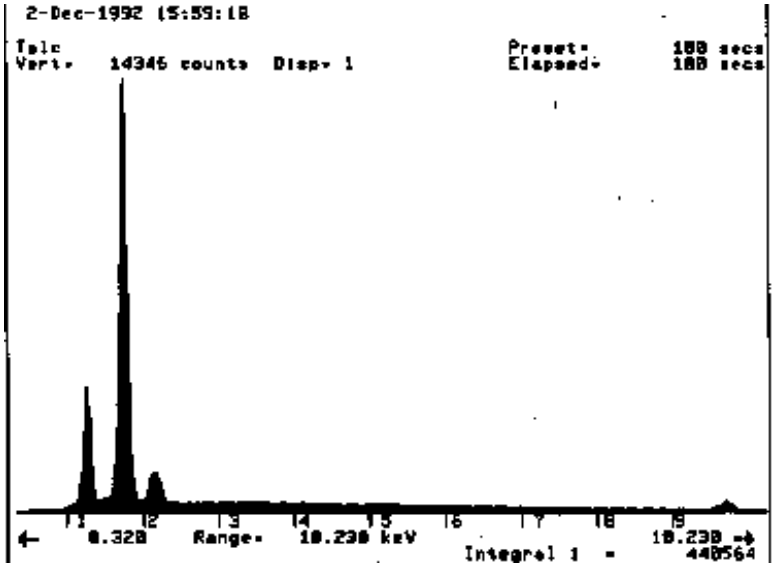
Monoclinic; 2/m
C2/c; a=5.27; b=9.12; c=18.85; β=100º0' a:b:c=0.578:1:2.067
Z= 4. d's: 9.34(10), 4.66(9), 3.12(10), 2.48(7), 1.87(4)
Orientation: 2V=6-30º, Z=b, X⊥{001}; r>v
Composition: MgO 31.7, SiO_2 63.5, H_2O 4.8
Al or Ti may substitute for Si. Fe may substitute for Mg.

Three Strongest Diffraction Lines:

(Monoclinic)		(Triclinic)	
9.35	(100)	9.34	(100)
1.53	(55)	4.56	(80)
4.59	(45)	3.12	(80)

Distinguishing features: TEM usually shows platy or rhombic outline with near perfect cleavage. Diffraction analysis yields distinct hexagonal patterns, which remain constant during tilting.

Care should be taken during TEM analysis. The chemical analysis may yield a breakdown similar to chrysotile asbestos and the platy aggregates can easily lie upon one another in a way as to appear tubular. The distinguishing feature is the hexagonal diffraction pattern quite unlike that of chrysotile.



WOLLASTONITE

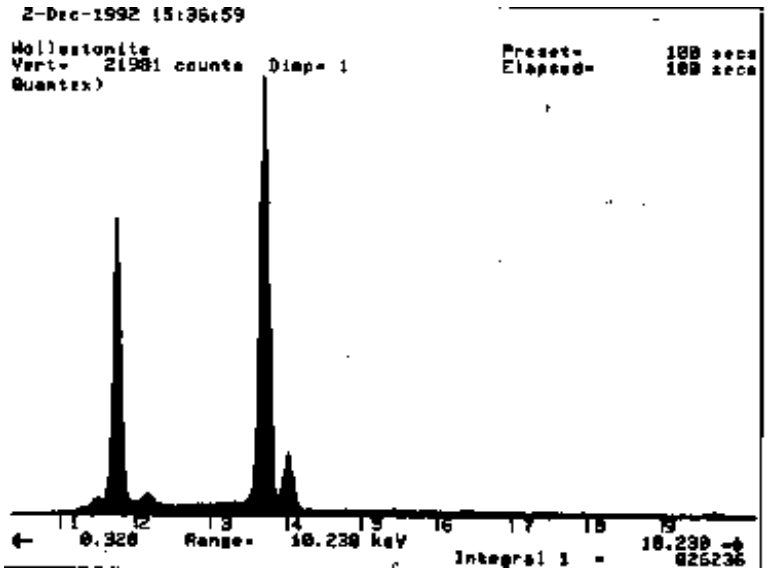
Class: Inosilicate
Group: Pyroxenoid

Wollastonite Ca Si O_3

Morphology: Acicular, fibrous
SAED:
EDX: S and Ca only

Triclinic; (-1)
P-1; a=7.94; b=7.32; c=7.07; α=90°2', β=95°22', γ=103°26'; a:b:c=1.084:1:0.966
Z=6. d's: 3.83(8), 3.52(8), 3.31(8), 2.97(10), 2.47(6)

Composition: CaO 48.3%, SiO_2 51.7% for pure $CaSiO_3$. Some Fe, Mn, & Mg may replace Ca. Pseudowollastonite is stable above 1120°C, has a larger unit cell (Z=24) as compared to (Z=6) of wollastonite.



VERMICULITE

Class: Phyllosilicate
Group: Clay Mineral

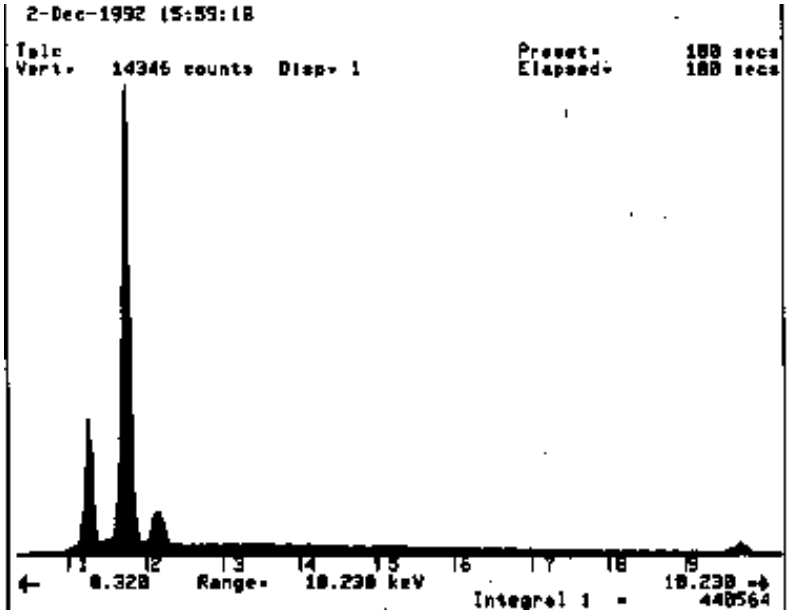
Vermiculite $K(Mg, Fe)_3(AlSi_3O_{10})(OH)_2$

Morphology: Lamellar, plate-like, non-fibrous
SAED: Hexagonal spot pattern
EDX: Presence of Al and possibly K

Monoclinic; C2/m
C2/m; a=5.31; b=9.23; c=10.18; β=99 º18' a:b:c=0.575:1:1.103
Z= 2. d's: 10.1(10), 3.37(10), 2.66(8), 2.54(8), 2.18(8)

Distinguishing features: TEM usually shows platy or rhombic outline with near perfect cleavage. Diffraction analysis yields distinct hexagonal patterns, which remain constant during tilting.

Vermiculite is altered Biotite Mica similar to talc.



APPENDIX III
EMSL's CAPACITY LETTER

USACE Corp of Engineers' Project

Date: **September 17, 2001**

To: **Mr. Aaron Everson**
Company: **Jacobs Sverdrup Companys**
Address: **13723 River Port Drive**
Maryland Heights, MO 63043

EMSL Account Manager:
John C. Van Voorhees (800) 220-3675 x1202

Parameter	Method	Quantity	TAT	Duration	Start Time
PCM	NIOSH 7400	15/Day	12Hr.	66 Days	April 2002
PLM Bulk	NOB 198.1(prep)	35 ea.	24Hr.	1 Set	April 2002
PLM Bulk	NOB 198.1(analy)	10 ea.	24Hr.	1 Set	Jan. 2002
PLM Bulk	NY Strat	98 ea.	24Hr.	1 Set	Jan. 2002
TEM		5/Sets	24Hr.	10 Sets	April 2002
TEM Bulk	198.4	7 ea.	24Hr.	1 Set	Jan. 2002

Statement of laboratory assurance that EMSL can deliver the aforementioned analyses as stated.

The above outline of analysis methods, turnaround times and other stated project specifications have been reviewed with the asbestos manager, Mr. Steven Siegel, and it was concluded that the EMSL corporate laboratory in Westmont, NJ can meet all project requirements within the specified turnaround times. The EMSL Long Island NY laboratory is also certified and capable of handling the analytical work-load as specified for this project.

APPENDIX IV
STL's QUALITY ASSURANCE PLAN

SEVERN TRENT SERVICES

STL St. Louis
13715 Rider Trail North
Earth City, MO 63045

Tel 314 298 8566
Fax 314 298 8757
www.stl-inc.com

STL St. Louis
Laboratory Quality Manual

August 2001

Approved by:

Laboratory Director: _____ Date: 8/17/01
William Deckelmann

QA Manager: _____ Date: 08-17-01
Jim Kleszczewski

Prepared By: _____ Date: 8/15/01
Nancy Julian

Table of Contents

Tables

Figures

Appendices

1. Introduction, Purpose, and Scope

1.1. STL St. Louis Overview

STL St. Louis is located in Earth City, Missouri and has been offering full-service environmental testing since 1985. Severn Trent Laboratories, Inc. acquired this facility in February of 2000. The 31,000 square foot laboratory offers inorganic, organic, and radiological analyses.

STL St. Louis is a part of the Severn Trent Laboratories, Inc. (STL), a national group of 35 laboratory facilities; 29 in the United States and six in the United Kingdom. STL is a part of Severn Trent Services Inc. (STS); a major group of U.S. based companies with 5,000 employees throughout U.S., Europe, and Asia pacific. Both companies are owned by Severn Trent Plc., a British water, waste and utility services company, one of the top publicly traded companies in the United Kingdom, employing some 13,500 people.

STL's group of laboratories offers a broad range of environmental testing services provided by over two thousand professionals in the US. STL's testing capabilities include chemical, physical, and biological analyses of a variety of matrices, including aqueous, solid, drinking water, waste, tissue, air and saline/estuarine samples. Specialty capabilities include dioxin and furan analysis, air toxics, radiological testing, geotechnical testing, tissue preparation and analysis, aquatic toxicology, asbestos analysis, microscopy services, High Resolution Mass Spectrometry (HRMS), Inductively Coupled Plasma/MS (ICP/MS), Liquid Chromatography/MS (LC/MS), and on-site technologies including mobile laboratories.

1.2. Quality Assurance Policy

It is STL St. Louis' policy to:

- provide high quality, consistent, and objective environmental testing services that meet all federal, state, and municipal regulatory requirements.
- generate data that are scientifically sound, legally defensible, meet project objectives, and are appropriate for their intended use.
- provide STL St. Louis clients with the highest level of professionalism and the best service practices in the industry.
- build continuous improvement mechanisms into all laboratory, administrative, and managerial activities.
- maintain a working environment that fosters open communication with both clients and staff.

1.3. Management Commitment to Quality Assurance

STL St. Louis management is committed to providing the highest quality data and the best service in the environmental testing industry. To ensure that the data produced and reported by STL St. Louis meet the requirements of its clients and comply with the letter and spirit of municipal, state and federal regulations, STL St. Louis maintains a Quality System that is clear, effective, well communicated, and supported at all levels in the company.

STL Mission Statement

We enable our customers to create safe and environmentally favorable policies and practices, by leading the market in scientific and consultancy services. We provide this support within a customer service framework that sets the standard to which others aspire. This is achieved by people whose professionalism and development is valued as the key to success and through continued investments in science and technology.

1.4. Purpose

The purpose of the St. Louis Laboratory Quality Manual (LQM) is to describe the STL St. Louis Quality System and to outline how that system enables all employees of STL St. Louis to meet the Quality Assurance (QA) policy. The LQM also describes specific QA activities and requirements and prescribes their frequencies. Roles and responsibilities of management and laboratory staff in support of the Quality System are also defined in the LQM.

1.5. Scope

The requirements set forth in this document are applicable to all STL St. Louis employees. Where the document uses the terms "must" and "shall", this denotes required activities. Where a practice is described, this denotes guidelines as to how those activities are generally performed.

STL St. Louis has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the Corporate Quality Management Plan (QMP) conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The STL St. Louis LQM shall take precedence over the QMP in those cases. Secondarily, STL St. Louis has the responsibility and authority to operate in compliance with documented client

requirements, where they do not conflict with regulatory requirements. STL St. Louis shall not enter any client agreements that conflict with regulatory requirements in the jurisdiction in which the work is performed. Where documented client agreements conflict with the LQM, but meet the regulatory requirements of the jurisdiction in which the work is performed, the client agreements shall supercede requirements in the LQM.

STL St. Louis operates under the regulations and guidelines of the following federal programs:

US Army Corp of Engineers, Hazardous, Toxic and Radioactive Waste (USACE HTRW)
Clean Water Act (CWA)
Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)
Department of Energy (DOE)
National Pollution, Discharge, and Elimination System (NPDES)
Nuclear Regulatory Commission (NRC)
Resource Conservation and Recovery Act (RCRA)
Safe Drinking Water Act (SDWA)
Toxic Substances Control Act (TSCA)

STL St. Louis also provides services under various state and local municipal guidelines. A current list of analytical methodologies is provided in Appendix A.

The St. Louis Laboratory Quality Manual (LQM) undergoes on annual review by the QA Manager, the Laboratory Director, and the Technical Director. It is a joint responsibility of the QA Manager, the Laboratory Director, the Technical Director, and the Group Leaders to ensure that all associates have a working knowledge of the LQM and its supporting documentation.

2. References

The following references were used in preparation of this document and as the basis of the STL St. Louis' Quality System:

STL Quality Management Plan, M-Q-001, revision 4, April 24, 2001.

EPA Guidance for the Preparation of Standard Operating Procedures (SOPs) for Quality Related Documents, US EPA, Office of Research and Development, EPA QA/G-6, November 1995.

EPA Requirements For Quality Management Plans, EPA QA/R-2, US EPA Management Staff, Washington, DC, Draft Interim Final, August 1994.

EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5, US EPA Quality Staff, Washington, DC, Interim Final, November 1999.

EPA Quality Manual for Environmental Programs, 5360, US EPA Office of Research and Development, National Center for Environmental Research and Quality Assurance, Quality Assurance Division, July 1998.

General Requirements for the Competence of Testing and Calibration Laboratories, ISO/IEC 17025, December 1999.

Good Automated Laboratory Practices, EPA 2185, 1995.

Quality Assurance Project Plan, HQ Air Force Center for Environmental Excellence, Version 3.0, March 1998.

National Environmental Laboratory Accreditation Conference, Constitution, Bylaws, and Standards, EPA600/R-98/151, US EPA Office of Research and Development, July 1999.

Quality Systems Manual for Environmental Laboratories, Department of Defense, Version 1, October 2000.

Model Statement of Work for Analytical Laboratories, Department of Energy, Revision 3, February, 2001.

Shell for Analytical Chemistry Requirements, US Army Corps of Engineers, December 1998.

3. Terms and Definitions

Accuracy: the degree of agreement between a measurement and true or expected value, or between the average of a number of measurements and the true or expected value.

Audit: a systematic evaluation to determine the conformance to specifications of an operational function or activity.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Chain of Custody (COC): an unbroken trail of accountability that ensures the physical security of samples, data and records.

Clean Air Act: legislation in 42 U.S.C. 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended.

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund): legislation (42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and reauthorization Act of 1986 (SARA), 42 U.S.C. 9601et seq.

Compromised Sample: a sample received in a condition that jeopardizes the integrity of the results. See Section 4.7.1 for a description of these conditions.

Confidential Business Information (CBI): information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products.

Confirmation: verification of the presence of a component using an additional analytical technique. These may include second column confirmation, alternate wavelength,

derivatization, mass spectral interpretation, alternative detectors, or additional cleanup procedures.

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect or other undesirable situation in order to prevent recurrence.

Data Audit: a qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Equipment Blank: a portion of the final rinse water used after decontamination of field equipment; also referred to as Rinsate Blank and Equipment Rinsate.

Document Control: the act of ensuring that documents (electronic or hardcopy and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA): legislation under 7 U.S.C. 135 et seq., as amended.

Federal Water Pollution Control Act (Clean Water Act, CWA): legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat. 816.

Field Blank: a blank matrix brought to the field and exposed to field environmental conditions.

Field of Testing (FOT): a field of testing is based on NELAC's categorization of accreditation based on program, matrix, analyte.

Good Laboratory Practices (GLP): formal regulations for performing basic laboratory operations outlined in 40 CFR Part 160 and 40 CFR Part 729 and required for activities performed under FIFRA and TSCA.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Instrument Blank: a blank matrix that is the same as the processed sample matrix (i.e. extract, digestate, condensate) and introduced onto the instrument for analysis.

Internal Chain of Custody: an unbroken trail of accountability that ensures the physical security of samples, data and records. Internal Chain of Custody refers to additional documentation procedures implemented within the laboratory that includes special sample storage requirements, and documentation of all signatures and/or initials, dates, and times of personnel handling specific samples or sample aliquots.

Instrument Detection Limit (IDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is $\pm$100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Laboratory Quality Manual (LQM): a document stating the quality policy, quality system and quality practices of the laboratory. The LQM may include by reference other documentation relating to the laboratory's quality system.

Limit of Detection (LOD): the minimum amount of a substance that an analytical process can reliably detect.

Matrix: the substrate of a test sample. Common matrix descriptions are defined in Table 2.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate; Laboratory Duplicate.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a replicate matrix spike.

Table 1. Matrix Descriptions

Matrix	Description
Air	Air samples as analyzed directly or as adsorbed into a solution or absorption matrix and desorbed.
Aqueous	Aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine source. Includes surface water, groundwater and effluents.
Drinking Water	Aqueous sample that has been designated a potable water source.
Saline	Aqueous sample from an ocean or estuary, or other salt-water source such as the Great Salt Lake.
Liquid	Liquid with <15% settleable solids.
Solid	Soil, sediment, sludge or other matrices with ≥15% settleable solids.
Waste	A product or by-product of an industrial process that results in a matrix not previously defined.
Tissue	Sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs using a specific method. Quantitative results are not produced in this range.

Non-conformance: an indication, judgement, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Precision: an estimate of variability. It is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical and/or biological integrity of the sample.

Proficiency Testing: determination of the laboratory calibration or testing performance by means of inter-laboratory comparisons.

Proficiency Test (PT) Sample: a sample, the composition of which is unknown to the analyst, that is provided to test whether the analyst/laboratory can produce analytical results within specified performance limits. Also referred to as Performance Evaluation (PE) Sample.

Proprietary: belonging to a private person or company.

Quality Assurance (QA): an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Assurance Project Plan (QAPP): a formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.

Quality Control (QC): the overall system of technical activities, the purpose of which is to measure and control the quality of a product or service.

Quality Control Sample: a control sample, generated at the laboratory or in the field, or obtained from an independent source, used to monitor a specific element in the sampling and/or testing process.

Quality Management Plan (QMP): a formal document describing the management policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an agency, organization or laboratory to ensure the quality of its product and the utility of the product to its users.

Quality System: a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA/QC.

Quantitation Limit (QL): the minimum amount of a substance that can be quantitatively measured with a specified degree of confidence and within the accuracy and precision guidelines of a specific measurement system. The QL can be based on the MDL, and is generally calculated as 3-5 times the MDL, however, there are analytical techniques and methods where this relationship is not applicable. Also referred to as Practical

Quantitation Level (PQL), Estimated Quantitation Level (EQL), Limit of Quantitation (LOQ).

Raw Data: any original information from a measurement activity or study recorded in laboratory notebooks, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic/optical media, including dictated observations, and recorded data from automated instruments. Reports specifying inclusion of "raw data" do not need all of the above included, but sufficient information to create the reported data.

Record Retention: the systematic collection, indexing and storing of documented information under secure conditions.

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Resource Conservation and Recovery Act (RCRA): legislation under 42 USC 321 et seq. (1976).

Safe Drinking Water Act (SDWA): legislation under 42 USC 300f et seq. (1974), (Public Law 93-523).

Sampling and Analysis Plan (SAP): a formal document describing the detailed sampling and analysis procedures for a specific project.

Selectivity: the capability of a measurement system to respond to a target substance or constituent.

Sensitivity: the difference in the amount or concentration of a substance that corresponds to the smallest difference in a response in a measurement system using a certain probability level.

Spike: a known amount of an analyte added to a blank, sample or sub-sample.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

Storage Blank: a blank matrix stored with field samples of a similar matrix.

Systems Audit: a thorough, systematic, on-site, qualitative review of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system.

Test Method: defined technical procedure for performing a test.

Toxic Substances Control Act (TSCA): legislation under 15 USC 2601 et seq., (1976).

Traceability: the property of a result of a measurement that can be related to appropriate international or national standards through an unbroken chain of comparisons.

Trip Blank: a blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Verification: confirmation by examination and provision of evidence against specified requirements.

4. Management Requirements

4.1. Organization and Management

4.1.1. Organization

STL's corporate organizational structure is presented in the STL Quality Management Plan (QMP). Corporate employees are located at various STL facilities as outlined in the organizational structure. The STL St. Louis QA Manager has an indirect reporting relationship to the QA Director. STL St. Louis' organizational chart, dated August 1, 2001, can be found in Figure 1 and current versions are available electronically in \\Qlstmo05\qa\Personnel\STL_ORG.

