

**Waste Management Division
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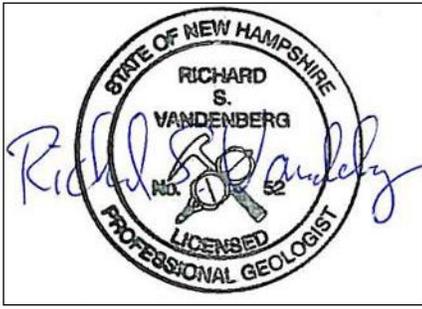
Type of Submittal (Check One-Most Applicable)

<input type="checkbox"/> Work Scope <input type="checkbox"/> Reimbursement Request	<input type="checkbox"/> Remedial Action <ul style="list-style-type: none"> • Remedial Action Plan • Bid Plans and Specifications • Remedial Action Implementation Report
<input type="checkbox"/> UST Facility Report <input type="checkbox"/> AST Facility Report	<input type="checkbox"/> Treatment System and POE O&M <input type="checkbox"/> Activity and Use Restriction
<input type="checkbox"/> Emergency/Initial Response Action <input type="checkbox"/> Groundwater Quality Assessment	<input type="checkbox"/> Temporary Surface Water Discharge Permit
<input type="checkbox"/> Initial Site Characterization <input type="checkbox"/> Site Investigation <ul style="list-style-type: none"> • Site Investigation Report • Supplemental Site Investigation Report • GMZ Delineation • Source Area Investigation • Data Submittal • Annual Summary Report <input checked="" type="checkbox"/> Unsolicited Brownfields SSQAPP Submittal <input type="checkbox"/> Closure Documentation	<input type="checkbox"/> Groundwater Management Permit <ul style="list-style-type: none"> • Permit Application • Renewal Application • Deed Recordation Documentation • Abutter Notification Documentation • Release of Recordation <input type="checkbox"/> Data Submittal <input type="checkbox"/> Annual Summary Report

DRAFT SITE-SPECIFIC QAPP ADDENDUM
 Ernie's Auto Sales Property
 180 East Main Street
 Tilton, New Hampshire
 NHDES # 199311019

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December 7, 2012

Recommended Risk Category (check one)		
<input type="checkbox"/> 1. Immediate Human Health Risk (Impacted water supply well, etc.)	<input type="checkbox"/> 4. Surface Water Impact	<input type="checkbox"/> 7. Alternate Water Available/Low Level Groundwater Contamination (<1,000 X AGQS)
<input type="checkbox"/> 2. Potential Human Health Risk (Water supply well within 1,000' or Site within SWPA)	<input type="checkbox"/> 5. No Alternate Water Available/No Existing Wells in Area	<input type="checkbox"/> 8. No AGQS Violation/No Source Remaining
<input type="checkbox"/> 3. Free Product or Source Hazard	<input type="checkbox"/> 6. Alternate Water Available/High Level Groundwater Contamination (>1,000 X AGQS)	<input type="checkbox"/> Closure Recommended

1. TITLE AND APPROVAL PAGE

SITE-SPECIFIC QUALITY ASSURANCE PROJECT PLAN (SSQAPP) ADDENDUM TO GENERIC QAPP RFA #08166 AND #09036

PROPERTY:

Ernie's Auto Sales
180 East Main Street, Tilton, New Hampshire
EPA Brownfields Cleanup Grant # BF-96162501
NHDES # 199311019

PREPARED BY:

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DRAFT December 7, 2012

Below is a listing of the names, titles, signatures, and signature dates of officials approving this SSQAPP Addendum:

Ms. Jerry Minor-Gordon EPA Brownfields Project Officer	Date
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EPA Quality Assurance Officer	Date
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Joyce Fulweiler, Town of Tilton Administrator Brownfields Grantee	Date
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Ms. Jennifer Marts, P.G. New Hampshire DES Project Manager	Date
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Mr. Vincent R. Perelli New Hampshire DES QA Manager	Date
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 Mr. Richard S. Vandenberg, CG, PG Credere Associates, LLC Project Manager & QA Manager	Date
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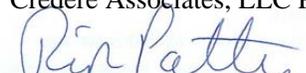
 Mr. Robert I Patten, PE, LEED-AP, LSP Credere Associates, LLC Program Manager	Date
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TABLE OF CONTENTS

1. TITLE AND APPROVAL PAGE	1
2. INTRODUCTION.....	4
3. PROJECT BACKGROUND INFORMATION.....	5
3.1 Ownership & Site Description	5
3.2 Summary of Phase I ESA Work	6
3.3 Summary of Phase II ESA and Supplemental Phase II ESA Work	7
4. REDEVELOPMENT SCENARIO.....	10
5. UPDATED CONCEPTUAL SITE MODEL	10
5.1 Physical Setting.....	10
5.2 Contaminants of Concern	11
5.3 Definitions of Exposure Pathways and Potential Receptors	11
5.4 Conceptual Site Model Summary	13
6. SAMPLING DESIGN.....	14
6.1 Asbestos Sampling.....	14
6.2 Waste Characterization Sampling	15
7. FIELD ACTIVITY METHODOLOGY	16
7.1 Asbestos Sampling.....	16
7.2 Waste Characterization Sampling	16
8. REGULATORY STANDARDS	18
8.1 Asbestos	18
8.2 Waste Characterization	18
9. PROPOSED PROJECT SCHEDULE	19

FIGURES

Figure 1	Site Location Plan
Figure 2	Proposed Sampling Plan
Figure 3	Project Organization Chart
Figure 4	Conceptual Site Model

TABLES

Table 1.....	Sample Reference Table
Table 2.....	Standard Operating Procedure Reference Table



APPENDICES

- Appendix A** Analytical Sensitivity and Project Criteria Tables
Appendix B EMSL Analytical, Inc. Asbestos Analytical SOP
Appendix C ASTM E1908-10: Standard Guide for Sample Selection of Debris Waste from a Building Renovation or Lead Abatement Project for Toxicity Characteristic Leaching Procedure (TCLP) Testing for Leachable Lead (Pb)



2. INTRODUCTION

The Town of Tilton, New Hampshire received a United States Environmental Protection Agency (EPA) Brownfield Cleanup Grant for the former Ernie's Auto Sales property located at 180 East Main Street in Tilton (the Site). The 0.8-acre Site is a former gasoline station, auto repair garage, and used car dealership. Credere Associates, LLC (Credere) is submitting this Site-Specific Quality Assurance Project Plan (SSQAPP) Addendum to execute the initial phase of work, which is scheduled to occur this upcoming winter. The initial phase of work involves razing the severely dilapidated buildings, which include a garage and a cottage. The Town received permission from the New Hampshire Department of Environmental Services (NHDES) and EPA to conduct the demolition work as a presumptive remedial measure. The request was made because there is significant concern that the garage building will collapse under the weight of winter snow, representing a safety concern to the public.

In order to demolish the Site buildings, known asbestos located inside and in the roofing materials of the garage, and in floor tiles located within the cottage, must be abated. In addition, there are containerized universal/hazardous wastes within both of the buildings and on the exterior of the property that must be removed and disposed.

This SSQAPP addendum includes provisions to allow Credere to implement building demolition at the Site and to address NC-1, NC-2, NC-3, and NC-4, including the following: sampling any previously unidentified asbestos materials if such materials are encountered during the demolition work; collecting air clearance samples after the asbestos remediation is complete; collecting samples of the building demolition debris if necessary for disposal facility approval; and collecting samples of the universal/hazardous containerized wastes if necessary for disposal facility approval.

Please note that REC-3 (The former use of the Site as an auto repair facility) will be addressed as part of subsequent work, and soil remediation is not addressed in this SSQAPP.

A significant amount of background information exists on the Site as documented in a November 2, 2010, Phase I Environmental Site Assessment (ESA), a June 2, 2011, Phase II ESA, and an October 3, 2012 Supplemental Phase II ESA. These reports were prepared by Credere and previously submitted to the NHDES, the EPA, and the Town of Tilton.

This SSQAPP presents the following information:

1. A summary of background information for the Site
2. The redevelopment plans for the Site
3. A Conceptual Site Model
4. The proposed sampling technique and rationale



5. Site-specific sampling methodology including proposed locations and analytical methods
6. Regulatory standards applicable to the Site
7. A proposed project schedule

This SSQAPP was prepared to be used in concert with Credere Associates, LLC's (Credere's) Generic Quality Assurance Project Plan (QAPP) EPA RFA#08166 and #09036 that was prepared for all of Credere's EPA work in New Hampshire. The quality assurance and quality control (QA/QC) procedures outlined in Credere's Generic QAPP will be followed for this investigation program including sample collection, handling, and analysis, chain-of-custody, data management and documentation, data validation, and data usability assessments.

Figure 1 shows the general location of the Site in Tilton, New Hampshire, and **Figure 2** presents the proposed sample locations and pertinent Site features. **Figure 3** is Credere's organization chart for the project team and **Figure 4** is a diagram depicting the updated conceptual model for the Site.

3. PROJECT BACKGROUND INFORMATION

The following is a summary of information obtained during previous environmental due diligence work conducted at the Site. Credere has completed a Phase I ESA, a Phase II ESA, and a Supplemental Phase II ESA under EPA Brownfield assessment grant # BF-96111801 received by the Lakes Region Planning Commission (LRPC). The following includes pertinent details from Credere's ESAs for the Site:

3.1 OWNERSHIP & SITE DESCRIPTION

The Site is owned by the Town of Tilton, New Hampshire. The Town acquired the Site on January 31, 2011, following the completion of Phase I and Phase II ESAs.

The Site is composed of one 0.8-acre parcel of land located at 180 East Main Street in Tilton, New Hampshire and is situated adjacent to the Winnepesaukee River. The Site is currently improved with a garage building and a cottage that are both unoccupied and in a state of disrepair. Both buildings were constructed prior to 1951, but the exact dates of construction are not known.

The Site was formerly operated as a gas station from approximately 1939 until the 1970s. An automobile body shop, used automobile repair shop, automobile salvage yard, used automotive sales, and a U-Haul truck rental business have also reportedly occupied the Site.



3.2 SUMMARY OF PHASE I ESA WORK

A Phase I ESA dated November 2, 2010, was completed by Credere for the Site in accordance with ASTM Standard Practice E 1527-05. The following represents the findings and recommendations from this report.

Based on the information obtained as a part of the Phase I ESA, the following recognized environmental conditions (RECs) were identified at the Site:

- REC-1 – The former use of the Site as a gas station between 1939 and the 1970s, past distribution, and past and present bulk storage of petroleum products (including a 275-gallon aboveground storage tank (AST) and a 55-gallon drum) may have resulted in releases of petroleum which may have impacted the environmental conditions of the Site.
- REC-2 – A release of petroleum was discovered on September 16, 1993, during the closure of two (2) 3,000-gallon and one (1) 4,000-gallon gasoline underground storage tanks (USTs) and one (1) 2,000-gallon waste oil UST. Though this release is considered by the New Hampshire Department of Environmental Services (NHDES) to be closed, the release represents a REC as impacted soil and/or groundwater may remain at the Site.
- REC-3 – The former use of the Site as an auto repair facility between the approximate dates of 1939 and 1978 represents a REC because hazardous materials and petroleum products were likely stored, used, and may have been disposed of on the Site and may have impacted the environmental conditions of the Site.
- REC-4 – The floor drain observed within the garage bay with an unknown discharge point represents a REC because the drain is a potential conduit to the environment whereby releases of petroleum products and hazardous substances from former activities may have impacted the environmental conditions at the Site.
- REC-5 – A suspected dump and fill area was observed along the southern portion of the Site including items such as, but not limited to, urban fill, automobile parts, and utility pole sections. Petroleum products and/or hazardous substances associated with these materials may have been released and impacted the environmental conditions at the Site.
- REC-6 – Stressed vegetation was observed below a pole mounted electrical transformer located along the northern Site boundary. This condition represents a REC because it could be indicative of a release of petroleum-based and/or polychlorinated biphenyl (PCB)-containing dielectric fluid that may have impacted the environmental conditions of the Site.

Additionally, Credere identified three (3) *de minimis environmental conditions* (DMEC) at the Site.

- DMEC-1 – Oil staining observed on the floor of the cottage represents a DMEC because it is evidence of a release; however, a pathway to the environment was not likely.



- DMEC-2 – Multiple small volume containers (less than 50-gallons each) of oil, gasoline, and automotive lubricants and cleaning materials represent a DMEC because of the poor conditions in which they were stored; however, a pathway to the environment was not likely.
- DMEC-3 – Multiple stains observed on the gravel parking lot represent a DMEC because they are evidence of small petroleum releases which may have impacted surficial soil at the Site. However, based on observed conditions, it was not likely that these small spills have significantly impacted environmental media at the Site.

The following four (4) ASTM *Non-Scope considerations* (NCs) were also noted during the Phase I ESA:

- NC-1 – Based on the age of the Site buildings, potential asbestos-containing materials (ACMs) may be present on the interior and exterior of the buildings.
- NC-2 – Based on the age of the Site buildings, lead-based paint may be present on the interior and exterior of the buildings.
- NC-3 – Based on the age of the Site buildings, PCB-containing bulk products may be present on the interior and exterior of the buildings.
- NC-4 – Based on the condition of the Site buildings and the collapsed roof of the garage, mold is likely present in the buildings.

3.3 SUMMARY OF PHASE II ESA AND SUPPLEMENTAL PHASE II ESA WORK

Phase II ESA

A June 2, 2011, Phase II ESA was completed by Credere for the Site. Phase II ESA activities included performing a ground penetrating radar (GPR) survey to locate potential subsurface structures, conducting lead-based paint and asbestos surveys of the two Site buildings, collecting samples of select building materials as a part of a PCB survey, and collecting surficial soil, subsurface soil, and groundwater samples to assess potential impacts from historical uses of the Site.

From the data collected during the Phase II ESA, REC-1, REC-2, REC-4, REC-5, and REC-6 were dismissed. The rationale for dismissing these RECs is detailed in the June 2, 2011 Phase II ESA report. NHDES concurred with these conclusions.

REC-3, which is associated with the former use of the Site as an auto repair facility, could not be conclusively confirmed or dismissed from the data collected. Arsenic and lead were detected in subsurface soil near the building in one sample (SB-5 4-6') at concentrations that exceeded applicable NHDES Soil Remediation Standards (SRS) and arsenic was detected in groundwater in two of the five wells sampled at a level that exceeded the applicable Ambient Groundwater Quality Standard (AGQS). However, based on the data collected, it was not clear if these contaminants were related to prior activities at the Site or associated with background conditions.



In particular, the arsenic in groundwater may have desorbed from the natural mineralogy caused by reducing conditions present in the groundwater associated with an old plume of petroleum contamination.

PAHs were detected in several surficial and one subsurface sample during the Phase II ESA. However, the detected PAHs were attributed to the presence of asphalt, coal, and ash materials that were identified in collected samples. The presence of PAHs in association with asphalt, coal, and ash materials meets the NHDES definition of "background" as defined by Env-Or 602.03. It was Credere's interpretation that PAHs identified onsite are not subject to the NHDES SRS [per Env-Or 606.19(f)], but they still represent a health risk that should be appropriately managed.

The following is a summary of Phase II ESA findings pertaining to the non-scope considerations being addressed as part of this SSQAPP:

NC-1, which is associated with potential presence of ACMs, was confirmed because ACMs were identified in both Site buildings.

- Asbestos (NC-1) was identified in the both garage building and cottage in the following locations:
 - Garage- Reception area floor tile
 - Garage- Reception area floor tile mastic
 - Garage-Flat Roof top layer of shingles
 - Garage-Flat Roof top layer shingle mastic
 - Cottage-Orange floor tile
- Lead paint (NC-2) was identified covering numerous surfaces in and on both the cottage and garage buildings.
- Detectible concentrations of PCBs (NC-3) were identified in the four bulk product samples collected from the Site buildings. Concentrations of PCBs that exceeded 1 part per million (ppm) were detected in sample BM-2 (pink paint from the interior of the garage) and BM-4 (blue exterior paint from rear door of cottage). PCBs concentrations in BM-2 and BM-4 were 2.2 ppm and 4.9 ppm, respectively. PCB concentrations that exceeded or approached 50 ppm were not detected during the Phase II ESA, and based on Credere's assessment of the sampled materials; no PCB Bulk Product Waste, as defined in 40 CFR 761.3, was identified at the Site. As such, NC-3 was dismissed, though once the buildings are demolished and the building materials are removed from use, the associated demolition debris waste stream warrants proper disposal consistent with the at-found concentrations of PCBs.
- The presence of Mold (NC-4) was not assessed because it was determined that the buildings needed to be demolished.



Supplemental Phase II ESA

A supplemental Phase II ESA was completed on October 3, 2012. The Supplemental Phase II ESA was performed to delineate the extent of the previously identified arsenic and lead concentrations in soil at sample location SB-5 and to perform a second round of groundwater sampling to verify the presence of dissolved arsenic concentrations in groundwater. Additional soil borings were drilled, soil was collected for field screening and laboratory analysis, and the existing monitoring wells were sampled and the collected samples were submitted for laboratory analysis.

Seven soil borings were installed during the Supplemental Phase II ESA work. Laboratory analysis of soil samples collected demonstrated that the extent of lead and arsenic contamination exceeding the SRS was limited to the area around previously drilled boring SB-5. However, x-ray fluorescence (XRF) field screening results of samples collected that were not laboratory analyzed revealed that some additional arsenic exceeding SRS may be present in the vicinity of soil borings (SB-12 and SB-14).

Dissolved arsenic was detected in three of the five existing wells during the Supplemental Phase II ESA. However, it was still inconclusive whether the detected arsenic in groundwater was the result of a release of hazardous substances related to Site activities, the result of changes in the geochemistry of groundwater related to past releases of petroleum, or were related to a background condition. The presence of arsenic in groundwater was determined to still represent a potential health risk to future Site workers and users.

Existing Environmental Conditions

Based on the findings of the Phase I ESA, Phase II ESA, and Supplemental Phase II ESA, the following conditions exist at the Site requiring cleanup and/or additional waste characterization:

- Arsenic and lead is present in soil in the area of SB-5 at depths of 4 to 6 feet below grade surface (bgs) at concentrations that exceed the NHDES SRS;
- Arsenic concentrations are present in groundwater at levels that exceed the NHDES AGQS;
- PAH concentrations that exceed the NHDES SRS are present in soil in the area of five surficial soil samples (SS-2, SS-4, SS-5, SS-7, and TP-1) and one subsurface soil sample (SB-5). The detected PAHs were determined to be associated with a background condition but still represent a health risk;
- Asbestos is present in both the garage building and cottage;
- Lead paint was identified covering numerous surfaces in and on both the cottage and garage buildings;
- Building materials sampled from within or on both buildings do not meet the definition of PCB Bulk Product waste as defined in 40 CFR 761.3 and are not regulated for disposal. However, once removed from use through building demolition, the waste stream associated



with these materials requires disposal at a facility that can accept demolition debris with PCBs consistent with at-found concentrations; and

- Universal/hazardous containerized wastes are located within the Site buildings and on the exterior portions of the property.

Please note that only asbestos lead-based paint, PCB-containing building materials, and Universal/hazardous materials in the building are being addressed as part of this SSQAPP.

4. REDEVELOPMENT SCENARIO

The Tilton Conservation Commission, in conjunction with the Winnepesaukee River Trails Association, will develop the Site into a trailhead and parking lot for the existing riverfront trail system.

5. UPDATED CONCEPTUAL SITE MODEL

The updated conceptual site model (CSM) describes the physical setting of the Site, the environmental conditions being addressed as part of this phase of the work, the potential contaminants of concern (COCs) associated with each environmental condition, migration pathways, impacted media, exposure pathways, and potential human and environmental receptors. The Town of Tilton successfully petitioned the NHDES and EPA to allow the demolition of the building as a presumptive remedial remedy. This allows the building to be demolished this winter and reduces the chances that the building will collapse due to snow load. In order to demolish the building the following environmental conditions must be addressed:

- Asbestos in both the garage building and cottage;
- Lead paint covering numerous surfaces in and on both the cottage and garage buildings; and
- PCBs that may be present in the demolition debris waste stream resulting from the razing of both buildings, which warrants proper disposal based on at-found concentrations.

The remainder of the SSQAPP only addresses the asbestos, lead based paint, and PCBs associated with the buildings. An Analysis of Brownfields Cleanup Alternatives will subsequently be prepared to identify the most appropriate remedial alternatives for the remaining environmental issues at the Site, including contaminated soil and groundwater.

5.1 PHYSICAL SETTING

Topography at the Site generally slopes in two directions. The north side of the Site slopes gently to the northwest, while the south side tends to slope radially to the south and west. Stormwater on the north side of the Site likely follows surficial topography resulting in a northwesterly flow which terminates at a catch basin located in the northwestern corner of the Site. This catch basin reportedly discharges via a culvert into a drainage ditch. The ultimate outfall of this drainage ditch is the Winnepesaukee River. Stormwater on the south side of the



Site generally follows the topography radially to the south and west and flows directly into the Winnepesaukee River.

According to the *Geohydrology and Groundwater Quality Data of Stratified-Drift Aquifers in the Winnepesaukee River Basin, Central New Hampshire*, United States Geological Survey (USGS), Water-Resources Investigations Report 94-4150, by Joseph D. Ayotte (1997), the surficial geology at the Site consists of glacial till over bedrock. Surficial materials observed at the Site during soil sampling activities revealed predominantly loose to dense sand with some gravel at deeper depths.

According to the *Generalized Bedrock Geologic Map of New Hampshire* compiled by the USGS, the Site is underlain primarily by metamorphic rocks of the Silurian age, consisting of aluminous schist, quartzite, calc-silicate granofels, and bimodal metavolcanic rocks. According to the USGS, the average depth to bedrock is 35-feet bgs, but can be up to 200-feet bgs in localized areas. Bedrock was not encountered during soil boring activities.

Groundwater in overburden materials at the Site was observed at depths ranging from 7.52 to 10.65 feet bgs during the July 26, 2011 sampling event. Based on groundwater elevations observed during the Phase II and Supplemental Phase II ESAs, groundwater at the Site generally flows to the southwest at a gradient of approximately 3%.

5.2 CONTAMINANTS OF CONCERN

The contaminants of concern (COCs) at the Site include the following:

- Asbestos;
- Lead-based paint; and
- PCBs in the demolition debris waste stream.

There are universal/hazardous containerized wastes (DMEC-2) present in the buildings that, for the purpose of this SSQAPP, are considered COCs. These wastes will need to be consolidated and properly disposed, which may require sampling the waste stream.

5.3 DEFINITIONS OF EXPOSURE PATHWAYS AND POTENTIAL RECEPTORS

To aid in a thorough understanding of the environmental concerns present at the Site, a graphical presentation of the identified COCs and potential migration pathways to receptors is included as **Figure 4**. Exposure Pathways and Potential Receptors depicted on the CSM figure are defined below.