4.1.2. Roles and Responsibilities

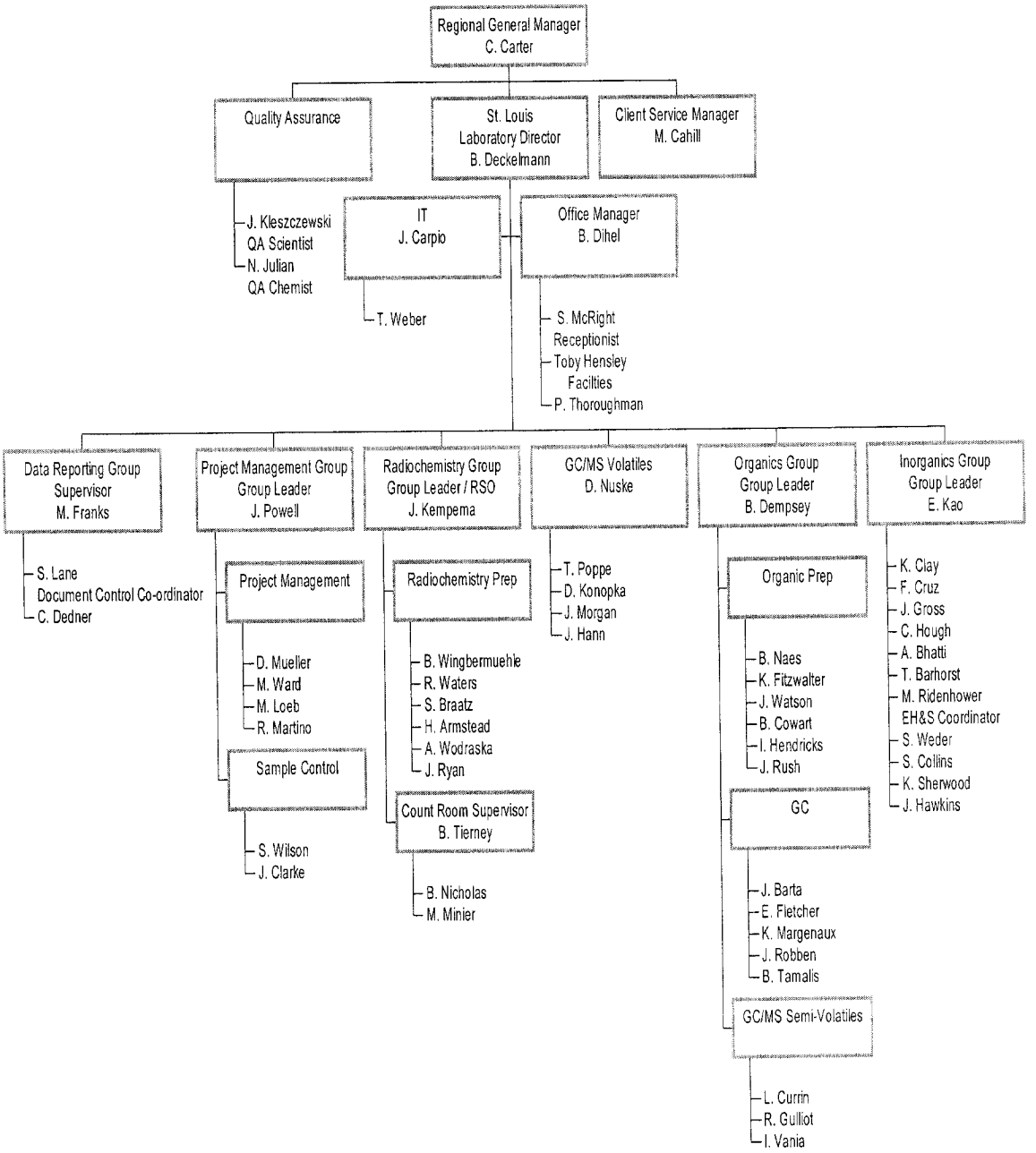
General Manager (GM)

The GM is directly responsible for the daily operations of one or more operating facilities within STL. The GM's responsibilities include allocation of personnel and resources, long term planning, setting goals, and achieving the financial, business, and quality objectives of STL. The GM ensures timely compliance with corporate management directives, policies, and management systems reviews.

Laboratory Director

The Laboratory Director oversees the daily operations of the laboratory. The Laboratory Director's responsibilities include supervision of staff, setting goals and objectives for both the business and the employees, and achieving the financial, business, technical and quality objectives of the facility. The Laboratory Director ensures timely compliance with audits and corrective actions, and is responsible for maintaining a working environment which encourages open, constructive problem solving and continuous improvement.

Figure 1. STL St. Louis Organizational Chart
August 1st, 2001



QA Manager

The QA Manager is responsible for ensuring that the laboratory's quality system and LQM meet the requirements set forth in the QMP, providing Quality Systems training to all new personnel, maintaining a Laboratory Quality Manual (LQM), and performing systems, data, special, and external audits. The QA Manager oversees the maintenance of QC records, maintains certifications, submits monthly QA Reports, and assists in reviewing new work as needed. The QA Manager shall have the final authority to accept or reject data, and to stop work in progress in the event that procedures or practices compromise the validity and integrity of analytical data. The QA Manager is available to any employee at the facility to resolve data quality or ethical issues. The QA Manager shall be independent of laboratory operations.

Technical Director

The Technical Director(s) of a laboratory has overall responsibility for a defined portion of the technical operations of the laboratory, and may or may not be the Laboratory Director. The Technical Director solves day to day technical issues, provides technical training and guidance to staff, project managers, and clients, investigates technical issues identified by QA, and directs evaluation of new methods.

Customer Service Manager

The Customer Service Manager (CSM) develops and manages responses to clients for bids, quotes, and proposals. The CSM conducts technical and business evaluations of bids and proposals. The CSM is responsible for the communication of information relative to clients, contacts, and status of opportunities to Account Executives, Business Development Directors, and Laboratory Directors.

Project Manager

The Project Managers (PM) are instrumental in assisting both laboratory and the client during the course of a project. The PM coordinates laboratory services directly with clients to ensure understanding of contractual requirements and effectively communicate client needs to laboratory personnel. The PM notifies clients regarding specific non-conformances and difficulties encountered in the laboratory. The PM monitors the analytical progress, compiles analytical reports

and reviews reports for completeness. The PM is designated by the Laboratory Director as a report approval signatory.

Group Leader

The Group Leader reports directly to the Laboratory Director. They are responsible for the daily activities of analyses. The Group Leader supervises the bench level chemists and data review within the group. They manger the group's laboratory operations including; work scheduling, sample tracking, and prompt reporting of results. The group leader is responsible for the technical secondary review of raw data for accuracy and completeness, checking calibrations and calculations and reconciling any non-compliant data. They also ensure that all instrumentation and equipment meets performance criteria and schedule instrument repairs.

Chemist/Analyst

Laboratory analysts are responsible for the generation of data by preparing and analyzing samples according to written SOPs and client requirements. They are responsible for understanding the requirements in the LQM and the SOPs associated with their specific function. The perform the initial technical review of sample preparation information, calculations, qualitative identifications and raw data with the authority to stop, accept, or reject data based on compliance with sell-defined QC criteria. The laboratory analyst also provides prompt documentation and notification to the Group Leader of problems or anomalies detected. They also monitor, calibrate, and maintain standard laboratory equipment such as refrigerators, ovens, water systems, and pipettes as necessary.

Sample Control Personnel

Sample Control Personnel is responsible for the receipt and handling of samples within the laboratory. They implement proper sample acceptance policies, sample receipt procedures, and sample preservation. They complete and review external and internal chain-of-custody as appropriate. Sample Control communicates and records anomalies associated with condition upon receipt of samples to the PM. The are responsible for assigning a laboratory identification number to each sample and the data entry of samples into the Laboratory Information Management System (LIMS). They secure and monitor sample storage and assist with sample disposal.

Data Reporting Staff

The Data Reporting Staff are responsible for compiling and archiving data results and analytical reports. They compile analytical reports and provide data packages and electronic data deliverables (EDD) according to client requirements. They also ensure that all aspects of data deliverable production, organization, contract compliance screening, archival storage, packaging and data delivery operations are performed according to client requirements.

Information Technology Department

The Information Technology (IT) Department is responsible for the design and maintenance of the laboratory's computer hardware and software. IT personnel implement and validate new data systems and provide hardware and software maintenance. They are responsible for the administration of the Network, e-mail, and LIMS systems. They provide support and training to all computer users.

For further details of roles and responsibilities, refer to the STL Intranet Web Site on the Human Resources-Job Descriptions web page.

Table 2. Essential Personnel List

Associate Name	Title	Degree	Years of Related Experience
Bill Deckelmann	Laboratory Director & Technical Director	BS, Biology	16
Jim Kleszczewski	QA Manager	Biology	17
Martha Cahill	Customer Service Manager	BS, Pre-Medicine	23
John Powell	Project Management Group Leader	MS, Engineering and Policy; BS, Chemistry	15
Ed Kao	Inorganic Chemistry Group Leader	BS, Biology	13
Bob Dempsey	Organic Chemistry Group Leader	BS, Chemistry	11
Joel Kempema	Radiochemistry Group Leader	BA, Chemistry	11
John Carpio	IT Manager	Biology	16

4.2. Quality System

4.2.1. Objectives of STL St. Louis Quality System

The goal of the STL St. Louis Quality System is to ensure that business and technical operations are conducted with the highest standards of professionalism in the industry.

To achieve this goal, it is necessary to provide STL St. Louis clients with not only scientifically sound, well documented, and regulatory compliant data, but also to ensure that STL St. Louis provides the highest quality service available in the industry. A well-structured and well-communicated Quality System is essential in meeting this goal. STL St. Louis' Quality System is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

The QMP is the basis and outline for STL's Quality System and contains requirements and general guidelines under which all STL facilities shall conduct their operations. The LQM is a continuation of the QMP. It further outlines the STL St. Louis Quality System and contains requirements and general guidelines under which STL St. Louis shall conduct operations.

4.3. Document Control

4.3.1. Document Type

The following documents, at a minimum, must be controlled STL St. Louis:

- STL St. Louis Laboratory Quality Manual
- STL St. Louis Standard Operating Procedures (SOP)
- STL Quality Management Plan
- STL Corporate Safety Manual

4.3.2. Document Control Procedure

Security and control of documents are necessary to ensure that confidential information is not distributed and that all current copies of a given document are from the latest applicable revision. Unambiguous identification of a controlled document is maintained by identification of the following items in the document

header: Document Name, Document Number, Revision Number, Effective Date, Number of Pages. Controlled documents are authorized by Management and/or the QA Department. Controlled documents are marked as such and records of their distribution are kept by the QA Department.

Controlled documents shall be available electronically or by hardcopy. The electronic versions of SOPs are located at \\Qlstmo05\SOPs. The hardcopy versions are located in the QA Office.

Electronic versions of SOPs may be printed out for laboratory use. Once printed, they will have a useful life of 30 days or until they are superceded by a new revision. This printout shall be recycled after use.

SOPs are numbered to identify the source and application of the document as follows:

CORP-XX-0000STL refers to SOPs that were formerly corporate SOPs, but have been altered for STL St. Louis.

STL-XX-0000 refers to SOPS that have been created by STL St. Louis.

Functions:

QA	Quality Assurance
IP	Inorganic Preparations
MT	Metals
WC	Wet Chemistry
OP	Organic Preparations
MS	Mass Spectrometry
GC	Gas Chromatography
LC	Liquid Chromatography
RC	Radiochemical Preparations
RD	Radiochemistry
HS	Environmental Health and Safety

4.3.3. Document Revision

Changes to documents occur when a procedural change warrants a revision of the document. When an approved revision of a controlled document is ready for distribution, obsolete copies of the document shall be replaced with the current

version of the document. The previous revision of the controlled document must be archived by the QA Department.

Notification of procedure changes will be e-mailed to all applicable STL St. Louis employees. Their reply to this e-mail will document their acceptance of the revision.

4.3.4. Official Documents

The STL Corporate Operations staff issues Corporate Manuals, Standard Operating Procedures, and Policies. These are collectively termed "Official Documents" and encompass the Policies and Procedures that all STL facilities are required to employ. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Official Documents is found in Corporate SOP S-Q-001.

4.4. Request, Tender, and Contract Review

4.4.1. Contract Review

For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is STL St. Louis' intent to provide both standard and customized environmental testing services to our clients. To ensure project success, technical staff shall perform a thorough review of technical and QC requirements contained in contracts. Contracts are reviewed for adequately defined requirements and STL St. Louis' capability to meet those requirements.

Contract review shall include a review of the client's requirements in terms of compound lists, test methodology requested, sensitivity, accuracy, and precision requirements. STL St. Louis ensures that the laboratory's test methods are suitable to achieve these requirements and must ensure that the laboratory holds the appropriate certifications and approvals to perform the work. The review also includes the laboratory's capabilities in terms of turnaround time, capacity, and resources to provide the services requested, as well the laboratory's ability to provide the documentation, whether hardcopy or electronic. If the laboratory cannot provide all services but intends to subcontract such services, whether to another STL facility or to an outside firm, this must be documented and discussed with the client prior to contract approval.

All contracts entered into by STL St. Louis shall be reviewed and approved by the appropriate personnel at the facility or facilities performing the work. Any contract requirement or amendment to a contract communicated to STL St. Louis verbally must be documented and confirmed with the client in writing. Any discrepancy between the client's requirements and STL St. Louis' capability to meet those requirements is resolved in writing before acceptance of the contract. Contract amendments, initiated by the client and/or STL St. Louis, are documented in writing for the benefit of both the client and STL.

All contracts, Quality Assurance Project Plans (QAPPs), Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the permanent project record as defined in Section 4.12.1.

4.4.2. Project Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, STL St. Louis assigns a Project Manager (PM) to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively communicated to the laboratory personnel before and during the project.

STL St. Louis shall have established procedures in order to ensure that communication is inclusive and effective. These include project memos, designation and meetings of project teams, and meetings between the laboratory staff and the client. STL St. Louis has found it very effective to invite the client into this process. STL St. Louis strongly encourages our clients to visit the laboratories and hold formal or informal sessions with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

4.4.3. Data Quality Objectives

Data Quality Objectives (DQO) are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application. Typically, DQOs are identified before project initiation, during the development of QAPPs and SAPs. The analytical DQOs addressed in this section are precision, accuracy, representativeness, completeness, and comparability.

The components of analytical variability (uncertainty) can be estimated when QC samples of the right types and at the appropriate frequency are incorporated into measurement process at the analytical laboratory. STL St. Louis incorporates numerous QC samples to obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The QC samples and their applications, described in Section 5.8.2, are selected based on regulatory, method- or client-specific requirements. Analytical laboratory QC samples for inorganic, organic, and radionuclide analyses may include calibration blanks, instrument blanks, method blanks, LCS, calibration standards, MS, MSD, surrogate spikes, and yield monitors.

The DQOs discussed below ensure that data are gathered and presented in accordance with procedures appropriate for its intended use, that the data is of known and documented quality, and are able to withstand scientific and legal scrutiny.

Precision is an estimate of variability. It is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions. Precision is expressed either as Relative Standard Deviation (RSD) for greater than two measurements or as Relative Percent Difference (RPD) for two measurements. Precision is determined, in part, by analyzing data from aggregate LCS results, MS, MSD, and MD. For radiochemical determinations, counting statistics can also provide an estimate of uncertainty.

Precision also refers to the measurement of the variability associated with the entire process, from sampling to analysis. Total precision of the process can be determined by analysis of duplicate or replicate field samples and measures variability introduced by both the laboratory and field operations.

Accuracy is the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value. It reflects the total error associated with a measurement.

Both random and systematic errors can affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100). Accuracy is determined, in part, by analyzing data from LCS, MS, and MSD. For radiochemical determinations, counting statistics can also provide an estimate of uncertainty.

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, a variation in a physical or chemical property at a sampling point, or an environmental condition. Data representativeness is primarily a function of sampling strategy; therefore, the sampling scheme must be designed to maximize representativeness. Representativeness also relates to ensuring that, through sample homogeneity, the sample analysis result is representative of the constituent concentration in the sample matrix. STL makes every effort to analyze an aliquot that is representative of the original sample, and to ensure the homogeneity of the sample before sub-sampling.

Completeness is defined as the percentage of measurements that are judged valid or useable. Factors negatively affecting completeness include the following: sample leakage or breakage in transit or during handling, loss of sample during laboratory analysis through accident or improper handling, improper documentation such that traceability is compromised, or sample result is rejected due to failure to conform to QC specifications. A completeness objective of greater than 90% of the data specified by the statement of work is the goal established for most projects.

Comparability is a measure of the confidence with which one data set can be compared to another. To ensure comparability, all laboratory analysts are required to use uniform procedures (e.g. SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data.

4.5. Subcontracting

Subcontracting must be arranged with the documented consent of the client, in a timely response which shall not be unreasonably refused. All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. The originating laboratory shall obtain proof of certification from the subcontract facility, and retain in STL records. Where applicable, specific QC guidelines, QAPPs, and/or SAPs are transmitted to the subcontract laboratory. Samples are subcontracted under formal Chain of Custody (COC).

Subcontract laboratories may receive an on-site audit by a representative of STL St. Louis' QA staff if it is deemed appropriate by the QA Manager. The audit involves a measure of compliance with the required test method, QC requirements, as well as any special client requirements. The originating laboratory may also perform a paper audit of

the subcontractor, which would entail reviewing the LQM, the last two PT studies, and a copy of any recent regulatory audits with the laboratory's responses.

Intra-company subcontracting may also occur between STL facilities. Intra-company subcontracting within STL must be arranged with the documented consent of the client, in a timely response which shall not be unreasonably refused. The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs.

Project reports from both STL St. Louis and external subcontractors are discussed in Section 5.9.4.

4.6. Purchasing Services and Supplies

Evaluation and selection of suppliers and vendors is done, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, all purchases from specific vendors are approved by a member of the supervisory or management staff.

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Purchasing guidelines for equipment and reagents meet with the requirements of the specific method and testing procedures for which they are being purchased.

4.7. Service to the Client

Sample representativeness and integrity are the foundation upon which meaningful analytical results rely. A documented and approved sampling plan reflecting data quality objectives should be in place at the sampling site. The integrity of the sample should be maintained though the use of preservation techniques specific to relevant protocols. Samples should be submitted to STL St. Louis under standard Chain of Custody (COC) procedures.

4.7.1. Sample Acceptance Policy

Samples shall be considered "compromised" if the following conditions are observed upon sample receipt:

- Cooler and/or samples are received outside of temperature specification.
- Samples are received broken or leaking.
- Samples are received beyond holding time.
- Samples are received without appropriate preservative.
- Samples are received in inappropriate containers.
- COC does not match samples received.
- COC is not properly completed or not received.
- Breakage of any Custody Seal.
- Apparent tampering with cooler and/or samples.
- Headspace in volatiles samples.
- Seepage of extraneous water or materials into samples.
- Inadequate sample volume.
- Illegible, impermanent, or non-unique sample labeling.

When "compromised" samples are received, it must be documented on a Condition Upon Receipt Form (CUR) for the project records and the client must be contacted for instructions. If the client decides to proceed with analysis, the project report shall clearly indicate any of the above conditions and the resolution. See STL-QA-0006, "Sample Receipt and Chain of Custody" for further details of this procedure.

4.7.2. Client Confidentiality and Proprietary Rights

Data and sample materials provided by the client or at the client's request, and the results obtained by STL St. Louis, shall be held in confidence (unless such information is generally available to the public or is in the public domain or client has failed to pay STL St. Louis for all services rendered or is otherwise in breach of the terms and conditions set forth in the STL St. Louis and client contract) subject to any disclosure required by law or legal process. STL St. Louis' reports, and the data and information provided therein, are for the exclusive use and benefit of client, and are not released to a third party without written consent from the client.

The audit reports supplied by federal, state, and local regulator agencies are considered public information and can be released without the written consent of those agencies. However, special project audits are confidential and must be approved by the client before release to a third party.

4.8. Complaints

Client complaints shall be documented, communicated to management, and addressed promptly and thoroughly. Client complaints are documented by the employee receiving the complaint. Client complaints are general received by the PM or CSM. Client complaints are documented through the Clouseau (NCM) System. The Laboratory Director, PM, Customer Service Manager, and QA Manager are informed of all client complaints, and assist in resolving the complaint.

The nature of the complaint is identified, documented, and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA department is required to conduct a special audit to assist in resolving the issue. A written confirmation, or letter to the client, outlining the issue and response taken is strongly recommended as part of the overall action taken. See QA Directive 00-0001, "The Tracking of Client Complaints" for further details on this policy.

The number and nature of client complaints shall be reported to the QA Director in the QA Monthly report submitted by each facility. The overall number of complaints received per facility is tracked and the appropriateness of the response to client complaints is assessed. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Management Systems Review.

4.9. Control of Non-conformances

Non-conformances include any out of control occurrence. Non-conformances may relate to client specific requirements, procedural requirements, or equipment issues. All non-conformances in the laboratory are documented at the time of their occurrence through the Clouseau (NCM) System.

All non-conformances that affect a sample and/or sample data become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, Manager, PM, Laboratory Director, or QA Manager for direction may be required. All records of reanalysis are kept with the project files.

Where non-conformances specifically affect a client's sample and/or data, the client shall be informed and action must be taken. Action can take the form of reporting and flagging the data, and including a description of the non-conformance in the project narrative or cover letter.

Where non-conformances are found after a report has been issued, such as, instrument calibration failure, data should be evaluated and if deemed compromised clients should be informed.

4.10. Corrective Action

4.10.1. General

A non-conformance is typically defined as an unplanned deviation from an established protocol. As occurrence of a non-conformance may be the result from STL St. Louis' action, which would be rendered as a deficiency or the result of events beyond STL St. Louis' control, which would be termed an anomaly. All non-conformances are documented through the Clouseau (NCM) system.

Deviations form established protocol require corrective action. Each corrective action is thoroughly investigated, and the investigation, outcome of the investigation, action taken, and follow-up is documented. Corrective action reports are reviewed, approved, and maintained by the QA department.

4.10.2. Initiation

Any employee in STL St. Louis shall be authorized to initiate a corrective action. The initial source of corrective action can also be external to STL St. Louis (i.e. corrective action because of client complaint, regulatory audit, or proficiency test). When a problem that requires corrective action is identified, the following items are identified by the initiator on the corrective action report: the nature of the problem, the name of the initiator, and the date. If the problem affects a specific client project, the name of the client and laboratory project number is recorded, and the PM is informed immediately.

4.10.3. Cause Analysis

The corrective action process must be embarked upon as a joint, problem solving, constructive effort. Identification of systematic errors, or errors that are likely to occur repetitively due to a defect or weakness in a system, is particularly valuable

in maintaining an environment of continuous improvement in laboratory operations.

When an NCM is initiated, the initiator works with the affected employee(s) and/or department(s) to identify the root cause of the problem. An essential part of the corrective action process is to identify whether the problem occurred due to a systematic or isolated error.

If the initiator of the corrective action report is uncertain as to what would constitute appropriate corrective action or is unable to resolve the situation, the problem is identified to the Supervisor, Manager, Laboratory Director or the QA Manager who provides assistance in the corrective action process.

The root cause of the problem and associated cause analysis is documented on the NCM.

4.10.4. Corrective Action

Once the root cause of a problem is identified, the initiator and affected employee(s) and/or department(s) examine potential actions that will rectify the present problem to the extent possible, and prevent recurrence of future, similar occurrences. An appropriate corrective action is then recommended. The corrective action must be appropriate for the size and nature of the issue.

If the corrective action concerns a specific project related issue, the PM or Customer Service Manager approves the corrective action before its implementation.

Implementation of the corrective action and the date of implementation are documented on the NCM.

The NCM is automatically routed to the appropriate Group Leader, Project Manager and QA Department. This ensures communication and awareness of the problem, the cause, and the action taken to prevent future occurrences and/or rectify the immediate problem.

4.10.5. Monitoring Corrective Action

All NCMs are maintained and automatically forwarded to the QA Department. The QA department reviews all corrective actions and selects one or more of the

more significant corrective actions for inclusion in the annual systems audit. The QA Department also may implement a special audit. The purpose of inclusion of the corrective action process in both routine and special audits is to monitor the implementation of the corrective action and to determine whether the action taken has been effective in overcoming the issue identified.