In general, exposure pathways describe how a human or environmental receptor comes into contact with contaminants that may be present at the Site. Exposure pathways presented in the updated CSM include the following:



- **Inhalation:** This pathway is primarily associated with groundwater contamination within 30 feet of an occupied structure when groundwater elevation is less than 15 feet below surface grade, or when depth to groundwater is unknown. This pathway is applicable when receptors may inhale impacted media in the form of vapor.
- **Dermal Absorption:** Exposure via dermal absorption occurs when receptors are exposed to chemical concentrations present in soil, groundwater, or surface water through direct contact with the skin.
- **Active Ingestion:** The active ingestion pathway represents exposure which may occur through the active ingestion of contaminant concentrations via a drinking water supply well or through agricultural products.
- **Incidental Uptake** This pathway is applicable when receptors may incidentally ingest or inhale impacted media in the form of dust or airborne particulates.

Potential Receptors are categorized by duration of exposure and intensity of use at the Site. The receptor categories described in the CSM include the following:

- **Resident:** The residential receptor is defined by high durational exposure and high intensity usage which may occur through gardening, digging, and recreational sports. This group includes the occupants of a residential property or a residential neighborhood.
- **Commercial:** Commercial receptors are those which are present at the Site for long durations but with low intensity exposure such as indoor office workers.
- **Site Worker:** Site workers are present at the Site for short durations though intensity of use is high, such as during non-routine activities including construction or utility work. Examples include outdoor commercial workers and construction workers.
- **Visitor:** Visitors are characterized by low duration, i.e. less than two hours per day, and low intensity usage such as that which would occur during activities such as walking, shopping, and bird watching.
- **Terrestrial and Aquatic Biota:** These receptors include flora and fauna which may be exposed to contaminants in their respective land-based or aquatic environments.



5.4 CONCEPTUAL SITE MODEL SUMMARY

As indicated above, only asbestos, lead-based paint, PCBs in the demolition debris waste stream, and containerized Universal/hazardous wastes have the potential to impact the Site Worker receptor group. This impact to human receptors would be via incidental uptake (inhaling or ingesting particles) and dermal uptake. Environmental receptors include terrestrial and aquatic biota. These receptor groups may be impacted through incidental uptake (inhaling or ingesting particles), and ingestion of contaminated materials.



6. SAMPLING DESIGN

This demolition phase of the cleanup will include the abatement of asbestos in the Site buildings, and the demolition and removal of the Site buildings. As indicated previously, lead-based paint and PCBs are currently present in the materials that comprise the buildings. Please note that no additional characterization sampling for lead or PCBs in building materials is being proposed as a part of this SSQAPP. However, this SSQAPP does include a provision for sampling the generated building demolition debris stream for lead and PCBs in order to receive facility acceptance and ensure proper disposal.

It is anticipated that the demolition work will be phased as described in the bullets below in order to facilitate safe and proper asbestos abatement and disposal:

1. Remove the garage roof and place it on the ground because it is currently in a slow state of collapse. This will be done so the asbestos removal contractor will be able to safely access asbestos on the roof of the garage. Additional asbestos characterization samples may be required and is detailed in **Section 6.1**.
2. Removal and disposal of select building walls thereby exposing the asbestos inside the building. This will be done so that the contractor has access to the asbestos and universal/hazardous containerized wastes. Waste characterization may be required as described in **Section 6.2**.
3. Abatement of the asbestos containing roof shingles and asbestos identified within the building footprint. If previously unknown asbestos containing materials are encountered, they will either be sampled to determine if they contain asbestos or will be presumptively determined to contain asbestos. Additional asbestos characterization samples may be required and is detailed in **Section 6.1**.
4. Consolidation, sampling, removal, and proper disposal of all containerized universal/hazardous wastes formerly inside the building. Waste characterization may be required as described in **Section 6.2**.
5. Removal and disposal of the concrete building foundation. Waste characterization may be required as described in **Section 6.2**.

The following describes the sampling tasks:

6.1 ASBESTOS SAMPLING

Prior to the demolition of the Site buildings, all identified asbestos will be removed by a New Hampshire certified Asbestos Abatement Contractor. During this abatement work, if previously unidentified suspect ACM is discovered, it will either be presumed to be ACM, or samples will be collected for laboratory analysis of asbestos content. This sampling will be performed in accordance with NHDES Env-A 1800: Asbestos Management and Control. The number of samples collected will be dependent on the number of suspect materials that are encountered and



determined to require sampling. However, of each suspect sample material, three samples will be collected for potential triplicate analysis.

After the abatement is complete, air clearance samples are required to confirm the efficacy of the abatement work. The collection of one per work/containment area will be collected (to be determined by the Contractor) according to NHDES Env-A 1800: Asbestos Management and Control.

Specific sampling methodologies are described in **Section 7**. **Table 1** includes the number and type of samples that are proposed to be collected, cross-referenced with the appropriate standard operating procedure (SOP) that will be used from Credere's Generic QAPP.

6.2 WASTE CHARACTERIZATION SAMPLING

Building Materials

Building demolition debris from the Site will be sampled and submitted for laboratory analysis for proper waste characterization prior to removal from the Site. **Table 1** includes the number and type of samples that are proposed to be collected, cross-referenced with the appropriate SOP that will be used from Credere's Generic QAPP. Specific sampling methodologies are described in **Section 7**.

Universal/Hazardous Containerized Wastes

Containerized Universal/hazardous wastes, or other hazardous or potentially regulated materials identified in containers within the Site building during the cleanup, may require waste characterization sampling prior to disposal. As necessary, samples will be collected and submitted for laboratory analysis prior to removal from the site. Alternately, these materials may be adequately labeled and identifiable, and may not warrant sampling in such instances. **Table 1** includes the number and type of samples that are proposed to be collected, cross-referenced with the appropriate SOP that will be used from Credere's Generic QAPP. Specific sampling methodologies are described in **Section 7**.

The data collected from these activities will serve as the basis for evaluating the efficacy of the cleanup and for ensuring compliance with applicable disposal regulations.

Requirements relative to Chain of Custody, Data Management and Documentation, Data Validation, and Data Usability Assessments contained in the Generic QAPP will be followed during the performance of the above scope of work.



7. FIELD ACTIVITY METHODOLOGY

Field activity methodologies are summarized in the following subsections. Field activities will be conducted in accordance with the SOPs included in Credere's Generic QAPP Rev. 3 (EPA RFA #08166 and #09036).

Where field observations and/or field screening results indicate the presence of additional source areas or potentially impacted media, additional exploration locations, or samples for approved methods may be added to determine the horizontal and/or vertical extent of contamination. The number and locations of these additional samples or exploration locations will be dependent on field data, Site constraints, and professional judgment. All decisions regarding delineation will be recorded in the field logbook, and all locations will be documented. Any additional samples obtained for the purpose of contamination delineation will be collected and field-analyzed in accordance with Credere's SOPs outlined on **Table 1** and the methodologies described in this section. If Credere determines that these additional sample locations require testing for analytes not described in **Table 1**, then the Town of Tilton, NHDES Project Officer (PO), EPA PO, and EPA QA Chemist will be contacted and Credere will prepare an amendment to this SSQAPP that includes a description of the additional samples for analysis, analytical methods, and sampling SOPs. Any amendments to the SSQAPP will include a Title and Approval page, similar to the one provided with this SSQAPP, to document that additional approval was obtained.

7.1 ASBESTOS SAMPLING

Any sampling of suspect ACM during the building demolition will be conducted by a certified Asbestos Inspector (retained by the Contractor, to be determined) according to NHDES Env-A 1800: Asbestos Management and Control. Samples will be analyzed by EMSL Analytical, Inc. (EMSL) of Woburn, Massachusetts using Polarized Light Microscopy (PLM) according to EPA Method 600/R-93/116.

Asbestos clearance air samples will be collected by an independent industrial hygienist according to NHDES Env-A 1800: Asbestos Management and Control. Air filter samples will be analyzed by EMSL using Phase Contrast Microscopy (PCM) according to National Institute of Occupational Safety and Health (NIOSH) Method 7400.

Laboratory analytical SOPs for methods that will be used for the Site samples are included as **Appendix B**.

7.2 WASTE CHARACTERIZATION SAMPLING

Building Materials

Building demolition debris from the Site scheduled for off-site disposal as part of the cleanup will be sampled for disposal characterization laboratory analysis. Consistent with known Site conditions, disposal characterization will include analysis of the waste stream for total lead and PCBs.



The demolition waste stream will be sampled in accordance with ASTM E1908-10, which is included at **Appendix C**. It is anticipated that one (1) composite sample will be collected from the demolition debris piles generated from the removal of the garage building and one (1) composite sample will be collected from the demolition debris piles generated from the removal of the cottage. It is expected that at least two (2) samples will be collected for analyses.

Based on initial analytical results and disposal facility requirement, the collected samples may also be analyzed according to test Method 1311 in EPA Publication SW-846 for toxicity characteristic leaching procedure (TCLP) lead.

All building demolition debris sampling will be completed in accordance with **Table 1**.

Universal/Hazardous/Containerized Wastes

As required, containerized universal/hazardous wastes, or other hazardous or potentially regulated materials identified within the Site building during the cleanup may be sampled for disposal characterization laboratory analysis. Disposal characterization may include the following laboratory analyses according to methods in EPA Publication SW-846: TPH, VOCs, SVOCs, PCBs, RCRA 8 metals, pesticides, herbicides, ignitability, corrosivity, and reactivity. Alternately, these materials may be adequately labeled and identifiable, and may not warrant sampling in such instances. All waste sampling will be done in accordance with **Table 1**.



8. REGULATORY STANDARDS

Sample results will be compared to the applicable state and/or federal standards/guidelines described below.

8.1 ASBESTOS

Laboratory analytical results for asbestos bulk or air clearance samples will be compared to limits specified in NHDES Env-A 1800: Asbestos Management and Control.

8.2 WASTE CHARACTERIZATION

Waste characterization sample results for building debris will be compared to limits in 40 CFR 261.24.



9. PROPOSED PROJECT SCHEDULE

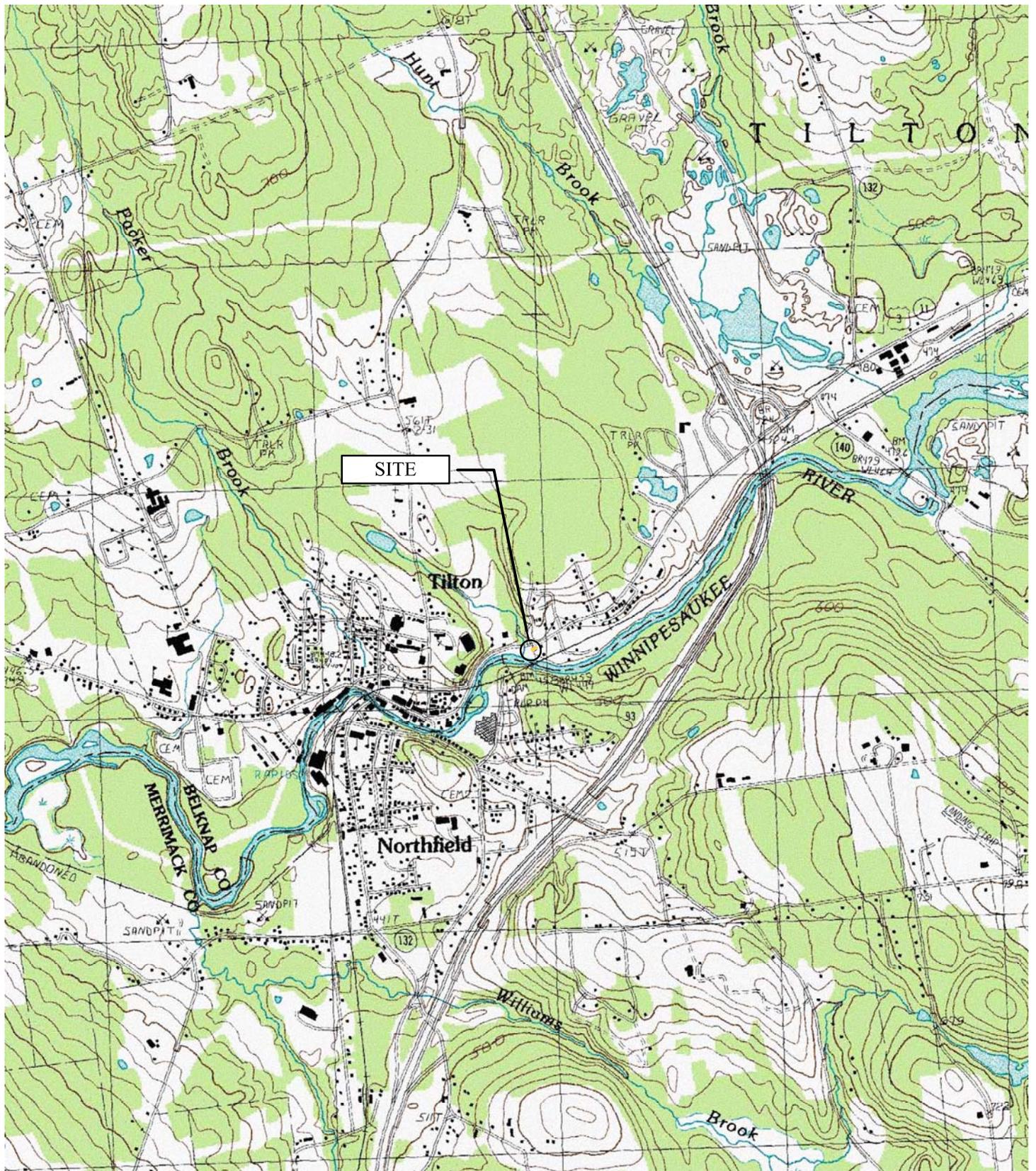
The following schedule is proposed for the clean-up. This is a dynamic schedule and tasks may be performed later based on document regulatory review time and contractor availability. Please note that the final cleanup completion report will not be submitted until June 2103 because additional cleanup activities that are not included within the scope of this SSQAPP must be accounted for in another SSQAPP, and must be completed before the report will be submitted.

TENTATIVE DATE	ACTION
December 30, 2012	Finalize SSQAPP
December 30, 2012	Begin Building Demolition, Asbestos, and Universal & Hazardous Waste Removal
June 2013	Submit Final Cleanup Completion Report



FIGURES





USGS 7.5 MINUTE NORTHFIELD, NH QUADRANGLE (1987)

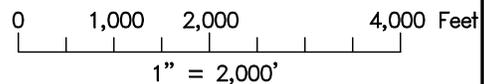
DRAWN BY: SWC DATE: 8/30/10
 CHECKED BY: RSV/JSS PROJECT: 12001162

FIGURE 1 - SITE LOCATION MAP



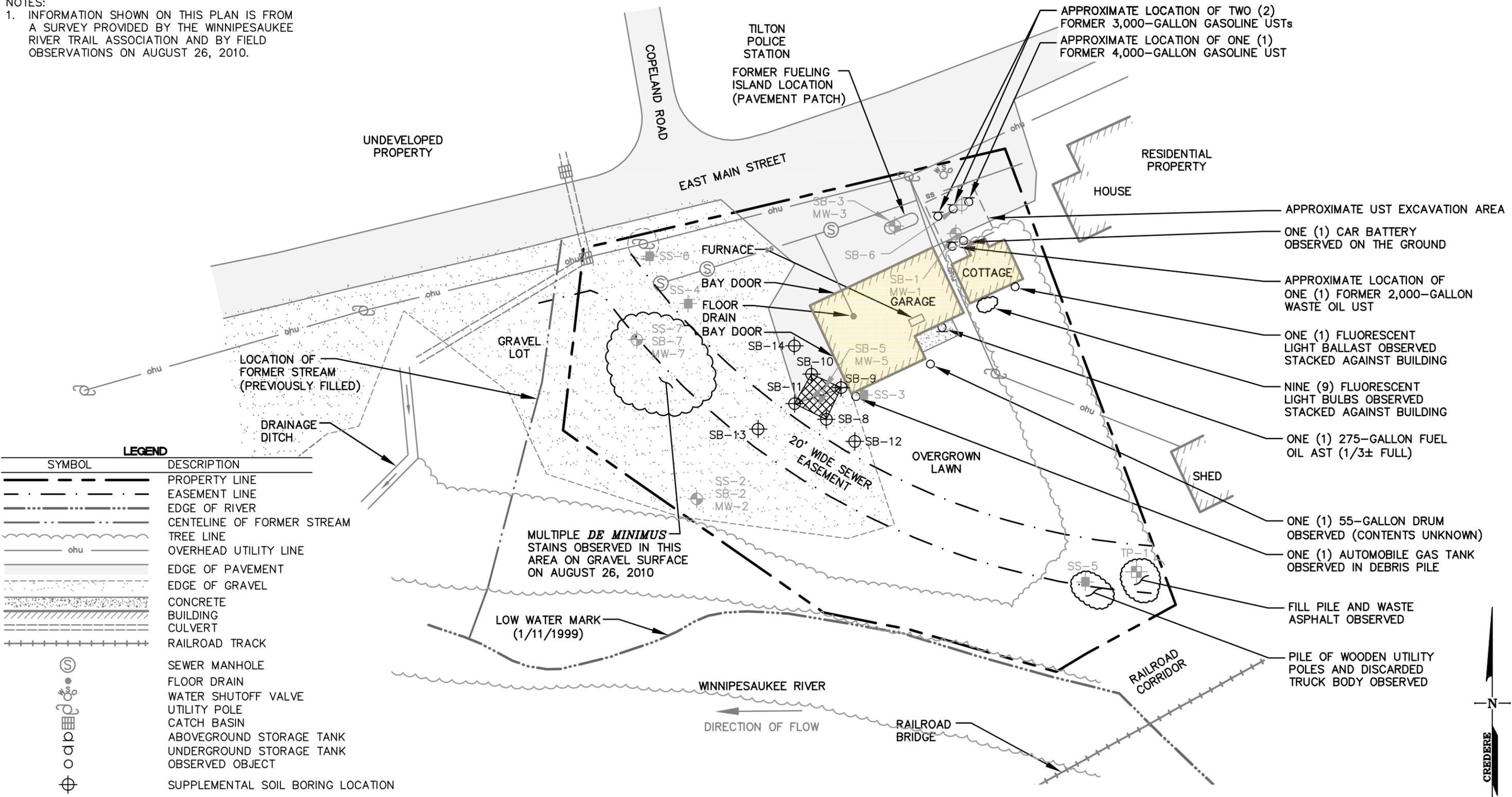
Creder Associates, LLC
 776 Main Street
 Westbrook, Maine 04092
 Tel. (207) 828-1272
 www.crederllc.com

**ERNIE'S AUTO
 SALES PROPERTY**
 180 EAST MAIN STREET
 TILTON, NH
 NHDES #199311019



NOTES:

1. INFORMATION SHOWN ON THIS PLAN IS FROM A SURVEY PROVIDED BY THE WINNIPESAUKEE RIVER TRAIL ASSOCIATION AND BY FIELD OBSERVATIONS ON AUGUST 26, 2010.



SYMBOL	DESCRIPTION
---	PROPERTY LINE
- - - -	EASEMENT LINE
~ ~ ~ ~	EDGE OF RIVER
— · — ·	CENTELINE OF FORMER STREAM
o o o o	TREE LINE
ohu	OVERHEAD UTILITY LINE
=====	EDGE OF PAVEMENT
-----	EDGE OF GRAVEL
	CONCRETE
	BUILDING
	CULVERT
+++++	RAILROAD TRACK
⊙	SEWER MANHOLE
⊙	FLOOR DRAIN
⊙	WATER SHUTOFF VALVE
⊙	UTILITY POLE
⊙	CATCH BASIN
⊙	ABOVEGROUND STORAGE TANK
⊙	UNDERGROUND STORAGE TANK
⊙	OBSERVED OBJECT
⊕	SUPPLEMENTAL SOIL BORING LOCATION
⊕	PREVIOUS SOIL BORING LOCATION
⊕	PREVIOUS SOIL BORING/MONITORING WELL LOCATION
⊕	PREVIOUS SURFICIAL SOIL SAMPLE LOCATION
⊕	PREVIOUS TEST PIT LOCATION
	ESTIMATED EXTENT OF LEAD AND ARSENIC CONTAMINATED SOIL EXCEEDING SOIL REMEDIATION STANDARDS (6'± DEPTH)
	BUILDING TO BE DEMOLISHED

DRAWN BY: SWC/WTE DATE: 12/14/14
 CHECKED BY: RSV/JSS PROJECT: 12001162

CREDERE ASSOCIATES, LLC
 776 MAIN STREET
 WESTBROOK, MAINE 04092
 TEL: 207.828.1272
 FAX: 207.887.1051
 WWW.CREDERELLC.COM