4.11. Preventative Action

Preventative action is defined as noting and correcting a problem before it happens, because of a weakness in a system, method, or procedure. Preventative action includes analysis of the Quality System to detect, analyze, and eliminate potential causes of non-conformances. When potential problems are identified, preventative action is initiated to effectively address the problem to eliminate or reduce the risk identified. The preventative action process takes the same format as the corrective action process.

In order to prevent system down time, minimize corrective maintenance costs and ensure data validity, the laboratory employs a system of preventative maintenance. General preventative maintenance procedures, many of which are unique to particular instruments are outlined in each instruments' operation manual. All routine maintenance is performed as recommended by the manufacturer. The manuals also assist in the identification of commonly needed replacement parts, so that an inventory of these parts can be maintained at the laboratory. It is the Group Leader's responsibility to make sure that the most current version of the operator's manual is available in the laboratory. Routine maintenance is performed by the analyst, while an external technician may be called for major repairs. Certain instruments are on service contracts for major repairs. See STl-QA-0024, "Preventative Maintenance" for additional information about this procedure.

A bound maintenance logbook is kept with each instrument to record all routine and non-routine maintenance. Notation of the data and maintenance activity is recorded every time service procedures are preformed. This includes routine service checks by laboratory personnel, as well as, factory service calls. The return to analytical control following instrument repair is also noted in laboratory maintenance logbooks.

4.12. Records

4.12.1. Record Types

Record types are described in Table 3.

Table 3. STL Record Types

Raw Data	Controlled Documents	QC Records	Project Records	Administrative Records
Calibration, Computer Tapes and Discs, QC Samples, Sample Data	LQM	Audits/ Responses	COC Documentation	Accounting
	QMP	Certifications, MDL/RL/QC Limits	Contracts and Amendments	EH&S Manual, Permits, Disposal Records
	SOPs	NCMs	Correspondence	Employee Handbook
		Logbooks*	QAPP	Personnel files, Employee Signature & Initials, Training Records
		Method & Software Validation, Verification data	SAP	
		Standards Log and Certificates		Technical and Administrative Policies

*Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature.

4.12.2. Record Retention

Table 4 outlines STL St. Louis' standard record retention time. For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QC, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 5 have lengthier retention requirements and are subject to the requirements in Section 4.12.3.

Table 4. STL Record Retention

Record Type		Archival Requirement
Raw Data	All*	5 Years from project completion
Controlled Documents	All*	5 Years from document retirement date
QC	All*	5 Years from archival
Project	All*	5 Years from project completion
Administrative	Personnel/Training	7 years
Accounting	All*	See Accounting and Control Procedures Manual

* Exceptions listed in Table 5.

Table 5. Special Record Retention Requirements

Program	Retention Requirement
Colorado – Drinking Water	10 years
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Minnesota – Drinking Water	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
NY Potable Water NYCRR Part 55-2	10 years
OSHA - 29 CFR Part 1910	30 years
Pennsylvania – Drinking Water	10 years
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

4.12.3. Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the STL St. Louis standard record retention time. These are detailed in Table 5 with their retention requirements. In these cases, the longer retention requirement must be implemented and noted in the archive. If special instructions exist such that

client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

4.12.4. Archives and Record Transfer

Archives must be indexed such that records are accessible on either a project or temporal basis. Archives are protected against fire, theft, loss, deterioration, and vermin. Electronic records are protected from deterioration caused by magnetic fields and/or electronic deterioration. Removal of archived records is controlled and documented. On-site and/or off-site facilities may be used. See STL-QA-0022, "Off-Site Documentation" for further details on this procedure.

STL St. Louis ensures that all records are maintained as required by the regulatory guidelines and per the QMP and LQM upon facility location change or ownership transfer. Upon STL St. Louis facility location change, all archives are retained by STL St. Louis in accordance with the QMP and LQM. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established.

4.13. Internal Audits

4.13.1. Audit Types and Frequency

A number of types of audits shall be performed at STL St. Louis. Audit type and frequency are categorized in Table 6.

Table 6. Audit Types and Frequency

Audit Type	Performed by	Frequency
Systems/Spot	QA Department or Designee	Annual
Data	QA Department	5% of all projects or as agreed upon with Corporate QA. The USACE requires 10%.
Special	QA Department or Designee	As Needed

4.13.2. Systems Audits (Spot Assessments)

Facility systems audits are technical in nature and are conducted on an ongoing basis by the QA Manager or his/her designee. Systems audits cover all departments of the facility, both operational and support. Systems review can be in the form of SOP compliance audits or general audits of functional areas. See STL-QA-0021, "Internal Surveillances" for additional information on this procedure.

The audit report is issued by the QA Manager of the facility within 21 calendar days of the audit. The audit report is addressed to the Group Leader and copied to the Laboratory Director.

Written audit responses are required within 21 calendar days of audit report issue. The audit response follows the format of the audit report, and corrective actions and time frames for their implementation are included for each deficiency. The audit response is directed to all individuals copied on the audit report. Where a corrective action requires longer than 21 days to complete, the target date for the corrective action implementation is stated and evidence of the corrective action is submitted to the QA Department in the agreed upon time frame.

4.13.3. Data Audits

Data audits are focussed to assess the level of customer service, method compliance, regulatory compliance, accuracy and completeness of test results and reports, documentation, and adherence to established QC criteria, laboratory SOPs, technical policy, and project specific QC criteria.

A data auditing frequency target of 5% has been established. The QA Department provides feedback and/or corrections and revisions to project reports where necessary. Data audits must include spot-checking of manual integrations by QA personnel in order to determine that the manual integration is appropriate and documented according to Section 5.3.6.

Records of the data audits shall be kept, and the frequency of data audits shall be included in the monthly QA report. In performing data audits, it is essential that data be assessed in terms of differentiating between systematic and isolated errors. Upon noting anomalous data or occurrences in the data audits, the QA Department is responsible for seeking clarification from the appropriate

personnel, ascertaining whether the error is systematic or an isolated error, and overseeing correction and/or revision of the project report if necessary. Errors found in client project reports are revised and the revision sent to the client. The QA Department is also responsible for assisting in the corrective action process where a data audit leads to identification of the need for permanent corrective action.

Where specific clients and regulatory programs require more frequent data auditing, the individual facility must meet the data auditing frequency for that program.

4.13.4. Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, proficiency testing results, data audits, systems audits, validation comments, or regulatory audits. Special audits are focussed on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

4.14. External Audits

STL St. Louis is routinely audited by clients and external regulatory authorities. STL St. Louis is available for these audits and makes every effort to provide the auditors with the personnel, documentation, and assistance required by the auditors. STL St. Louis recommends that the audits be scheduled with the QA Department so that all necessary personnel are available on the day of the audit.

4.15. Management Reviews

4.15.1. QA Reports to Management

A monthly QA report shall be prepared by the QA Manager and forwarded to the Laboratory Director and the QA Director. The reports include statistical results that are used to assess the effectiveness of the Quality System. The format of the monthly report is shown in Figure 2.

A Corporate QA Monthly Report containing a compilation of the STL St. Louis QA reports statistics, information on progress of the Corporate QA program, and a narrative outlining significant occurrences and/or concerns shall be prepared by

the QA Director and forwarded to the General Manager of Operational and Technical Services and the COO.

4.15.2. Management Systems Review

STL St. Louis shall perform a management systems review at least annually. The management systems review ensures that the laboratory's quality system is adequate to satisfy the laboratory's policies and practices, regulatory requirements, certification, accreditation, approval requirements, and client expectations. Management systems reviews are accomplished through monthly quality assurance reporting, goal setting and an annual LQM review.

Figure 2. Monthly QA Report Format

1.	Audits Internal systems audits performed. External systems audits performed. Data audits performed (in percent).
2.	Revised Reports/Client Complaints Revised reports in percent. Total number of client complaints, reason, and resolution.
3.	Certifications/Parameters Changes
4.	Proficiency Testing Score for each PT as a percent. Note repeat failures and/or significant problems.
5.	Miscellaneous QA and Operational Issues Narrative outlining improvements, regulatory compliance issues, general concerns, and assistance required from Corporate QA.

5. Technical Requirements

5.1. Personnel

5.1.1. General

STL St. Louis management believes that its highly qualified and professional staff is the single most important aspect in assuring the highest level of data quality and service in the industry.

STL St. Louis staff consists of about 65 professionals and support personnel that include the following positions:

- Laboratory Director
- Technical Director
- Customer Service Manager
- Quality Assurance Manager
- Quality Assurance Chemist
- Group Leader
- Group Supervisor
- Information Technology Manager
- Information Technology Specialist
- Office Manager
- Project Manager
- Chemist/Analyst
- Sample Control

5.1.2. Training

STL St. Louis is committed to furthering the professional and technical development of employees at all levels. Minimum training requirements for STL St. Louis employees are outlined in Table 7.

Table 7. STL Employee Minimum Training Requirements

Required Training	Time Frame*	Employee Type
Environmental Health & Safety	Month 1/Annually	All
Quality Assurance	Quarter 1/Annually	All
Radiation Worker Training	Quarter 1/Annually	All
Demonstration of Capability (DOC)	Prior to unsupervised performance/Annually	Technical

*From date of initial employment unless otherwise indicated.

Technical training is accomplished within each laboratory by the Group Leader or their designee to ensure method comprehension. All new personnel shall be required to demonstrate competency in performing a particular method by successfully completing a Demonstration of Capability (DOC) before conducting analysis independently on client samples.

DOCs are performed by analysis of four replicate QC samples. Results of successive LCS analyses can be used to fulfill the DOC requirement. The accuracy and precision, measured as average recovery and standard deviation (using n-1 as the population), of the 4 replicate results are calculated and compared to those in the test method (where available). If the test method does not include accuracy and precision requirements, the results are compared to target criteria set by the laboratory. The laboratory sets the target criteria such that they reflect the DQOs of the specific test method or project. A DOC Certification Statement is recorded and maintained in the employee's training or personnel file. Figure 3 shows an example of a DOC Certification Statement.

The following evidence must be on file at the laboratory for each technical employee:

- DOC.
- The employee has read and understood the latest version of the laboratory's quality documentation.
- The employee has read and understood the latest, approved version of all test methods and/or SOPs for which the employee is responsible.
- Annual evidence of continued DOC that may include successful analysis of a blind sample on the specific test method, or a similar test method, or an annual DOC, or four successive, successful LCS.

Figure 3. Example Demonstration of Capability Certification Statement

Demonstration of Capability
Certification Statement

Date: Matrix:
Laboratory Name: Method:
Laboratory Address:
Analyst Name:

We the undersigned certify that:

1. The analyst identified above, using the cited test method, which is in use at this facility for the analysis of samples under the National Environmental Laboratory Accreditation Program, has met the Demonstration of Capability.
2. The test method was performed by the analyst identified on this certification.
3. Copies of the test method and SOP are available for all personnel on site.
4. The data associated with the DOC are true, complete and representative.
5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is available for review by authorized inspectors.

Laboratory Manager/Supervisor _____ Signature _____ Date _____

5.1.3. Ethics Policy

Establishing and maintaining a high ethical standard is an important element of a Quality System. In order to ensure that all personnel understand the importance the company places on maintaining high ethical standards at all times; STL St. Louis has established an Ethics Agreement (Figure 4). Each employee shall sign the Ethics Agreement, signifying agreed compliance with its stated purpose.

Ethics is also a major component of the STL St. Louis QA training program. Each employee must be trained in ethics within three months of hire in a standardized QA training program that includes an overview of regulatory programs and program goals, a review of the ethics statement, and group discussions about data integrity and data misrepresentation. Employees must be trained as to the legal and environmental repercussions that result from data misrepresentation. A data integrity hotline is maintained by STL and administered by the QA Director.

Figure 4. STL Ethics Agreement

It is the policy of STL to incorporate the highest standard of quality with all analytical programs by adhering to the following practices:

STL will only offer environmental analyses for which it can consistently demonstrate compliance with high quality, traceable and legally defensible performance standards. All STL staff is committed to the practice of complete honesty in the production and reporting of data.
Staff who are aware of misrepresentation of facts or data manipulation to bypass established QA/QC requirements, are required to immediately inform their supervisor or any member of the upper management.

All employees are asked to sign a copy of the statement below upon their first day of employment.

I, _____(print name) understand that high standards of integrity are required of me with regard to the duties I perform and the data I report in connection with my employment at the Company. I agree that in the performance of my duties at the Company:
I will not intentionally report data values that are not the actual values obtained;
I will not intentionally report the dates, times, sample or QC identifications, or method citations of data analyses that are not the actual dates, times, sample or QC identifications, or method citations;
I will not intentionally misrepresent another individual's work; and
If a supervisor or a member of STL management requests me to engage in or perform an activity that I feel is compromising data validity or quality, I will not comply with the request and report this action immediately to a member of the upper management, up to and including the president of Severn Trent Laboratories Inc.
I will not intentionally report data values that do not meet established quality control criteria as set forth in the Method and/or Standard Operation Procedures, or as defined by Company Policy.

I agree to inform my Supervisor of any accidental reporting of non-authentic data by me in a timely manner. I agree to inform my Supervisor of any accidental or intentional reporting of non-authentic data by other employees. I have read this Ethics Agreement and understand that failure to comply with the conditions stated above will result in disciplinary action, up to and including termination from the Company.

Compliance with this policy of business ethics and conduct is the responsibility of every STL employee. Disregard or failing to comply with this standard of business ethics and conduct could lead to disciplinary action, up to and including possible termination of employment.

5.2. Facilities

5.2.1 General

The 31,000-square-foot facility is designed to provide inorganic, organic, and radiochemical analyses of various environmental samples that include wastewater, soil, sediment, sludge, oil, and hazardous waste. The design of the laboratory ensures data quality, safety, efficiency, automation, and security. Instrument laboratories are separate from the sample preparation laboratories to eliminate the potential for cross contamination. Ventilation of the instrument laboratory for volatile organic constituents (VOC) analyses is setup to prevent solvent contamination. The reagent water system provides water of the required quality for standard and QC sample preparations and laboratory operations.

STL St. Louis is designed for efficient, automated high-quality operations. All laboratories are equipped with Heating, Ventilation, and Air Conditioning (HVAC) systems appropriate to the needs of environmental testing laboratories. Environmental conditions in the facilities, such as hood flow, are routinely monitored and documented.

STL St. Louis is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. STL St. Louis also provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc.

STL St. Louis must be secure and access must be controlled and documented. Access is controlled by various measures including locked doors, passwords, electronic access cards, security codes, and staffed reception areas. All visitors sign in and are escorted by STL St. Louis personnel while at an STL St. Louis facility.

5.2.2 Contingency Planning

An effective QA Program must emphasize contingency planning, actions to prevent problems from recurring, and to ensure timely and effective completion of a measurement effort. The following are considered relative to contingency planning:

Staffing – A primary objective is to ensure that qualified staff are always available to perform the necessary analytical work, regardless of employee turnover, vacation, illness, or other absence.

Redundant Instrument or Equivalent Methods – In certain areas of the laboratory, redundant instrumentation is available to ensure uninterrupted workflow. In circumstances where a catastrophic instrument failure occurs, alternative but equivalent methods may be recommended to the client approval and implementation.

Preventative Maintenance – Preventative maintenance program is designed to minimize analytical instrument malfunctions, permit simple adjustments, and to ensure fewer and shorter breakdowns of critical analytical equipment.

Network Laboratories & Subcontractor Laboratories – To support the laboratory during peak periods or in the event of a critical malfunction, STL has the capability to arrange the use of other network laboratories or qualified analytical laboratories as subcontractors for short-term backup analytical support. Any use of a subcontractor laboratory is approved by the client prior to award of a contract or sample shipment for existing contracts.

Uninterruptable Power Supply - Unexpected power surges and power outages can lead to loss of data or even catastrophic failure of computer systems. To prevent such an occurrence, data on all local area network servers and certain mission-critical desktop computers are protected by an uninterruptable power supply (UPS) system. A UPS system contains batteries that maintain a constant charge to be used as a backup power supply in the event of power failure. When a power failure occurs, power to critical computer systems is maintained until normal electrical current is restored. If it appears that the duration of the power outage may exceed the expected life of the UPS system's energy stores, all affected computer systems are shut down gracefully before the backup power supply is depleted. To ensure the integrity of the UPS system, scheduled maintenance is performed by a third party vendor.

5.3. Test Methods

5.3.1. Method Selection

Most of the test methods performed at STL St. Louis originate from test methods published by a regulatory agency such as the US EPA and other state and federal regulatory agencies. These include, but are not limited to, the following published compendiums of test methods:

Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water.

Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.

Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.

Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991.

Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992.

NIOSH Manual of Analytical Methods, 4th ed., August 1994.

Statement of Work for Inorganics Analysis, ILM04.0, USEPA Contract Laboratory Program Multi-media, Multi-concentration.

Statement of Work for Organics Analysis, OLM03.2, USEPA Contract Laboratory Program, Multi-media, Multi-concentration.

Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration, OLMO4.1, USEPA Contract Laboratory Program, September 1998.

Standard Methods for the Examination of Water and Wastewater, 18th/19th edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.

Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.

Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.

Prescribed Procedures for Measurement of Radioactivity in Drinking Water, No. EPA-600/4-80-032, USEPA, Cincinnati, Ohio, 1980.

U.S. Department of Energy, consolidated by Los Alamos National Laboratory, Los Alamos, New Mexico, 1993.

Eastern Environmental Radiation Facility Radiochemistry Procedures Manual, No. 520/5-84-006, USEPA, 1984.

National Academy of Sciences, Nuclear Science Series.

The Determination of Technetium-99 By Liquid Scintillation Counting, C.R. Walker, H.S. Short, H.S. Spring, Technical Division, Goodyear Atomic Corp., Piketon, Ohio.

Environmental Measurements Laboratory Procedures Manual, No. HASL-300, HASL.

Appendix A lists the analytical methods performed at STL St. Louis. Also indicated are the methods accredited by NELAP.

5.3.2. SOPs

STL St. Louis shall maintain an SOP Index for both Method and Process SOPs. Method SOPs are maintained to describe a specific test method. Process SOPs are maintained to describe function and processes not related to a specific test method.

Appendix C is a list of current, active SOPs in use at STL St. Louis.

Method SOPs contain the following information:

Title Page with Document Name, Document Number, Revision Number, Effective Date, Page Numbers and Total # of Pages, Authorized Signatures, Dates and Proprietary Information Statement (Figure 5).

1. Identification of Test Method
2. Applicable Matrix
3. Reporting Limit
4. Scope and Application, including test analytes
5. Summary of the Test Method
6. Definitions
7. Interferences

8. Safety
9. Equipment and Supplies
10. Reagents and Standards
11. Sample Collection, Preservation, Shipment and Storage
12. Quality Control
13. Calibration and Standardization
14. Procedure
15. Calculations
16. Method Performance
17. Pollution Prevention
18. Data Assessment and Acceptance Criteria for Quality Control Measures
19. Corrective Actions for Out-of-Control Data
20. Contingencies for Handling Out-of-Control or Unacceptable Data
21. Waste Management
22. References
23. Tables, Diagrams, Flowcharts and Validation Data

Process SOPs may contain the following information:

Title Page with Document Name, Document Number, Revision Number, Effective Date, Page Numbers and Total # of Pages, Authorized Signatures, Dates and Proprietary Information Statement (Figure 5).

1. Scope
2. Summary
3. Definitions
4. Responsibilities
5. Safety
6. Procedure
7. References
8. Tables, Diagrams, and Flowcharts

The QA Department is responsible for maintenance of SOPs, archival of SOP historical revisions, maintenance of an SOP index, and records of controlled distribution. SOPs, at a minimum, must undergo biennial review. Where an SOP is based on a published method, the laboratory must maintain a copy of the reference method.

Figure 5. Proprietary Information Statement

This documentation has been prepared by Severn Trent Laboratories (STL) solely for STL's own use and the use of STL's customers in evaluating its qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to STL upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions. **THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF SEVERN TRENT LABORATORIES IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY STL IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY: ©COPYRIGHT 2001 SEVERN TRENT LABORATORIES, INC. ALL RIGHTS RESERVED.**

Deviations from the Method

In some cases, a standard laboratory procedure is modified slightly for a specific client or project at the client or regulatory agency's request. In these cases, a subsection in the References is included which indicates the modifications to the SOP which are specific to that project.

SOPs are written procedures for standardized methods (i.e. SW-846, EPA-200, 500, and 600 series methods) to document specific laboratory procedures which satisfy the general requirements specified in the individual methods and to explain any differences between the application of the established method and the published procedure. If any differences exists between STL St. Louis' SOP and a standard method's specific procedures, method validation studies are performed to document the fact that the change does not adversely affect the applicability of the method. In general, every effort is made to adhere to the protocols of the standard method.

5.3.3. Method Validation

Laboratory developed methods are validated and documented according to the procedure described in Section 5.3.5.

5.3.4. Method Verification

Method verification is required when a validated standard test method or a method modification is implemented. The level of activity required for method verification is dependent on the type of method being implemented, or on the level of method modification and its affect on a method's robustness. Method modification often takes advantage of a method's robustness, or the ability to make minor changes in a method without affecting the method's outcome. Method verification may require some, but not all, of the activities described in Section 5.3.5.

5.3.5. Method Validation and Verification Activities

Before analyzing samples by a particular method, method validation and/or method verification must occur. A complete validation of the method is required for laboratory developed methods. While method validation can take various courses, the following activities can be required as part of method validation.

Method validation records are designated QC records and are archived accordingly.

Determination of Method Selectivity
Method selectivity is demonstrated for the analyte(s) in the specific matrix or matrices. In some cases, to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

Determination of Method Sensitivity
Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

IDLs are performed where required by specific data quality objectives or requirements.

Method Detection Limit Study – (USEPA, Glaser etal, 1981)
This approach establishes a procedure for estimation of the MDL at a single concentration using a minimum of seven successive determinations of samples or spikes containing the analyte to be determined.

Where a single analyte has been used to represent a group of target analytes in demonstrating sensitivity, it is recommended that the analyte be varied amongst that group in demonstrating ongoing sensitivity.

See STL-QA-0016, "IDL/MDL Determination" for further information on this procedure.

Determination of Interferences
A determination that the method is free from interferences in a blank matrix is performed.

Determination of Range
Where appropriate, a determination of the applicable range of the method may be performed. In most cases, range is determined and demonstrated by comparison of the response of an analyte in a curve to established or targeted criteria. The curve is used to establish the range of quantitation and the lower and upper values

of the curve represent upper and lower quantitation limits. Curves are not limited to linear relationships.

Demonstration of Capability
DOCs are performed prior to method performance.

Determination of Accuracy and Precision
Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

Documentation of Method
The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Appendix describing the specific differences in the new method is acceptable in place of a separate SOP.

Continued Demonstration of Method Performance
Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS and Method Blanks.