**FIGURE 2
 DETAILED SITE PLAN**

ERNIE'S AUTO SALES PROPERTY
 180 EAST MAIN STREET
 TILTON, NH
 NHDES #199311019

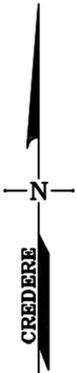
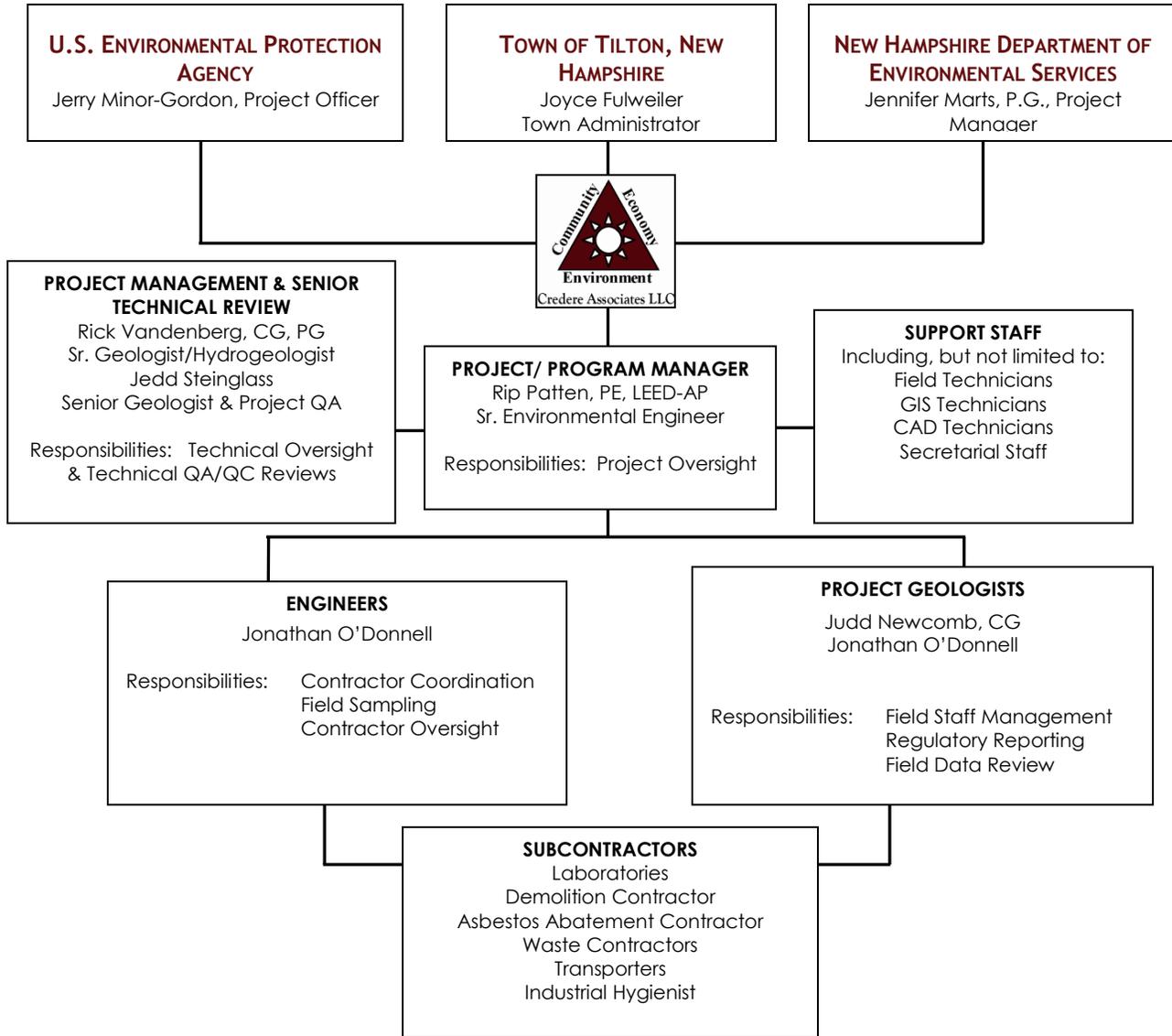
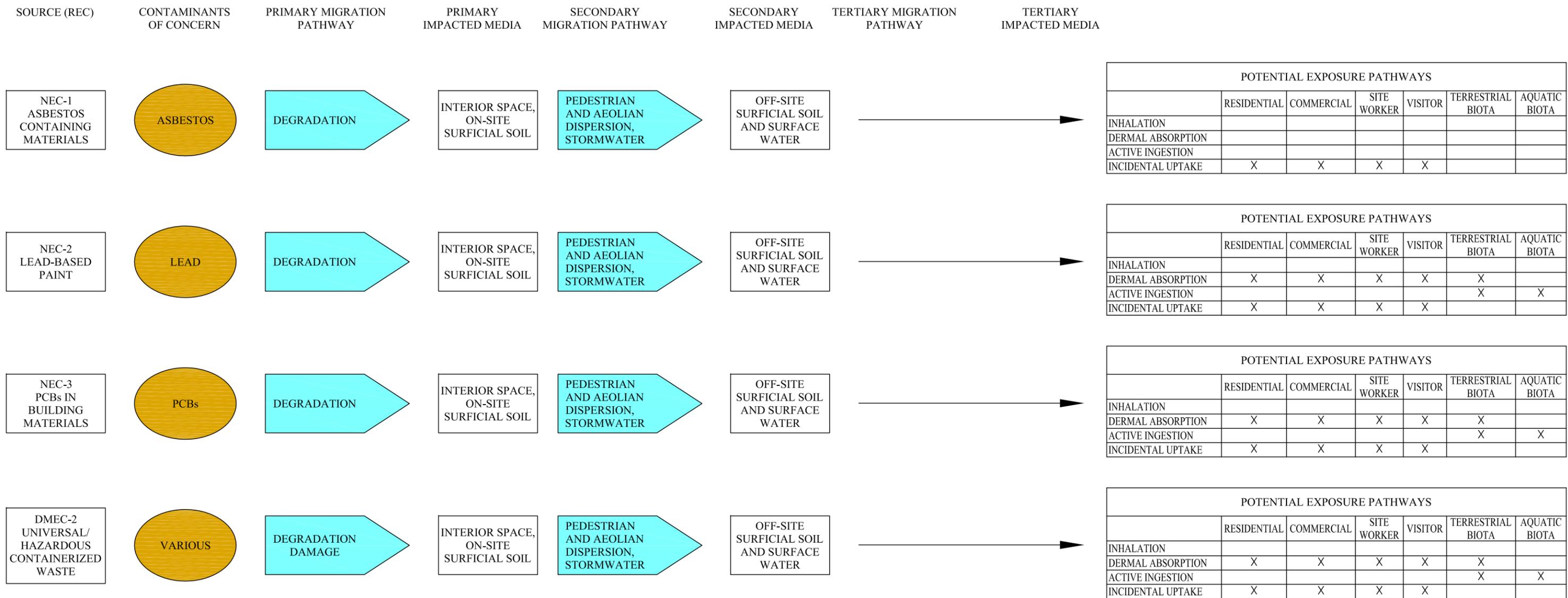


Figure 3 - Credere Organization and Responsibility Chart





NOTE: THIS CONCEPTUAL SITE MODEL COVERS ONLY NEC-1 THROUGH NEC-3 AND DEMC-2. REMAINING ENVIRONMENTAL CONDITIONS WILL BE ADDRESS THROUGH A SUBSEQUENT UPDATED CONCEPTUAL SITE MODEL.

DRAWN BY: SWC	DATE: 8/22/11
CHECKED BY: RSV/JSS	PROJECT: 10001087



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FIGURE 4 UPDATED CONCEPTUAL SITE MODEL

ERNIE'S AUTO SALES PROPERTY
 180 EAST MAIN STREET
 TILTON, NH
 NHDES #199311019

TABLES



Table 1: Sample Reference Table
Ernie's Auto Sales
180 East Main Street
Tilton, New Hampshire
NHDES # 199311019

Media to be Collected	Proposed Sample IDs	Sample Type	Sample Design	Sample Depth (ft bgs)	Field SOPs to be Used	Field Analysis/Observations	No. of Samples for Analysis	No. of Field Dups	Analytical Method	Sample Container Information & Preservative (per location)	Lab SOPs	Laboratory To be Used
Asbestos	TBD	Bulk Materials	Three samples will be collected for analysis from any previously unidentified suspected asbestos-containing material	N/A	Crederre-004 DR-12 HWRB-15 Sampling as outlined in NHDES Env-A 1800:	Visual	3 per material, laboratory stop upon positive result	None	Polarized Light Microscopy EPA 600/R-93/116	Plastic zipper baggies	EMSL PLM SOP (Previously submitted by not numbered)	EMSL Analytical, Inc., Woburn, MA
	CA-AC-1 CA-AC-2	Air Sample on Filter Media	Clearance air sampling will be conducted prior to releasing the asbestos work area and before building demolition.		Crederre-004 EPA SOP #2015 DR-12 HWRB-15		1 per work/containment area		Phase Contrast Microscopy NIOSH Method 7400.	Filter in owm case/container	EMSL NIOSH 7400 SOP (see attached SOP)	
Building Demolition Debris	BMWC-1 BMWC-2	Bulk materials	Composite sampling for waste characterization	N/A	Crederre-004 DR-12 HWRB-15 ASTM 1908-10 (as needed)	Visual	At least 2	None Anticipated	Total Lead - EPA 6010 PCBs - EPA 8082 TCLP Lead - EPA 1311	Total or TCLP Lead - One 8 oz. glass jar with Teflon-lined cap per location (Chilled to 4°C) PCBs - One 4 oz. glass jar with Teflon-lined cap per location (Chilled to 4°C)	RL-4 RL-5 RL-8 RL-22	Absolute Resource Associates, Portsmouth, NH
Universal/Hazardous/Containerized Wastes	UW -1	Misc. consolidated universal/hazardous containerized materials	Sampling for waste characterization	N/A	Crederre-004 EPA SOP#2009 DR-12 HWRB-15	Visual	TBD (only as necessary)	None Anticipated	TPH - EPA 8015 Metals - EPA 6010 VOCs - EPA 8260 Semi VOCs - EPA 8270 PCBs - EPA 8082 Herbicides - EPA 8150 Pesticides - EPA 8081 Ignitability - EPA 1030 Corrossivity - SW-846 7.2 Sulfide/Cyanide Reactivity - EPA 9045C, 7.3.4.2, 7.3.3.2	TPH - One 8 oz. glass jar with Teflon-lined cap per location (Chilled to 4°C) RCRA 8 Metals - One 8 oz. glass jar with Teflon-lined cap per location (Chilled to 4°C) VOCs - One 40 ml VOA with 5 ml methanol and one 40 ml VOA for percent solids (Chilled to 4°C) Semi-VOCs - One 4 oz. glass jar with Teflon-lined cap per location (Chilled to 4°C) Pesticides/Herbicides - One 4 oz. glass jar with Teflon-lined cap per location (Chilled to 4°C) PCBs - One 4 oz. glass jar with Teflon-lined cap per location (Chilled to 4°C) Ignitability - One 4 oz. glass jar with Teflon-lined cap per location (Chilled to 4°C) Corrossivity - One 4 oz. glass jar with Teflon-lined cap per location (Chilled to 4°C) Sulfide Reactivity - One 4 oz. glass jar with Teflon-lined cap per location (Chilled to 4°C) Cyanide Reactivity - One 4 oz. glass jar with Teflon-lined cap per location (Chilled to 4°C)	RL-4 RL-5 RL-6 RL-7 RL-9 RL-11 RL-13 RL-18 RL-19 RL-20 RL-24 RL-25	Absolute Resource Associates, Portsmouth, NH

Notes:
TBD - To be determined in the field
Metals* = RCRA 8 (arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver).

Table 2: Standard Operating Procedure (SOP) Reference Table

**Ernie's Auto Sales
180 East Main Street
Tilton, New Hampshire
NHDES # 199311019**

Field SOPs		
SOP	SOP Description	Date
Crede-re-004	SOP for log book entries	October 2006
EPA SOP#2009	Drum Sampling	November 1994
EPA SOP#2015	Asbestos Sampling	November 1994
DR#12	Chain of Custody Protocol	April 3, 2009
HWRB-15	Decontamination	July 2007
ASTM 1908-10	Standard Guide for Sample Selection of Debris Waste from a Building Renovation or Lead Abatement Project for Toxicity Characteristic Leaching Procedure (TCLP) Testing for Leachable Lead (Pb)	2010 (See Appendix C)
Laboratory SOPs		
SOP	SOP Description	Date
EMSL PLM SOP	Asbestos in Bulk materials by PLM	November 2010
EMSL NIOSH 7400 SOP	Asbestos and Other Fibers by PCM	October 2011
RL-4	Analysis of Polychlorinated Biphenyls in Soil and Water Extracts by EPA 8082, SOP 5303	December 2010
RL-5	Trace Metals Analysis ICP-AES Using EPA Method 200.7/6010-SOP 5603	August 2007
RL-6	Mercury analysis by Cold Vapor Extraction Methods 245.1, 7470A	September 2007
RL-7	Method for Determining TPH by SW846 Method 8015, SOP 5501	November 2008
RL-8	TCLP Extraction Method 1311, SOP 5604, rev1	June 2003
RL-9	VOCs by EPA Method 8260	June 2012
RL-11	Cyanide by EPA Method 9014 4500CN-E	August 2011
RL-13	PAHs, Base/Neutrals, and Acids by EPA Method 8270D	May 2009
RL-18	pH by Method SM 4500 H+B	August 2011
RL-19	Sulfide by method SM 4500-S2D+F	August 2011
RL-20	Ignitability/Flashpoint	August 2011
RL-22	Microwave Extraction by EPA method 3546	August 2011
RL-24	Preparation & Analysis of Organo-Chlorine Pesticides in Soil and Water by Method 8081B	August 2011
RL-25	Chlorinated Herbicides by GC Using Methylation Derivatization	August 2010

APPENDIX A

Analytical Sensitivity and Project Criteria Tables

As of the date of this Site Specific Quality Assurance Project Plan Addendum, the current state and/or federal standards have been reviewed for accuracy.

PCBs in Building Materials by EPA Method 8082

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard (40 CFR 761.3)
PCB-1016	0.2	50 (Total)
PCB-1221	0.2	
PCB-1232	0.2	
PCB-1242	0.2	
PCB-1248	0.2	
PCB-1254	0.2	
PCB-1260	0.2	

Notes:

All values are in mg/kg.

PCBs in Solids by EPA Method 8082

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard ¹
PCB-1016	0.2	1 (Total)
PCB-1221	0.2	
PCB-1232	0.2	
PCB-1242	0.2	
PCB-1248	0.2	
PCB-1260	0.2	

Notes:

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Soil Remediation Standards.

All concentrations in mg/kg

NE = Regulatory guideline not established

Metals in Solids by EPA Methods 6010 and 7471

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard ¹
Arsenic	0.5	11
Barium	2	1,000
Cadmium	0.2	33
Chromium	2	130
Lead	0.5	400
Mercury	0.06	6
Selenium	2	180
Silver	0.4	89

Notes:

All values are in mg/kg.

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Soil Remediation Standards unless marked with an *.

* - United States Environmental Protection Agency Regions 3, 6, and 9. (accessed 4/12/12). Regional Screening Levels for Chemical Contaminants at Superfund Sites (Residential Soil). http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm

** - The chromium VI standard was used because it is the lowest and most conservative standard.

Metals in Water by EPA Methods 6010 and 7471

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard ¹
Arsenic	8	10
Barium	50	2,000
Cadmium	4	5
Chromium	50	100
Lead	8	15
Mercury	0.2	2
Selenium	50	50
Silver	7	100

Notes:

All values are in ug/L.

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Ambient Groundwater Quality Standards for groundwater, unless marked with an *.

* - United States Environmental Protection Agency Regions 3, 6, and 9. (accessed 4/12/12). Regional Screening Levels for Chemical Contaminants at Superfund Sites.

http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm

** - The chromium VI standard was used because it is the lowest and most conservative chromium standard.

TPH in Solids by EPA Method 8100

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard¹
Total Petroleum Hydrocarbons	200	10,000

Notes:
All values are in mg/kg.
1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Soil Remediation Standards.

VOCs in Solids by EPA Method 8260

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard ¹
dichlorodifluoromethane	0.1	1,000
chloromethane	0.1	3
vinyl chloride	0.1	1
bromomethane	0.1	0.3
chloroethane	0.1	NE
trichlorofluoromethane	0.1	1,000
diethyl ether	0.1	3,900
acetone	2	75
1,1-dichloroethene	0.1	2
methylene chloride	0.1	0.1
carbon disulfide	0.1	460
methyl t-butyl ether (MTBE)	0.1	0.2
trans-1,2-dichloroethene	0.1	9
diisopropyl ether (DIPE)	0.1	10
ethyl t-butyl ether (ETBE)	0.1	0.7
1,1-dichloroethane	0.1	3
t-butanol (TBA)	2	2
2-butanone (MEK)	0.3	51
2,2-dichloropropane	0.1	NE
cis-1,2-dichloroethene	0.1	NE
chloroform	0.1	3
bromochloromethane	0.1	160*
tetrahydrofuran (THF)	0.5	200
1,1,1-trichloroethane	0.1	78
1,1-dichloropropene	0.1	NE
t-amyl-methyl ether (TAME)	0.1	3
carbon tetrachloride	0.1	12
1,2-dichloroethane	0.1	0.1
benzene	0.1	0.3
trichloroethene	0.1	5
1,2-dichloropropane	0.1	0.1
bromodichloromethane	0.1	0.1
1,4-dioxane	2	0.3
dibromomethane	0.1	25*
4-methyl-2-pentanone (MIBK)	0.4	29
cis-1,3-dichloropropene	0.1	NE
toluene	0.1	100
trans-1,3-dichloropropene	0.1	NE
2-hexanone	0.5	210*
1,1,2-trichloroethane	0.1	0.1
1,3-dichloropropane	0.1	1,600*
tetrachloroethene	0.1	2
dibromochloromethane	0.1	1
1,2-dibromoethane (EDB)	0.1	0.1
chlorobenzene	0.1	6
1,1,1,2-tetrachloroethane	0.1	0.8
ethylbenzene	0.1	140
m&p-xylenes	0.1	500**
o-xylene	0.1	500**
styrene	0.1	17

VOCs in Solids by EPA Method 8260

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard ¹
bromoform	0.1	0.1
isopropylbenzene	0.1	330
1,1,2,2-tetrachloroethane	0.1	4
1,2,3-trichloropropane	0.1	0.2
n-propylbenzene	0.1	85
bromobenzene	0.1	300*
1,3,5-trimethylbenzene	0.1	96
2-chlorotoluene	0.1	15
4-chlorotoluene	0.1	2,400
tert-butylbenzene	0.1	100
1,2,4-trimethylbenzene	0.1	130
sec-butylbenzene	0.1	130
1,3-dichlorobenzene	0.1	150
4-isopropyltoluene	0.1	3,400
1,4-dichlorobenzene	0.1	7
1,2-dichlorobenzene	0.1	88
n-butylbenzene	0.1	110
1,2-dibromo-3-chloropropane (DBCP)	0.1	0.1
1,2,4-trichlorobenzene	0.1	19
1,3,5-trichlorobenzene	0.1	340
hexachlorobutadiene	0.1	7
naphthalene	0.1	5
1,2,3-trichlorobenzene	0.1	49*

Notes:

All values are in mg/kg.

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Soil Remediation Standards unless marked with an *.

* - United States Environmental Protection Agency Regions 3, 6, and 9. (accessed 4/12/12). Regional Screening Levels for Chemical Contaminants at Superfund Sites (Residential Soil). http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm

** NDHES mixed isomer standard.

NE = Regulatory guideline not established

VOCs in Water by EPA Method 8260

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard ¹
dichlorodifluoromethane	2	1,000
chloromethane	2	30
vinyl chloride	2	2
bromomethane	2	10
chloroethane	2	21,000*
trichlorofluoromethane	2	2,000
diethyl ether	5	1,400
acetone	50	6,000
1,1-dichloroethene	1	7
methylene chloride	5	5
carbon disulfide	2	70
methyl t-butyl ether (MTBE)	2	13
trans-1,2-dichloroethene	2	100
isopropyl ether (DIPE)	2	120
ethyl t-butyl ether (ETBE)	2	40
1,1-dichloroethane	2	81
t-butanol (TBA)	30	40
2-butanone (MEK)	10	4,000
2,2-dichloropropane	2	NE
cis-1,2-dichloroethene	2	2
chloroform	2	70
bromochloromethane	2	83*
tetrahydrofuran (THF)	10	154
1,1,1-trichloroethane	2	200
1,1-dichloropropene	2	NE
t-amyl-methyl ether (TAME)	2	140
carbon tetrachloride	2	5
1,2-dichloroethane	2	5
benzene	2	5
trichloroethene	2	5
1,2-dichloropropane	2	5
bromodichloromethane	0.6	0.6
1,4-dioxane	0.25***	3
dibromomethane	2	7.9*
4-methyl-2-pentanone (MIBK)	10	2,000
cis-1,3-dichloropropene	2	NE
toluene	2	1,000
trans-1,3-dichloropropene	2	NE
2-hexanone	10	34*
1,1,2-trichloroethane	2	5
1,3-dichloropropane	2	290*
tetrachloroethene	2	5
dibromochloromethane	2	60
1,2-dibromoethane (EDB)	0.05***	0.05
chlorobenzene	2	100
1,1,1,2-tetrachloroethane	2	70
ethylbenzene	2	700
m&p-xylenes	2	10,000**
o-xylene	2	10,000**
styrene	2	100
bromoform	2	4
isopropylbenzene	2	800

VOCs in Water by EPA Method 8260

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard ¹
1,1,2,2-tetrachloroethane	2	2
1,2,3-trichloropropane	2	40
n-propylbenzene	2	260
bromobenzene	2	54*
1,3,5-trimethylbenzene	2	330
2-chlorotoluene	2	100
4-chlorotoluene	2	190*
tert-butylbenzene	2	260
1,2,4-trimethylbenzene	2	330
sec-butylbenzene	2	260
1,3-dichlorobenzene	2	600
4-isopropyltoluene	2	260
1,4-dichlorobenzene	2	75
1,2-dichlorobenzene	2	600
n-butylbenzene	2	260
1,2-dibromo-3-chloropropane (DBCP)	0.2***	0.2
1,2,4-trichlorobenzene	2	70
1,3,5-trichlorobenzene	2	40
hexachlorobutadiene	0.5	0.5
naphthalene	5	20
1,2,3-trichlorobenzene	2	5.2*

Notes:

All values are in ug/L.

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Ambient Groundwater Quality Standards for groundwater, unless marked with an *.

* - United States Environmental Protection Agency Regions 3, 6, and 9. (accessed 4/12/12). Regional Screening Levels for Chemical Contaminants at Superfund Sites. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm

***- Reporting limit utilizing EPA Method 8260 SIM.

NHDES mixed isomer standard.

NE = Regulatory guideline not established.

SVOC in Solids by EPA Method 8270

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard ¹
N-nitrosodimethylamine	0.2	0.0023*
aniline	0.2	85*
phenol	0.2	56
2-chlorophenol	0.5	2
bis(2-chloroethyl)ether	0.2	0.7
1,3-dichlorobenzene	0.2	150
1,4-dichlorobenzene	0.2	7
1,2-dichlorobenzene	0.2	88
benzyl alcohol	0.2	6,100*
2-methylphenol	0.2	0.9
bis(2-chloroisopropyl) ether	0.2	5
hexachloroethane	0.2	0.7
N-nitroso-di-N-propylamine	0.2	0.069*
4-methylphenol	0.2	0.7
nitrobenzene	0.2	4.8*
isophorone	0.5	1
2-nitrophenol	0.2	NE
2,4-dimethylphenol	0.2	4
bis(2-chloroethoxy)methane	0.2	180*
2,4-dichlorophenol	0.5	0.7
1,2,4-trichlorobenzene	0.5	19
naphthalene	0.05	5
benzoic acid	5	350
4-chloroaniline	0.2	1.3
hexachlorobutadiene	0.2	7
4-chloro-3-methylphenol	0.2	6,100*
2-methylnaphthalene	0.05	96
hexachlorocyclopentadiene	1	200
2,4,6-trichlorophenol	0.2	0.7
2,4,5-trichlorophenol	0.2	24
2-chloronaphthalene	0.5	NE
2-nitroaniline	0.2	610*
acenaphthylene	0.05	490
dimethylphthalate	0.5	700
2,6-dinitrotoluene	0.2	61*
2,4-dinitrotoluene	0.2	0.7
acenaphthene	0.05	340
3-nitroaniline	0.2	NE
2,4-dinitrophenol	5	0.7
dibenzofuran	0.05	78*
4-nitrophenol	2	NE
fluorene	0.05	77
diethyl phthalate	0.5	1000
4-chlorophenyl phenyl ether	0.5	NE
4-nitroaniline	0.5	24*
4,6-dinitro-2-methylphenol	2	4.9*
azobenzene	0.2	5.1*
N-nitrosodiphenylamine	0.2	99*
4-bromophenyl phenyl ether	0.2	NE
hexachlorobenzene	0.2	0.8

SVOC in Solids by EPA Method 8270

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard ¹
pentachlorophenol	1	3
phenanthrene	0.05	960
anthracene	0.05	1000
carbazole	0.2	NE
di-n-butylphthalate	0.5	2,600
fluoranthene	0.05	960
benzidine	3	0.004
pyrene	0.05	720
butyl benzyl phthalate	0.5	260*
benzo(a)anthracene	0.05	1
chrysene	0.05	120
3,3'-dichlorobenzidine	3	0.7
bis(2-ethylhexyl)phthalate	0.5	72
di-n-octyl phthalate	0.5	NE
benzo(b)fluoranthene	0.05	1
benzo(k)fluoranthene	0.05	12
benzo(a)pyrene	0.05	0.7
indeno(1,2,3-cd)pyrene	0.05	1
dibenzo(a,h)anthracene	0.05	0.7
benzo(g,h,i)perylene	0.05	960

Notes:

All values are in mg/kg.

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Soil Remediation Standards, unless marked with an *.

NE = Regulatory guideline not established

* - United States Environmental Protection Agency Regions 3, 6, and 9. (accessed 4/12/12). Regional Screening Levels for Chemical Contaminants at Superfund Sites. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm

SVOC in Water by EPA Method 8270

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard ¹
N-nitrosodimethylamine	2	0.00042*
aniline	2	12*
phenol	2	4000
2-chlorophenol	5	35
bis(2-chloroethyl)ether	2	10
1,3-dichlorobenzene	2	600
1,4-dichlorobenzene	2	75
1,2-dichlorobenzene	2	600
benzyl alcohol	2	1,500*
2-methylphenol	2	40
bis(2-chloroisopropyl) ether	2	300
hexachloroethane	2	1
N-nitroso-di-N-propylamine	2	0.0093*
4-methylphenol	2	40
nitrobenzene	2	0.12*
isophorone	5	100
2-nitrophenol	2	NE
2,4-dimethylphenol	2	140
bis(2-chloroethoxy)methane	5	47*
2,4-dichlorophenol	5	21
1,2,4-trichlorobenzene	5	70
naphthalene	0.5	20
benzoic acid	50	28,000
4-chloroaniline	2	28
hexachlorobutadiene	2	0.5
4-chloro-3-methylphenol	2	1,100*
2-methylnaphthalene	0.5	280
hexachlorocyclopentadiene	10	50
2,4,6-trichlorophenol	2	5
2,4,5-trichlorophenol	2	700
2-chloronaphthalene	5	550*
2-nitroaniline	2	150*
acenaphthylene	0.5	420
dimethylphthalate	5	50,000
2,6-dinitrotoluene	2	15*
2,4-dinitrotoluene	2	10
acenaphthene	0.5	420
3-nitroaniline	2	NE
2,4-dinitrophenol	50	14
dibenzofuran	0.5	5.8*
4-nitrophenol	10	NE
fluorene	0.5	280
diethyl phthalate	5	1,000
4-chlorophenyl phenyl ether	5	NE
4-nitroaniline	5	3.3*
4,6-dinitro-2-methylphenol	20	1.2*
azobenzene	2	0.10*
N-nitrosodiphenylamine	2	10*
4-bromophenyl phenyl ether	2	NE
hexachlorobenzene	2	1
pentachlorophenol	10	1
phenanthrene	0.5	210

SVOC in Water by EPA Method 8270

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard ¹
anthracene	0.5	2100
carbazole	2	NE
di-n-butylphthalate	5	2,600
fluoranthene	0.5	280
benzidine	30	0.8
pyrene	0.5	210
butyl benzyl phthalate	5	14*
benzo(a)anthracene	0.5	0.1
chrysene	0.5	5
3,3'-dichlorobenzidine	30	1.3
bis(2-ethylhexyl)phthalate	5	6
di-n-octyl phthalate	2	NE
benzo(b)fluoranthene	0.5	0.1
benzo(k)fluoranthene	0.5	0.5
benzo(a)pyrene	0.2	0.2
indeno(1,2,3-cd)pyrene	0.5	0.1
dibenzo(a,h)anthracene	0.5	0.1
benzo(g,h,i)perylene	0.5	210

Notes:

All values are in ug/L.

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Ambient Groundwater Quality Standards for groundwater, unless marked with an *.

* United States Environmental Protection Agency Regions 3, 6, and 9. (accessed 4/12/12). Regional Screening Levels for Chemical Contaminants at Superfund Sites. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm

**Asbestos in Bulk Materials by PLM by EPA Method
600/R**

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard¹
Asbestos	0.20%	1%

Notes:
Values ar % by Volume
1 - New Hampshire Department of Environmental Services Chapter 1800:
Asbestos Management Control, October 21, 2008.

Asbestos in Air via Filter Media by PCM by NIOSH Method 7400		
Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard¹
Asbestos	7.0 fibers/mm ² on filter	0.01 fibers/cc air
Notes: Sample volume will be adjusted to give 100 to 1300 fibers/mm ² where possible. 1 - New Hampshire Department of Environmental Services Chapter 1800: Asbestos Management Control, October 21, 2008.		

Herbicides in Solids by MADEP Method EPH-04-1.1

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard ¹
Dalapon	0.04167	3
Dicamba	0.08333	1,800*
MCPP	18.75	26
MCPA	18.75	13
Dichloroprop	0.04167	NE
2,4-D	0.04167	300
Pentachlorophenol	0.04167	3
2,4,5-TP (silvex)	0.04167	60
2,4,5-T	0.04167	610*
2,4-DB	0.41667	490*
Picloram	0.08333	6
Dinoseb	0.08333	1

Notes:

All values are in mg/kg.

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Soil Remediation Standards.

NE = Regulatory guideline not established

Pesticides in Soil by 8081

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard ¹
alpha-BHC	0.03	0.06
beta-BHC	0.03	0.06
delta-BHC	0.03	NE
gamma-BHC (Lindane)	0.03	0.09
Heptachlor	0.03	0.2
Aldrin	0.03	0.09
Heptachlor Epoxide	0.03	0.1
Endosulfan I	0.03	NE
Dieldrin	0.03	0.06
4,4'-DDE	0.03	4
Endrin	0.03	8
Endosulfan II	0.03	NE
4,4'-DDD	0.03	6
Endosulfan Sulfate	0.03	NE
4,4'-DDT	0.03	4
Methoxychlor	0.03	130
Endrin Ketone	0.03	NE
Endrin Aldehyde	0.03	NE
alpha-Chlordane	0.03	NE
gamma-Chlordane	0.03	NE
Toxaphene	0.2	1

Notes:

All values are in mg/kg.

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Soil Remediation Standards.

NE = Regulatory guideline not established

Pesticides in Water by 8081

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard ¹
alpha-BHC	0.05	0.1
beta-BHC	0.05	0.1
delta-BHC	0.05	
gamma-BHC (Lindane)	0.05	0.2
Heptachlor	0.05	0.4
Aldrin	0.05	0.1
Heptachlor Epoxide	0.05	0.2
Endosulfan I	0.05	
Dieldrin	0.05	0.1
4,4'-DDE	0.05	
Endrin	0.05	2
Endosulfan II	0.05	
4,4'-DDD	0.05	
Endosulfan Sulfate	0.05	
4,4'-DDT	0.05	
Methoxychlor	0.05	40
Endrin Ketone	0.05	
Endrin Aldehyde	0.05	
alpha-Chlordane	0.05	
gamma-Chlordane	0.05	
Toxaphene	0.4	3

TCLP Analysis and Total Constituent Analysis in Solids per EPA Publication SW-846

EPA HW No. 1	Contaminant	Laboratory Practical Quantitation Limit for TCLP Analysis (mg/L)	TCLP Regulatory Limit (mg/L)	Laboratory Practical Quantitation Limit for Total Constituent Analysis (mg/kg)	Regulatory Limit for Total Constituent Analysis (Equal to 20 times TCLP Limit) (mg/kg)	Method for Total Constituent Analysis
D004	Arsenic	< 0.08	5.0	< 0.5	100	EPA 6010
D005	Barium	< 0.5	100	< 2.5	2000	EPA 6010
D006	Cadmium	< 0.04	1.0	< 0.2	20	EPA 6010
D007	Chromium	< 0.5	5.0	< 2.5	100	EPA 6010
D008	Lead	< 0.08	5.0	< 0.5	100	EPA 6010
D009	Mercury	< 0.2	0.2	< 4	4	EPA 7470A/7471B
D010	Selenium	< 0.5	1.0	< 2.5	20	EPA 6010
D011	Silver	< 0.07	5.0	< 0.35	100	EPA 6010
D012	Endrin	< 0.008	0.02	< 0.16	0.4	EPA 8081
D013	Lindane	< 0.008	0.4	< 0.16	8	EPA 8081
D014	Methoxychlor	< 0.008	10	< 0.16	200	EPA 8081
D015	Toxaphene	< 0.008	0.5	< 0.16	10	EPA 8081
D016	2,4-D	< 10	10	< 200	200	EPA 8151
D017	2,4,5-TP (Silvex)	< 1.0	1.0	< 20	20	EPA 8151
D018	Benzene	< 0.2	0.5	< 4	10	EPA 8260
D019	Carbon tetrachloride	< 0.2	0.5	< 4	10	EPA 8260
D020	Chlordane	< 0.03	0.03	< 0.6	0.6	EPA 8081
D021	Chlorobenzene	< 0.2	100	< 4	2000	EPA 8260
D022	Chloroform	< 0.2	6.0	< 4	120	EPA 8260
D023	o-Cresol	< 0.5	200	< 10	4000	EPA 8270
D024	m-Cresol	< 0.5	200	< 10	4000	EPA 8270
D025	p-Cresol	< 0.5	200	< 10	4000	EPA 8270
D026	Cresol	< 0.5	200	< 10	4000	EPA 8270
D027	1,4-Dichlorobenzene	< 0.2	7.5	< 4	150	EPA 8260
D028	1,2-Dichloroethane	< 0.2	0.5	< 4	10	EPA 8260
D029	1,1-Dichloroethylene	< 0.2	0.7	< 4	14	EPA 8260
D030	2,4-Dinitrotoluene	< 0.1	0.13	< 2	2.6	EPA 8270
D031	Heptachlor (and its epoxide)	< 0.008	0.008	< 0.16	0.16	EPA 8081
D032	Hexachlorobenzene	< 0.1	0.13	< 2	2.6	EPA 8270
D033	Hexachlorobutadiene	< 0.5	0.5	< 10	10	EPA 8270
D034	Hexachloroethane	< 0.5	3.0	< 10	60	EPA 8270
D035	Methyl ethyl ketone	< 2	200	< 40	4000	EPA 8260
D036	Nitrobenzene	< 0.5	2.0	< 10	40	EPA 8270
D037	Pentachlorophenol	< 0.5	100	< 10	2000	EPA 8270
D038	Pyridine	< 0.5	5.0	< 10	100	EPA 8270
D039	Tetrachloroethylene	< 0.2	0.7	< 4	14	EPA 8260
D040	Trichloroethylene	< 0.2	0.5	< 4	10	EPA 8260
D041	2,4,5-Trichlorophenol	< 0.5	400	< 10	8000	EPA 8270
D042	2,4,6-Trichlorophenol	< 0.5	2.0	< 10	40	EPA 8270
D043	Vinyl chloride	< 0.2	0.2	< 4	4	EPA 8260

Reactivity in Solids for Cyanide (Method E9014) and Sulfide (Method SM4500-S)

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard¹
Cyanide (total)	0.5	
Sulfide (soluble)	0.4	
Notes: Values are in fibers/cubic centimeter (f/cc)		

EPH in Solids by MADEP Method EPH-04-1.1

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard ¹
naphthalene	0.1	5
2-methylnaphthalene	0.1	96
phenanthrene	0.1	960
acenaphthene	0.1	340
acenaphthylene	0.1	490
fluorene	0.1	77
anthracene	0.1	1000
fluoranthene	0.1	960
pyrene	0.1	720
benzo(a)anthracene	0.1	1
chrysene	0.1	120
benzo(b)fluoranthene	0.1	1
benzo(k)fluoranthene	0.1	12
benzo(a)pyrene	0.1	0.7
indeno(1,2,3-cd)pyrene	0.1	1
dibenzo(a,h)anthracene	0.1	0.7
benzo(g,h,i)perylene	0.1	960
C9-C18 Aliphatics	10	NE
C19-C36 Aliphatics	10	NE
C11-C22 Aromatics	10	NE

Notes:

All values are in mg/kg.

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Soil Remediation Standards.

NE = Regulatory guideline not established

EPH in Water by MADEP Method EPH-04-1.1

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard ¹
naphthalene	0.5	20
2-methylnaphthalene	0.5	280
phenanthrene	0.5	210
acenaphthene	0.5	420
acenaphthylene	0.5	420
fluorene	0.5	280
anthracene	0.5	2100
fluoranthene	0.5	280
pyrene	0.5	210
benzo(a)anthracene	0.5	0.1
chrysene	0.5	5
benzo(b)fluoranthene	0.5	0.1
benzo(k)fluoranthene	0.5	0.5
benzo(a)pyrene	0.2	0.2
indeno(1,2,3-cd)pyrene	0.5	0.1
dibenzo(a,h)anthracene	0.5	0.1
benzo(g,h,i)perylene	0.5	210
C9-C18 Aliphatics	50	NE
C19-C36 Aliphatics	50	NE
C11-C22 Aromatics	50	NE

Notes:

All values are in ug/L.

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Ambient Groundwater Quality Standards for groundwater.

NE = Regulatory guideline not established

VPH in Solids by MA DEP Method VPH-04-1.1

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard ¹
methyl t-butyl ether (MTBE)	0.1	0.2
benzene	0.1	0.3
toluene	0.1	100
ethylbenzene	0.1	140
m&p-xylenes	0.1	500
o-xylene	0.1	500
naphthalene	0.2	5
C5-C8 Aliphatics	5	NE
C9-C12 Aliphatics	5	NE
C9-C10 Aromatics	5	NE

Notes:

All values are in mg/kg.

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Soil Remediation Standards.

NE = Regulatory guideline not established

VPH in Water by MA DEP Method VPH-04-1.1

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard ¹
methyl t-butyl ether (MTBE)	2	13
benzene	1	5
toluene	2	1000
ethylbenzene	2	700
m&p-xylenes	2	10,000
o-xylene	2	10,000
naphthalene	5	20
C5-C8 Aliphatics	100	NE
C9-C12 Aliphatics	100	NE
C9-C10 Aromatics	100	NE

Notes:

All values are in ug/L.

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Ambient Groundwater Quality Standards for groundwater.

NE = Regulatory guideline not established

PAHs in Solid by 8270

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard ¹
naphthalene	0.5	5
2-methylnaphthalene	0.5	96
acenaphthylene	0.5	490
acenaphthene	0.5	340
dibenzofuran	0.5	78*
fluorene	0.5	77
phenanthrene	0.5	960
anthracene	0.5	1000
fluoranthene	0.5	960
pyrene	0.5	720
benzo(a)anthracene	0.5	1
chrysene	0.5	120
benzo(b)fluoranthene	0.5	12
benzo(k)fluoranthene	0.5	12
benzo(a)pyrene	0.5	0.7
indeno(1,2,3-cd)pyrene	0.5	1
dibenzo(a,h)anthracene	0.5	0.7
benzo(g,h,i)perylene	0.5	960

Notes:

All values are in mg/kg.

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Soil Remediation Standards.

* - United States Environmental Protection Agency Regions 3, 6, and 9. (accessed 4/12/12). Regional Screening Levels for Chemical Contaminants at Superfund Sites. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm

NE = Regulatory guideline not established

PAHs in Water by 8270

Analyte	Laboratory Practical Quantitation Limit	Regulatory Standard ¹
naphthalene	0.5	20
2-methylnaphthalene	0.5	280
acenaphthylene	0.5	420
acenaphthene	0.5	420
dibenzofuran	0.5	5.8*
fluorene	0.5	280
phenanthrene	0.5	210
anthracene	0.5	2100
fluoranthene	0.5	280
pyrene	0.5	210
benzo(a)anthracene	0.5	0.1
chrysene	0.5	5
benzo(b)fluoranthene	0.5	0.1
benzo(k)fluoranthene	0.5	0.5
benzo(a)pyrene	0.2	0.2
indeno(1,2,3-cd)pyrene	0.5	0.1
dibenzo(a,h)anthracene	0.5	0.1
benzo(g,h,i)perylene	0.5	210

Notes:

All values are in ug/L.

1 - New Hampshire Department of Environmental Services (NHDES) Chapter 600 Ambient Groundwater Quality Standards for groundwater.

* United States Environmental Protection Agency Regions 3, 6, and 9. (accessed 4/12/12). Regional Screening Levels for Chemical Contaminants at Superfund Sites. http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm

NE = Regulatory guideline not established

APPENDIX B

EMSL Analytical, Inc. Asbestos Analytical SOPs





EMSL Analytical S.O.P.

PLM SOP

EPA 600/R-93/116, EPA 400 Point Count, EPA 1000 Point Count, EPA PLM NOB, NYSDOH ELAP 198.1 & 198.6, NIOSH 9002

1.0 Method Description

1.1 Applicable Matrix

This method is applicable to friable, non-friable and non-friable organically bound building materials. Sample should be of sufficient quantity to ensure reliable quantitation through the use of multiple preparations.

1.2 Scope and application

This procedure should be used to determine the asbestos content of bulk building materials and includes procedures for various prep techniques and quantitation methods. Due to the procedure's use of multiple preps and reprep, as well as numerous preparation options, bulk building materials are specified due to their inherent gross homogeneity that cannot be assumed in debris or other non-manufactured materials. Various accreditation agencies may require only specific parts of this procedure and exclude others. This procedure, in its entirety can be viewed as a set of tools to handle a wide range of materials.

1.3 Summary of Method

Samples are initially examined under low magnification using stereo microscopy. Initial observations note gross material appearance (homogeneity, fibrous/non-fibrous) and physical characteristics (color, texture, friable/non-friable). Preparation using various techniques, including but not limited to pinch mounts and gravimetric reduction, follow by analysis by polarized light microscopy (PLM) are used for the positive identification of suspect fibers and quantitation. Positive identification of asbestos requires the determination of optical property characteristics of the six types of asbestos: chrysotile, amosite (grunerite), crocidolite (riebeckite), anthophyllite, tremolite and actinolite asbestos. Quantitation follows using calibrated visual estimation or, depending on the method used, one of a number of point counting techniques. Final results are generated incorporating gravimetric preparation data when applicable. This procedure will generate aerial percentages, or in the case when combined with gravimetric reduction, a combined aerial / weight percentage.

1.4 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%. Similarly when EPA 600/R-93/116 is combined with gravimetric reduction the preparation will lower the detection limit of this procedure. This detection limit is based on the limits as referenced in the documented methodologies. When a detection limit of 1% is appropriate for an analysis the results are reported for samples containing asbestos as "less than" one percent (<1%).



2.0 Interferences

Interferences for this method include but are not limited to:

- 2.1 Non-regulated asbestos minerals such as the two polymorphs of Chrysotile, Lizardite and Antigorite.
- 2.2 Non-regulated amphiboles such as winchite and richterite, and pyroxenes.
- 2.3 Cleavage fragments of the regulated asbestos types which may at times have aspect ratios similar to the true asbestiform varieties.
- 2.4 Clay minerals that can have similar morphology to asbestos such as sepiolite and pallygorskite.
- 2.5 All non-asbestos particulate, fibrous or not, which can partially or wholly obscure asbestos fibers.
- 2.6 Coatings applied during sample manufacturing, or while the building material application was in place, may obscure the optical properties of fibers.

3.0 Definitions

- 3.1 Asbestiform - A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility. Asbestiform morphology is characterized by high mean aspect ratio (generally >20:1 for fibers > 5 µm) and thin fibril widths (usually < 0.5µm).
- 3.2 Asbestos - A collective mineralogical term encompassing the asbestiform varieties of various minerals that are typically hydrated silicates. Regulated asbestos refers to 6 specific asbestiform minerals belonging to two classes of silicate minerals.
 - 3.2.1 Serpentine Asbestos - only one serpentine mineral is classified as a regulated asbestos variety. Serpentine asbestos is typically less dense and more flexible than amphibole varieties.
 - 3.2.1.1 Chrysotile - a serpentine, layered mineral that forms long, silky fibrils and bundles. Chrysotile is the most prevalent of all regulated asbestos varieties in the US.
 - 3.2.2 Amphibole Asbestos - double chain silicates that are typically more rigid than chrysotile and are all members of a solid solution series. Of the amphibole varieties, amosite is the most prevalent in the US.
 - 3.2.2.1 Actinolite - an amphibole solid solution member in the series of the Tremolite - Actinolite - FerroActinolite.
 - 3.2.2.2 Amosite - mineralogically known as grunerite, it is an amphibole solid solution mineral. The trade name "AMOSITE" is rooted in the terminology "amosa" describing Asbestos Mines Of South Africa.
 - 3.2.2.3 Anthophyllite - amphibole silicate solid solution end member.
 - 3.2.2.4 Crocidolite - mineralogically known as riebeckite, it is an amphibole solid solution mineral.
 - 3.2.2.5 Tremolite - an amphibole solid solution end member in the series of Tremolite - Actinolite - FerroActinolite.
- 3.3 Birefringence - the largest difference in refractive index along the various axes of a biaxial particle
- 3.4 Calibrated Visual Estimate - a concentration estimate based on, and calibrated for, samples with known quantities of asbestos. This may be actual samples, permanent mounts or pictures.



- 3.5 Crossed Polarizers - the normal condition of the polarized light microscope when the polarizer (sub stage polar) and the analyzer (post stage polar) are set so that their privileged directions are perpendicular to each other.
- 3.6 Dispersion Staining - method of "staining" particles while in refractive index liquids. This staining effect is a function of the particle's and liquid's physical properties in transmitting the individual wavelengths of light differing amounts. When particles and liquid have larger differences in refractive index at the red (short) and blue (long) ends of the visible spectrum, their dispersion colors will be brighter and more intense.
- 3.7 Extinction - when all transmitted light is blocked by both the polarizer and the particle of interest, this will appear as if the light in the particle has gone out or has become extinguished.
- 3.8 Fiber - An asbestos or non-asbestos particle that may be described as being elongated with substantially parallel sides on a gross scale.
- 3.9 Fiber Color - the color of the fiber in plane polarized light.
- 3.10 Fibrous - The description of a bulk building material describing the presence of fibers, asbestos or non-asbestos, in the bulk sample.
- 3.11 Plane Polarized Light - light that is transmitted through a sub stage polarizer only. No post stage polar (analyzer) is present.
- 3.12 Pleochroism - the change in color of a particle as the fiber's alignment changes (rotates) in comparison to the plane polarized light.
- 3.13 Point Count - quantitation technique in which a series of points is randomly superimposed on the sample's field of views. The material that lies under the point(s) is classified and counted. The points are totaled and percentages generated.
- 3.14 Polarized Light - light that passes through a polarizer, is reduced to only the light that has the same, radial direction, vibration direction as the polar it passes through. Polarized light is light that has only one, known vibration direction.
- 3.15 Refractive Index - the speed at which light is transmitted through a medium. The refractive index of air is 1.0.
- 3.16 Refractive Index Liquid - a liquid of known refractive index used to determine the refractive index of unknown particles through comparison.
- 3.17 Sign of Elongation - due to the change in refractive index along a particle's different optical axes, biaxial particles with an elongation (fibers) will retard light differently in directions defined by their elongation. The use of a 530-550nm gypsum retardation plate allows this to be easily seen by changing the fiber color from blue to yellow over a 90° rotation.