Determination of Reporting Limits
The MDL is the approximate limit at which an analyte can be qualitatively detected using a specific method at a 99% confidence interval. The MDL is a statistically calculated value and measures the sensitivity of an entire method and is independent of device. The Reporting Limit (RL) or Limit of Quantitation is the limit at which a compound can be qualitatively detected and quantified at a 99% confidence interval. The RLs are also set based on specific knowledge about the analyte, project specific requirements and/or regulatory requirements. The RL is always greater than the MDL and is typically set based on 2-10 times the MDL.

STL St. Louis reports results to the sample-specific RLs. For most methods, the low calibration standard is set at the laboratory RL to monitor method sensitivity per instrument per calibration. Sample specific RLs are derived by taking into account various sample specific data, which can include the amount of the sample subject to testing, percent moisture, dilution factors, interferences and the base RLs for the analysis.

In some cases, it is appropriate to report values between the MDL and the RL. In this region, an analyte can be qualitatively detected, but not accurately quantified. Any data point reported in this is flagged with "J" for organics and a "B" for inorganics to indicate that it is an estimated value.

5.3.6. Data review

All data, regardless of regulatory program or level of reporting, shall be subject to a thorough review which involves a primary, secondary, and completeness review process. All levels of the review must be documented.

Primary Review

The primary review is often referred to as a "bench-level" review. In most cases, the analyst who generates the data (i.e. logs in, prepares and/or runs the samples) is the primary reviewer. In some cases, an analyst may be reducing data for samples run by an auto-sampler set up by a different analyst. In this case, the identity of both the analyst and the primary reviewer is identified in the raw data.

One of the most important aspects of primary review is to make sure that the test instructions are clear, and that all project specific requirements have been understood and followed.

Once an analysis is complete, the primary reviewer must ensure that:

- Sample preparation information is complete, accurate, and documented.
- Calculations have been performed correctly.
- Quantitation has been performed accurately.
- Qualitative identifications are accurate.
- Manual integrations are appropriate.
- Data flags to indicate manual integrations are recorded.
- Manual integrations are authorized by a date and signature or initials of primary analyst.
- Client specific requirements have been followed.
- Method and process SOPs have been followed.
- Method QC criteria have been met.
- QC samples are within established limits.
- Dilution factors are correctly recorded and applied.
- Non-conformances and/or anomalous data have been properly documented and appropriately communicated.

- COC procedures have been followed.
- Primary review is documented by date and initials/signature of primary analyst.

Any anomalous results and/or non-conformances noted during the Primary Review are communicated to the Supervisor and the PM for resolution. Resolution can require sample reanalysis, or it may require that data be reported with a qualification. Non-conformances are documented using the Clouseau (NCM) System. Revisions are never erased, deleted or written over. They are corrected by drawing a single line through the error and entering the correction alongside. The correction is then initialed and dated by the person who edited the data.

Secondary Review

The secondary review shall be a complete technical review of a data set. The secondary review must be documented and the secondary reviewer identified. The following items are reviewed:

- Qualitative Identification
- Quantitative Accuracy
- Calibration
- QC Samples
- Method QC Criteria
- Adherence to method and process SOPs
- Accuracy of Final Client Reporting Forms
- Manual Integrations – Minimal requirement is to spot-check raw data files for manual integration, as verified by date and initials or signature of secondary data reviewer. Some regulatory programs require 100% secondary review of manual integrations.
- Completeness
- Special Requirements/Instructions

If problems are found during the secondary review, the reviewer must work with the appropriate personnel to resolve them. If changes are made to the data, such as alternate qualitative identifications, identifications of additional target analytes, re-quantitation, or re-integration, the secondary reviewer must contact the laboratory analyst and/or primary reviewer of the data so that the primary analyst and/or reviewer is aware of the appropriate reporting procedures.

Completeness Review

The completeness review shall include the generation of a project narrative and/or cover letter which outlines anomalous data and non-compliances using project narrative notes and non-compliance reports generated during the primary and secondary review. The completeness review addresses the following items:

- Is the project report complete?
- Does the data meet with the client's expectations?
- Were the data quality objectives of the project met?
- Are QC outages and/or non-conformances approved and appropriately explained in the narrative notes?

Logbook Review

All logbooks and records of routine monitoring are reviewed monthly to ensure accuracy and compliance with its SOP and this policy. This review is performed by a Group Leader or a designee. At a minimum, The review is documented by a signature or initials and the date reviewed. See STL-QA-0020, "Laboratory Logbook Control and Maintenance" and QA Directive 00-0008, "Logbook Reviews" for additional information on this procedure and policy.

5.3.7. Data Integrity and Security

This section details those procedures that are relevant to computer systems that collect, analyze, and process raw instrumental data, and those that manage and report data.

Security and Traceability

Access to computer systems that collect, analyze, and process raw instrumental data, and those that manage and report data must be both controlled and recorded. There are various systems at STL St. Louis to which this applies, which include the Laboratory Information Management System (LIMS), as well as specific systems such as Target and Central.

Control of the system is accomplished through limitation of access to the system by users with the education, training and experience to perform the task knowledgeably and accurately. System users are granted privileges that are commensurate with their experience and responsibilities.

Computer access is tracked by using unique login names and passwords for all employees that have access to the computer system. "General" or "multi-user" account access to computer systems that collect, analyze and process raw instrumental data, and those that manage and report data shall not be permitted. Entries and changes are documented with the identity of the individual making the entry, and the time and date. Where a computer system is processing raw instrumental data, the instrument identification number as described in Section 5.4.1 is recorded. Many of these systems, such as Target, have the capability of maintaining audit trails to track entries and changes to the data. This function is activated on any computer system that has that capability.

Verification

All commercially obtained software shall be verified prior to use and after version upgrade. Verification involves assessing whether the computer system accurately performs its intended function. Verification generally is accomplished by comparing the output of the program with the output of the raw data manually processed, or processed by the software being replaced. The records of the verification are required to contain the following information: software vendor, name of product, version, comparison of program output and manual output, raw data used to verify the program, date, and name of the individual performing the verification. Records of verification are retained as QC records.

Validation

Software validation involves documentation of specifications and coding as well as verification of results. Software validation is performed on all in house programs. Records of validation include original specifications, identity of code, printout of code, software name, software version, name of individual writing the code, comparison of program output with specifications, and verification records as specified above. Records of validation are retained as QC records.

Auditing

The QA Department systems audit includes review of the control, security, and tracking of IT systems and software.

Version Control

The laboratory shall maintain copies of outdated versions of software and associated manuals for all software in use at the laboratory for a period of five years from its retirement date. The associated hardware, required to operate the software, must also be retained for the same time period.

5.4. Equipment

5.4.1. Equipment Operation

STL St. Louis is committed to routinely updating and automating instrumentation. STL St. Louis maintains state of the art instrumentation to perform the analyses within the QC specifications of the test methods. STL St. Louis maintains an equipment list that includes the following information:

- Identity
- Date Installed
- Manufacturer's Name, Model Number, Serial Number
- Current Location
- Preventative Maintenance Schedule

All equipment is subject to rigorous checks upon its receipt, upgrade, or modification to establish that the equipment meets with the selectivity, accuracy, and precision required by the test method for which it is to be used. All manufacturer's operations and maintenance manuals are kept up to date and accessible for the use of the equipment operator. Documentation of equipment usage is maintained using analytical run and maintenance logbooks.
A comprehensive list of major instrumentation available, along with supportive and miscellaneous equipment can be found on Table 8.

Table 8. Instrument List

Instrument Type	Instrument Description	Manufacturer	Model	Date Purchased	Autosampler
GC/MS	Gas Chromatograph/Mass Spectrometer RTE Data System and Library (Wiley/NBS) 9-track magnetic tape Autosampler	Hewlett Packard	5970	1989	Yes
	GC System	Hewlett Packard	5890	1989	
	Purge and Trap	Tekmar	LSC2000	1989	
	ALS/Heated Purge	Tekmar	LSC2016	1989	
GC/MS	Gas Chromatograph/Mass Spectrometer RTE Data System and Library (Wiley/NBS) 9-track magnetic tape Autosampler	Hewlett Packard	5970	1987	Yes
	GC System	Hewlett Packard	5890	1987	
	Sample Concentrator	O-I-Analytical	4460A	1987	
	Mult. Purging Module	O-I-Analytical	MHC-16	1987	

Instrument Type	Instrument Description	Manufacturer	Model	Date Purchased	Autosampler
GC/MS	Gas Chromatograph/Mass Spectrometer RTE Data System and Library (Wiley/NBS) 9-track magnetic tape Autosampler	Hewlett Packard	5973	1987	Yes
	GC System	Hewlett Packard	6890	1987	
	Purge and Trap Concentrator	Tekmar	LSC3000	1987	
	Purge and Trap	Tekmar	ALS2016	1987	
GC/MS	Gas Chromatograph/Mass Spectrometer RTE Data System and Library (Wiley/NBS) 9-track magnetic tape Autosampler	Hewlett Packard	5970	1987	Yes
	GC System	Hewlett Packard	5890	1987	
	Purge and Trap Concentrator	Tekmar	LSC3000	1987	
	Precept II	Tekmar	Precept II	1987	
GC/MS	Gas Chromatograph/Mass Spectrometer RTE Data System and Library (Wiley/NBS) 9-track magnetic tape Autosampler	Hewlett Packard	5973	1998	Yes
	GC System	Hewlett Packard	6890	1998	
	Series Injector	Hewlett Packard	7683	1998	
GC/MS	Gas Chromatograph/Mass Spectrometer RTE Data System and Library (Wiley/NBS) 9-track magnetic tape Autosampler	Hewlett Packard	5973	1998	Yes
	GC System	Hewlett Packard	6890	1998	
	Series Injector	Hewlett Packard	7683	1998	
GC	Gas Chromatograph Capillary/Packed Column Autoinjector, Autosampler	Hewlett Packard	5890 Series II	1987	Yes
	Series Interface	PE Nelson	900	1987	
	Precept II	Tekmar	Precept II	1997	
	Purge and Trap Concentrator	Tekmar	LSC3000	1997	
GC	Gas Chromatograph Capillary/Packed Column Autoinjector, Autosampler	Hewlett Packard	5890	1987	Yes
	Series Interface	PE Nelson	900	1987	
	Purge And Trap Concentrater	Tekmar	LSC3000	1987	
	Purge And Trap Autosampler	Tekmar	LSC2016	1996	
HPLC	High Pressure Liquid Chromatograph Series 1100	Hewlett Packard	ALS G1329A	1999	Yes
	ALS Therm	Hewlett Packard	ALS THERM G1330A	1999	
	COLCOM	Hewlett Packard	COLCOM G1316A	1999	

Instrument Type	Instrument Description	Manufacturer	Model	Date Purchased	Autosampler
	DAD	Hewlett Packard	DAD G1315A	1999	
	Degasser	Hewlett Packard	DEGASSER G1322A	1999	
	Quat Pump	Hewlett Packard	QUAT PUMP G1311A	1999	
	FLD	Hewlett Packard	FLD G1321A	1999	
GC	Gas Chromatograph Capillary/Packed Column Autoinjector, Autosampler	Hewlett Packard	5890	1987	Yes
	Integrator	Hewlett Packard	3392A	1987	
	Controller	Hewlett Packard	7673A	1987	
	Wheel	Hewlett Packard	7673A	1987	
	Series Integrator	PE Nelson	900	1987	
GC	Gas Chromatograph Capillary/Packed Column Autoinjector, Autosampler	Hewlett Packard	5890	1987	Yes
	AutoSampler	Hewlett Packard	7673A	1987	
	Controller	Hewlett Packard	7673A	1987	
	Wheel	Hewlett Packard	7673A	1987	
	Integrator	Hewlett Packard	3392A	1987	
	Series Interface	PE Nelson	900	1987	
GC	Gas Chromatograph Capillary/Packed Column Autoinjector, Autosampler	Hewlett Packard	5890	1987	Yes
	Autosampler	Hewlett Packard	7673A	1987	
	Autosampler	Hewlett Packard	7673A	1987	
	Wheel	Hewlett Packard	7673A	1987	
	Controller	Hewlett Packard	7673A	1987	
	Integrator	Hewlett Packard	3392A	1987	
	Series Interface	PE Nelson	900	1987	
	Series Interface	PE Nelson	900	1987	
GC	Gas Chromatograph Capillary/Packed Column Autoinjector, Autosampler	Hewlett Packard	5890	1987	Yes
	Controller	Hewlett Packard	7673A	1987	
	Autosampler	Hewlett Packard	7673A	1987	
	Series Interface	PE Nelson	900	1987	

Instrument Type	Instrument Description	Manufacturer	Model	Date Purchased	Autosampler
	Integrator	Hewlett Packard	3392A	1987	
GC	Gas Chromatograph Capillary/Packed Column Autoinjector, Autosampler	Hewlett Packard	5890	1987	Yes
	Controller	Hewlett Packard	7673A	1987	
	Autosampler	Hewlett Packard	7673A	1987	
	Wheel	Hewlett Packard	7673A	1987	
	Integrator	Hewlett Packard	3392A	1987	
GC	Gas Chromatograph Capillary/Packed Column Autoinjector, Autosampler	Hewlett Packard	5880	1987	Yes
	Autosampler	Hewlett Packard	7673A	1987	
	Controller	Hewlett Packard	7673A	1987	
	Wheel	Hewlett Packard	7673A	1987	
	Integrator	Hewlett Packard	3392A	1987	
	Series Interface	PE Nelson	900	1987	
HPLC	High-Pressure Liquid Chromatograph	Hewlett-Packard	1090	1988	Yes
	Spectrometer	Perkin Elmer	LC 90UV	1998	
	Series Interface	PE Nelson	900	1998	
	Integrator	Hewlett-Packard	9133	1983	
HPLC	High-Pressure Liquid Chromatograph	Hewlett-Packard	1090 SeriesII	1988	Yes
ICP-MS	ICPMS	Perkin Elmer	ELAN 6000	1999	Yes
	Autosampler	Perkin Elmer	AS-91	1999	
	Chiller-Recirc EPR	Perkin Elmer	PD2 60 HZ	1999	
ICP	Inductively-Coupled Argon Plasma Vacuum Spectrometer	Thermo Jarrell Ash	1100	1987	Yes
ICP	Inductively-Coupled Argon Plasma Vacuum Spectrometer	Thermo Jarrell Ash	61E	1994	Yes
GFAA	Atomic Absorption Spectrometer, Graphite Furnace	Perkin Elmer	5100Z-PC	1991	Yes
GFAA	Atomic Absorption Spectrometer, Graphite Furnace	Perkin Elmer	5100Z-PC	1991	Yes
GFAA / FLAA	Atomic Absorption Spectrometer, Graphite Furnace/Flame	Perkin Elmer	5100Z-PC	1991	Yes
CVAA	Automated Mercury Analyzer	Leeman Labs	PS200	1993	Yes
FLAA / CVAA	Atomic Absorption Spectrometer, Cold Vapor Assembly	Thermo Jarrell Ash	12E	1987	No

Instrument Type	Instrument Description	Manufacturer	Model	Date Purchased	Autosampler
TOC	Total Organic Carbon Analyzer	Dohrmann-Xertex	DC180	1987	No
TOX	Total Organic Halogen Analyzer	Dohrmann-Xertex	DX20A	1987	No
IC	Ion Chromatograph HPLC Module	Dionex	4000i	1987	Yes
UV Spec	UV Spectrophotometer	Milton Roy	601	1987	No
IR	IR Spectrophotometer, Ratio Recording	Perkin Elmer	1420	1988	No
TRAACS-1	Auto Analyzer	Technicon	Traacs 800	1988	Yes
TRAACS-2	Auto Analyzer	Technicon	Traacs 800	1988	Yes
TOC-5050A	Total Organic Carbon Analyzer	Shimadzu	TOC-5050A	1999	Yes
1200 TOX	Total Organic Halide Analyzer	EuroGlass	1200 TOX	1999	Yes
GPC	Gel Permeation Chromatograph with UV Detector (GPC)	ABC Laboratories	1002B	1993	Yes
Alpha Spectrometer	Alpha Spectrometer Counting System (48 detectors)	EG&G Ortec/Canberra	Multi-component	1987-1992	No
Gamma Spectrometer	Gamma Spectrometer, Intrinsic Germanium Detectors	Princeton	Multi-component	1988	No
Gamma Spectrometer	Gamma Spectrometer, Intrinsic Germanium Detectors	Tennelec/Ortec	Multi-component	1991-1992	No
Gamma Spectrometer	Gamma Spectrometer, Intrinsic Germanium Detectors	Tennelec/Ortec	Multi-component	1991-1992	No
Gamma Spectrometer	Gamma Spectrometer, Intrinsic Germanium Detectors	Tennelec/Ortec	Multi-component	1991-1992	No
Gamma Spectrometer	Gamma Spectrometer, Intrinsic Germanium Detectors	Tennelec/Ortec	Multi-component	1991-1992	No
Gamma Spectrometer	Gamma Spectrometer, Intrinsic Germanium Detectors	Tennelec/Ortec	Multi-component	1991-1992	No
Gamma Spectrometer	Gamma Spectrometer, Intrinsic Germanium Detectors	Tennelec/Ortec	Multi-component	1991-1992	No
Gamma Spectrometer	Gamma Spectrometer, Intrinsic Germanium Detectors	Canberra	Multi-component	1991	No
GPC	Gas Proportional Counter, Low Background, 16 Detectors	Tennelec	LB4000	1988	No
GPC	Gas Proportional Counter, Low Background, 12 Detectors	Tennelec	LB4100 (Blue)	1994	No
GPC	Gas Proportional Counter, Low Background, 16 Detectors	Tennelec	LB4100 (Red)	1993	No
LSD	Liquid Scintillation Detector TriCarb 2000CA	Packard	2000CA	1985	Yes
KPA	Kinetic Phosphorescence Analyzer (KPA)	Chemchek	Multi-component	1991	No
KPA	Kinetic Phosphorescence Analyzer (KPA)	Chemchek	Multi-component	1989	No

Information Systems

- All terminals, PCs, and printers are connected on an integrated MS Windows NT Local Area Network (LAN) via network switches, routers and wide area links.
- Extensive variety of software to aid in data analysis and presentation: AS/400 database system, Microsoft Office 97 Products (access, Excel, Word, etc.) and Visual Basic programming environment.
- The GC division utilized TurboChrom data systems. The Target System is used by the GC/MS labs. All other systems have been custom designed internally. The data are autoloaded to a central LIMS where it undergoes a QC review.
- Personnel have access to the World Wide Web for additional resources.

5.4.2. Equipment Maintenance

STL St. Louis employs a system of preventative maintenance in order to ensure system up time, minimize corrective maintenance costs and ensure data validity. All routine maintenance is performed as recommended by the manufacturer and may be performed by an analyst, instrument specialist or outside technician. Maintenance logbooks are kept on all major pieces of equipment in which both routine and non-routine maintenance is recorded. Notation of the date and maintenance activity is recorded each time service procedures are performed. The return to analytical control following instrument repair is documented in the maintenance logbook. Maintenance logbooks are retained as QC records. The Group Leaders are responsible for the review of the maintenance records.

Maintenance contracts are held on specific pieces of equipment where outside service is efficient, cost-effective, and necessary for effective operation of the laboratory. See STL-QA-0024, "Preventative Maintenance" for further details on this procedure.

5.4.3. Equipment Verification and Calibration

All equipment shall be tested upon receipt to establish its ability to meet the QC guidelines contained in the test method for which the instrumentation is to be used. This testing shall be documented. Once an instrument is placed in routine service, ongoing instrument calibration is demonstrated at the appropriate frequency as defined in the test method. Any instrument that is deemed to be

malfunctioning is clearly marked and taken out of service. When the instrument is brought back into control, this is documented in the instrument maintenance log.

5.5. Measurement Traceability

5.5.1. General

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy.

At a minimum, these must include procedures for checking specifications for balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. With the exception of Class A Glassware (including glass microliter syringes that have a certificate of accuracy), quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balances are calibrated on each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

Laboratory DI and RO water systems have documented preventative maintenance schedules and the conductivity of the water is recorded on each day of use. Refer to STL-QA-0028, "Water System Maintenance and Monitoring" for additional information on this procedure.

5.5.2. Reference Standards

The receipt of all reference standards must be documented through the electronic standards log program, except for Radiochemistry, which documents through a paper logbook. References standards are labeled with a unique Standard

Identification Number, date received, and the expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number. See STL-QA-0002, "Standards Preparation" and QA Directive 00-0009, "Standards Log Entry Reviews" for additional information on this procedure and policy.

All standards should be purchased with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The documentation of standard purity is archived, and references the Standard Identification Number.

All efforts are made to purchase standards that are $\geq$ 97.0% purity. If this is not possible, the purity is used in performing standards calculations.

The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a different lot is acceptable for use as a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or Laboratory Control Sample (LCS) is used as the second source confirmation.

5.5.3. Reagents

Reagents are, in general, required to be analytical reagent grade unless otherwise specific in method SOPs. Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the date the reagent was opened are documented. Solvents and acids are verified by one of the STL Network Laboratories before being received at STL St. Louis.

5.6. Sampling

Sample representativeness and integrity are the foundations upon which meaningful analytical results rely. Where documented and approved SAPs and/or QAPPs are in place, they must be made available to the laboratory before sample receipt, and approved by laboratory management before sample receipt.

5.7. Sample Handling, Transport, and Storage

5.7.1. General

Chain of Custody (COC) can be established either when bottles are sent to the field, or at the time of sampling. STL St. Louis can provide all of the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory.

Samples are received at the laboratory by a designated sample custodian and a unique Laboratory Project Identification Number is assigned. The following information is recorded for each sample shipment: Client/Project Name, Date and Time of Laboratory Receipt, Laboratory Project Number, and Signature or initials of the personnel receiving the cooler and making the entries.

Upon inspection of the cooler and custody seals, the sample custodian opens and inspects the contents of the cooler, and records the cooler temperature. If the cooler arrival temperature exceeds the required or method specified temperature range by $\pm 2^{\circ}C$ (for samples with a temperature requirement of $4^{\circ}C$, a cooler temperature of just above the water freezing temperature to $6^{\circ}C$ is acceptable); sample receipt is considered "compromised" and the procedure described in Section 4.7.1 is followed. All documents are immediately inspected to assure agreement between the test samples received and the COC.

Any non-conformance, irregularity, or compromised sample receipt as described in Section 4.7.1 must be documented on the Condition Upon Receipt Form (CUR) and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the permanent project record.

Samples that are being tested at another STL facility or by an external subcontractor shall be appropriately packaged, and sent out under COC.

Following sample labeling as described in Section 5.7.2, the sample is placed in storage. Sample storage is required to be access controlled. All samples are stored according to the requirements outlined in the test method and in a manner such that they are not subject to cross contamination or contamination from their environment. Unless specified by method or state regulation, a tolerance range of $4 \pm 2^{\circ}C$ is used. Sample storage temperatures are monitored daily.