4.0 Safety

All personnel performing preparation and/or analysis of samples must be familiar with the EMSL Chemical Hygiene Plan (EMSLChemHygiene 200.0). Specific hazards and precautions associated with this analysis include:

4.1 Asbestos

- 4.1.1 Prudent measures must be taken to prevent any possible airborne asbestos fiber release from occurring during sample handling.
- 4.1.2 Ensure that work stations and surrounding floors are clutter free and dust free.
- 4.1.3 All handling of samples, including gravimetrically reduced samples should take place in a safety hood.
- 4.1.4 All intermediate preparation steps (acid dissolution, slide preparation etc.) should also take place in a safety hood.



4.1.5 After an analyst completes work for the day or shift, or as needed, the HEPA hood, the surrounding work surfaces and floors in the immediate vicinity below the work area should be cleaned as follows:

4.1.5.1 The hood and all surfaces (including floor) should be vacuumed with a HEPA filtration equipped vacuum.

4.1.5.2 The hood and all surfaces (including floors) should be wiped down with either disinfecting wipes or for the floor areas a "Swiffer" type, handled floor cleaner that utilizes disposable, wet cleaning pads.

4.2 Muffle Furnace

4.2.1 The muffle furnace should be vented out of the building due to organic vapors generated during the ashing process.

4.2.2. Care should be used and appropriate safety clothing worn, during handling of samples in the muffle furnace due to high temperatures (480°C).

4.3 Solvent Dissolution

4.3.1 Solvent (including acid) dissolution should only take place under a safety, fume hood.

4.3.2 Acid is a corrosive, proper clothing should be worn.

4.3.3 Exposure to all solvents should be avoided, proper safety precautions should be taken at all times.

5.0 Equipment and Supplies

5.1 Centrifuge and tubes (optional)

5.2 Cover slips, glass

Note: ideal coverslip thickness is matched to the objectives of a particular microscope. This information is inscribed on the objectives with a number such as 0.17.

5.3 Crucibles w/ tops

5.4 Filtration apparatus including:

5.4.1 Vacuum pump

5.4.2 Tubing

5.4.3 Vacuum flask

5.4.4 Rubber stoppers appropriate for frits of filtration setup

5.5 Filtration setups including:

5.5.1 47mm disposable filtration funnels w/ supports and frits

5.5.2 47mm absorbent pads

5.6 Forceps

5.7 Fume safety hood capable of ≥ 75 lpm

5.8 HEPA filtered safety hood capable of ≥ 75 lpm

5.9 Microscope slides, glass, 3x1

5.10 Mortar and pestles, agate or porcelain

5.11 Muffle furnace capable of 480°C

5.12 Petri dishes, 47mm disposable

5.13 Polycarbonate filters, 47mm, 0.4 μ m pore size

5.14 Polarized Light Microscope including:

5.14.1 360° rotating stage

5.14.2 Analyzer (post stage polar)

5.14.3 Central stop dispersion objective

5.14.4 Eyepiece reticule - cross hair

5.14.5 Eyepiece reticule - point array such as Chalkley



- 5.14.6 Gypsum retardation compensator plate, 530-550nm
- 5.14.7 Light source
- 5.14.8 Objective lenses, 4x-10x-20x-40x
- 5.14.9 Oculars 10x
- 5.14.10 Polarizer (sub stage polar)
- 5.14.11 Port for retardation plate
- 5.14.12 Sub stage Condenser
- 5.14.13 Sub stage field iris
- 5.15 Probes and other prep tools
- 5.16 Scalpel handles and blades
- 5.17 Stereoscopic microscope of low power (4-40x)
- 5.18 Thermometer capable of ambient room temperature displayed in °C
- 5.19 Weigh boats and drying tins
- 5.20 Wylie mill (optional)

6.0 Reagents and Standards

All Reagents should be ACS Reagent Grade or better.

- 6.1 Acetone
- 6.2 Calibrated Refractive Index Glass Beads such as Cargille M-25 set (M-7 is still acceptable if already on-site)
- 6.3 Hydrochloric Acid - concentrated at least 3N
- 6.4 NIST standard asbestos (1866/1866a and 1867) on grids
- 6.5 Non-Asbestos standards on grids
- 6.6 Refractive Index Liquids
 - 6.6.1 $N_D = 1.550, 1.605, 1.625$ and 1.680 for common use
 - 6.6.2 $N_D = 1.490 - 1.720$ in 0.005 increments or less

7.0 Sample Collection, Preservation, Shipment and Storage

- 7.1 Samples are collected in a manner which produces no fiber release. Samples are commonly collected using a strong 'zip lock' bag.
- 7.2 Only one sample per bag should be submitted.
- 7.3 No preservation is required.
- 7.4 Bulk samples are not to be shipped or stored with air samples.
- 7.5 Samples are retained in an easily retrievable manner for a minimum of 60 days.

8.0 Calibration and Standardization

Each major component of the method is calibrated and/or standardized including the analyst. Examples follow:

- 8.1 The analytical balance is checked daily, or on next use, using 2 weights over a range that represents the normal use of the balance.
- 8.2 The muffle furnace is calibrated quarterly at 3 points over the range of normal temperature use.
- 8.3 The PLM analyst is calibrated as follows; details are listed in EMSL's QA Manual Module A section A.19:
 - 8.3.1 Using past proficiency samples and samples of known quantity, analyst precision and accuracy are tracked and monitored.



8.3.2 Analysts calibrate their visual estimates using standards (8.3.1 above) and also against point counts on the same sample.

8.4 The polarized light microscope is aligned daily, or prior to first use as follows, these instructions are specific for a binocular polarized light microscope (PLM) equipped with dispersion staining (DS) objective:

8.4.1 Alignment of the Illumination/Field Diaphragm

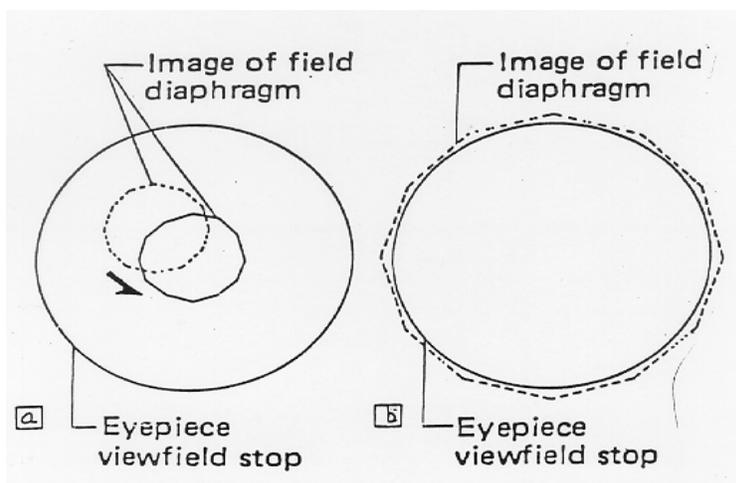
This is not able to be adjusted on all Polarized Light Microscopes. For proper illumination the filament should be able to be adjusted in all directions (up/down, left/right, forwards/backwards and rotationally). The microscope, to this end, may have the filament housed externally from the microscope itself, this housing will be adjustable if this is the case.

8.4.1.1 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.

8.4.1.2 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.

8.4.1.3 The condenser diaphragm should be open and the field diaphragm closed just until its image is visible in the object field of the microscope (Figure I). The condenser should be moved up and down until the image of the field diaphragm is as sharp as possible.

FIGURE I: OBJECTIVE DIAGRAMS



8.4.1.4 If the scope is able to have the illumination adjusted, remove the specimen if necessary, insert the Bertrand lens or telescoping ocular and view the filament image through a 10x objective. If using a telescoping ocular, focus the image of the lens by rotating the ocular threads clockwise or counter clockwise.

8.4.1.5 Adjust the filament:



- 8.4.1.5.1 Rotationally: so that the filament coils are perpendicular to the direction the light travels in the microscope from the light source to the first mirror where it is reflected up.
- 8.4.1.5.2 Horizontally and vertically: so that the filament is centered within the field of view.
- 8.4.1.5.3 Backward and forward: so that the filament completely fills the field of view.
- 8.4.1.6 Remove the Bertrand lens or telescoping ocular and view the field. The field should be of equal illumination over its entirety, without bright or dark regions.
- 8.4.1.7 For microscopes without adjustable filaments, verify the filament is illuminated.
- 8.4.1.8 Check the illumination column of the PLM Calibration & Contamination worksheet.

8.4.2 Stage / Objective Alignment

The microscope will most likely have a nosepiece that allows the objectives to be centered on the rotational axis of the stage. If this is not the case the stage is generally able to be centered for an objective. However if stage centering must be used it can truly be centered on only one (1) objective, even if it may be very close for the others. When centering the stage, the 10x Dispersion Staining objective should be the one chosen for centering.

8.4.2.1 Objective Centering

- 8.4.2.1.1 Starting with the lowest power objective, bring a particle of a slide to the center of the field of view, at the crosshairs of the ocular.
- 8.4.2.1.2 Rotate the stage through a full 360° rotation. Without moving the slide, bring the stage to the rotational orientation where the particle was furthest from the cross hair center.
- 8.4.2.1.3 Using a set/pair of objective adjustment keys (may look like inverted hex wrenches) adjust the set screw adjustments for the objective being calibrated so that the particle is approximately ½ way back to the cross hair center.
- 8.4.2.1.4 Move the slide so that the particle is now back in the center of the crosshair field of view.
- 8.4.2.1.5 Repeat steps 8.4.2.1.1 to 8.4.2.1.4 until the particle rotates at the crosshair center without moving off of the crossed hairs.
- 8.4.2.1.6 Repeat steps 8.4.2.1.1 to 8.4.2.1.5 until all objectives have been centered, making sure to adjust the objectives in order of increasing power (ie. 4x - 10x - 20x - 40x).

8.4.2.2 Stage Centering

- 8.4.2.2.1 As stated before, the stage can only be truly centered on one (1) objective at a time; this must be the 10x DS Objective.
- 8.4.2.2.2 Bring a particle of a slide to the center of the field of view, at the crosshairs of the ocular.
- 8.4.2.2.3 Rotate the stage through a full 360° rotation. Without moving the slide, bring the stage to the rotational orientation where the particle was furthest from the cross hair center.



- 8.4.2.2.4 Using a set/pair of stage adjustment keys (may look like inverted hex wrenches) adjust the set screw adjustments for the stage so that the particle is approximately $\frac{1}{2}$ way back to the cross hair center.
- 8.4.2.2.5 Move the slide so that the particle is now back in the center of the crosshair field of view.
- 8.4.2.2.6 Repeat steps 8.4.2.2.2 to 8.4.2.2.5 until the particle rotates at the crosshair center without moving off of the crossed hairs.
- 8.4.2.2.7 Check the Stage/Objective Alignment column of the PLM Calibration & Contamination worksheet.

8.4.3 Central Stop / Condenser Alignment

- 8.4.3.1 With the microscope focused on a particle with the DS Objective, insert the Bertrand Lens or telescoping ocular.
- 8.4.3.2 Focus on the image of the central stop and adjust the central stop (if possible) so that the stop is at the center of the cross hairs.
- 8.4.3.3 Slowly close the sub stage condenser until it comes into view and is just larger than the central stop.
- 8.4.3.4 Using the adjustments on the sub stage condenser (either set screw or adjustment key), center the condenser on the crosshairs. The sub stage condenser should now be centered on both the cross hairs and the central stop.
Note: If the central stop is not adjustable, the substage condenser should be centered on the central stop, not the crosshairs to enable the best dispersion colors possible.
- 8.4.3.5 Close the condenser until it is almost the same size as the stop, and verify that light is allowed to pass evenly in the same amount, 360° around the stop.
- 8.4.3.6 Open the condenser back up, until it is just as big as the field of view. All sides of the substage condenser should touch the ocular field of view at the same time. Remove the Bertrand Lens / telescoping ocular.
- 8.4.3.7 Check the Condenser Alignment column of the PLM Calibration & Contamination worksheet.

8.4.4 Analyzer / Polarizer Alignment

PLM microscopes will have different ways of achieving the Polarizer and Analyzer alignment; however the objective of this alignment is to:

- Set the polarizer and analyzer privileged directions at 90° angles to each other and
 - To set the polarizer at a known orientation; horizontally across the stage. Some microscopes may have fixed polarizers, fixed analyzers or neither fixed lens. It is important to check with the microscope manual to determine which type of system is used and the type of marking the manufacturer used for this denotation.
- 8.4.4.1 Rotate the polarizer until it is set to have its privileged direction be horizontal across the stage. If the polarizer is moveable there will generally be markings on the polarizer housing either indicating radial measurements (0° - 90° - 180° - 270°) or a mark denoting the 0° measurement. If the polarizer is not moveable it will be set to 0° by the manufacturer.



- 8.4.4.2 Once the polarizer is set, adjust the analyzer so that its privileged direction is 90° off from the polarizer. Again there will be markings on the analyzer such as radial measurements (0°-90°-180°-270°) or a mark denoting the 90° measurement. If the analyzer is fixed this is not necessary.
- 8.4.4.3 Check this by slightly uncrossing either a polarizer or analyzer (but not both). The field of view should be at its darkest at the 90 ° setting. If this is not the case the microscope may need servicing.
- 8.4.4.4 Check the Polarizer / Analyzer 90° column of the PLM Calibration & Contamination worksheet.

8.4.5 Ocular Alignment

The cross hairs of the ocular should be set so they coincide with the privileged directions of both the analyzer and polarizer only after they have been adjusted to 90° offsets in section 8.4.4.

- 8.4.5.1 Using NIST 1867 Anthophyllite (only anthophyllite is compatible with this step) bring an anthophyllite fiber to the center of the field of view at the crosshairs.
- 8.4.5.2 Rotate the Anthophyllite fiber until it comes to extinction horizontally.
- 8.4.5.3 Rotate the ocular containing the crosshair so it coincides with this extinction.
- 8.4.5.4 Rotate the anthophyllite fiber fully through 360° and check that the fiber is extinct at each of the crosshair orientations (0°-90°-180°-270°).
- 8.4.5.5 Check the Ocular Alignment column of the PLM Calibration & Contamination worksheet.

8.4.6 Daily Contamination Check

- 8.4.6.1 In the PLM hood, with a clean mortar and pestle, place a small portion of fine grained table salt (or other non-fibrous, isotropic, confirmed negative friable material) in the mortar.
- 8.4.6.2 Crush the material using the pestle.
- 8.4.6.3 Place a small portion of the material on a clean slide and place a small drop of all oils to be used during the day's analyses on the slide.
- 8.4.6.4 Mix the crushed material, using sequentially all tools that will be used for prepping that day, and place a cover slip on the prep.
- 8.4.6.5 Scan the entire area under the cover slip.
- 8.4.6.6 Check the Daily Contamination Check column of the PLM Calibration & Contamination worksheet.
- 8.4.6.7 *Additionally:* after every 20 uses of any homogenization equipment (ie. mortar and pestle) run a negative material (as described in 8.4.6.1) prep to monitor contamination of the homogenization equipment. It is not necessary to use all tools and liquids for this step, just what is necessary to produce the prep.

8.5 Refractive Index Liquid Calibration

- 8.5.1 Refractive index liquids are calibrated to a precision of ± 0.004.
- 8.5.2 Refractive index calibrations are corrected for temperatures other than 25°C.
- 8.5.3 Refractive Index liquids are calibrated at least upon:
 - 8.5.3.1 Opening the bottle and



8.5.3.2 Additionally all refractive index liquids, including small bottle Cargille sets, are calibrated upon next use if the last calibration has been 3 months ago or longer.

8.5.3.3 For analysis of samples by the NIOSH 9002 method, refractive index liquids, including small bottle Cargille sets, must be calibrated before next use if the last calibration has not been within the last week.

8.5.4 Refractive Index liquids are calibrated using EMSL's RI Liquid Calibration SOP.

8.6 Dispersion Staining Color Verification

8.6.1 At least monthly, the dispersion staining colors or NIST 1866 Amosite are obtained and recorded to verify the microscope's ability to produce dispersion staining colors.

8.6.2 After microscope alignment (following section 8.4 above) place a permanent mount of NIST 1866 Amosite in a 1.68 refractive index medium on the microscope stage.

8.6.3 Follow section 9.6.7.1, obtain dispersion staining colors from the amosite prep.

8.6.4 Translate the observed dispersion staining colors to wavelengths, following step 9.6.7.1.4.

8.6.5 Record the wavelengths corresponding to the parallel and perpendicular directions of the amosite fiber on the PLM Calibration and Contamination record for the microscope used.

9.0 Procedure

9.1 Sample Receipt

9.1.1 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.

9.1.2 If the samples do not have a COC then one is completed at the time of log in. Have the client fill out the necessary information completely.

9.1.3 Information required on the Chain of Custody includes:

9.1.3.1 Client name, address, telephone number, contact person, fax #, e-mail.

9.1.3.2 Project number/ name, state where samples were taken

9.1.3.3 Number of samples sent and sample ID's

9.1.3.4 Type of analysis requested

9.1.3.5 Sample volumes or areas if applicable

9.1.3.6 Turn around time. "RUSH" is not acceptable

9.1.3.7 A date and signature of the person relinquishing the samples

9.1.3.8 All samples MUST be accounted for with the proper sample ID's

9.1.3.9 All samples MUST be sealed, properly bagged and undamaged.

9.1.4 All samples must be clocked in at the time of receipt and signed and dated by an EMSL employee. If the lab does not have a clock for sample receipt the receiving employee should record the time of receipt also.

9.1.5 Check to see if the samples match the COC and if the containers are open, damaged, or contaminated. If the samples are damaged or if the COC does not match, notify the client.

9.2 Sample Log In

If all of the above criteria for sample receiving are met then the samples can be logged in to Sample Master (LIMS) as per the Sample Master SOP.

9.2.1 This process will assign a unique EMSL order number for the project as well as unique lab sample ID's.



9.2.2 Sample Master generates an Internal Chain of Custody and the appropriate bench sheets for the analysis.

9.3 Macroscopic Observation

9.3.1 Regardless of the requested analytical method, all samples are observed both using the naked eye and using a stereo microscope prior to further processing.

9.3.2 In a HEPA and/or fume hood, remove the sample from the sample container (zip lock bag, jar, etc.) and set it on a clean piece of scrap paper or paper towel.

9.3.3 Using a stereoscope when necessary, make and record observations pertaining to the sample including:

9.3.3.1 Sample Color - the sample color of the hand specimen

9.3.3.2 Homogeneity - whether the sample is homogenous or heterogeneous with respect to discernable sample layers. If there is more than one discreet sample layer observed, each layer is analyzed and reported as a separate sample. Compositing, except in the following circumstances is not done:

9.3.3.2.1 Joint compound / Drywall system - are allowed to be composited only if the joint compound is part of the wall system (used for joint and holes only) and not a separate application / layer. This information would only be available to the field sampling entity.

9.3.3.2.2 The sample layers are inseparable to the point that it would be impossible to separate the layers without substantial contamination between the layers.

9.3.3.2.3 When the client requests samples be composited, but only then when accompanied by a report / sample comment indicating the client requested this non-compliant procedure.

9.3.3.3 Whether fibers are visible in the sample hand specimen and the percentage of suspect asbestos fibers present as viewed through the stereoscope.

9.3.3.4 Any other observation that may be helpful in analysis is also recorded.

9.3.4 When samples are to be separated into layers, an aliquot is added to the sample ID in Sample Master and additional worksheets (if needed) are generated. These layers will have unique EMSL lab sample ID numbers for tracking purposes.

9.4 Preliminary Fiber Identification (excluding NYSELAP 198.6)

All prep work should take place in a HEPA / fume hood. Samples proceeding directly with gravimetric reduction (NYS ELAP 198.6, EPA PLM NOB, client's request, etc.) may proceed directly to section 9.5. Preliminary fiber identification may be better performed on the final residue after gravimetric reduction

Note: for those labs adhering to NYS 198.6: following the NYS ELAP regulations, fibers protruding from the matrix may be removed for PLM analysis. If the fibers are identified as asbestos, the client may be notified that the NOB contains an unquantifiable amount of asbestos. However, analysis must continue with a full gravimetric reduction in order to determine the specific percentages.

9.4.1 When possible, fibers observed in the previous macroscopic observation step are mounted in an appropriate RI liquid and identified as part of the qualitative analysis of the sample.

9.4.2 Using clean tools (forceps, probes, scalpel, etc.) isolate a fiber and remove it from the sample, place the fiber on a clean microscope slide.



- 9.4.3** Place a drop or two of an appropriate RI liquid on top of the fiber. The appropriate RI liquid is determined by the suspect fiber identification. If the fiber is suspected to be:
- 9.4.3.1** Chrysotile - use 1.550 RI oil
 - 9.4.3.2** Amphibole - use 1.680 RI oil
 - 9.4.3.2** Non-Asbestos - use 1.550 RI oil
- 9.4.4** Using prep tools immerse and modify (chop, cut up, etc.) the fibers to disperse them in the RI liquid. Place a cover slip on the fiber prep and tap gently with a pencil eraser or other tool to expel any remaining air bubbles under the cover slip.
- 9.4.5** Mount the slide on the PLM stage and following the steps in Section 9.6 Fiber Identification, determine if the fiber's identity was consistent with the suspected identification.
- 9.4.6** After observation of the fiber's optical properties, determine if the fiber was indeed the suspected fiber type.
- 9.4.6.1** If the fiber was consistent with the suspected identification, record all required optical properties (as specified in section 9.6) on the analytical worksheet. Repeat steps 9.4.2 to 9.4.5 until all fibers present in the sample have been identified.
 - 9.4.6.2** If the fiber was not consistent with the suspected fiber type, but the optical properties of the fiber were able to be measured, leading to a positive identification of another fiber type, record all required optical properties (as specified in section 9.6) on the analytical worksheet. Repeat steps 9.4.2 to 9.4.5 until all fibers present in the sample have been identified.
 - 9.4.6.3** If the fiber's optical properties were not able to be measured satisfactorily because the oil chosen in step 9.4.3 was not conducive to obtaining the optical properties, return to step 9.4.3 and remount more of the suspect fibers in another oil. Suspected amphibole fibers that are not identified as amosite or crocidolite but remain suspected amphiboles, should be mounted in 1.605 RI oil. Repeat steps 9.4.2 to 9.4.5 until all fibers present in the sample have been identified.
 - 9.4.6.4** If the fibers' optical properties were not able to be measured satisfactorily due to interferences of matrix, coatings, etc. an alternate prep technique (such as gravimetric reduction) may be necessary. Alternate prep techniques are discussed in section 9.5. Solvent washing (acetone, RI liquid, etc.) may be required to clean fibers or bundles sufficiently prior to identification. Fresh RI liquid must be used after the cleaning for identification.

9.5 Alternate / Specialized Preparations - Gravimetric Reduction

A full gravimetric reduction is required by NYS ELAP 198.6, and as requested by the client (ie. EPA PLM NOB). Certain material may require parts or all of this procedure for correct identification and quantitation of the sample. Preparation steps where the sample is outside of a sealed container (zip lock bag, crucible with top, etc.) should take place in either a safety or fume hood. All steps that involve the use of acid should take place in a fume hood.



- 9.5.1 Place the samples into the order noted on the COC, initial the EMSL internal chain of custody in the space "Prepped By" and date. All personnel involved in the prep process should add their initials and appropriate date here.
- 9.5.2 For each sample assign a clean, pre-weighed crucible and a pre-weighed petri dish with a 0.4 μ m PC filter. Record the weight of the empty crucible and petri dish on the analytical bench sheet corresponding to the sample in locations A and D respectively.
- 9.5.3 Label the petri dish with the EMSL order ID and sample number, or optionally the EMSL order ID and client sample number, and the crucible ID or crucible location in the tray/rack. Mount each filter in a filtration funnel setup and store with the labeled petri dish.
- 9.5.4 Following steps in section 9.3 above, and after stereoscopic examination, record the following sample observations on the analytical bench sheet.
- 9.5.4.1 Sample color.
- 9.5.4.2 Whether the sample is homogeneous or heterogeneous.
- 9.5.4.3 Whether the sample is fibrous or non-fibrous.
- 9.5.5 Always separate layers (ie. floor tile and mastic, etc.) and prepare separately, if possible without significant cross contamination.
- 9.5.6 If the client requests sample layers to be combined:
- 9.5.6.1 Verify that the client's request is in writing or otherwise documented.
- 9.5.6.2 Combine layers during preparation, prior to gravimetric reduction.
- 9.5.6.3 Sample a representative amount of each layer when combining layers.
- 9.5.6.4 Note this clearly on the analytical bench sheet. **This must be commented on the final report.**
- 9.5.7 Remove between 100 to 500 mg of sample and place into the crucible. The sub sample should be taken so that it represents the overall composition of the sample and is treated in such a way (shaving of tiles, crushing, etc) to maximize the surface area of the material being placed in the crucible.
- 9.5.8 If the sample does not consist of at least 100mg of material, this is considered to be "Insufficient Sample" and should not be processed. It can sometimes be difficult to acquire this much mastic from the back of a tile but is crucial for accurate results. Comment the analytical bench sheet to indicate that there was "Insufficient Sample". The client may be contacted and asked to submit more sample. However if the client requests, we will process the sample "as is" provided the following:
- 9.5.8.1 There is at least 20mg of sample to process and
- 9.5.8.2 The sample result on the report is noted "Sample below method recommended minimum weight, analyzed at client's request"
- 9.5.9 Weigh the crucible in the analytical balance and record the weight of the crucible and unashed sample on the corresponding analytical bench sheet in location B.
- 9.5.10 Place the crucible in a labeled rack or tray, this may be a specialized crucible rack or simply a 'muffin tin'. A crucible map is filled out for each of the trays and the tray ID and sample numbers are filled in on the map. Samples are placed in the rack or tin beginning at the top left and proceeding across and down (as if reading a book). Alternately, crucibles may be labeled with special marking ink and the individual crucible IDs used to track location. Labeling in this manner requires crucibles to be baked at extremely high temperatures before use.
- 9.5.11 Repeat this process (steps 9.5.2 to 9.5.10) for all samples to be processed.



- 9.5.12** Place the crucibles into a pre-heated muffle furnace at 480°C for 12 hours. *(note: In general 12 hours is a good starting point, but for smaller sample sets and samples of amenable matrix compositions, shorter ashing times may be possible. NYS ELAP item 198.6 suggests anywhere between 1-12 hours for the mass to stabilize and EPA 600/R-93/116 offers six hours at 450°C is usually sufficient. In practice, 4 hours is insufficient for most roofing and some other types of samples. The ashing time is dependent on available sample surface area as a result of the sample preparation techniques used.)*
- 9.5.13** Remove samples from the furnace and allow to cool to room temperature, re-weigh the crucible with sample and record this weight on the analytical bench sheet in location C.
- 9.5.14** In the fume hood, arrange the crucibles and filtration funnel setups that were loaded with the pre-weighed filters, in sample order. Add approximately 0.5 ml of water to each crucible and grind the sample in the crucible using the flat end of a clean pestle. After grinding, rinse the pestle with a minimum of water to wash any remaining sample on the pestle back into the crucible.
- 9.5.15** Carefully add 2-5 ml of concentrated HCl to the crucible so that any reaction between the sample and the acid does not overflow the crucible. Allow the sample and acid to react for 15 minutes. Prolonged exposure of chrysotile fiber to acid will result in fiber damage that will adversely affect the ability to identify these fibers.
- 9.5.16** Dilute the sample with more water and filter through the associated pre-weighed funnel setup. Rinse the crucible with water to ensure that all sample has been transferred. Rinse the sides of the funnel so that no sample is left on the funnel after the filter is removed.
- 9.5.17** Remove the filter from the funnel setup and place it in its associated labeled petri dish. Allow the filter to dry in the petri dish in a drying oven or slide warmer for 15 minutes at about 40-60°C. Check for dryness by inspecting the petri dish cover for condensation.
- 9.5.18** Once dry, remove from the heat sources and allow to cool to room temperature. Re-weigh the petri dish and record the weight on the analytical bench sheet in location E.
- 9.5.19** Perform calculations on the analytical bench sheets to determine the weight of the original sub-sample (W1), weight of the post-ash residue (W2) and the weight of the post-acid residue (W3) Also, the calculations for percent organics, percent acid soluble and final residue should be performed, see Section 10 Calculations for details.

9.6 Fiber Identification

- 9.6.1** Suspect fibers classified as asbestos must be identified using the following properties.
- 9.6.1.1** Morphology
 - 9.6.1.2** Sign of Elongation
 - 9.6.1.3** Color
 - 9.6.1.4** Pleochroism
 - 9.6.1.5** Extinction
 - 9.6.1.7** Refractive Index
 - 9.6.1.8** Birefringence



- 9.6.2** For suspect fibers classified as non-asbestos, at least one of the above properties must be measured and recorded that would fall outside of normal asbestos classification ranges.
- 9.6.3** Morphology
- 9.6.3.1** The morphology of suspect fibers should be such that fibrocity is evident from its morphology. This may be including the value of aspect ratio and three dimensional aspects of morphology that may not be readily noticeable at first.
- 9.6.3.2** Suspect Chrysotile: will have morphology that is generally silky and wavy. Chrysotile fibrils are never visible under light microscopy and all chrysotile under the PLM are bundles. Larger bundles may exhibit large waves and/or kink bands. The ends of bundles may be splayed; portions of extremely long bundles may split and rejoin at a later point in the bundle.
- 9.6.3.3** Suspect Amphiboles: will have morphology that is straighter and more rod like than chrysotile. The end of amphibole bundles may have broom like properties with a small amount of splaying. Very long amphibole asbestos will show curvature and bending, but with more rigidity than chrysotile.
- 9.6.3.4** For EPA 600/R-93/116 and NYS ELAP 198.1 & 198.6, Asbestiform Morphology is generally recognized by the following:
- 9.6.3.4.1** Mean aspect ratio from 20:1 to $\geq 100:1$
 - 9.6.3.4.2** Thin fibrils, usually $< 0.5\mu\text{m}$ in width
 - 9.6.3.4.3** Two or more of the following:
 - 9.6.3.4.3.1** Parallel fibers occurring in bundles
 - 9.6.3.4.3.2** Bundles displaying splayed ends
 - 9.6.3.4.3.3** Matted masses of individual fibrils
 - 9.6.3.4.3.4** Fibers showing curvature

Note: (From EPA 600/R-93/116 Appendix A. Glossary Of Terms, Asbestiform Morphology (last paragraph) also applying to NYSDOH ELAP 198.1 & 198.6.

"These characteristics refer to the population of fibers as observed in a bulk sample. It is not unusual to observe occasional particles having aspect ratios of 10:1 or less, but it is unlikely that the asbestos component(s) would be dominated by particles (individual fibers) having aspect ratios of $< 20:1$ for fibers longer than $5\mu\text{m}$. If a sample contains a fibrous component of which most of the fibers have aspect ratios of $< 20:1$ and that do not display the additional asbestiform characteristics, by definition the component should not be considered asbestos."

- 9.6.3.5** For NIOSH 9002 asbestos fiber aspect ratios are:
- 9.6.3.5.1** Typically $> 10:1$ for fibers found to be chrysotile, amosite and/or crocidolite.
 - 9.6.3.5.2** Typically $< 10:1$ for fibers found to be anthophyllite or actinolite/tremolite.
- 9.6.3.6** Other morphological types (bladed, acicular, etc.) may at first appear to be fibrous but careful examination will show the morphology to be other than fibrous (thinning thickness across the width of bladed particles, tapered ends on acicular particles).
- 9.6.4** Sign of Elongation
- 9.6.4.1** While viewing the fiber the sign of elongation of a fiber may be measured in crossed polarized light.



- 9.6.4.2** Insert the first order red 1 plate into the microscopes accessory port (at 45° to the analyzer and polarizer).
- 9.6.4.3** When the fiber exhibits a blue color in the NE-SW orientation and yellow in the NW-SE orientation the sign of elongation is positive. For these fibers the refractive index of the fiber parallel to the elongation (n_{\parallel}) is greater than the refractive index perpendicular to the elongation (n_{\perp}). Chrysotile, amosite, anthophyllite, tremolite and actinolite have positive signs of elongation.
- 9.6.4.4** When the fiber exhibits a yellow color in the NE-SW orientation and blue in the NW-SE orientation the sign of elongation is negative. For these fibers the refractive index of the fiber parallel to the elongation (n_{\parallel}) is less than the refractive index perpendicular to the elongation (n_{\perp}). Crocidolite has a negative sign of elongation.
- 9.6.4.5** Isotropic fibers (fibers in which n_{\parallel} and n_{\perp} are equal) do not exhibit colors while being viewed with the red plate, but remain red. They should be the same color as the background area of the slide.

9.6.5 Fiber Color / Pleochroism

- 9.6.5.1** Fiber color is the color that the fiber has in plane polarized light. That is light that is polarized with a sub stage polarizer but without the analyzer or red plate in place during fiber viewing. Pleochroism is when the fiber will change color while rotated on the PLM stage.
- 9.6.5.2** While viewing the fiber observe the fiber color present. Many fibers are clear in plane polarized light.
- 9.6.5.3** Rotate the stage slowly 360° until the fiber has returned to its original orientation. Observe if the fiber exhibited pleochroism and the colors present along with their orientations (parallel or perpendicular to the fiber length).

9.6.6 Extinction

- 9.6.6.1** When the crystallographic axis of a fiber (anisotropic) coincides with the privileged vibration directions of the polarizer and analyzer (in crossed polar orientation), the fiber will appear dark. When rotating the fiber from other orientations to, and through, one where the axes coincide with the polar / analyzer privileged directions the fibers will first be light and then appear dark and then light again. This is called an extinction because it appears that a light within the fiber has been extinguished. An anisotropic fiber will have 4 extinctions during a 360° rotation.
- 9.6.6.2** Extinction angles are measured by rotating the fiber so that fiber length is parallel to one of the cross hairs and noting the angular reading on the stage.
- 9.6.6.3** Next rotate the stage until the fiber comes to extinction. The extinction is always <45°, if the extinction is >45°, return to the original angular measurement and rotate the stage in the opposite direction. Note the angular reading of the stage. The difference in the two angular readings is the extinction angle.
- 9.6.6.4** Extinctions may be classified as follows:
- 9.6.6.4.1** Parallel extinction is when a fiber that goes extinct when the fiber is parallel to the vibration directions of the polarizer and analyzer. Chrysotile and anthophyllite have parallel

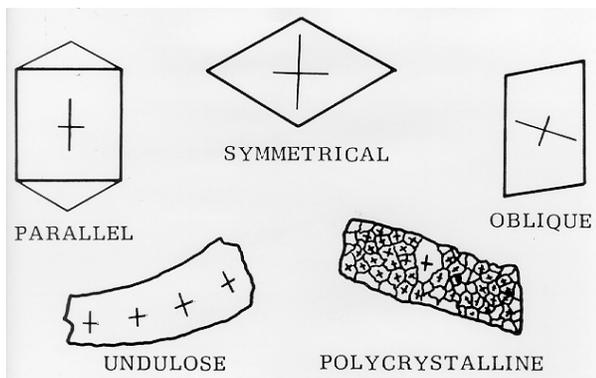


extinction. Amosite and crocidolite may often exhibit parallel extinction due to twinning.

9.6.6.4.2 Oblique or Inclined extinction is when a fiber goes extinct at an angle greater than 0° but less than 45° of the fiber's elongation being parallel to the vibration direction of the polarizer and analyzer. All of the monoclinic amphibole asbestos varieties possess inclined extinction, however due to twinning; amosite and crocidolite rarely exhibit inclined extinction and usually present parallel extinction.

9.6.6.4.3 Symmetrical extinction is when the extinctions bisect the prominent interfacial angles. Some gypsum fibers exhibit symmetrical extinction.

9.6.6.4.4 Undulose extinction may occur in a curved particle, it is truly a parallel extinction at any given point but due to the curve, the extinction band has the effect of fanning across the fiber as it is rotated Chrysotile may show good undulose extinction.



9.6.7 Refractive Index

Refractive index may be measured either using dispersion staining or Becke lines. Practically, dispersion staining is used for most samples for its speed and ease of use. Dispersion Staining determinations can usually be made from a single prep (provided the right oil is selected) as opposed to Becke line determination that may need to use multiple (up to 5 or more) preps from different RI liquids. Dispersion staining will be outlined following Rapidly and Accurately Determining RIs of Fibers Using Dispersion Staining - Dr. Su 1994. The use of the more recent version (2003) of this procedure is optional, and has significant changes.

9.6.7.1 Dispersion Staining (Central Stop)

9.6.7.1.1 Mount the suspect fiber in the appropriate RI oil for the suspected asbestos type (see section 9.4.3 to 9.4.6). Oil selections to keep in mind for fiber identification are:

- 9.6.7.1.1.1** 1.550 for suspect chrysotile
- 9.6.7.1.1.2** 1.680 for amosite
- 9.6.7.1.1.3** 1.700 for crocidolite (optionally 1.680)
- 9.6.7.1.1.4** 1.625 for anthophyllite, actinolite, tremolite (1.605 optional)



- 9.6.7.1.2** Using the dispersion staining objective (central stop) bring the fiber to the center of the screen and focus the fiber. If inserted, remove the red compensator plate. Decrease the illumination of the microscope and close the sub stage condenser so it is just as big as the central stop as viewed through the Bertrand lens.
- 9.6.7.1.3** Remove the Bertrand lens and the analyzer, view the fiber. Increase the illumination as needed and/or adjust the sub stage condenser to yield the fullest, most vibrant colors.
- 9.6.7.1.4** Rotate the stage fully through 360°, then stop at the perpendicular fiber direction. Using a wavelength color chart for central stop dispersion staining (McCrone or other), translate the observed dispersion color to its corresponding wavelength from this chart. DS colors should be obtained on several fibers. Repeat this process for the fiber in the parallel direction. The longest (higher) wavelength in each direction should be used for conversion to a refractive index in the following steps.
- 9.6.7.1.5** Using the applicable chart corresponding to the suspect asbestos type and RI oil used, as available in Su 1994, look up the corresponding refractive index for the fiber's perpendicular orientation DS color wavelength and the ambient room temperature at the time of DS color observation. For wavelengths between listings and/or temperatures, extrapolate the RI for the non-listed wavelength. Repeat for the RI of the fibers parallel orientation.
- 9.6.7.2** Becke Line
- 9.6.7.2.1** Mount the suspect fiber in the appropriate RI oil for the suspected asbestos type (see section 9.4.3 to 9.4.6)
- 9.6.7.2.2** View the fiber using an objective that magnifies the fibers so it is large enough to be easily viewed in the center of the field of view. In general, the 20x objective is adequate, but if they provide better viewing the 10x or 40x objectives may also be used. Center the fiber in the field of view, remove the red compensator (if present), remove the analyzer and focus.
- 9.6.7.2.3** While observing the fiber, rotate the fine focus knob back and forth and notice the bright shadow or halo, emanating near the fiber. This is a Becke Line.
- 9.6.7.2.4** Orient the fiber in the perpendicular orientation and with continued movement notice if the halo appears to move out of, or into the fiber during the fine focus rotation. When the stage is lowered the Becke line will move into the higher refractive index.
- 9.6.7.2.5** Note the RI Oil used and whether the fiber was > or < the oil in this perpendicular orientation.



- 9.6.7.2.6** Change the fiber orientation to parallel and repeat step 9.6.7.2.4. Note the RI Oil used and whether the fiber was > or < the oil in this parallel orientation.
- 9.6.7.2.7** At this point we have only determined whether the fiber is > or < than the oil in both orientation. Next it is necessary to make additional preps of suspect fibers. The oil used for these preps are not predetermined; it is based on the results from our Becke line determination from the original prep. It may be helpful at this time to address only one orientation at a time. In this manner, choose an orientation (perpendicular or parallel is not important, this will be the first orientation) to perform RI determination on using Becke lines, and only measure this orientation until the determination is complete. After this determination is done, the second orientation can be measured and further preps made for this second orientation. Also, Becke line determination will require the use of many other RI oils besides the typically used common oils. The use of small bottled Cargille oil sets will be necessary, and it should be noted that each oil must be calibrated prior to use if the last calibration for the particular oil was more than three (3) months prior, weekly if performing NIOSH 9002 analysis. RI Oil calibration should be done following EMSL's RI Calibration SOP 2008 05 05.
- 9.6.7.2.8** In the first orientation, if the fiber was < then the oil, prep another suspect fiber in an oil that is lower than the original oil, otherwise choose a higher oil. The amount of relief (the amount that the fiber "stands out" in the prep) should indicate what range of oil to use. The more the fiber stands out, the farther away from the original oil the new oil should be.
- 9.6.7.2.9** Orient the fiber in the first orientation and focus back and forth, notice the direction of the Becke line movement when the stage is lowered. Note whether the fiber is > or < than the oil.
- 9.6.7.2.10** Repeat steps 9.6.7.2.8 and 9.6.7.2.9 until the fiber in the first orientation has been bracketed by two oils. The Cargille RI oil sets from 1.460 to 1.700 are in 0.004 RI increments and from 1.700 to 1.720 are in 0.005 RI increments.
- 9.6.7.2.11** Once the fiber is bracketed between two oils (first orientation only) look at the fiber in both oil preps again. Observe the amount of relief that is present in both preps.
- 9.6.7.2.11.1** If the relief is almost identical in both preps the refractive index of the fiber in the first orientation can be assumed to be mid-point between the two oils.
- 9.6.7.2.11.2** If the relief is vastly different and the fiber is almost completely unnoticeable in one of the preps the refractive index of the fiber is very close to that oil, and can safely be recorded "as" that oil's RI value.



9.6.7.2.11.3 If the relief is closer to one oil than the other, extrapolate the refractive index between the two bracketing values (ie. the bracketing values are 1.692 and 1.696, and the fiber is closer to 1.692 but not exactly there, report value as 1.693).

9.6.7.2.12 Repeat steps 9.6.7.2.8 to 9.6.7.2.12, substituting the second orientation for the first orientation.

9.6.8 Birefringence

9.6.8.1 Birefringence is defined as the difference between the highest and lowest refractive index of a particle.

9.6.8.2 The highest refractive index is always γ regardless of orientation \parallel or \perp .

9.6.8.3 Subtract $n_{\gamma} - n_{\alpha}$ to determine birefringence.

9.6.9 Classification

9.6.9.1 All characteristics should fall within the known acceptable limits for regulated asbestos species. These references may be found in section 16 of this procedure. Known regulated asbestos varieties are:

- 9.6.9.1.1** Chrysotile
- 9.6.9.1.2** Amosite (Grunerite)
- 9.6.9.1.3** Crocidolite (Riebeckite)
- 9.6.9.1.4** Anthophyllite
- 9.6.9.1.5** Actinolite
- 9.6.9.1.6** Tremolite

9.6.9.2 If any one of the characteristics falls outside of, or is inconsistent with a suspected asbestos type, the fiber must be characterized as non-asbestos.

Note: Through many processes, asbestos may be altered during its life as a building material. Some of these processes may alter some of the physical characteristics and properties of the fibers in building material samples. Even if the fiber is suspected to have at one time been a regulated asbestos variety, if at the time of analysis the fiber's properties have been altered to such an extent that one, some or all of them fall outside accepted ranges, the fiber may not be categorized as a regulated asbestos variety for the purpose of analytical reporting. Please see EMSL Policy Statement, 03/08/2007, Heat or Otherwise Altered Asbestos.

9.7 Slide / Sub Sample Preparation

The following sections outline possible prep procedures. There are numerous different ways to prepare samples for analysis and other procedures, while not outlined here, are not excluded from use.

9.7.1 Homogenization

9.7.1.1 Samples that are not so already should be homogenized before sub sample preparation. A number of homogenization procedures are available, including, forceps, scalpel, probes, mills and mortar and pestle.

9.7.1.2 The object of homogenization is to alter the sample so that all areas are represented, representatively in the sub sample preps.

9.7.1.3 Mortar and pestle are the most useful for cementitious samples. This is useful in both homogenization and in grain size reduction to help cover slips sit parallel to the slide. A representative sub sample may be placed in the mortar and crushed with the pestle until the sample is of substantially uniform distribution and grain size.



9.7.1.4 Another process of homogenization may be milling. If available at the lab this may be used and it takes the use of a mortar and pestle a step further.

9.7.1.5 Additionally, probes, forceps and scalpels may be used to rip, tear and cut portions of sample and they continually work the portions until the sub sample is homogeneous in nature.

Note: When grinding samples that contain amphibole minerals or vermiculite, care should be taken during analysis. Non-asbestiform amphiboles may be ground into cleavage fragments that are >3:1 in aspect ratio. One way to distinguish these fragments from asbestiform amphiboles is by using their morphological properties to determine the presence or absence of asbestiform habit in the fiber population. Asbestos mineral species that do not exhibit asbestiform habit are not considered regulated asbestos. Grinding of vermiculite may produce vermiculite fibers (scrolls) that may appear to look similar morphologically to chrysotile, but which may be distinguish on the basis of their optical properties.

9.7.2 Pinch Mount

9.7.2.1 After homogenization (if required) the sample should be of uniform grain size and distribution. A "pinch" of the sample may be taken and combined with a drop or two of 1.550 oil on a clean glass microscope slide. Fiber identification has already been performed so unless other oils are necessary for fiber distinction, 1.550 oil is used for sample prep.