5.7.2. Sample Identification and Traceability

Each sample container shall be assigned a unique Sample Identification Number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a sample identification label. Access to samples is documented, identifying the identity of the sample handler, date, and time of sample access.

The laboratory utilizes a custom designed LIMS to uniquely identify and track samples and analytical data throughout the facility. The following information is entered into the LIMS:

- Quote number (unique to the job or set of samples)
- Sample number (a sequential 6 digit number)
- Date received
- Date analytical results are due
- Sample description
- Client's name and address
- Client's job number (if available)
- Billing information – purchase order numbers
- Analyses requested
- Notation of special handling information

This information then becomes the Project Receipt Records. Once these records are generated, method-specific analytical worksheets from LIMS are generated for distribution to the appropriate analysts along with the sample receiving information. A secondary review of the project receipt records is carried out by the PM to ensure compliance with project requirements.

All unused portions of samples, including empty sample containers, are returned to the sample control area.

5.7.3. Sample Preparation

Holding times for every analysis are established in the method SOPs or on a project specific basis. Holding times are tracked throughout the facility using the LIMS and the NCM database. Work is scheduled by Group Leaders to avoid expiration of any sample prior to analysis. If any holding times are not met, the laboratory informs the PM as soon as possible and the PM notifies the client, through the use of the Clouseau (NCM) system.

Samples are prepared according to standardized methods. Batches are generated in the prep lab according to preparation method, analytical method, and matrix. In general, batches do not exceed 20 field samples of the same matrix and are defined as samples prepared at the same time. Subsampling ensures representativeness.

5.7.4. Sample Storage and Disposal

Samples are stored according to preservation protocols and per method or manufacture's guidelines. Samples are stored away from standards, reagents, and potentially contaminating sources in such a manner as to prevent cross contamination.

Samples should be retained in STL storage facilities for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Samples may be returned to the client per written request. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

Samples shall be disposed of in accordance with federal, state and local regulations.

5.8. Assuring the Quality of Test Results

5.8.1. Proficiency Testing

STL St. Louis analyzes Proficiency Test (PT) samples as required for accreditation. As required by NELAC, STL St. Louis participates in the PT program semi-annually for each PT field of testing for which it is accredited; according to the NELAC PT field of testing published guidelines. Under SDWA, the laboratory also analyzes a PT sample by each method once per year, if the laboratory uses more than one method for the analyte.

In addition to the PT program required for NELAC accreditation, STL participates in a number of additional PT programs, as appropriate for the specific facility. Refer to Table 9 for a complete listing.

Table 9. PE Program Participation

PE Program	Analyses	Frequency
ERA	Organics, Inorganics, Wet Chemistry, Radiochemistry	As Required
DOE MAPEP	Organics, Inorganics, Radiochemistry	Semi-Annually
DOE QAP	Radiochemistry	Annually
NYELAP	Organics, Inorganics, Wet Chemistry, Radiochemistry	Semi-Annually
APG	Organics, Inorganics, Wet Chemistry	Semi-Annually

PT samples must be handled and tested in the same manner (procedural, equipment, staff) as environmental samples. PT test sample data is archived using the requirements for project and raw data record retention.

Double Blind Performance Evaluation

STL St. Louis also participates in a double blind performance evaluation annually. An external vendor is contracted to submit double blind samples to the STL St. Louis. Both the level of customer service and the accuracy of the test results are assessed objectively by the external contractor, who provides a detailed report to the QA Director and to each of the STL facilities. This is administered as a double blind program in order to assess all facets of STL operations.

5.8.2. Control Samples

Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity, selectivity, and interferences. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch. Control samples must be uniquely identified and correlated to unique batches. There are also a number of QC sample types that monitor field sampling

accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Control Sample types and typical frequency of their application are outlined in Table 10 for environmental analyses and in Table 11 for radiochemical analyses. Note that frequency and use of control samples vary with specific regulatory, methodology and project specific criteria. Table 9 does not define STL's approach to application of QC samples for each regulatory program or test method.

5.8.3. Establishing QC Acceptance Limits

Foe new procedures, published method limits can be used until sufficient QC data are acquired (minimum of 20 to 30 data points recommended). However, the published limits may not be appropriate if they are based on a single-operator or single-laboratory study. In this case, the QA Department may establish default limits until enough data are collected for laboratory established limits to be determined.

For existing procedures, data collected over time can be used. Control charts are used with the calculated mean and standard deviation to determine if the data set being considered free of trends and are representative. If it appears that data include gross outliers, outlier tests such as the Grubbs Test, can be used to justify eliminating individual data points. Laboratory established limits must be reevaluated at least annually.

For organic analysis, "real-time" control limits may be used to evaluate the recovery limits for spikes and surrogates. The recovery limits that are set in the LIMS system are derived from historic LCS data. When sample recoveries are outside the LCS limits, additional evaluation is required. Control charts limits are reviewed for historic field samples. If, after comparison to "real-time" limits, the sample surrogate and spikes recoveries are in control, the field sample in question is deemed in control. See QA Directive 01-0001, "Use of Real Time Control Limits" for further details.

Table 10. Control Samples

Laboratory QC Sample Type	Use	Required Frequency
Laboratory Control Sample (Laboratory Fortified Blank)	Measures accuracy of method in blank matrix	1 per batch of 20 or less samples per matrix type per sample extraction or preparation method[1]
Method Blank	Measures method contribution to any source of contamination	1 per batch of 20 or less samples per matrix type per sample extraction or preparation method[1]
Instrument Blank	Measures instrumental contribution to any source of contamination	As specified in test method
Storage Blank	Measures storage contribution to any source of contamination (Volatiles only)	As specified in test method or SOP
Field QC Sample Type	**Use**	**Typical Frequency**
Matrix Duplicate	Measures effect of site matrix on precision of method	Per 20 samples per matrix or per SAP/QAPP[1,2]
Matrix Spike	Measures effect of site matrix on accuracy of method	Per 20 samples per matrix or per SAP/QAPP[1]
Matrix Spike Duplicate	Measures effect of site matrix on precision of method	Per 20 samples per matrix or per SAP/QAPP[1,2]
Equipment Blank (Equipment Rinsate)	Measures field equipment contribution to any source of contamination	Per SAP/QAPP
Trip Blank	Measures shipping contribution to any source of contamination (Volatiles only)	Per Cooler
Field Blank	Measures field environment contribution to any source of contamination	Per SAP/QAPP
Field Duplicate	Measures representativeness of sampling and effect of site matrix on precision	Per SAP/QAPP

[1] Denotes an STL required frequency
[2] Either an MSD or an MD is required per 20 samples per matrix or per SAP/QAPP.

Table 11. Minimum Radiological Quality Control Samples

Analytical Parameters	QC Sample	Quality Control
Tracerless Analysis	Method Blank	Frequency: 1 per analytical batch of 20 samples of the same matrix Criteria: Activity is Contract Detection Limit Corrective Action: Evaluate data, recount blank, reanalyze batch
	Laboratory Control Sample	Frequency: 1 per analytical batch of 20 samples of the same matrix Criteria: +/- 3s of the mean recovery as determined by control tables Corrective Action: Evaluate data, recount/reanalyze LCS, and/or associate sample batch
	Duplicate Sample	Frequency: 1 per analytical batch of 20 samples of the same matrix Criteria: <40% RPD or the uncertainties of the results overlap at the 95% confidence level, RER less than or equal to 1 Corrective Action: Evaluate data, reanalyze batch, or flag data for client evaluation
Gravimetric Yield Analysis	Method Blank	Frequency: 1 per analytical batch of 20 samples of the same matrix Criteria: Activity is Contract Detection Limit Corrective Action: Evaluate data, recount blank, reanalyze batch
	Laboratory Control Sample	Frequency: 1 per analytical batch of 20 samples of the same matrix Criteria: +/- 3s of the mean recovery as determined by control tables Corrective Action: Evaluate data, recount/reanalyze LCS, and/or associate sample batch
	Duplicate Sample	Frequency: 1 per analytical batch of 20 samples of the same matrix Criteria: <40% RPD or the uncertainties of the results overlap at the 95% confidence level, RER less than or equal to 1 Corrective Action: Evaluate data, reanalyze batch, or flag data for client evaluation
	Carrier	Frequency: added to each sample, blank, and QC sample Criteria: recovery shall be 35-120% Corrective Action: Evaluate data, reanalyze batch if yield for blank or LCS is outside acceptance criteria. Reanalyze samples with yield outside acceptance criteria. Check samples for high levels of carrier.
Analysis with Radiometric Tracers	Method Blank	Frequency: 1 per analytical batch of 20 samples of the same matrix Criteria: Activity is Contract Detection Limit Corrective Action: Evaluate data, recount blank, reanalyze batch
	Laboratory Control Sample	Frequency: 1 per analytical batch of 20 samples of the same matrix Criteria: +/- 3s of the mean recovery as determined by control tables Corrective Action: Evaluate data, recount/reanalyze LCS, and/or associate sample batch
	Duplicate Sample	Frequency: 1 per analytical batch of 20 samples of the same matrix Criteria: <40% RPD or the uncertainties of the results overlap at the 95% confidence level, RER less than or equal to 1 Corrective Action: Evaluate data, reanalyze batch, or flag data for client evaluation
	Tracer Recovery	Frequency: added to each sample, blank, and QC sample Criteria: recovery shall be 20-120% Corrective Action: Evaluate data, reanalyze batch if blank or LCS fail. Reanalyze samples with yield outside acceptance limits. Document matrix effect.

5.8.4. Calibration

Calibration of instrumentation is required to ensure that the analytical system is operating correctly and functioning at the proper sensitivity to meet established reporting limits. Each instrument is calibrated with standard solutions appropriate to the type of instrument and the calibration range established for the analytical method.

Method-specific SOPs discuss in detail how each instrument is calibrated, including frequency for calibration and re-calibration, and the source or grade of the calibration materials for environmental analyses. Table 12 discusses the calibration requirements for radiochemical analyses.

Table 12. Summary of Radiological Instrument Calibrations

Detector	Type	Minimum Frequency	Criteria
Gas Proportional Counter	Initial Calibration	Approximately Annually Or As needed	±10% of NIST traceable standard
	Self-Absorption Curve	As needed	Correlation coefficient (r^2) ≥ 0.90
	Check Source	Daily/PTU	±3 Standard Deviations from Control Values
	Plateau Check	Approximately Annually	Technical Review
	Background measurement (for subtraction)	Approximately Monthly 1000 minutes	$\alpha \leq 0.2$ cpm $\beta \leq 2.0$ cpm
	Background Check	Daily/PTU and after sample with ≥ 250 cpm	±3 Standard Deviations from Control Values
Alpha Spectrometry	Initial Calibration (eff., energy and resolution)	Approximately Annually Or As Needed	Sufficient Counts to Guarantee ≤ 2.0% Counting Uncertainty
	Calibration Check	Approximately Monthly Efficiency And Energy	±3 Standard Deviations from Control Values
	Pulser Check	Daily/PTU	±100 KeV of true value Peak Resolution at FWHM (Full Width at Half Maximum Height) ≤ 50 KeV 80-90 cps

Detector	Type	Minimum Frequency	Criteria
	Subtracted Background	Approximately Monthly	1,000 minutes
	Background Check	Approximately Monthly	±3 Standard Deviations from Control Values
KPA	Initial Calibration	Per Batch	Standard curve; $r^2 > 0.96$; lifetime between 100-340 ms
	Calibration Check	1 mid-range Per Batch	Must be within 10% of Curve
	Continuing Calibration	Every 10^{th} sample and/or at end of sequence	must be within 10% of curve or reanalyze all samples since the last acceptable calibration
Gamma Spectrometer	Initial Calibration (efficiency, energy and resolution)	Approximately Annually	± 10% of NIST source
	Check Source (efficiency, energy and resolution)	Daily	±3 Standard Deviations from Control Values
	Subtracted Background	Approximately Monthly	1000 minutes
	Background Check	Daily	±3 Standard Deviations from Control Values
Liquid Scintillation	Initial Calibration	Approximately Annually	Per manufacturer Quench curves targets 10,000 counts per source
	Check Source	Daily or per manufactures suggestion (C-14, 90% eff; H^3, 60% eff)	> 10,000 counts
	Background	Daily	±3 Standard Deviations from Control Values
	Efficiency	Daily	±3 Standard Deviations from Control Values

5.8.5. Permitting Departures from Documented Procedure

Where a departure from a documented SOP, test method, or policy is determined to be or perceived to be necessary, or is unavoidable, the departure is documented through the Clouseau (NCM) System. The departure from the procedure must be reviewed and authorized by the QA Manager and the department supervisor. Where a departure affects a specific client project, the PM must be informed of the deviation. In some instances, it is appropriate to inform the client before permitting a departure. Any such occurrence is documented in the cover letter and/or project narrative.

5.9. Project Reports

5.9.1. General

All STL St. Louis Project reports that are generated under NELAC requirements must contain the content as described in Section 5.9.2. The criteria described in Section 5.9.3 and 5.9.4 apply to all Project Reports.

5.9.2. Project Report Content

- Title
- Laboratory name, address, telephone number, contact person
- Unique Laboratory Project Number
- Total Number of Pages (report must be paginated)
- Name and address of Client
- Client Project Name (if applicable)
- Laboratory Sample Identification
- Client Sample Identification
- Matrix and/or Description of Sample
- Dates: Sample Receipt, Collection, Preparation and/or Analysis Date
- Definition of Data Qualifiers
- Reporting Units
- Test Method

The following are required where applicable to the specific test method or matrix:

- Solid Samples: Indicate Dry or Wet Weight

- Whole Effluent Toxicity: Statistical package used
- If holding time $\leq$ 48 hours, Sample Collection, Preparation and/or Analysis Time
- Indication by flagging where results are reported below the quantitation limit.

5.9.3. Project Narrative

A Project Narrative and/or Cover Letter shall be included with each project report and at a minimum includes an explanation of any and all of the following occurrences:

- Non-conformances
- "Compromised" sample receipt (see Section 4.7.1)
- Method Deviations
- QC criteria failures

Project Release

The Laboratory Director or his/her designee must authorize the release of the project report with a signature.

Where amendments to project reports are required after issue, these shall be in the form of a separate document and/or electronic data deliverable. The revised report is clearly identified as revised with the date of revision and the initials of the person making the revision. Specific pages of a project report may be revised using the above procedure with an accompanying cover letter indicating the page numbers of the project revised. The original version of the project report must be kept intact and the revisions and cover letter included in the project files.

5.9.4. Subcontractor Test Results

Project reports from external subcontract shall not be altered, and shall be included in original form in the final project report provided by STL St. Louis. Data from subcontractors' reports may be added to an STL St. Louis electronic deliverable.

Subcontracted data shall be clearly identified as such, and the name, address, and telephone number for the laboratory performing the test is included in the project report. If the report is being generated under NELAC requirements, all

information outlined in Section 5.9.2 are required for both the originating laboratory and the subcontracting laboratory.

Data subcontracted within STL may be reported on the originating laboratory's report forms provided the following mandatory requirements are met:

- The name, address, and telephone number of the facility are provided.
- Analytical results produced by the STL intra-company subcontractor are clearly identified as being produced by the subcontractor facility.
- The intra-company subcontractor's original report, including the chain of custody is retained by the originating laboratory.
- Proof of certification is retained by the originating laboratory.
- All information as outlined in Section 5.9.2 is included in the final report where the report is required to be compliant with NELAC, for both the originating and subcontracting laboratory.

5.9.5. Electronic Data Deliverables

Electronic Data Deliverables (EDD) are routinely offered as part of STL St. Louis' services. STL St. Louis offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), New Agency Standard (NAS), Format A, Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process in Section 4.4.1. Once the facility has committed to providing diskettes in a specific format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained as a QC record.

EDDs shall be subject to a secondary review to ensure their accuracy and completeness.

5.9.6. Project Report Format

STL St. Louis offers a wide range of project reporting formats, including EDDs, short report formats, and complete data deliverable packages modeled on the Contract Laboratory Protocol (CLP) guidelines. Regardless of the level of reporting, all projects must undergo the levels of review as described in Section 5.3.6.

Appendix A. Analytical Methodologies Performed at STL St. Louis

Method	NELAP Approved	Description
EPA 305.1	Yes	Acidity
EPA 310.1 SM 18 2300-B	Yes	Alkalinity
EPA 350.1	Yes	Ammonia
EPA 405.1	Yes	BOD/CBOD (Biochemical Oxygen Demand)
EPA 300.0		Bromide
EPA 300.0	Yes	Chloride
EPA 410.4	Yes	COD (Chemical Oxygen Demand)
EPA 120.1 SW846 9050	Yes	Conductivity
EPA 335.1 SW846 9010		Cyanide (Amenable)
EPA 335.2 SW846 9010	Yes	Cyanide
EPA 415.1		DOC (Dissolved Organic Carbon)
SW846 1010		Flashpoint
EPA 300.0	Yes	Fluoride
EPA 130.2	Yes	Hardness
SW846 7196	Yes	Hexavalent Chromium
EPA 300.0	Yes	Nitrite
EPA 300.0 EPA 353.1	Yes	Nitrate
EPA 353.1	Yes	Nitrate/Nitrite
EPA 413.1	Yes	Oil and Grease
EPA 365.1 EPA 300.0	Yes	Orthophosphate
SW846 9095		Paint Filter
EPA 150.1 SW846 9040 SW846	Yes	pH
EPA 420.2 SW846 9066	Yes	Phenols
EPA 365.1	Yes	Phosphate
EPA 365.1 SM 18 4500-P B&E	Yes	Phosphorus

Method	NELAP Approved	Description
SW846 Section 7.3	Yes	Reactivity
EPA 330.1		Residual Chlorine
EPA 300.0 EPA 375.4	Yes	Sulfate
EPA 376.1 SW846 9030		Sulfide
EPA 415.1	Yes	TOC (Total Organic Carbon)
EPA 450.1 SW846 9020		TOX (Total Organic Halides)
EPA 160.3	Yes	Total Solids
EPA 160.1	Yes	Total Dissolved Solids (TDS)
EPA 160.2	Yes	Total Suspended Solids (TSS)
EPA 180.1		Turbidity
SW846 1311	Yes	TCLP (Toxicity Characteristic Leaching Procedure)
EPA 200.7 SW846 6010B	Yes	ICP Metals
EPA 200.8 SW6020		ICP/MS Metals
EPA 245.1 EPA 245.2 SW846 7470A SW846 7471A	Yes	CVAA Mercury
EPA 200.0 SW846 3000/7000 Series	Yes	GFAA Metals
EPA 524.2 EPA 624 SW846 8260B	Yes	Volatiles by GC/MS
EPA 608 SW846 8082	Yes	PCBs by GC
SW846 3510B California Luft		Total Petroleum Hydrocarbons (TPH)
SW846 8151A	Yes	Herbicides by GC
EPA 625 SW846 8270C	Yes	Semivolatiles by GC and GC/MS
EPA 608 SW846 8081A	Yes	Pesticides/PCBs by GC
SW846 8040		Phenols by GC
SW846 8310	Yes	Polynuclear Aromatic Hydrocarbons (PAH)
SW846 8330		Nitroaromatics by HPLC

Method	NELAP Approved	Description
EPA 900.0 SW846 9310	Yes	Gross Alpha/Beta
EPA 904.0 SW846 9320	Yes	Radium 228
EPA 901.0 EPA 906.0 SM 18 7500-3H B	Yes	Tritium
ASTM D5174-91		Total Uranium by KPA
NAS-NS-3050A		Isotopic Uranium (U-238, -234, -235)
NAS-NS-3058		Isotopic Plutonium (Pu-239/240, -238)
NAS-NS-3004		Isotopic Thorium (Th-228, -230, -232)
Goodyear Atomic Corp		Technetium
EPA EERF, 00-02		Gross Alpha by Coprecipitation
NAS-NS-3006		Amercicium-241
EPA 903.0 SW846 9315		Total Alpha Emitting Isotopes of Radium
EPA 905.0	Yes	Strontium 89/90
EPA 901.1 ASTM D3649-85	Yes	Radium 226/228
EPA 901.1 ASTM D3649-85	Yes	Gamma Emitters
EPA 907.0	Yes	Plutonium
EPA 908.0 DOE 1990 U-02	Yes	Uranium

Appendix B. List of Certifications and Accreditations

Agency (Program)	Certification No.	Expiration Date	Comments
NELAP	11616	06/30/01	
South Carolina	85002	10/31/99[3]	Need to complete response.
Pennsylvania	68-540	09/01/2001	
Illinois	100309	3/31/00	Need to send application.
New York ELAP	11616	06/30/01	
California	2093	10/31/2001	Secondary accreditation due 07/02/01
Washington	C118	08/30/2001	Contingent on USACE
Utah	E-94	01/31/02	
Missouri	00780	8/19/00	Renewal granted. Response to audit sent 5/21/01.
Nevada	N/A	06/30/2001	Reapplication in process. Nevada requested that application be sent after 07/01/01.
New Jersey			Have project specific authorization to perform rad work. Need to complete 2002 application, NJ 2002 starts July 1, 2001.
Arkansas	N/A	06/19/02	
Kansas	Requested		Need new NY certification to reflect that NELAP/NY ELAP are connected.
Kentucky	90125	12/31/01	
Connecticut	PH0241	03/31/03	
USACE		01/14/02	

Appendix C. List of Standard Operating Procedures

Quality Assurance			
Number	**Rev #**	**Date**	**Title**
STL-QA-0002	3	4/20/01	Standards Preparation
STL-QA-0003	2	4/20/01	Thermometer Calibration
STL-QA-0004	3	4/20/01	Mechanical Pipette Calibration
STL-QA-0005	3	4/20/01	Balance Calibration and Weight Verification
STL-QA-0006	3	4/20/01	Sample Receipt and Chain of Custody
STL-QA-0007	1	4/20/01	Release of Samples for Analysis Prior to Complete Login
STL-QA-0009	2	4/19/01	Data Reports
STL-QA-0010	2	4/19/01	Data Packaging
STL-QA-0011	3	4/22/01	Data Review and Verification
STL-QA-0014	1	4/19/01	Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
STL-QA-0016	2	4/22/01	IDL/MDL Determination
STL-QA-0018	2	4/22/01	Quality Assurance Records Maintenance
STL-QA-0019	1	4/22/01	Autosampler Loading Verification
STL-QA-0020	2	4/22/01	Laboratory Logbook Control and Maintenance
STL-QA-0021	2	4/22/01	Internal Surveillance
STL-QA-0022	2	4/19/01	Off-Site Storage Documentation
STL-QA-0023	2	4/19/01	Project Records and Document Control
STL-QA-0024	2	4/20/01	Preventative Maintenance
STL-QA-0025	2	5/10/01	Temperature Monitoring
STL-QA-0028	3	4/20/01	Water System Maintenance and Monitoring
STL-QA-0029	2	4/20/01	Laboratory Security Systems
STL-QA-0030	2	4/20/01	Preventing Sample Contamination
STL-QA-0031	2	4/20/01	VOA Holding Blank Analysis
STL-QA-0032	1	4/20/01	Maintaining Time and Data Integrity
STL-QA-0033	1	4/20/01	Handling Confidential Information: USEPA CLP