9.7.2.2 Disperse the sample in the oil with a clean scalpel blade. Further reduce the sample grain size if necessary with the scalpel blade.

9.7.2.3 Once the sample has become completely and evenly dispersed in the oil, place a cover slip on top of the sample. Lightly tap the top of the cover slip with the back of the scalpel handle or pencil eraser to help even the prep out.

9.7.2.4 If there are air pockets under the cover slip, that do not go away with tapping, additional oil may be added at the side of the cover slip. Capillary action should wick the oil under the cover slip filling the air pockets. Once again, as in step 9.7.2.3, lightly tap the cover slip to disperse the new oil throughout the prep.

9.7.3 Melting

9.7.3.1 The sample may contain significant organically bound matrix. If this is the case the best path for preparation is a full gravimetric reduction (see section 9.5 above).

9.7.3.2 If this is not an option, the organically bound matrix may be melted and the encumbered material may be freed for analysis.

9.7.3.3 It is most likely that homogenization will not be effective on these types of samples (floor tiles, mastics, caulks, roofing, etc.) so homogenization should be skipped. It is imperative however, to fully sample all portions of the hand sample to account for any sample compositional differences within the material. In general this is dependent on the material.

9.7.3.3.1 Floor tiles should be broken to reveal a fresh edge and cross scraped. Place a small portion of the scrapings on a clean slide.

9.7.3.3.2 Mastics and caulks may be assumed to be fairly homogeneous, but if available, fresh surfaces should still be



chosen for sampling. The material chosen should be placed on a clean slide.

9.7.3.3.3 Separable roofing material, as with all samples, individual layers are prepared and analyzed separately. Various areas of the layer should be selected in representative amounts to their presence in the sample. The sample must both (a) be sufficient in size and quantity to adequately represent the sample during analysis and (b) not be of overloading quantity so that the prep is unreadable due to excess material on the slide.

9.7.3.3.4 Inseparable roofing may contain many layers (layered, rolled roofing) however, due to the nature of the matrix (thick and sticky binding material) separation may not always be accomplished without significant contamination. In this case the sample should be representatively sampled throughout all materials. These samples are possibly the most challenging material to prepare representatively, and as above care should be taken so the sample prep will both (a) be sufficient in size and quantity to adequately represent the sample during analysis and (b) not be of overloading quantity to that the prep is unreadable due to excess material on the slide. Sampled material should be placed on a clean microscope slide.

9.7.3.4 Smearing a small amount of mastic on a slide prior to adding RI oil may expose fibers to be examined.

9.7.3.5 Place a drop or two of oil on the samples and disperse the sample in the oil with a clean scalpel blade.

9.7.3.6 Place a cover slip on the sample and with the back of an eraser, push the cover slip both downward and laterally around to both even the cover slip so it lays flat and to distribute the material under the cover slip.

9.7.3.7 Place the slide on a slide warmer / hot plate inside the hood and allow it to sit for a minute or two, until the organically bound material has been melted slightly. The temperature of the slide warmer should be only warm enough to melt the matrix. Excess heat will cause the oil to boil and should be avoided.

Note: If suspect fibers were not able to be identified previously, care should be used in letting the sample/oil cool to room temperature before RI determinations are made. Dissolved mastic/tar will change the refractive index of the RI liquid in which the fiber is mounted. If discoloration of the liquid is noted it may be "wicked" out from under the cover slip with a piece of paper towel. Fresh liquid can then be drawn under the cover slip by capillary action.

9.7.3.8 Once the matrix material had melted, and cooled slightly, rub the sample between the cover slip and slide to help the hidden particles free themselves from the organic binder.

9.7.4 Dissolve

9.7.4.1 Samples that contain high percentages of calcium carbonate or other acid soluble material may cause sample components to be obscured and difficult



to work with. After homogenization one way to quickly remove this component is by using a low concentration (10-50%) acid solution to dissolve the acid soluble fraction of the sample before observation. Two cautions are necessary for this process

- 9.7.4.1.1** If suspect material is found, quantitation must include gravimetric reduction to determine the percent of acid soluble material removed prior to quantitation.
- 9.7.4.1.2** Use of acid must take place in vented hood.
- 9.7.4.2** Acid removal of soluble material may take place in two ways; with the use of gravimetric tracking or with out. If gravimetric tracking is to be used, refer to the procedure outlined in section 9.5 of this method, skipping steps 9.5.11 and 9.5.12, and then proceed with fiber identification (9.6) and prepping via pinch mount (9.7.2).
- 9.7.4.3** The removal of acid soluble matrix material without gravimetric tracking should be considered qualitative at best. This is however a good tool to guard against false negatives. To remove acid soluble matrix material without gravimetric reduction, take a pinch or two of sample and place it on a clean microscope slide.
- 9.7.4.4** Place a drop or two of diluted acid (10-50%) on the sample, mix the sample and acid with the scalpel.
- 9.7.4.5** Allow the reaction to come to completion, no further bubbling, and afterwards place a cover slip on the preparation. It should be noted that even when there is no further noticeable reaction, there may in fact be a slight reaction progressing. This will be evident after the cover slip has been placed on the sample if the preparation develops numerous and enlarging air bubble/pockets. If this is the case, prepare another slide and allow the reaction to proceed further before a cover slip is placed on the slide.
- 9.7.4.6** It is absolutely necessary to place a cover slip on the preparation before observation. Failure to do so will subject the microscope to acid vapors and cause corrosion of the objectives, stage, etc. This will be considered purposeful damage of company property, and is in fact very poor lab practice, and use of acid in this manner is forbidden.
- 9.7.4.7** The slide is now ready for quantitative evaluation as detailed in section 9.8.

9.8 Quantitation

- 9.8.1** Quantitation may take place using a number of methods as required by particular methods or by the client. Some analytical methods may be combined with gravimetric reduction, the quantitation methods available are:
 - 9.8.1.1** Calibrated Visual Estimate
 - 9.8.1.2** EPA 400 Point Count
 - 9.8.1.3** EPA 1000 Point Count
 - 9.8.1.4** EPA PLM NOB may utilize either calibrated visual estimate or one of the EPA Point Count procedures above.
 - 9.8.1.5** NYS Stratified Point Count
 - 9.8.1.6** NYSDOH ELAP 198.6 utilizes NYS Stratified Point Count
 - 9.8.1.7** NIOSH 9002 Estimation Reported as a Range



Note: For any quantitation method, when samples of problematic matrices are determined to be <1% and approach or are near the 1% level, further / alternative analysis may be recommend when other technologies and/or procedures are likely to produce more accurate results.

9.8.2 Identification of fibrous components should already have been completed in section 9.4 and 9.6 above. If new fibrous species are encountered, all required criteria in section 9.6 must be met.

9.8.3 Calibrated Visual Estimate

9.8.3.1 For calibrated visual estimate scan the entire area under the cover slip on the slide prepared in section 9.7 above. Scanning should be done with a combination of 4x-10x-20x-40x objectives as dictated by the sample. Higher magnifications are appropriate for finer grained samples that are difficult to see at lower magnification. Lower magnification may be more appropriate for large grain sizes. The object is to be able to see good representation of the sample in any given field of view. The process of switching back and forth between objectives is encouraged, however, the entire slide should be scanned in it's entirety at least once using one magnification.

9.8.3.2 Observe the slide and determine the percentage of the regulated asbestos portion of the sample to the rest of the sample, both fibrous and non-fibrous. It may be helpful to keep in mind these relative ratios when doing this:

- 9.8.3.2.1** Fibrous to Non-Fibrous components
- 9.8.3.2.2** Regulated Asbestos to other Fibrous components
- 9.8.3.2.3** Do not take empty space into account
- 9.8.3.2.4** Refer to any standards you may need (daily reference slides, old proficiencies, manufactured predetermined asbestos containing samples, drawings, point counted percentages, etc.) where the percentage of target fibers is know. Refer to samples with the same asbestos type(s) that are present in the sample.

9.8.3.3 Once you have completed observation of the first slide, return to step 9.7 and prepare another slide and repeat section 9.8.3.1 to 9.8.3.2 above

9.8.3.4 Repeat step 9.8.3.3 until you have completed estimation of the sample using at least (more if you feel it necessary) three (3) slide preps of the sample. If you are unsure of the final sample quantitation, more preparations may be processed.

Note: For friable samples and ashed residues this will ensure that the equivalent of at least 1000 non-empty points have been counted and will ensure an analytical sensitivity of $\leq 0.1\%$ for None Detected samples.

- 9.8.3.4.1** Estimated percentage of total asbestos (all types) in the sample
- 9.8.3.4.2** Estimated percentage of each asbestos component
- 9.8.3.4.3** Estimated percentage of each non-asbestos fibrous component



- 9.8.3.4.4** Estimated percentage of each non-fibrous component (if categorized)
- 9.8.4 EPA 400 Pt. Ct.**
- 9.8.4.1** Point counting is a petrographic technique in which a single point (cross hairs) or a point array (Chalkley for example) is/are superimposed on the sample.
 - 9.8.4.2** Slides prepared for point counting should be preferably only 1 layer thick of sample. The sample should be disperse enough so overlapping areas of the slide are rare, but the slide is still mostly covered with sample.
 - 9.8.4.3** Point counting should be done at a magnification that is dictated by fiber visibility. Higher magnifications are appropriate for finer grained samples that are difficult to see at lower magnification. Lower magnification may be more appropriate for large grain sizes. The object is to be able to see good representation of the sample in any given field of view.
 - 9.8.4.4** The sample is moved to a random field of view and the area that is directly below each point or points is counted as a point. Only points that are superimposed on a particle (non-empty point) are counted. Points that fall on / are superimposed over an unoccupied area (empty point) should not be counted; the closet particle identity is not used to count empty points.
 - 9.8.4.5** Each non-empty point is classified as a regulated asbestos point or a non-asbestos particle. Categories of non-asbestos particles may be subcategorized during the point count (ie. as cellulose, fiberglass, matrix, etc.) but it in not necessary to point count non-asbestos fiber types, their quantity may be estimated.
 - 9.8.4.6** Points that are superimposed over two individual particle types (ie. asbestos and non-asbestos) should be counted as 1 point for each particle type.
 - 9.8.4.7** When a point(s) are superimposed on an area that has several overlapping particles, the slide should be moved to another field.
 - 9.8.4.8** After the point / all points in a field of view are counted move to the next field of view. This should be done dependent on the type of reticule in use:
 - 9.8.4.8.1** Cross-hair: When using a cross hair reticule, new fields should be selected along multiple parallel traverses of the sample preparation.
 - 9.8.4.8.2** Point Array: When using a point array reticule, new fields should be selected on the basis of sampling as much of preparation as possible.
 - 9.8.4.9** If asbestos is encountered, but does not happen to be counted as a point, its presence should be noted.
 - 9.8.4.10** Point counting should continue until 400 non-empty points are counted. These should be evenly spread over a minimum of two (2) preps to a maximum of 8 preps. Once the required number of counts has been tallied from the first prep, return to step 9.7 and prepare and analyze additional slides as needed until 400 points have been reached.
 - 9.8.4.10.1** EPA 600/R-93/116 is the most recent method for the identification of asbestos in bulk building materials and stipulates the requirements stated above in section 9.8.4.10



- 9.8.4.10.2** EPA 600/M4-82-020 was the interim method prior to EPA 600/M-82-020. If this method is being used the point count must take place using 8 slide preparations.
- 9.8.4.11** While point counting inherently assumes the presence of asbestos, a point count may be performed on a negative sample to demonstrate compliance with a desired analytical sensitivity. In this manner, 400 points are counted as outlined above in step 9.8.4.1 to 9.8.4.10, however, a minimum of 3 preparations must be point counted and scanned in their entirety as outline in section 9.8.3 to demonstrate a None Detected result.
- 9.8.5 EPA 1000 Pt Ct**
- 9.8.5.1** When it is desirable, either by agency regulation, or by client request, to provide a lower analytical sensitivity the number of points counted during a point count (as described in section 9.8.4 above) can be increased. This is effective in increasing the accuracy of results under 1% and can also meet depressed action limits (ie. 0.1% asbestos).
- 9.8.5.2** Slides prepared for point counting should be preferably only 1 layer thick of sample. The sample should be disperse enough so overlapping areas of the slide are rare, but the slide is still mostly covered with sample.
- 9.8.5.3** Point counting should be done at a magnification that is dictated by fiber visibility. Higher magnifications are appropriate for finer grained samples that are difficult to see at lower magnification. Lower magnification may be more appropriate for large grain sizes. The object is to be able to see good representation of the sample in any given field of view.
- 9.8.5.4** The sample is moved to a random field of view and the area that is directly below each point or points is counted as a point. Only points that are superimposed on a particle (non-empty point) are counted. Points that are that fall on / are superimposed over an unoccupied area (empty point) should not be counted; the closet particle identity is not used to count empty points.
- 9.8.5.5** Each non-empty point is classified as a regulated asbestos point or a non-asbestos particle. Categories of non-asbestos particles may be subcategorized during the point count (ie. as cellulose, fiberglass, matrix, etc.) but it in not necessary to point count non-asbestos fiber types, their quantity may be estimated.
- 9.8.5.6** Points that are superimposed over two individual particle types (ie. asbestos and non-asbestos) should be counted as 1 point for each particles type.
- 9.8.5.7** When a point(s) are superimposed on an area that has several overlapping particles, the slide should be moved to another field.
- 9.8.5.8** After the point / all points in a field of view are counted move to the next field of view. This should be done dependent on the type of reticule in use:
- 9.8.5.8.1** Cross-hair: When using a cross hair reticule, new fields should be selected along multiple parallel traverses of the sample preparation.
- 9.8.5.8.2** Point Array: When using a point array reticule, new field should be selected on the basis of sampling as much of preparation as possible.
- 9.8.5.9** If asbestos is encountered, but does not happen to be counted as a point, its presence should be noted.



9.8.5.10 Point counting should continue until 1000 non-empty points are counted. These should be evenly spread over a minimum of 2 preps. Once the required number of counts has been tallied from the first prep, return to step 9.7 and prepare and analyze additional slides as needed until 1000 points have been reached. Additional preps (beyond 2) may provide better accuracy and representation of the entire sample, especially when performing extended point counts.

9.8.5.10.1 EPA 600/R-93/116 is the most recent method for the identification of asbestos in bulk building materials and stipulates the requirements stated above in section 9.8.5.10

9.8.5.10.2 EPA 600/M4-82-020 was the interim method prior to EPA 600/R-93/116. This method requires 400 points be counted and cannot be used for the 1000 point count.

9.8.5.11 While point counting inherently assumes the presence of asbestos, a point count may be performed on a negative sample to demonstrate compliance with a desired analytical sensitivity. In this manner, 1000 points are counted as outlined above in step 9.8.5.1 to 9.8.5.10, however, a minimum of 3 preparations must be point counted and scanned in their entirety as outline in section 9.8.3 to demonstrate a None Detected result.

9.8.6 NYS DOH ELAP Stratified Point Count

9.8.6.1 While similar to EPA point counting, NYS DOH ELAP's specified method of point counting (often referred to as Stratified or NYS Stratified Point Counting) differs in significant ways. While the precision of NYS Stratified point count is lower than traditional 400 point counting at higher concentrations, is it is important to realize however that at levels at 1% or below, 400 points are counted using this scheme. Differences include:

9.8.6.1.1 Point counting must be performed at 100x, although other magnifications may be used to clarify fibers, counting must be conducted at 100x.

9.8.6.1.2 4 to 8+ slides are required for analysis.

9.8.6.1.3 The scanning negative option for None Detected samples requires 4 slide preparations

9.8.6.1.4 The point counting procedure utilized a "positive stop" form of point counting.

9.8.6.2 Point counting is a petrographic technique in which a single point (cross hairs) or a point array (Chalkley for example) is/are superimposed on the sample. Initially 4 slides should be prepared for analysis. Slides prepared for point counting should be preferably only 1 layer thick of sample. The sample should be disperse enough so overlapping areas of the slide are rare, but the slide is still mostly covered with sample.

9.8.6.3 Point counting should be done at a 100x magnification. During the point count it may be advantageous to switch to other magnification for better visualization / characterization; however it is necessary to return to 100x for counting.

9.8.6.4 The slide (initially the first slide) is scanned to determine its gross composition and compatibility for point counting. A random field of view is selected and the microscope (if not already there) is at 100x. Point counting



may use either a cross hair reticule or point array (Chalkley) reticule. During point counting each slide is counted until the following

- 9.8.6.4.1** The first asbestos point is counted or
- 9.8.6.4.2** 50 non-empty points have been counted regardless
- 9.8.6.4.3** No more than 1 asbestos point may be counted per slide
- 9.8.6.4.4** If the sample is judged to have no asbestos in it, the entire area of the sample prep should be scanned at 100x. If at any time asbestos is encountered, scanning should stop and point count commence. All slides, regardless of which slide the asbestos was encountered on, are to be point counted.

Note: If a cross hair reticule is chosen, count the point under the reticule cross hairs and move to a new, random field of view. If a point array reticule is chosen, counting should commence at the same point in every field of view. The Chalkley array has a total of 25 points in the reticule as well as a cross hair. The cross hair should be ignored and one of the points should be picked to be the first counted each time. A systematic path should then be used to traverse the points in the Chalkley array and the same path used for every field of view during the analysis.

- 9.8.6.5** Only points that are superimposed on a particle (non-empty point) are counted. Points that fall on / are superimposed over an unoccupied area (empty point) should not be counted; the closest particle identity is not used to count empty points.
- 9.8.6.6** Each non-empty point is classified as a regulated asbestos point or a non-asbestos particle. Categories of non-asbestos particles may subcategorized during the point count (ie. as cellulose, fiberglass, matrix, etc.) but it is not necessary to point count non-asbestos fiber types, their quantity may be estimated.
- 9.8.6.7** Points that are superimposed over two individual particle types (ie. asbestos and non-asbestos) should be counted as 1 point for each particle type.
- 9.8.6.8** When a point(s) are superimposed on an area that has several overlapping particles, the slide should be moved to another field.
- 9.8.6.9** After the point / all points in a field of view are counted move to the next field of view. The next field of view should be selected at random, with the analyst looking away temporarily while moving the slide.
- 9.8.6.10** If asbestos is encountered, but does not happen to be counted as a point, its presence should be noted.
- 9.8.6.11** The remaining three (3) slides should be processed using steps 9.8.6.4 through 9.8.6.10.
- 9.8.6.12** After completion of point counting all four slides, and if the point count has yielded 4 asbestos points the point count is complete.
- 9.8.6.13** If less than 4 asbestos points have been counted after the first four slides are processed, additional slides should be prepped following section 9.7, and point counted following steps 9.8.6.4 through 9.8.6.10. Preps should be made and point counting should continue until either:
 - 9.8.6.13.1** 4 asbestos points are counted or
 - 9.8.6.13.2** 400 non-empty points are counted or



9.9 Data Recording

9.9.1 Following method requirements, specific information is collected and recorded during analysis. Information is recorded on EMSL's PLM worksheet, or in EMSL's Direct Data entry database interface "iLab". For information on the use of "iLab" please see EMSL's Asbestos DDE Manual.

Regardless of the method of recording used, all information detailed below is required to be recorded. The microscopist observes and documents macroscopic and microscopic characteristics, concentrations of materials, optical properties and any other pertinent information. Along with the analyst's signature, date of analysis and temperature (in °C), the information recorded includes:

Macroscopic Characteristics	Sample Treatment	Component Types	Microscopic Characteristics
Color	Teased	Asbestos	Morphology
Texture (fibrous, non fibrous, other)	Crushed	Other fibrous	Sign of Elongation
Homogeneity	Dissolved	Non-fibrous	Pleochroism
	Ashed		Birefringence
	Heated/Melted		Fiber color
			Refractive Index
			Extinction

9.9.2 At least the first 4 fibers of asbestos in each sample shall be positively identified by each of these microscopic parameters. When more than one asbestos type is identified in a sample, at least 4 fibers of each asbestos type must be positively identified. Many of EMSL's worksheets are designed to utilize codes in order to conserve space and make data entry more efficient. The following is an example of the header of the PLM analytical worksheet and the codes are listed below. If a component is not explicitly listed in the codes, an option of "Other" is available for recording purposes. The material should still be correctly identified and properties recorded:

Macroscopic			Treatment	Asbestos		Fibrous		Non-Fibrous		Optical Properties			
COLOR (C) 1 Brown 4 White 7 Black 2 Gray 5 Red 8 Silver 3 Tan 6 Various 9 Blue 10 Yellow TEXTURE (T) 1 Fibrous 2 Non-Fibrous 3 Other HOMOGENEITY (H) 1 Homogeneous 3 OTHER 2 Heterogeneous 4 Layers (#)			1 Teased 2 Crushed 3 Dissolve 4 Ashed 5 Heated 6 Melted	1 Chrysotile 2 Amosite 3 Anthophyllite 4 Tremolite 5 Actinolite 6 Crocidolite	7 Cellulose 8 Glass 9 Min. Wool 10 Synthetic 11 Other 12 Mollastonite 13 Hair	14 Quartz 15 Mica 16 Gypsum 17 Cal. Carbonate 18 Matrix 19 Perlite 20 Other	Morphology (M) 1. Wavy 2. Scaled 3. Splayed 4. Straight 5. Frayed 6. Variable 7. Unif. Diameter 8. Multiaxial 9. Curved Shape 10. Other Pleochroism (P) 1. No 2. Yes Birefringence (B) 1. Low 2. High Fiber Color (FC) 1. White 2. Brown Extinction (E) 1. Parallel 2. Perpendicular 3. Oblique 4. Uniaxial 5. Biaxial 6. Circular						
Sample	Macrosc.	Treat	Stereo Asbestos Est. %	Asbestos Type	% of Asbestos	Other Fibrous Type	%	Non-Fibrous Type	%	Non-Asb Char. Ex. E4	Optical Properties		
1-1	(C) 4	1	10	1	10	7	10	20	75	m4	1.547	1.555	1.708
	(T) 1			2	5						1	2	M 1 / 1 S
	(H) 1										2	1	1 / 1 (FC) 10 / 10 1 / 1 E

Optical Property codes can be seen more clearly here:



Morphology (M)		Sign of Elongation (S)	
1. Wavy	6. Scaled	1. +	
2. Straight	7. Pitted	2. -	
3. Uniform Diameter	8. Medulla	3. Variable	
4. Ribbon-Like	9. Exotic Shapes		
5. Tapered Ends	10. Other		
Pleochroism (P)	Birefringence (B)	Fiber Color (FC)	Extinction (E)
1. Yes	1 Low: 0.010	1 White	1. Parallel
2. No	2 Med 0.010-0.050	2 Brown	2. Symmetrical
	3 High >0.050	3 Beige	3. Oblique
	4 None 0.00 or Isotropic	4 Blue	4. Undulose
		5 Green	
		6 Colorless	

9.9.3 Worksheets are completed by inserting the appropriate code for each observation. In the example above the sample 1-1 was:

9.9.3.1 White (C=4)

9.9.3.2 Fibrous (T=1)

9.9.3.3 Homogeneous (H=1)

9.9.3.4 Teased (Treat=1)

9.9.3.5 Contained approximately 10% suspect asbestos by stereomicroscope

9.9.3.6 Contained 10 % chrysotile (Asbestos Type=1, 10%)

9.9.3.6 and 5% amosite (Asbestos Type=2, 5%)

9.9.3.7 10% cellulose (Other Fibrous=7, 10%)

9.9.3.8 and 75% other non-fibrous material (Non-Fibrous=20, 75%).