Inorganic Preparations			
Number	**Rev #**	**Date**	**Title**
CORP-IP-0001STL	1.1	10/31/01	Acid Digestion of Waters and Soils, CLP SOW ILM03.0
CORP-IP-0002STL	2.1	10/31/01	Acid Digestion of Soils, SW846 Method 3050B
STL-IP-0001	4	03/26/01	Reactive Cyanide
STL-IP-0002	3	03/26/01	Reactive Sulfide
STL-IP-0004	2	03/15/01	Glassware Preparation for Inorganic and Trace Metal Analysis
STL-IP-0005	3	04/03/01	Cyanide Distillation
STL-IP-0006	1	04/03/01	Distillation of Phenols
STL-IP-0007	1	02/02/01	Bomb Preparation of Solids for Inorganic Analysis
STL-IP-0008	3	02/02/01	Aqueous Sample Preparation for ICP and GFAA CLP Analysis
STL-IP-0009	2	02/02/01	Soil Sample Preparation for ICP and GFAA CLP Analysis
STL-IP-0011	2	02/02/01	Acid Digestion of Aqueous and Extracts for Total Metals by Graphite Furnace Atomic Absorption (GFAA) Spectroscopy (method 3020A, 7740, and 7060A)
STL-IP-0013	3	07/13/01	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA, ICP Spectroscopy, or ICP/MS (Method 3010A)
STL-IP-0014	0	05/29/01	Alkaline Digestion for Hexavalent Chromium

Metals			
Number	**Rev #**	**Date**	**Title**
CORP-MT-OOO1STL	2.2	10/31/00	Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analyses, SW-846 Method 6010B and EPA Method 200.7
CORP-MT-0002STL	2.2	11/03/00	Inductively Coupled Plasma-Atomic Emission Spectroscopy, Method 200.7 CLP-M, SOW ILM03.0
CORP-MT-0005STL	0.2	10/31/00	Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW846 7470A and MCAWW 245.1
CORP-MT-0006STL	1.1	8/5/00	Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, Method 245.1 CLP-M, SOW ILM03.0
CORP-MT-0007STL	1.2	10/31/00	Preparation and Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption Spectroscopy, SW846 7471A and MCAWW 245.5
CORP-MT-0008STL	0.2	8/5/00	Preparation and Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption Spectroscopy, Method 245.5 CLP-M, SOW ILM03.0
STL-MT-0001	0	8/17/01	Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry
STL-MT-0002	2	8/5/00	Graphite Furnace Atomic Absorption Spectrophotometry by SW-846 Method 7000A, EPA 200 Series and EPA 200.9

WET CHEMISTRY			
Number	**Rev #**	**Date**	**Title**
CORP-WC-0001	1.1	10/30/00	TOTAL ORGANIC HALIDES IN WATER BY SW-846 METHOD 9020B
CORP-WC-0002 STL	1.2	9/20/00	DETERMINATION OF SOLIDS IN WATER AND WASTES
STL-WC- 0001	0	9/20/00	TURBIDITY
STL-WC-0002	0	8/25/00	CYANIDE ANALYSIS BY THE TECHNICON TRAACS 800 AUTOANALYZER
STL-WC-0003	1	9/20/00	HARDNESS
STL-WC-0004	0	8/25/00	CHEMICAL OXYGEN DEMAND
STL-WC-0005	1	9/20/00	PERCENT SOLIDS DETERMINATION
STL-WC-0009	2	9/20/00	OIL AND GREASE (PARTITION-GRAVIMETRIC METHOD)
STL-WC-0011	2	9/20/00	ANALYSIS OF pH in WATER
STL-WC-0012	1	9/20/00	ANALYSIS OF SULFIDE IN WATER
STL-WC-0013	1	9/20/00	PHOSPHOROUS, ALL FORMS
STL-WC-0014	1	8/25/00	ANALYSIS OF AMMONIA AS NITROGEN IN WATER AND SOIL
STL-WC-0015	0	8/25/00	BIOCHEMICAL OXYGEN DEMAND
STL-WC-0016	1	4/20/01	TOTAL ORGANIC CARBON
STL-WC-0017	0	8/25/00	PHENOLICS, TOTAL RECOVERABLE
STL-WC-0018	2	9/20/00	ACIDITY OF WATER AND WASTEWATER
STL-WC-0019	1	9/20/00	ALKALINITY IN WATER AND SOIL
STL-WC-0021	2	9/20/00	ANALYSIS OF pH IN SOIL
STL-WC-0023	2	9/20/00	NITRATE/NITRITE ANALYSIS BY TRAAC 800 (HYDRAZINE REDUCTION)
STL-WC-0025	0	9/20/00	CONDUCTIVITY IN WATER AND SOLIDS
STL-WC-0026	1	9/20/00	FLASHPOINT BY PENSKY-MARTENS CLOSED CUP TESTER
STL-WC-0028	1	8/25/00	THE ANALYSIS OF ANIONS BY ION CHROMATOGRAPHY
STL-WC-0029	2	9/20/00	RESIDUAL CHLORINE
STL-WC-0031	1	9/20/00	PAINT FILTER LIQUIDS TEST
STL-WC-0033	2	9/20/00	HEXAVALENT CHROMIUM (COLORIMETRIC)
STL-WC-0034	1	9/20/00	HEAT OF COMBUSTION (BTU)
STL-WC-0035	1	9/20/00	COLOR

Organic Preparations			
Number	**Rev #**	**Date**	**Title**
CORP-OP-0001STL	3.2	04/30/01	Extraction and Cleanup of Organic Compounds from Water and Soils, Based on SW-846 3500 Series, 3600 Series, 8151A and 600 Series
CORP-OP-0002STL	1.2	10/31/01	Toxicity Characteristic Leaching Procedure and Synthetic Precipitation Leaching Procedure
STL-OP-0001	4	5/17/01	Glassware Preparation for Organic Analysis
STL-OP-0003	1	03/20/01	Extraction of PCB in Oil
STL-OP-0004	1	03/20/01	Extraction of Semivolatile Organic Compounds Using the Dionex Accelerated Solvent Extractor (ASE) Model 200
STL-OP-0005	1	03/20/01	California Wet Leaching Procedure for Metals

GC and HPLC			
Number	**Rev #**	**Date**	**Title**
CORP-GC-0001 STL	5.1	4/20/01	Gas Chromatographic Analysis Based on Method 8000B, 8021B, 8081A, 8082 and 8151A, SW-846
STL-GC-0005	4	11/14/00	Extractable Total Petroleum Hydrocarbons (TPH) by GC
STL-GC-0009	2	03/08/01	Analysis of Chlorinated Pesticides and PCBs by EPA Method 608
STL-GC-0010	2	03/12/01	LBH Purge and Trap Analysis by Modified EPA Method 8015A or the California LUFT
STL-GC-0013	4	03/12/01	Extraction and Analysis of Phenols by SW846-8040A
STL-LC-0001	2	04/23/01	HPLC Analysis of PAH's (PNA's) by SW-846-8310
STL-LC-0002	1	11/14/00	Analysis of Nitroaromatic and Nitramine Explosives by HPLC

GC/MS Semivolatiles			
Number	**Rev #**	**Date**	**Title**
CORP-MS-0001STL	2.2	04/23/01	GC/MS Analysis Based on Methods 8270C and 625
STL-MS-0008	2	04/23/01	Extraction and Analysis of Semivolatile Organics by CLP 3/90

GC/MS Volatiles			
Number	**Rev #**	**Date**	**Title**
CORP-MS-0002STL	1.2	10/31/01	Determination of Volatile Organics by GC/MS Based on Method 8260B, 624 and 524.2
STL-MS-0007	1	06/06/95	Analysis of Volatile Organics by Gas Chromatography/Mass Spectroscopy: USEPA CLP, 3/90, Scope of Work
STL-MS-0015	1	03/8/99	The Low Level Analysis of Volatile Organics by Gas Chromatography/Mass Spectroscopy: USEPA CLP, 3/95, Scope of Work

Radiochemistry			
Number	**Rev #**	**Date**	**Title**
STL-RC-0002	2	10/30/00	PLANCHET PREPARATION FOR RADIOCHEMISTRY AND RADIOLOGICAL SCREENING ANALYSIS
STL-RC-0003	3	04/24/01	DRYING AND GRINDING OF SOIL AND SOLID SAMPLES
STL-RC-0004	3	10/30/00	PREPARATION OF SOIL, SLUDGE, AND FILTER PAPER SAMPLES FOR RADIOCHEMICAL ANALYSIS
STL-RC-0020	2	10/30/00	DETERMINATION OF GROSS ALPHA/BETA ACTIVITY
STL-RC-0021	1	10/30/00	GROSS ALPHA RADIATION IN WATER USING COPRECIPITATION
STL-RC-0025	2	10/30/00	PREPARATION OF SAMPLES FOR GAMMA SPECTROSCOPY
STL-RC-0030	2	10/30/00	THE DETERMINATION OF TRITIUM IN WATER AND OTHER FLUIDS
STL-RC-0040	0	4/13/01	TOTAL ALPHA EMITTING ISOTOPES OF RADIUM
STL-RC-0041	0	4/13/01	RADIUM 228 IN WATER
STL-RC-0050	1	10/30/00	PREPARATION OF STRONTIUM 89 AND 90
STL-RC-0090	2	10/30/00	PREPARATION OF SAMPLES FOR SEQUENTIAL DETERMINATION OF ISOTOPIC AMERICIUM, CURRIUM, NEPTUNIUM, PLUTONIUM, THORIUM, AND URANIUM IN AQUEOUS SAMPLES
STL-RC-0100	2	10/30/00	ACTINIDE COPRECIPITATION
STL-RC-0110	1	10/30/00	ANALYSIS OF TOTAL URANIUM BY LASER-INDUCED PHOSPHORIMETRY
STL-RC-0120	0	4/13/01	DETERMINATION OF TECHNETIUM-99
STL-RC-0232	2	10/30/00	ISOTOPIC THORIUM AND NEPTUNIUM BY EICHROM SEPARATION RESIN
STL-RC-0238	1	10/30/00	ISOTOPIC URANIUM BY EICHROM SEPARATION RESIN
SL 13012	2	10/30/00	EVALUATION OF THE SAMPLE TRANSMISSION FACTOR
STL 13019	2	10/30/00	CALIBRATION OF THE LOW BACKGROUND GAS FLOW PROPORTIONAL COUNTING SYSTEM
STL 13021	2	10/30/00	DAILY OPERATION OF THE LOW BACKGROUND GAS FLOW PROPORTIONAL COUNTING SYSTEM
STL-RC-5006	2	10/30/00	DECONTAMINATION OF LABORATORY GLASSWARE AND ELECTRODEPOSITION EQUIPMENT
STL-RC-5016	0	7/04/99	PREPARATION OF VEGETATION AND TISSUE MATRICES
STL-RC-5048	0	7/4/99	RADIOCHEMICAL DETERMINATION OF TRITIUM IN SOIL, VEGETATION AND OTHER BIOLOGICAL SAMPLES AZEOTROPIC
STL-RD-0101	2	3/16/01	DAILY OPERATIONS OF A GERMANIUM SPECTROSCOPY SYSTEM
STL-RD-0103	0	10/30/99	MAINTENANCE OF THE GERMANIUM SPECTROSCOPY SYSTEM
STL-RD-0201	2	10/30/00	DAILY OPERATIONS OF AN ALPHA SPECTROSCOPY SYSTEM
STL-RD-0203	1	10/30/00	CALIBRATION AND MAINTENANCE OF A ALPHA SPECTROSCOPY SYSTEM
STL-RD-0403	2	10/1/00	DAILY CALIBRATION VERIFICATION AND MAINTENANCE OF THE LOW BACKGROUND GAS FLOW PROPORTIONAL COUNTING SYSTEM

APPENDIX V
STL's GROSS ALPHA STANDARD OPERATING PROCEDURE

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SOP No.:	**STL-RC-0020**
Revision No.:	2
Revision Date:	10/30/00
Page:	1 of 26
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OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE:
Determination of Gross Alpha/Beta Activity

(SUPERCEDS SL13002 REVISION 2)

Prepared by: _____

Approved by: _____
Technical Specialist

Approved by: _____
Quality Assurance Manager

Approved by: _____
Environmental Health and Safety Coordinator

Approved by: _____
Laboratory Director

1 SUMMARY OF METHOD

1.1 This SOP is applicable for the preparation and analysis of samples for gross alpha and/or beta radioactivity.

1.2 This method is applicable to determination of gross alpha and/or gross beta activity in air filters, water, soil/sediment, and vegetation samples.

1.2.1 For total sample activity, an aliquot of aqueous sample is evaporated to a small volume, treated with nitric acid to convert any chlorides to nitrates, and transferred quantitatively to a tarred counting planchet. The sample residue is dried, and then counted for alpha and/or beta radioactivity using a Gas Flow Proportional Counter.

1.2.2 For the activity of dissolved matter, an aliquot of aqueous sample is filtered through a 0.45-mm membrane filter. The filtrate is evaporated to a small volume, treated with nitric acid to convert any chlorides to nitrates, and transferred quantitatively to a tarred counting planchet. The sample residue is dried, and then counted for alpha and/or beta radioactivity using a Gas Flow Proportional Counter.

1.2.3 For the activity of suspended matter, an aliquot of aqueous sample is filtered through a 0.45-mm membrane filter. The filter is transferred to a counting planchet. The sample residue is dried, and then counted for alpha and/or beta radioactivity using a Gas Flow Proportional Counter.

1.2.4 Air filter samples are counted for gross alpha and/or beta activity without further processing if the filter is less than 2 inches diameter. If the filter is greater than 2-inch diameter, the sample is digested per STL-RC-0004, "Preparation of Soil, Sludge and Filter Paper Samples for Radiochemical Analysis," and then an aliquot prepared like a liquid.

1.2.5 Solid samples are analyzed for gross alpha and/or beta activity as a dry powder unless DOE Method RP710 is requested. When the QAS shows that RP710 is required, an acid leach is performed per STL-RC-0004, "Preparation of Soil, Sludge and Filter Paper Samples for Radiochemical Analysis". The digestate is then treated like a liquid.

1.2.6 Oil samples are ashed in a muffle furnace, then dissolved in nitric acid. The sample is then transferred to a tarred planchet, dried, and counted for alpha and/or beta radioactivity using a Gas Flow Proportional Counter.

1.2.7 Gross Alpha and Gross Beta activity does not identify the radionuclide that is present. Instead, the activity is referenced as equivalent to Am-241 for Gross Alpha and Sr-90/Y-90 for Gross Beta.

2 SCOPE AND APPLICATION

2.1 This procedure applies to the preparation and analysis of samples for gross alpha and/or beta radioactivity.

2.2 Responsibilities:

2.2.1 Counting Lab Supervisor: to confirm that this procedure is followed for the determination of gross alpha and/or gross beta activity in air filters, water, soil/sediment, and vegetation samples.

2.2.2 Radiochemistry Group Leader: (or designee) to delegate the performance of this procedure to personnel who are experienced with this procedure and with the equipment associated with the implementation of this procedure.

2.2.3 Analyst/Technician performing this procedure, to follow the instructions and to report any abnormalities to Counting Lab Team Leader immediately. To confirm that all equipment used is working properly prior to starting this procedure.

2.3 This SOP is applicable to EPA 600/4-80-032, "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," Method 900.0; APHA, "Standard Methods for Water and Wastewater," Method 7110; SW846, Method 9310; and DOE/EM-0089T, "DOE Methods for Evaluating Environmental and Waste Management Samples," Method RP710.

2.4 Quality control limits (accuracy and precision for spikes) are also maintained in QuantIMS, and are also dynamic. Therefore, they are not specifically listed in this document, but can be retrieved at any time using TraQAr tools.

2.5 Method detection limits are maintained in the Information Management System (QuantIMS). Because of their dynamic nature, they are not specifically listed in this document, but can be retrieved at any time using TraQAr tools. Reporting limits will be proportionately higher for sample extracts that require dilution and for soil samples that require concentration adjustments to account for percentage moisture.

2.6 The Reporting Limits (RL) under routine operating conditions are summarized in the following table:

Matrix	Sample Volume (Note 1)	Count Time (Min)	Gross Alpha Reporting Limit	Gross Beta Reporting Limit
Air	1 filter	100	1 pCi/filter	1 pCi/filter
Water	0.200 L	100	5 pCi/L	3 pCi/L
Drinking Water	0.500 L	100	2 pCi/L	1.5 pCi/L
Soil, Sediment, Vegetation	0.100 g	100	10pCi/g	10 pCi/g

Note 1: Sample volume may need to be adjusted in order not to exceed 100 mg of dried residue on planchet. Volume shown is "typical" maximum volume used provided Total Solids does not exceed 500 ppm for waters and 200 ppm for drinking waters.

3 DEFINITIONS

3.1 See Quality Assurance Management Plan (QAMP) for glossary of common terms.

3.2 Minimum Detectable Activity (MDA) - The smallest amount of activity that can be detected given the conditions of a specific sample. It is reported at the 95% confidence interval, meaning that there is a 5% chance that a false signal was reported as activity, and a 5% chance that true activity went undetected. The MDA that is reported is a combination of counting error as well as preparation errors.

4 INTERFERENCES

4.1 Since, in this method for gross alpha and gross beta measurement, the radioactivity of the sample is not separated from the solids of the sample, the solids concentration is a limiting factor in the sensitivity of the method for any given sample.

4.2 For a 2-inch diameter counting planchet (20 cm2), an aliquot containing 100 mg of dissolved solids would be the maximum aliquot size for that sample which should be evaporated and counted for gross alpha or gross beta activity.

4.3 Radionuclides that are volatile under the sample preparation conditions of this method can not be measured. Other radioactivities may also be lost during the sample evaporation and drying (such as tritium and some chemical forms of radioiodine). Some radioactivities, such as the cesium and technetium radioisotopes, may be lost when samples are heated to dull red color. Such losses are limitations of the test method.

4.4 Moisture absorbed by the sample residue increases self absorption and, if uncorrected, leads to low-biased results. For hygroscopic sample matrices, the nitrated water solids (sample evaporated with nitric acid present) will not remain at a constant weight after being dried and exposed to the atmosphere before and during counting. Those types of water samples need to be heated to a dull red color for a few minutes to convert the salts to oxides.

4.5 Inhomogeneity of the sample residue in the counting planchet interferes with the accuracy and precision of the method.

5 SAFETY

5.1 Procedures shall be carried out in a manner that protects the health and safety of all associates.

5.2 The Chemical Hygiene Plan (CHP) gives details about the specific health and safety practices, which are to be followed in the laboratory area. Personnel must receive training in the CHP, including the written Hazard Communication plan, prior to working in the laboratory. Consult the CHP, the Health and Safety Policies and Procedures Manual, and available Material Safety Data Sheets (MSDS) prior to using the chemicals in the method.

5.3 Consult the Health and Safety Policies and Procedures Manual for information on Personal Protective Equipment. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately. VITON gloves may be worn when halogenated solvents are used for extractions or sample preparation. Nitrile gloves may be used when other solvents are handled. [Note: VITON is readily degraded by acetone; all solvents will readily pass through disposable latex rubber gloves.]

5.4 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

5.4.1 Nitric acid is known to be an oxidizer and corrosive.

5.5 Exposure to chemicals will be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples will be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.6 The preparation of all standards and reagents and glassware cleaning procedures that involve solvents will be conducted in a fume hood with the sash closed as far as the operations will permit or by other means of mechanical ventilation.

5.7 All work must be stopped in the event of a known, or potential, compromise to the health or safety of any associate. The situation must be reported immediately to a laboratory supervisor.

5.8 Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices outlined in the Health and Safety Manual. These employees must have training on the hazardous waste disposal practices initially upon assignment of these tasks, followed by annual refresher training.

6 EQUIPMENT AND SUPPLIES

6.1 Equipment and supplies for this procedure consist of the following:

6.1.1 Analytical Balance (4 - or 5 - place).

6.1.2 Beakers: borosilicate glass, various sizes.

6.1.3 Bottle, wash.

6.1.4 Counting planchets, stainless steel, 5.0 cm (2.0"), cleaned per STL-RC-0002, "Preparation of Stainless Steel Planchets for Radiochemistry Analyses."

6.1.5 Desiccator with desiccant, Dri-Rite or equivalent.

6.1.6 Drying oven with thermostat set at 105° C ± 2 °C.

6.1.7 Filter paper: ash less, Whatman #41 or ash less paper pulp, and 0.45-mm membrane, 5.0 cm.

6.1.8 Gas flow proportional counting system.

6.1.9 Graduated cylinder - size appropriate to sample volume.

6.1.10 Propane torch.

6.1.11 Hot plate-stirrer or heat lamp.

6.1.12 Calibrated pipettes, Eppendorf or equivalent.

6.1.13 Policeman: rubber or plastic

6.1.14 Porcelain crucibles with lids, approximately 30-ml. capacity.

6.1.15 Muffle furnace, programmable preferred.

6.1.16 Tongs or forceps.

7 REAGENTS AND STANDARDS

7.1 All reagent preparation is documented in the reagent logbook. All reagents are labeled with their unique ID, name (and concentration, if applicable) of the reagent, the date prepared and the expiration date.

7.2 Deionized Water Type II as described in ASTM Part 31, 1193-74, obtained from the Milli-Q unit.

CAUTION: Refer to Material Safety Data Sheets (MSDS) for specific safety information on chemicals and reagents prior to use or as needed.

7.3 Reagents

7.3.1 ASTM Type II water.

7.3.2 Nitric acid, concentrated (16N HNO3) - WARNING: Corrosive liquid and hazardous vapor; oxidizer.

7.3.3 Sodium Sulfate.

7.4 Prepared Reagents

NOTE: Reagents are prepared from reagent grade chemicals, unless otherwise specified below, and reagent water.

NOTE: Replace lab-prepared reagents annually, unless otherwise specified below. As a minimum, label all reagents with chemical name, concentration, date prepared and preparer's initials, and expiration date.

7.4.1 4N Nitric acid (4N HNO3) - Add 250 ml of 16N HNO3 to 750 ml of reagent water and mix well.

7.4.2 Sodium Sulfate (100 mg/ml. Na2SO4). Dissolve 10g of Na2SO4 in 80 ml. of DI water. Dilute to 100 ml and mix well.

7.5 Standards

7.5.1 All standards preparation, documentation and labeling must follow the requirements of STL-QA-0002.

CAUTION: Radioactive

7.5.1.1 Americium-241, calibrated - NIST traceable, diluted to approximately 20 dpm/ml.

7.5.1.2 Strontium-90, calibrated - NIST traceable, in equilibrium with Yttrium 90, diluted to approximately 20 dpm/ml. CAUTION: Radioactive.

8 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Aqueous samples should be preserved at the time of collection by adding sufficient nitric acid to a pH < 2.

8.2 Preserve soil samples by maintaining at 4° ± 2°C from the time of sample collection.

8.3 Samples must be analyzed within 28 days of sample collection

8.4 If samples are collected without acidification, they should be brought to the laboratory within 5 days, nitric acid added to bring the pH to 2 or less, the sample shaken, and then held for a minimum of 16 hours in the original container before analysis or transfer of sample. If dissolved or suspended material is to be analyzed separately, do not acidify the sample before filtering the sample. The filtering may be performed in the field by the customer or by the laboratory.

8.5 Samples may be collected in either plastic or glass containers.

8.6 The maximum holding time is 180 days from sample collection for all matrices.

9 QUALITY CONTROL

9.1 QC requirements

9.1.1 Each analytical batch may contain up to 20 environmental samples, a method blank, a single Laboratory Control Sample (LCS, an MS/MSD pair, or a sample duplicate. In the event that there is not sufficient sample to analyze an MS/MSD or duplicate pair, a LCS duplicate (LCSD) is prepared and analyzed.

9.1.2 Samples that have assigned QC limits different than the standard limits contained in QuantIMS QC code 01 (unless permitted by QA) must be batched separately, but can share the same QC samples.

9.1.3 Additional MS/MSDs or sample duplicates do not count towards the 20 samples in an analytical batch.

9.1.4 A method blank must be included with each batch of samples. The matrix for aqueous analyses is organic-free water and salt for solids.

9.1.5 The LCS is spiked with all of the standard target compounds and is used to monitor the accuracy of the analytical process, independent of matrix effects. The matrix for aqueous analyses is organic-free water and sodium sulfate for solids.

9.1.6 All LCS and MS/MSD and results -- whether they pass criteria or not -- are uploaded into the QuantIMS system for maintenance and periodic update of limits.

9.1.7 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.8 All data will be reviewed by the analyst (1st review level) and then by a peer or supervisor (2nd level review).

9.2 Analyze quality control samples concurrent with routine samples. The frequency of quality control samples is based on a batch of 20 samples or less of a similar matrix processed at the same time. The minimum QC samples will consist of one method blank, one LCS, and one duplicate per batch.

9.3 Activity in the method blank must be less than the Reporting Limit (RL). Samples associated with a noncompliant method blank must be reprepared with an acceptable method blank. An exception to this corrective action is made if the method blank activity is above the RL but less than 10X the sample activity. A comment should be made on the Data Review Sheet if the blank has an activity above the MDA calculated for the blank.

9.4 The acceptable criteria for the LCS is $\pm 3\sigma$ from the mean as determined by laboratory control charts. Samples associated with a noncompliant LCS must be reprepared with a compliant Laboratory Control Sample.

9.5 A duplicate sample is analyzed every 20 samples or less. Calculate the Relative Percent Difference (RPD) for all duplicate analyses. The acceptance limit for the RPD is 25% for water samples and 40% for soils if the concentration in the sample and duplicate are [3] 5 times the CRDL. If the concentrations are below the CRDL the duplicate concentration should be within the 3s error of the sample. If the duplicate does not meet the acceptance criteria, the data for the batch will be flagged.

9.6 Matrix spike shall be included with each batch of samples. Exceptions are allowed only for samples in which Project Management clearly indicates, either during project planning with the client, or in the data report, that the QC is not acceptable for Utah compliance. The criteria for the MS is $\pm 3s$ from the mean as determined; by historical data. Data associated with recoveries outside the limit will be flagged

9.7 Additional quality control measures will be performed if they are requested by the client. The requirements of a client Quality Assurance Project Plan (QAPP) will have precedence over the requirements of this SOP in cases where they differ.

9.8 Documentation

9.8.1 Measure and record a method blank and laboratory control sample with every analytical batch.

9.8.2 The LCS and the ICVS must be made from a different stock than that used for the working calibration standards.

9.8.3 The acceptable limits for the quality control samples/standards are as follows:

9.8.3.1 The correlation coefficient of calibration must be ^{3} 0.995.

9.8.3.2 The CCVSs and ICVSs must be ± 15 of the true value.

9.8.3.3 Laboratory Control Samples must be ± 20 of the true value or as determined by control charts if so specified.

9.8.3.4 Matrix spikes should be ± 25% of the true value or as determined by control charting.

9.8.3.5 Relative Percent Differences should be within 20% for aqueous samples and within 35% for solid samples, or laboratory generated control charts may be used for control limits.

9.8.3.6 The Method Blank, ICB and CCB concentrations must always be less than the method or contract required detection limit.

9.9 Procedural Variations

9.9.1.1 One time procedural variations are allowed only if deemed necessary in the professional judgment of the analyst to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager.

9.9.1.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

9.10 Nonconformance and Corrective Action

9.10.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.11 QC Program

9.11.1 Further details of QC and corrective action guidelines are presented in the QC Program policy document (QA-003).

10 CALIBRATION AND STANDARDIZATION

10.1 The ratio of count rate to disintegration rate of the detectors shall be obtained through calibration of the detectors for both alpha and beta activity determinations using geometries and weight ranges similar to those encountered when performing the gross analyses. Refer to SOP SL13019 (to be replaced by STL-RD-0001), " Calibration of the Low Background Gas Flow Proportional Counting System."

10.2 The acceptable limits for the quality control samples/standards are as follows:

10.3 Laboratory Control Samples must be ± 20 of the true value or as determined by control charts if so specified.

10.4 Matrix spikes should be ± 25% of the true value or as determined by control charting.

10.5 Relative Percent Differences should be within 20% for aqueous samples and within 35% for solid samples, or laboratory generated control charts may be used for control limits.

10.6 The Method Blank, ICB and CCB concentrations must always be less than the method or contract required detection limit.

11 PROCEDURE

11.1 One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.1.1 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and a corrective action described.

11.2 Water Sample Preparation

11.2.1 If total sample activity of an aliquot of aqueous sample is to be determined, proceed to section 11.3.

11.2.2 If the activity of dissolved matter in an aliquot of aqueous sample is to be determined, filter the desired aliquot through a 0.45-mm membrane filter. Proceed to section 11.3 using the filtrate.

11.2.3 If the activity of suspended matter of an aliquot of aqueous sample is to be determined, filter the desired aliquot through a 0.45-mm membrane filter. The filter is analyzed per section 11.6.

11.3 Aqueous Sample - Total Solid Screen

NOTE: If the sample was previously radiation screened as per SOP SL13015 (to be superseded by STL-RC-0010), "Screening Samples for the Presence of Radioactive Materials," the total solid content can be determined from this procedure and this section omitted.

11.3.1 Record all sample preparation data on a sample worksheet or on the Weight file (Appendix 1) for the batch.

11.3.2 Agitate the sample container thoroughly.

NOTE: If alpha and beta are to be determined simultaneously from a single aliquot, the net residue weights for alpha apply.

11.3.3 Pipette a 10 ml. aliquot on to a tared planchet.

11.3.4 Evaporate to dryness using a hot plate or heat lamp on low to medium heat.

11.3.5 Allow to cool in desicator for a minimum of 30 minutes.

11.3.6 Reweigh the planchet to estimate solids content of the sample.

11.3.7 From the net residue weight and sample volume used, determine the sample volume required to meet the target residue weight using the formula given in step 12.1, with a target weight of 90 mg (not to exceed 100 mg) alpha/beta dried residue on the planchet. If only Gross Beta is being performed, the target weight may be increased to 140 mg. Compare the calculated volume to meet the weight limitation with the volume required to ensure that the MDA is below the Reporting Limit (Tables 17.4). The volume for analysis is the smaller of the two volumes.

11.4 Aqueous Sample Total Activity

11.4.1 Initiate appropriate sample worksheet for the samples to be analyzed and complete as required or begin a Weight file for recording planchet weights (Appendix 1).

11.4.2 Shake the sample container thoroughly. Measure a volume of sample, previously determined in section 11.3, into an appropriately sized beaker. Record volume of sample used.

11.4.3 If it is determined in step 11.3.6 that only a small volume of sample is required, the additional volume may be added in small aliquots directly to the planchet used to determine the volume needed to achieve the target sample weight. If this is done, skip steps 11.4.6 through 11.4.8 below.

11.4.4 Prepare a method blank from an aliquot of reagent water equivalent to the sample volumes. Prepare LCS with a similar aliquot of reagent water spiked with 1 ml. of the standards described in 7.3.1 and 7.3.2. Note: separate LCS's must be prepared for alpha and beta analysis due to the beta emitting decay products of Am-241.

11.5 Add 5 ml of 16N nitric acid to blank and LCS.

11.5.1 Evaporate to near dryness using a medium to low temperature hot plate or heat lamp. DO NOT ALLOW LIQUID TO SPLATTER. Do not allow residue to "bake" on hot plate.

11.5.2 Quantitatively transfer the sample to a tared, stainless steel planchet.

11.5.3 Use a policeman, if needed, to complete the transfer. Wash down the beaker wall with small portions of dilute HNO3 and add to the planchet.

11.5.4 Evaporate to dryness on a warm hot plate so that the sample does not boil. Do not allow residue to "bake" on hot plate. Remove sample from hot plate.

NOTE: Do not allow liquid to splatter.

11.5.5 Dry planchets in an oven at 105 ± 2 °C for a minimum of 2 hours. This step may be eliminated if planchet is flamed as described in Section 11.4.12.

11.5.6 Cool planchets in a desiccator for a minimum of 30 minutes. Weigh the cooled planchets and record final weight.

NOTE: If alpha and beta are to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; 100 mg (2.0" planchet).

11.5.7 If moisture is present in the sample residue due to hygroscopic salts, heat the planchet using a propane torch to a dull red color for a few minutes to convert the salts to oxides.

11.5.8 Store dry sample in a desiccator until counted for gross alpha and/or beta activity.

11.5.9 Submit the sample for counting..

11.5.10 Calculate the activity, the total error, and the MDA per section 12.0.

11.6 Oil

11.6.1 Initiate appropriate sample worksheet for the samples to be analyzed and complete as required or begin a Weight file for recording planchet weights (Appendix 1).

11.6.2 Fill a 30 ml porcelain crucible ¼ full with confetti made from Whatman No. 41 filter paper or ashless paper pulp.

11.6.3 Place crucible on analytical balance, then tare the balance.

11.6.4 Weigh to the nearest 0.0001 g, approximately 1 to 2 gm sample of the oil onto the shredded filter paper. Record the sample weight. Cover with a crucible lid.

11.6.5 Prepare a method blank from shredded filter paper in a crucible. Prepare a LCS from shredded filter paper that has been spiked with 1 ml. of the solutions described in 7.3.1 and 7.3.2 in a crucible. Note: separate LCS's must be prepared for alpha and beta analysis due to the beta emitting decay products of Am-241.

11.6.6 If the sample is a mixture of oil and water,or is a sample spiked with an aqueous solution, evaporate the water on a hot plate or under a heat lamp before muffling. Do not allow residue to "bake" on hot plate. A programmable muffle program may also be used to dry the water before ramping the temperature.

NOTE: Do not allow liquid to splatter.

11.6.7 Heat the sample in a muffle oven for one hour at 750° C.

11.6.8 Remove the sample from the muffle oven and allow the sample to cool to room temperature.

11.6.9 Add approximately 2 ml of 4 N HNO3 to the residue in the crucible.

11.6.10 Quantitatively transfer the sample to a tared, stainless steel planchet, with 4 N HNO3.

11.6.11 Use a policeman, if needed, to complete the transfer. Wash down the crucible walls with small portions of dilute HNO3 and add to planchet from step 11.4.9.

11.6.12 Evaporate to dryness on a warm hot plate or heat lamp so that the sample does not boil. DO NOT ALLOW LIQUID TO SPLATTER. Do not allow residue to "bake" on hot plate.

11.6.13 Dry the planchet in an oven at 105 ± 2 °C for a minimum of two hours. This step may be eliminated if planchet is flamed as described in Section 11.4.14.

11.6.14 Weigh the cooled planchet and record final weight.

CAUTION: Ensure that the solids content do not exceed the maximum allowed weight for the determination and planchet used.

11.6.15 If a green residue from chlorides which may be present forms, obtain technical assistance on how to proceed.

11.6.16 Store dry sample in a desiccator until counted for gross alpha and/or beta activity.

11.6.17 Submit the sample for counting.

11.6.18 Calculate the activity, the total error, and the MDA per section 12.0.

11.7 Filter Samples

11.7.1 Initiate appropriate sample worksheet for the samples to be analyzed and complete as required or begin a Weight file for recording planchet weights (Appendix 1).

11.7.2 If the filter is 2" diameter or less, secure the air filter in a stainless steel planchet with double-sided cellophane tape such that no portion of filter extends above the lip of the planchet. Then proceed to step 11.5.13.

11.7.2.1 Prepare a method blank for filter samples £ 2 " by securing a blank filter into a planchet. Prepare a LCS by securing a blank filter which has been spiked with 1 ml of the solutions described in 7.3.1 and 7.3.2 in a planchet. Note: separate LCS's must be prepared for alpha and beta analysis due to the beta emitting decay products of Am-241. Dry the planchets which have been spiked with the aqueous solutions under a heat lamp or in an oven (105 ± 2 °C) before proceeding to 11.5.13.

11.7.3 If the filter is greater than 2 inches diameter, digest the sample per STL-RC-0004. Prepare a method blank and LCS from blank filters, spiked as above, which are digested in the same manner.

11.7.4 Shake the digested sample thoroughly. Measure a volume of sample into an appropriately sized beaker. Record volume of sample used.

11.7.5 Add 5 ml of 16N nitric acid.

11.7.6 Evaporate to near dryness using a medium to low temperature hot plate or heat lamp. DO NOT ALLOW LIQUID TO SPLATTER. Do not allow residue to "bake" on hot plate. Do not allow residue to "bake" on hot plate.

11.7.7 Quantitatively transfer the sample to a tared, stainless steel planchet.

11.7.8 Use a rubber policeman, if needed, to complete the transfer. Wash down the beaker wall with small portions of dilute HNO3 and add to the planchet.

11.7.9 Evaporate to dryness on a warm hot plate so that the sample does not boil. DO NOT ALLOW LIQUID TO SPLATTER. Do not allow residue to "bake" on hot plate. Remove sample from hot plate. Allow to cool.

11.7.10 Dry the planchet in an oven at 105 ± 2 °C for a minimum of two hours. This step may be eliminated if planchet is flamed as described in Section 11.5.12.

CAUTION: Ensure that the solids content do not exceed the maximum allowed weight for the determination and planchet used.

11.7.11 Weigh the cooled planchet and record final weight.

11.7.12 If moisture is present in the sample residue, heat the planchet to a dull red color for a few minutes to convert the salts to oxides.

11.7.13 Store dry sample in a desiccator until counted for gross alpha and/or beta activity.

11.7.14 Submit the sample for counting .

11.7.15 Calculate the activity, the total error, and the MDA per section 12.0.

11.8 Solid and/or Soil Samples

11.8.1 Initiate appropriate sample worksheet for the samples to be analyzed and complete as required or begin a Weight file for recording planchet weights (Appendix 1).

11.8.2 If the sample has already been prepared per STL-RC-0003, "Drying and Grinding of Soil and Solid Samples," proceed to step 11.8.7. If the sample is to be leached per DOE Method RP710, proceed to STL-RC-0004, "Preparation of

Soil, Sludge and Filter Paper Samples for Radiochemical Analysis." The digestate is then treated like a liquid (section 11.3).

11.8.3 Remove an aliquot (typically 1 - 5 gm.) with a spatula and place into a clean, labeled aluminum pan." (Aluminum weighing pans work well).

11.8.4 Place sample on a hot plate or in a drying oven at approximately 105° C and evaporate any moisture.

11.8.5 When dry, remove from hot plate or oven and allow the sample to cool.

11.8.6 Using a metal spatula, reduce the solid sample to a fine particle size.

NOTE: Sample size is restricted to 100 mg for alpha/beta analysis.

11.8.7 Self adhesive label dots of the chosen planchet size can be used advantageously to hold finely divided solid material uniformly for gross alpha and/or beta analysis. Tare the prepared planchet.

11.8.8 Distribute the sample ash evenly in a tared stainless steel planchet.

11.8.9 Weigh and record the gross sample weight.

11.8.10 Prepare a method blank from 1 ml. of the Na2SO4 solution.

11.8.10.1 Pipette directly into a tared planchet. Prepare LCS in the same manner, spiking with 1 ml. of the standards described in 7.3.1 and 7.3.2. Note: separate LCS's must be prepared for alpha and beta analysis due to the alpha emitting decay products of Sr-90. Evaporate on a hot plate. DO NOT ALLOW LIQUID TO SPLATTER. Do not allow residue to "bake" on hot plate.

OR:

11.8.10.2 Use table salt for the blank and a soil standard reference material, i.e. NIST Traceable Rocky Flats Soil, for the LCS. Prepare in the same fashion as the samples.

11.8.11 Store dry sample in a desiccator until counted for gross alpha and/or beta activity.

11.8.12 Submit the sample for counting .

11.8.13 Calculate the activity, the total error, and the MDA per section 12.0.

11.9 Reprocessing planchets which are over the weight limit.

11.9.1 Rinse residue from planchet with 4 N HNO3 into a beaker. Use a policeman, if needed, to complete the transfer.

11.9.2 Redissolve the residue into 4 N HNO3. Dilute the sample to a known volume.

11.9.3 Remove an aliquot which will keep the residue weight under the limit and transfer to the tared planchet.

11.9.4 Evaporate to dryness on a warm hot plate so that the sample does not boil. Remove sample from hot plate. Allow to cool.

11.9.5 Weigh the cooled planchet and record final weight.

12 DATA ANALYSIS AND CALCULATIONS

12.1 To calculate the aqueous sample volume required (ml), use the following equation;

$$\text{ml required} = \frac{\text{target net residue weight (mg)} * \text{initial aliquot volume (ml)}}{\text{initial aliquot net residue weight (mg)}}$$

12.2 To calculate the density (mg/cm2), use the following equation;

$$\text{mg} / \text{cm}^2 = \frac{\text{net residue weight (mg)}}{20.27\text{cm}^2 \ (2\text{" planchet})}$$

12.3 To calculate the Activity (pCi/unit mass or volume) use the following equation;

$$A_S = \frac{R_S - R_B}{2.22 * E * TF * V_A}$$

Where:

A_S	=	Activity of the sample
R_S	=	Count rate of the sample (in cpm)
R_B	=	Count rate of detector background (in cpm)
E	=	Detector efficiency
TF	=	Transmission factor
V_A	=	Sample aliquot volume or mass

12.4 To calculate the total 2s error, use the following equation;

$$U_{AC} = 1.96 * A_S * \sqrt{\frac{(R_S * t_S) + (R_B * t_S)}{\left|(R_S * t_S) - (R_B * t_S)\right|^2} + .0025}$$

Where:

A_S	=	Activity of the sample
R_S	=	Count rate of the sample (in cpm)
R_B	=	Count rate of detector background (in cpm)
t_s	=	Count time for analysis

12.5 To calculate the Minimum Detectable Activity, use the following equation;

$$MDA = \frac{4.65 * \sqrt{R_B * t_S} + 2.71}{2.22 * E * TF * V_A * t_S}$$

Where:

MDA	=	Minimum Detectable Activity of the sample
RB	=	Count rate of detector background (in cpm)
E	=	Detector efficiency
t_s	=	Count time for analysis
TF	=	Transmission factor
V_A	=	Sample aliquot volume

12.6 To calculate the Relative Percent Difference, use the following equation;

$$RPD = \frac{|A_S - A_{DUP}|}{\left(\frac{A_S + A_{DUP}}{2}\right)} * 100\%$$

Where:

As	=	Sample activity
AD_{UP}	=	Sample activity of the duplicate

12.7 To calculate the LCS recovery, use the following equation;

$$\%R = \frac{A_{LCS}}{TV_{LCS}} * 100\%$$

Where:

A_{LCS}	=	LCS Observed activity
TV_{LCS}	=	True Value of the LCS

12.8 To calculate the MS(MSD) recovery, use the following equation;

$$\%R_{MS(MSD)} = \frac{A_{MS(MSD)} - A_S}{TV_{MS}} * 100\%$$

Where:

$A_{MS(MSD)}$ = MS(MSD) observed activity
$TV_{MS(MSD)}$ = True Value of the MS(MSD)
A_s = Sample activity

13 DATA ASSESSMENT AND ACCEPTANCE CRITERIA

The following represent data assessment for samples and acceptance criteria for QC measures and corrective actions for any failure in QC measurements.

13.1 QC sample acceptance criteria

13.1.1 Method Blank

13.1.1.1 No target analyte may be present in the method blank above the reporting limit.

13.1.2 Laboratory Control Sample (LCS).

13.1.2.1 All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD). Exceptions are allowed only with QA and project management approval.

13.1.3 Matrix Spike/Matrix Spike Duplicate (MS/MSD).

13.1.3.1.1 All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD). Deviations from this may be the results of matrix effects, which are confirmed by passing LCSs.

13.1.3.2 No specific corrective actions are required in the evaluation of the MS/MSD results provided that the batch LCS is in control. Analysts should use sound judgment in accepting MS/MSD results that are not within control limits, especially if the LCS results are borderline.

14 CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

14.1 Method Blank

14.1.1 The samples in the batch associated to the defective method blank are evaluated.

14.1.1.1 If the analyte found in the method blank is confirmed to not be present in any of the associated samples at any level, the contamination did not affect those samples. This represents a non-impact situation and no specific corrective action is taken. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.1.1.2 If the analyte found in the method blank is confirmed to be present in one or more of the associated samples, the concentrations are compared.

14.1.1.2.1 If the analyte concentration in the method blank exceeded 10% of concentration found in one or more samples, the prescribed corrective action is to re-analyze all affected samples.

14.1.1.2.2 If the concentration in the method blank was less than 10% of the concentration found in one or more samples, the sample can be reported by qualifying the affected analytes. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.2 Laboratory control sample

14.2.1 If any control analyte is out of control for accuracy (% Recovery), the associated samples are evaluated.

14.2.1.1 If the recovery is biased high and the associated samples have no positive results for that analyte, a non-impact situation ensues. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.2.1.2 If the recovery is biased low and the associated samples have positive results for that analyte, a minimal impact situation ensues. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.2.1.3 If the recovery is biased high and the associated samples have positive results for that analyte, the prescribed corrective action is reanalysis of the affected sample(s).

14.2.1.4 If the recovery is biased low and the samples have no positive results for that analyte, the prescribed corrective action is reanalysis of the affected sample(s).

14.2.2 If any control analyte is out of control for precision (RPD), the associated samples are evaluated. All decisions about corrective action(s) are made in consultation with the project manager.

14.2.2.1 If there are no positive results in one or more samples, a non-impact situation ensues for those samples. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.2.2.2 If there are positive results for one or more analytes, the likelihood of poor reproducibility increases and corrective action must be evaluated. A nonconformance memo is written to notify project management of the situation for a project decision on whether the affected sample(s) should be reanalyzed.

15 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

15.1 Method blanks

15.1.1 If there is insufficient sample to perform re-analysis, the analyst must notify the project manager for consultation with the client. In this situation, all associated samples are flagged with a "C" qualifier and appropriate comments in the narrative.

15.2 LCS

15.2.1 If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report (can be accomplished with an NCM). Acceptable matrix spike recoveries are not sufficient justification to preclude re-extraction of the batch.

15.2.2 If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

15.3 Insufficient sample

15.3.1 If there is insufficient sample to repeat the analysis, the project manager is notified via NCM for consultation with the client.

16 METHOD PERFORMANCE

16.1 Training Qualification:

16.1.1 The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

16.1.2 The analyst has the responsibility that he/she has successfully completed the necessary demonstration of proficiency samples, read the SOP, and understand the basic operation and theory of the instrumentation.

17 POLLUTION PREVENTION

17.1 This procedure will be carried out in a manner consistent with all applicable federal, state and local regulations regarding pollution control and prevention. Specific controls due to the accidental release of hazardous materials can be found in the STL Chemical Hygiene Plan and facility attachments.

18 WASTE MANAGEMENT

18.1 Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedure. The Environmental Health and Safety Coordinator should be contacted if additional information is required.

19 CLARIFICATIONS, MODIFICATIONS AND ADDITIONS TO REFERENCE METHOD

19.1 Corrective actions for quality control samples/standards not meeting the criteria stated in Section 9 are as follows:

19.1.1 Correlation coefficient: stop analysis and rerun the standard curve,

19.1.2 CCVS, ICVS: stop analysis and reanalyze all samples analyzed after the last acceptable calibration verification standard,

19.1.3 Laboratory Control Sample: re-prep and reanalyze all samples associated with the unacceptable LCS,

19.1.4 Matrix Spike: matrix spike recoveries outside the suggested limit will be assumed to be due to matrix effect and will be flagged,

19.1.5 Duplicate: RPDs outside the suggested limit will be assumed to be due to, matrix effect and will be flagged,

19.1.6 Method Blank, ICB, CCB: re-prep and reanalyze all samples associated with the unacceptable method blank. For an unacceptable ICB, investigate instrument

contamination and reanalyze the samples. For an unacceptable CCB, investigate instrument contamination and reanalyze all samples analyzed after the last acceptable CCB.

19.2 Nonconformance and Corrective Action

19.2.1 Procedural variations are allowed only if deemed necessary in the professional judgement of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by the Technical Director and QA/QC coordinator. If contractually required the client will be notified. The Nonconformance Memo will be filed in the project file.

19.2.2 Any unauthorized deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance Memo shall be used for this documentation. The original Nonconformance Memo will be filed in the project file.

19.3 Records Management/Documentation

19.3.1 Record all analysis data on a sample data sheets or in computer weighing file. Include all method blanks, LCSs, duplicates, and MS/MSDs.

19.3.2 All raw data, data run logs, copies of standard logs, and quality control charts are released to the Document Control Coordinator after review and approval.

19.4 MDA Table

19.4.1 The following tables show the MDA's achievable with this procedure when varying amounts of sample and varying count times are used with this SOP.

19.4.2 Liquids

	Highest Background (cpm)	Lowest Efficiency	Transmission Factor	Minimum Volume (L)	Count Time (minutes)	Achievable MDA (pCi/L)
Gross Alpha	0.100	27%	29%	0.100	1000	2.79
Gross Alpha	0.100	27%	29%	0.200	100	4.94
Gross Alpha	0.100	27%	29%	0.200	200	3.33
Gross Alpha	0.100	27%	29%	0.200	1000	1.39
Gross Beta	1.500	40%	90%	0.100	1000	2.29
Gross Beta	1.500	40%	90%	0.200	100	3.73

Gross Beta	1.500	40%	90%	0.200	200	2.60
Gross Beta	1.500	40%	90%	0.200	1000	1.14

19.5 Solids

	Highest Background (cpm)	Lowest Efficiency	Transmission Factor	Minimum Weight (g)	Count Time (minutes)	Achievable MDA (pCi/g)
Gross Alpha – Leached	0.100	27%	20%	1.000	100	1.43
Gross Alpha	0.100	27%	20%	0.100	100	14.31
Gross Alpha	0.100	27%	20%	0.100	200	9.66
Gross Alpha	0.100	27%	20%	0.100	1000	4.05
Gross Beta - Leached	1.500	40%	78%	1.000	100	0.86
Gross Beta	1.500	40%	78%	0.100	100	8.61
Gross Beta	1.500	40%	78%	0.100	200	6.01
Gross Beta	1.500	40%	78%	0.100	1000	2.64

19.6 Associated SOPs

19.6.1 SL13019, "Calibration of the Low Background Gas Flow Proportional Counting System."

19.6.2 STL-RD-0403, "Daily Calibration Verification and Maintenance of the Low Background Gas Flow Proportional Counting System."

20 REFERENCES

20.1 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

20.2 EPA 600/4-80-032, "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," Method 900.0, August, 1980.

20.3 APHA/AWWA/WEF, "Standard Methods for Water and Wastewaters,", 18th Edition, Method 7110, 1992.

20.4 "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, Method 9310, Rev. 0, September, 1986.

20.5 DOE/EM-0089T Rev. 2, "DOE Methods for Evaluating Environmental and Waste Management Samples," Method RP710, October 1994.

20.6 STL Quality Assurance Management Plan.

20.7 STL St. Louis Quality Assurance Management Plan, Laboratory Specific Attachment.

20.8 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", L.G. Kanipe, EPA-600/7-77-088, August 1977.

20.9 Quanterra St. Louis Laboratory, STL-QA-0002, "Standards Preparation Procedure."

20.10 Quanterra St. Louis Laboratory, STL-QA-0004, "Automatic Pipetter Calibration."

20.11 Quanterra St. Louis Laboratory, STL-QA-0006, " Sample Receipt and Chain-of-Custody."

20.12 Quanterra St. Louis Laboratory, STL-QA-0013, "Personnel Training and Evaluation."

20.13 Quanterra St. Louis Laboratory, STL-RC-0002, "Preparation of Stainless Steel Planchets for Radiochemistry Analyses."

20.14 Quanterra St. Louis Laboratory, SL13021, "Operation of Low Background Gas Flow Proportional Counting System."

APPENDIX VI
STL's USACE APPROVAL LETTER

DEPARTMENT OF THE ARMY
CORPS OF ENGINEERS
HTRW CENTER OF EXPERTISE
12565 WEST CENTER ROAD
OMAHA, NEBRASKA 68144-3869

REPLY TO
ATTENTION OF:

10 May 2000

Hazardous, Toxic and Radioactive Waste
Center of Expertise

STL St. Louis
13715 Rider Trail North
Earth City, MO 63045-1205

Gentlemen:

This correspondence addresses the recent evaluation of STL St. Louis of Earth City, MO by the U.S. Army Corps of Engineers (USACE) for chemical analysis in support of the USACE Hazardous, Toxic and Radioactive Waste Program.

Your laboratory is now validated for the parameters listed below:

METHOD	PARAMETERS	MATRIX[1]
9010B/9012A	Cyanide	Water[2]
9013	Cyanide	Solids
8151A	Herbicides	Water[2]
8151A	Herbicides	Solids
8081A	Organochlorine Pesticides	Water[2]
8081A	Organochlorine Pesticides	Solids
8270C	Semivolatile Organics	Water[2]
8270C	Semivolatile Organics	Solids[2]
SW-846	TAL Metals[3]	Water[2]
SW-846	TAL Metals[3]	Solids[2]
8260B	Volatile Organics	Water[2]
8260B	Volatile Organics	Solids
STL-RC-0004	Preparation of Soil, Sludge, and Filter Paper Samples for Radiochemical Analysis	Solids[4]
STL-RC-0090	Preparation of Samples for Sequential Determination of Isotopic Americium, Neptunium, Plutonium, Thorium, and Uranium	Water[4]
STL-RC-0090	Preparation of Samples for Sequential Determination of Isotopic Americium, Neptunium, Plutonium, Thorium, and Uranium	Solids[4]

STL-RC-0100	Actinide Coprecipitation	Water[4]
SL13013	Operation and Calibration of the Alpha Spectrometer	N/A
SL13030	Preparation of Samples for Gamma Spectroscopy	Water[4]
SL13030	Preparation of Samples for Gamma Spectroscopy	Solids[4]
STL-RD-0101	Daily Operations of a Germanium Spectroscopy System	N/A
STL-RD-0103	Calibration and Maintenance of a Germanium Spectroscopy System	N/A
STL-RC-0020	Determination of Gross Alpha/Beta Activity	Water[4]
STL-RC-0020	Determination of Gross Alpha/Beta Activity	Solids[4]
STL-RC-0110	Analysis of Total Uranium by Laser-induced Phosphorimetry	Water[4]
STL-RC-0110	Analysis of Total Uranium by Laser-induced Phosphorimetry	Solids[4]
STL-RD-0201	Daily Operations of an Alpha Spectroscopy System	N/A

Remarks:
1) 'Solids' includes soils, sediments, and solid waste.
2) The laboratory has successfully analyzed a performance evaluation sample for this method/matrix.
3) TAL Metals: Aluminum, antimony, arsenic, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, mercury, nickel, potassium, selenium, silver, sodium, thallium, vanadium, and zinc.
4) Approval for this parameter is based on review of SOPs only.

Enclosed for your information is a copy of the Laboratory Inspection and Evaluation Report. Your laboratory has responded to the deficiencies as noted in the report. No further responses are necessary.

Based on the successful analysis of the performance evaluation samples and the results of the laboratory inspection, your laboratory will be validated for sample analysis by the methods listed above. The period of validation is 24 months and expires on January 14, 2002.

A request for validation of parameters currently in a "FAIL" status may be pursued in six months, only after improvements have been made to correct general performance. Validation for these additional parameters must originate from a USACE Contracting Officer Representative.

The USACE reserves the right to conduct additional laboratory inspections or to suspend validation status for any or all of the listed parameters if deemed necessary. It should be noted that your laboratory may not subcontract USACE analytical work to any other laboratory location without the approval of this office. This laboratory validation does not guarantee the delivery of any analytical samples from a USACE Contracting Officer Representative.

Any questions or comments can be directed to Dr. Jan W. Dunker at (402) 697-2566. General questions regarding laboratory validation may be directed to the Laboratory Validation Coordinator at (402) 697-2574.

Sincerely,

Marcia C. Davies

Marcia C. Davies, Ph.D.
Director, USACE Hazardous,
Toxic and Radioactive Waste
Center of Expertise

APPENDIX VII
STL's CAPACITY LETTER

SEVERN
TRENT
SERVICES

STL St. Louis

September 25, 2001

Skip Mann
Jacobs Engineering
13723 Riverport Drive
Maryland Heights, MO 63043

RE: Gross Alpha capacity- One sample per day for three weeks

Dear Mr. Mann:

Severn Trent Laboratories St. Louis has capacity to provide analysis of up to (20) gross alpha samples per day. STL-St. Louis will maintain sufficient capacity to complete the above referenced project scheduled for April 2002. Please contact Ron Martino, STL-St. Louis, one week prior to sample arrival to confirm sample numbers and required turnaround.

Should you have further questions please do not hesitate to call.

Sincerely,

Richard H. Mannz
Account Executive

CC: Ron Martino

APPENDIX VIII
SAMPLE LABORATORY REPORTS

EMSL Analytical, Inc.
107 Haddon Ave., Westmont, NJ 08108
Phone: (856) 858-4800 Fax: (856) 858-4960 Email: ssiegel@EMSL.com

Attn:

Fax:

Project:

Customer ID:
Customer PO:
Received:

EMSL Order:
EMSL Project ID:

Analysis Date:

Asbestos Fiber Analysis by Transmission Electron Microscopy (TEM) Performed by EPA 40 CFR Part 763 Final Rule (AHERA)

Sample	Volume	Area Analyzed	Non Ash	Asbestos Type(s)	# Structure 0.5μ -5	# Structure 5μ	Analytical Sensitivity (S/cc)	Asbestos Concentration (S/mm²)	Asbestos Concentration (S/cc)	Notes
										PRELIMINARY RESULT
09-26-MS-11 040115247-0011										

Analyst

Stephen Siegel, CIH
or other approved signatory

EMSL Analytical, Inc.
107 Haddon Ave., Westmont, NJ 08108
Phone: (856) 858-4800 Fax: (856) 858-4960 Email: ssiegel@EMSL.com

Attn:

Customer ID:
Customer PO:
Received:

Fax:
Project:

EMSL Order:
EMSL Project ID:
Analysis Date:

Asbestos Analysis of Bulk Materials via EPA 600/R-93/116 Method using Polarized Light Microscopy

Sample	Location	Appearance	Treatment	Non-Asbestos % Fibrous	Non-Asbestos % Non-Fibrous	Asbestos % Type
03 040115249-0003						

PRELIMINARY RESULT

Analyst(s)

Stephen Siegel, CIH
or other approved signatory

PLM has been known to miss asbestos in a small percentage of samples which contain asbestos. Negative PLM results cannot be guaranteed. Samples reported as <1% or none detected should be tested with TEM. The above test report relates only to the items tested. This report may not be reproduced, except in full, without written approval by EMSL Analytical, Inc. The above test must not be used by the client to claim product endorsement by NVLAP nor any agency of the United States Government.
Analysis performed by EMSL Westmont (NVLAP #101049-0), NY ELAP 10872

EMSL Analytical, Inc.
107 Haddon Ave., Westmont, NJ 08108
Phone: (856) 858-4800 Fax: (856) 858-4960 Email: ssiegel@EMSL.com

Attn:

Customer ID:
Customer PO:
Received:

Fax:
Project: ,05

EMSL Order:
EMSL Project ID:
Analysis Date:

Fiber Analysis of Air Samples via NIOSH 7400, Revision 3, Issue 2, 8/15/94

Sample	Location	Sample Date	Volume	Fibers	Fields	LOD (fib/cc)	Fibers/ mm²	Fibers/ cc	Notes
									PRELIMINARY RESULT
9/27/01-01									
040115242-0001									

Analyst

Stephen Siegel, CIH
or other approved signatory

Limit of detection is 7 fibers/mm². The laboratory is not responsible for data reported in fibers/cc, which is dependent on volume collected by non-laboratory personnel. This report relates only to the samples reported above. This report may not be reproduced, except in full, without written approval by EMSL.
Analysis performed by EMSL Westmont (NY State ELAP #10872)

PCM-1

[redacted]

Client Sample ID: [redacted]

Severn Trent Laboratories - Radiochemistry

Lab Sample ID: [redacted]
Work Order: ELE32
Matrix: WATER

Date Collected: 09/27/01 1543
Date Received: 10/02/01 0835

Parameter	Result	Qual	Total Uncert. (2 σ+/-		MDC	Prep Date	Analysis Date	Batch #	Yld %
GROSS A/B BY GFPC EPA 900.0 MOD				pCi/L		900.0 MOD			
Gross Alpha	93		21		13	10/03/01	10/06/01	1276189	
Gross Beta	69		12		13	10/03/01	10/06/01	1276189	
Gamma Cs-137 & Hits by EPA 901.1 MOD				pCi/L		901.1 MOD			
Cesium 137	0.2	U	6.7		13	10/04/01	10/04/01	1277314	
Cobalt 60	0.004	U ?	9.1		18	10/04/01	10/04/01	1277314	
Europium 152	12	U	42		92	10/04/01	10/04/01	1277314	
Europium 154	-62	U	60		89	10/04/01	10/04/01	1277314	
Europium 155	4	U	22		40	10/04/01	10/04/01	1277314	
TRITIUM (Distill) by EPA 906.0 MOD				pCi/L		906.0 MOD			
Tritium	70	U	150		250	10/04/01	10/05/01	1277389	
SR-90 BY GFPC EPA-905 MOD				pCi/L		905 MOD			
Strontium 90	0.0	J	0.0		0.0	10/09/01		1282417	0.0

NOTE(S)

Data are incomplete without the case narrative.

MDC is determined by instrument performance only.
Bold results are greater than the MDC
? For informational purposes only. The result does not follow significant figures SOP
J Result is greater than sample detection limit but less than stated reporting limit.
U Result is less than the sample detection limit.

APPENDIX IX
EMSL'S CERTIFICATIONS

STATE OF NEW YORK
DEPARTMENT OF HEALTH

Wadsworth Center The Governor Nelson A. Rockefeller Empire State Plaza P.O. Box 509 Albany, New York 12201-0509

Antonia C. Novello, M.D., M.P.H., Dr. P.H.
Commissioner

Dennis P. Whalen
Executive Deputy Commissioner

Dear Laboratory Director:

Enclosed are the ELAP Certificate(s) of Approval for permit year 2001-2002 issued to your environmental laboratory. The Certificate(s) supersede any previously issued and are in effect through March 31, 2002. Please carefully examine the Certificate(s) to insure that the categories, subcategories, analytes and methods for which your laboratory is approved are listed correctly, as well as verifying your laboratory's name, address, director and identification number.

Please notify this office of any corrections required.

Sincerely,

Linda L. Madlin

Linda L. Madlin
Administrative Assistant
Environmental Laboratory
Approval Program

LLM:mes
Enclosure

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
Antonia C. Novello, M.D., M.P.H., Dr.P.H. Commissioner

Expires 12:01 AM April 01, 2002
Issued August 23, 2001

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

DR. PETER FRASCA
EMSL ANALYTICAL
107 HADDON AVE
WESTMONT NJ 08108 USA

NY Lab Id No: 10872
EPA Lab Code: NJ00337

is hereby APPROVED as an Environmental Laboratory for the category
ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved subcategories and/or analytes are listed below:

Miscellaneous

Asbestos in Friable Material	Method Not Specified
Asbestos in Non-Friable Materi	Method Not Specified

Serial No.: 13430

Property of the New York State Department of Health. Valid only at the address shown. Must be conspicuously posted. Valid certificates have a raised seal and may be verified by calling (518)485-5570.

DOH-3317 (3/97)

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
Antonia C. Novello, M.D., M.P.H., Dr.P.H. Commissioner

Expires 12:01 AM April 01, 2002
Issued August 23, 2001

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

DR. PETER FRASCA
EMSL ANALYTICAL
107 HADDON AVE
WESTMONT NJ 08108 USA

NY Lab Id No: 10872
EPA Lab Code: NJ00337

is hereby APPROVED as an Environmental Laboratory for the category
ENVIRONMENTAL ANALYSES AIR AND EMISSIONS
All approved subcategories and/or analytes are listed below:

Miscellaneous Air

Asbestos	40 CFR APX A No. III
	YAMATE,AGARWAL GIBB
Fibers	40 CFR 763.121 APX B
	Method Not Specified
	NIOSH 7400 A RULES

Serial No.: 13431

Property of the New York State Department of Health. Valid only at the address shown. Must be conspicuously posted. Valid certificates have a raised seal and may be verified by calling (518)485-5570.

DOH-3317 (3/97)

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
Antonia C. Novello, M.D., M.P.H., Dr.P.H. Commissioner

Expires 12:01 AM April 01, 2002
Issued August 23, 2001

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

DR. PETER FRASCA
EMSL ANALYTICAL
107 HADDON AVE
WESTMONT NJ 08108 USA

NY Lab Id No: 10872
EPA Lab Code: NJ00337

is hereby APPROVED as an Environmental Laboratory for the category
ENVIRONMENTAL ANALYSES POTABLE WATER
All approved subcategories and/or analytes are listed below:

Drinking Water Miscellaneous

Asbestos	EPA 100.1

Serial No.: 13429

Property of the New York State Department of Health. Valid only at the address shown. Must be conspicuously posted. Valid certificates have a raised seal and may be verified by calling (518)485-5570.

DOH-3317 (3/97)

The American Industrial Hygiene Association

is proud to acknowledge that

EMSL Analytical, Inc.

Westmont, NJ

has fulfilled the requirements for and has been formally recognized by AIHA
and is technically competent to perform the analyses listed in the following

SCOPE OF ACCREDITATION

INDUSTRIAL HYGIENE Originally Accredited: 01/01/98		ENVIRONMENTAL LEAD		ENVIRONMENTAL MICROBIOLOGY
___Metals	_X_Silica	___Paint Chips	___Air	___Bacteria
_X_Asbestos PCM	_X_Asbestos PLM	___Dust Wipes	___Soil	___Fungi
___Organic Solvents	___Diffusive Samples			

The above named laboratory agrees to perform all analyses listed above in the scope of accreditation according to applicable policy requirements and acknowledges that continued accreditation is dependent on successful participation in the appropriate proficiency testing programs. This laboratory may be contacted to verify the current scope of accreditation, proficiency testing performance and accreditation status. Accreditation by AIHA is not a guarantee of the validity of the data generated by the laboratory.

Laboratory # 100192

Certificate #

Accreditation Expires: 02/01/04

Dave Sandusky, CIH
Chair, Analytical Accreditation Board

Steven P. Levine, Ph.D., CIH
President, AIHA

United States Department of Commerce
National Institute of Standards and Technology

NVLAP®

ISO/IEC GUIDE 25:1990
ISO 9002:1987

Certificate of Accreditation

EMSL ANALYTICAL, INC.
WESTMONT, NJ

is recognized under the National Voluntary Laboratory Accreditation Program for satisfactory compliance with criteria established in Title 15, Part 285 Code of Federal Regulations. These criteria encompass the requirements of ISO/IEC Guide 25 and the relevant requirements of ISO 9002 (ANSI/ASQC Q92-1987) as suppliers of calibration or test results. Accreditation is awarded for specific services, listed on the Scope of Accreditation for:

AIRBORNE ASBESTOS FIBER ANALYSIS

June 30, 2002
Effective through

David F. Alderman
For the National Institute of Standards and Technology

NVLAP Lab Code: 101048-0

NVLAP-01C (11-95)

United States Department of Commerce
National Institute of Standards and Technology

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ISO/IEC GUIDE 25:1990
ISO 9002:1987

Certificate of Accreditation

DEPARTMENT OF COMMERCE ★ UNITED STATES OF AMERICA ★

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is recognized under the National Voluntary Laboratory Accreditation Program for satisfactory compliance with criteria established in Title 15, Part 285 Code of Federal Regulations. These criteria encompass the requirements of ISO/IEC Guide 25 and the relevant requirements of ISO 9002 (ANSI/ASQC Q92-1987) as suppliers of calibration or test results. Accreditation is awarded for specific services, listed on the Scope of Accreditation for:

BULK ASBESTOS FIBER ANALYSIS

June 30, 2002
Effective through

David F. Alderman
For the National Institute of Standards and Technology

NVLAP Lab Code: 101048-0

NVLAP-01C (11-95)