9.9.3.9 The distinguishing characteristic for the non-asbestos fiber detected (cellulose) was ribbon-like morphology (Non-Asb Char.=M4).

Note: For sample analyzed using NIOSH 9002, asbestos concentrations should be recorded as a range.

9.9.4 The optical properties are filled out similarly and for the purposes of filling out the PLM analytical bench sheet when more than one asbestos type is encountered, the Optical Properties may be recorded as follows:

9.9.4.1 Each sample space/block on the worksheet allows for the listing of up to 3 asbestos types on three distinct lines within the space.

9.9.4.2 List each asbestos type and percent, in order of decreasing quantity, on the lines within the sample.

9.9.4.3 In the Optical Properties section of the worksheet, separate each box in the section using a slash or slashes. This will mark individual sections of each box for entry of optical properties of each asbestos type (ie. if there are 2 asbestos types, each box will be divided into two spaces to allow for recording of two sets of optical properties)

9.9.4.4 The first (leftmost) section of each Optical Properties box is for the first asbestos type, the next section is for the second asbestos type and so on. In this example, chrysotile is the first asbestos type (asbestos type #1) and amosite is the second (asbestos type #2). The optical properties for chrysotile are the first or leftmost property in each box. In this case, the \perp Refractive Index (RI) for chrysotile is 1.547 and the \perp RI for amosite is 1.692.



Asbestos Type	% of Asbestos
1	10
2	5

Optical Properties			
1.547	/	1.692	[⊥] R.I.
1.555	/	1.709	R.I.
1	/	2	M
2	/	2	^P
1	/	2	^B
6	/	6	(FC)
1	/	1	^E

- 9.9.5** The full set of properties for chrysotile above is:
- 9.9.5.1** Refractive Index is \perp RI=1.547, \parallel RI=1.555
 - 9.9.5.2** Fiber morphology is wavy (M=1)
 - 9.9.5.3** Sign of elongation is positive (S=1)
 - 9.9.5.4** No pleochroism observed (P=2)
 - 9.9.5.5** Birefringence is low (B=1)
 - 9.9.5.6** Fiber color in plane polarized light is clear (FC=6)
 - 9.9.5.7** Extinction is parallel (E=1).

10.0 Calculations

All calculations are where:

- %A - Percent asbestos
- %Ave- Percent asbestos Visual Estimate
- %Apt- Percent Asbestos from Point Count
- a - asbestos points counted
- n - total (asbestos + non-asbestos) points counted
- W1 - Weight of unashed sample
- W2 - Weight of post-ash sample
- W3 - Weight of Final (post-ash, post-acid) sample
- O - % Organics of sample
- AS - % Acid soluble material of sample
- R - % non-organic, non-soluble of sample

10.1 Percentage Asbestos Concentrations

10.1.1 Point Count (EPA 400 & 1000 and NYSDOH ELAP Stratified 198.1)

$$\% Apt = (a/n) \cdot 100$$

10.1.2 NOB

10.1.2.1 EPA NOB

$$\% A = (\% Ave \cdot R) / 100$$

10.1.2.2 NYSDOH 198.6

$$\% A = (\% Apt \cdot R) / 100$$

10.2 Gravimetric Reduction Calculation

10.2.1 The percent of organics removed in the ashing process is calculated as:

$$O = [(W1 - W2) / W1] \cdot 100$$



10.2.2 The percent of acid soluble material removed in the acid dissolution is calculated as:

$$AC = [(W2 - W3)/W1] \cdot 100$$

10.2.3 The Final Residue is the percent of non-organic, non-soluble material in the sample is calculated as:

$$R = (W3/W1) \cdot 100$$

11.0 Reporting

- 11.1 *A note on the use of the term "trace": It is EMSL policy not to use the term 'trace' when reporting sample results, as this terminology is ill defined and ambiguous. When applying the point count technique, the New York State Environmental Laboratory Approval Program (NYSELAP) methods refer to the term 'trace' where asbestos is observed in the sample slide but is not directly under a point. EMSL reports <1 % in these cases.*
- 11.2 *A note for sample results at 1%: It is EMSL policy to avoid having to report asbestos concentrations at the 1% level. In these instances additional analytical effort should be applied. This may include additional preps for estimation, sampling more areas of the sample, observation by a second analyst, counting more points during point counts, or other means to determine whether the sample may be greater or less than 1%.*
- 11.3 Certain specific reporting criteria must be taken into account when reporting asbestos concentrations in this SOP. The following items are included in the final report (on EMSL letterhead) to the client:
- 11.3.1 EPA 600
 - 11.3.1.1 Asbestos concentrations should be reported to the nearest whole integer.
 - 11.3.1.2 Concentrations below 1% are reported as <1%.
 - 11.3.1.3 Samples in which no asbestos was encountered are reported as "None Detected".
 - 11.3.2 EPA 400 Point Count
 - 11.3.2.1 Asbestos concentrations are reported to the nearest ¼ percent (1/400 points), including concentrations below 1%. If EPA 600/M4/92/020 is being used results below 1% should be reported as <1%.
 - 11.3.2.2 Samples in which asbestos is encountered but not landed on in point counting are reported as < 0.25%.
 - 11.3.2.3 Samples in which no asbestos was encountered are reported as "None Detected".
 - 11.3.3 EPA 1000 Point Count
 - 11.3.3.1 Asbestos concentrations are reported to the nearest tenth (1/10) percent (1/1000 points), including concentrations below 1%.
 - 11.3.3.2 Samples in which asbestos is encountered but not landed on in point counting are reported as < 0.1%.
 - 11.3.3.3 Samples in which no asbestos was encountered are reported as "None Detected".
 - 11.3.4 EPA NOB



- 11.3.4.1 Asbestos concentrations are reported to the two significant figures, to a lower bound of 0.25%.
- 11.3.4.2 Concentrations below 0.25% are reported as <0.25%
- 11.3.4.3 Sample in which no asbestos was encountered are reported as "None Detected".
- 11.3.4.4 Reporting of "Insufficient Residue" samples:
 - 11.3.4.4.1 This designation is regardless of actual residue weights and typically would be utilized for Final Residue percentages around or below 1%.
 - 11.3.4.4.2 When the preparation of slide preparations from the final residue yields noticeably low loading background particulate but does not possess sufficient material to be able to reliably make a confident qualitative or quantitative determination or
 - 11.3.4.4.3 The absence of said low loading background particulate indicates that there is no appreciable particulate loading that has been transferred from the sample filter indicating the possibility that the only transferred material was actual filter.
 - 11.3.4.4.4 This may be determined by noticing if in fact there is a particulate loading of approx. <1% of total area or that there is no loading at all on the prepared slides.
 - 11.3.4.4.5 Report the sample using the designation "Insufficient Residue" as well as the sample's Gravimetric Breakdown (% organics, % carbonate and Final Residue %) in a sample comment.
- 11.3.5 NYS 198.1
 - 11.3.5.1 Asbestos concentrations should be reported to 2 significant figures.
 - 11.3.5.2 For asbestos concentration below 1%, including samples in which asbestos was noted but not counted during point counting, are reported as <1%.
 - 11.3.5.3 Samples in which no asbestos was encountered are reported as "None Detected".
- 11.3.6 Sample reported using NYSDOH ELAP 198.6:
 - 11.3.6.1 Asbestos concentrations should be reported to 2 significant figures.
 - 11.3.6.2 Total asbestos results (for samples containing more than one (1) asbestos type) are also reported.
 - 11.3.6.3 For asbestos concentration below 1%, including samples in which asbestos was noted but not counted during point counting, are reported as <1%.
 - 11.3.6.4 Samples in which no asbestos was encountered are reported as "None Detected".
 - 11.3.6.5 Samples reported as None Detected or $\leq 1\%$ should be analyzed by NYS 198.4 (TEM NOB)
 - 11.3.6.6 Samples where No Asbestos is detected or at levels $\leq 1\%$ are report with the additional comment of "Inconclusive" as per 198.6.
 - 11.3.6.7 Reporting of "Insufficient Residue" samples:
 - 11.3.6.7.1 This designation is regardless of actual residue weights and typically would be utilized for Final Residue percentages around or below 1%.
 - 11.3.6.7.2 When the preparation of slide preparations from the final residue yields noticeably low loading background particulate but does not



- possess sufficient material to be able to reliably make a confident qualitative or quantitative determination or
- 11.3.6.7.3 The absence of said low loading background particulate indicates that there is no appreciable particulate loading that has been transferred from the sample filter indicating the possibility that the only transferred material was actual filter.
- 11.3.6.7.4 This may be determined by noticing if in fact there is a particulate loading of approx. <1% of total area or that there is no loading at all on the prepared slides.
- 11.3.6.7.5 Report the sample using the designation "Insufficient Residue" as well as the sample's Gravimetric Breakdown (% organics, % carbonate and Final Residue %) in a sample comment.
- 11.3.6.8 If asbestos was identified during preliminary stereomicroscopic observation and the sample was eventually found to be None Detected or calculated to be $\leq 1\%$ asbestos by NYSDOH ELAP 198.4, report the sample as "Conflicting Results - additional sampling and analysis needed".
*Note: According to this procedure in section 9.4 above, fiber identification during preliminary stereomicroscopic observation is **not performed**.*
- 11.3.7 NIOSH 9002
- 11.3.7.1 Asbestos concentrations should be reported as a range representing the analyst's precision.
- 11.3.7.2 Concentrations below 1% are reported as <1%.
- 11.3.7.3 Samples in which no asbestos was encountered are reported as "None Detected".
- 11.3.8 Alternative reports - there may exist alternative reports (ie. NY 3 in 1, NJ 5 in 1, Ontario report, etc.), that are meant to summarize multiple analysis methodologies in one summary. In these cases the composite report may not include all required information as detailing in this section and should only be issued as a supplement to the individual reports for each analytical method. Uncertainty may also be reported at client request as an alternate report and is calculated from EMSL's Uncertainty Worksheet – Asbestos Excel spreadsheet. This may be done as follows:
Note: separate uncertainties for multiple concentration ranges are recommended.
- 11.1.8.1 The uncertainty range (at a 95% confidence level) for a result is defined as the reported result $\pm 2S_r$, where $S_r = S/\text{mean}$.
- 11.1.8.2 S_r values are typically derived using either relative percent difference (RPD) data from quality control samples or recovery data from standard reference materials (SRMs). In the latter case, the mean shall be the reference value (or 100% if using mean recovery), and thus $S_r = S$.
- PLM Example**
If the standard deviation (S) for a population of NVLAP PT data is 10.0% (calculated using % recovery data relative to the reference value), and the mean recovery is 90.0%, the relative standard deviation (S_r) is equal to 10.0% since $S_r = S$. Thus, in this example, the uncertainty range for a client result of 10% shall be



reported as 10% (not corrected for bias) \pm 2% ($2S_r = 20\%$, thus $2S_r \times 10\% = 2\%$)
with a probable negative bias of 1%.

- 11.1.8.3 Note that the reported result is not corrected for bias for any client sample. Rather, bias (available only when S_r is derived from SRM data) must be reported as a separate value.
- 11.1.8.4 When applied to a specific client result, both uncertainty and bias shall be provided in the appropriate units.
- 11.1.8.5 Alternatively, at the request of the client, the uncertainty, confidence level and bias may simply be provided to the client as the derived percentage values without application to a specific result.
- 11.4 As a service for our Canadian Clients a special report format is provided. For this report a statement as to the analytical sensitivity for None Detected samples will be included. This statement will correspond to Section 9.8.3.4 above.
- 11.5 Identification of each asbestos type in the sample.
- 11.6 Asbestos concentration in % asbestos of each asbestos type in the sample (upper and lower estimates should be reported for samples following NIOSH 9002).
- 11.7 Identification of non-asbestos fibers encountered.
- 11.8 Percentage of each non-asbestos fiber type identified.
- 11.9 Percentage, and optionally identification, of matrix material in the sample.
- 11.10 Sample Appearance (color, homogeneity, texture)
- 11.11 Sample Description or Location of the sample in the field
- 11.12 Client and Lab sample numbers
- 11.13 Client identification and contact information
- 11.14 EMSL Order ID
- 11.15 Client Project information (if supplied)
- 11.16 Analysis date, report date and date received.
- 11.17 Signature of Lab Manager
- 11.18 Report comments
- 11.19 Lab accreditations

12.0 Method Performance

12.1 MDL

- 12.1.1 EPA 600/R-93/116, NYSDOH ELAP 198.1 & 198.6, NIOSH 9002, EPA 400 & 1000 Point Count method detection limits are 1%.
- 12.1.2 EPA NOB method detection limit is dependant on the gravimetric reduction component of the analysis, in theory it could be as low as $0.1\% \times 1\%$.

12.2 DOC's

Demonstrations of Capability are required for each analytical method.

12.3 PTs

- 12.3.1 Proficiency tests are available from NVLAP and NYS ELAP for EPA 600/R-93/116 (with or without EPA point counts and gravimetry components) and NYSDOH 198.1.
- 12.3.2 All analysts are required to participate in each round of testing either before or after results have been submitted.

12.4 Accuracy

- 12.4.1 EPA 600/R-93/116 by visual estimate or 400 point count would expect precision and accuracy to be 0-3% at the 1 % range. Gravimetry may increase these levels.



12.4.2 NYSDOH ELAP 198.1 only offers that point counting is usually more accurate than visual estimate and by 198.6 the results negatively biased as compared to TEM with the possibility of false negatives.

12.4.3 Not determined for NIOSH 9002

12.5 Precision

12.5.1 EPA 600/R-93/116 by visual estimate or 400 point count would expect precision and accuracy to be 0-3% at the 1 % range. Gravimetry may increase these levels.

12.5.2 NYSDOH ELAP 198.1 produces Sr values of 25% or less for concentration of 1 to 3% and 24 to 45% for concentration of 5 to 7%. ELAP 198.6 applied to VAT samples in the 15 to 30% range (determined via TEM) produced Sr values of 60 to 100%.

12.5.3 Not determined for NIOSH 9002

13.0 Quality Control

13.1 All QC data must be maintained and available for easy reference and inspection

13.2 Blanks

13.2.1 Known negative materials are prepared, using all prep equipment and oils at a rate of 1 per 100 samples, or at least daily.

13.2.2 Known negative materials are prepared and processed with at least every 20 used of all homogenization equipment (ie. mortar and pestle, etc.)

13.2.3 For samples involving gravimetric preparation

13.2.3.1 Known negative material is processed, including gravimetric reduction at a rate of 1 per every 20 samples, and at least 1 with every batch of samples processed.

13.2.3.2 Blanks are both checked for contamination and for consistency in the gravimetric reduction process.

13.3 7% Inter-analyst QC analysis

13.4 2% Intra-analyst QC analysis

13.5 1% Standard analysis

Note: NIOSH 9002 requires analysis of 5% known quantitative standards.

13.6 0.5% Inter-laboratory analyses performed quarterly.

13.7 The selection of QC samples should be semi random and as blind as possible, so that the QC analyst does not know the sample identity or result before analysis. One suggested scenario for selecting and submitting QC samples is as follows:

13.7.1 For each batch of samples, the original analyst should determine the number of QC samples to be selected for QC. This is 2% (approximately 1/50) for intra-analyst analysis and 7% (approximately 1/15 sample) for inter-analyst analysis.

13.7.2 Small jobs may be combined to use as a sample pool, while it is important to keep in mind that not every batch of samples needs to have QC samples picked from it.

13.7.3 The original analyst will select the inter-analyst QC samples to be used and record the EMSL order ID and the sample numbers to be reanalyzed on a separate, blank analytical worksheet.

13.7.4 These samples are then submitted, along with the worksheet to the QC analyst for reanalysis.



- 13.7.5 The original analyst also inputs the Date, Order ID, Sample ID, Original Analyst Initials and Original Sample Results in the PLM QC spreadsheet.
- 13.7.6 After the inter-analyst QC samples have been analyzed, the QC analyst will input the QC results in the corresponding rows of the PLM QC spreadsheet as started by the original analyst.
- 13.7.7 The QC analyst will then pick the required amount of intra-analyst QC samples and as in step 13.7.3 above record the EMSL order ID and the sample numbers to be reanalyzed on a separate, blank analytical worksheet.
- 13.7.8 The QC analyst will also input the Date, Order ID, Sample ID, Original Analyst Initials and Original Sample Results in the PLM QC spreadsheet.
- 13.7.9 These samples are then submitted, along with the blank worksheet to the Original analyst for QC analysis.
- 13.7.10 After reanalysis by the original analyst, the results are entered in the PLM QC spreadsheet.

14.0 Data Assessment

14.1 Acceptance criteria for QC measures

These are addressed in EMSL's QA Manual Module A section A.13.

14.1.1 If a sample falls outside the acceptable limits it needs to be reconciled with participating analysts and/or a third analyst when necessary.

14.1.2 The Pass/Fail criteria for inter-analyst QC and formula for R (variance) is:

Pass $-1 \leq R \leq 1$
Fail $R < -1$ or $R > 1$

$$R = \frac{(A - B)}{((A + B)/2)} \quad \text{where } A = \text{analysis 1 and } B = \text{analysis 2}$$

14.1.3 The Pass/Fail criteria for intra-analyst QC and formula for R (variance) is:

Pass $R \leq 1$
Fail $R > 1$

$$R = \frac{|A - B|}{((A + B)/2)} \quad \text{where } A = \text{analysis 1 and } B = \text{analysis 2}$$

14.1.4 Standard analysis is tabulated for precision and accuracy.

14.2 Corrective actions

These policies are addressed fully in the EMSL's QA manual section 13.

14.2.1 All corrective actions should look for the root cause of the error.

14.2.2 All out of control or unacceptable data must be brought to the attention of the Laboratory Manager.

14.2.3 The Laboratory manager is responsible for generating a corrective action including an investigation of calibration procedures, a review of analytical technique and investigation of training policies and compliance.



14.2.4 Corrective actions will be reported to the QA Department by means of the Quarterly Management Report or sooner when appropriate.

14.3 Contingencies for handling out-of control or unacceptable data.
Any quality control requirements not met must have an explanation to their nonconformance.

15.0 Pollution Prevention / Waste Management

15.1 Pollution Prevention

EMSL Analytical makes all efforts to reduce the volume and toxicity of the waste generated by the laboratory. An effort to manage procurement of hazardous materials has been implemented in order to avoid over ordering. Hazardous waste is classified for proper disposal.

15.2 Waste Management

The waste generated during prep and analysis will be disposed of following safety procedures outlined in the chemical hygiene plan (EMSLChemHygiene 200.0).



16.0 Tables, Diagrams, Flowcharts, and Validation Data

16.1

CHEMICAL FORMULAS FOR TYPICAL ASBESTOS MINERALS

<u>Mineral</u>	<u>Chemical Formula</u>
Chrysotile	$Mg_3Si_2O_5(OH)_4$
Amosite	Approx $Mg_{2.8}Fe_{4.2}Si_8O_{22}(OH)_2$
Crocidolite	$Na_2Fe^{2+}_3Fe^{3+}_2Si_8O_{22}(OH)_2$
Anthophyllite	$Mg_7Fe_0Si_8O_{22}(OH)_2$ - $Mg_6Fe_1Si_8O_{22}(OH)_2$
Tremolite	$Mg_5Fe_0Ca_2Si_8O_{22}(OH)_2$ - $Mg_{4.5}Fe_{0.5}Ca_2Si_8O_{22}(OH)_2$
Actinolite	$Mg_{4.5}Fe_{0.5}Ca_2Si_8O_{22}(OH)_2$ - $Mg_{2.5}Fe_{2.5}Ca_2Si_8O_{22}(OH)_2$

16.2

ASBESTOS AND ANALOG NON-ASBESTIFORM VARIETIES

<u>Asbestos</u>	<u>Non-Asbestos Analog</u>
Chrysotile	Antigorite-lizardite
Crocidolite	Riebeckite
Amosite	Cummingtonite-grunerite
Asbestiform Anthophyllite	Anthophyllite
Asbestiform Tremolite	Tremolite
Asbestiform Actinolite	Actinolite



16.3 OPTICAL PROPERTIES OF ASBESTOS FIBERS
 As summarized from EPA 600/R-93/116 and ELAP 198.1

Mineral	Morphology, Color	Refractive Indices		Birefringence	Extinction	Sign of Elongation
		α	γ^1			
Chrysotile (asbestiform) serpentine	Wavy fibers. Fiber bodies have splayed ends and "kinks". Aspect ratio typically > 10:1, Colorless	1.493-1.560	1.517-1.567	0.004 - 0.017	Parallel / Undulose	+ (length slow)
Amosite (asbestiform) grunerite	Straight to curved, rigid fibers moderately flexible. Aspect ratio typically > 10:1. Colorless to tan / brown, nonpleochroic or weakly so. Opaque inclusions may be present. Easily splayed ends.	1.657-1.686	1.696-1.729	0.021 - 0.054	Usually Parallel / Infrequently 2°	+ (length slow)
Crocidolite (asbestiform) riebeckite	Straight to curved, flexible and rigid fibers, some kink bands, splayed ends. Thick fibers and bundles common, blue to dark blue in color. Pleochroic. Aspect ratio typically > 10:1.	1.654-1.701	1.668-1.717	0.003 - 0.022	Usually Parallel	- (length fast)
Anthophyllite	Straight to curved fibers and bundles, usually stiff, ends splayed to blunt. Aspect ratios typically >10:1, cleavage fragments may be present with aspect ratios < 10:1. Colorless to tan/ light brown	1.596-1.652	1.615 - 1.722	0.013 - 0.028	Parallel	+ (length slow)
Tremolite	Straight to curved fibers and bundles, usually stiff, large bundles may have splayed ends. Aspect ratio > 10:1, cleavage fragments may be present as single crystals with aspect ratios < 10:1. Colorless to pale green (rarely).	1.599-1.628	1.625-1.655	0.017 - 0.028	Parallel and oblique / inclined up to 21°	+ (length slow)
Actinolite	Straight to curved fibers and bundles, usually stiff, large bundles may have splayed ends. Aspect ratio > 10:1, cleavage fragments may be present as single crystals with aspect ratios < 10:1. Colorless to pale green. Pleochroic.	1.600-1.628	1.625-1.688	0.017 - 0.018	Parallel and oblique / inclined up to 21°	+ (length slow)

¹ γ is the parallel orientation for all asbestos minerals except in crocidolite γ is \perp .



16.4 Expected Dispersion Staining Colors Using Central Stop Lens

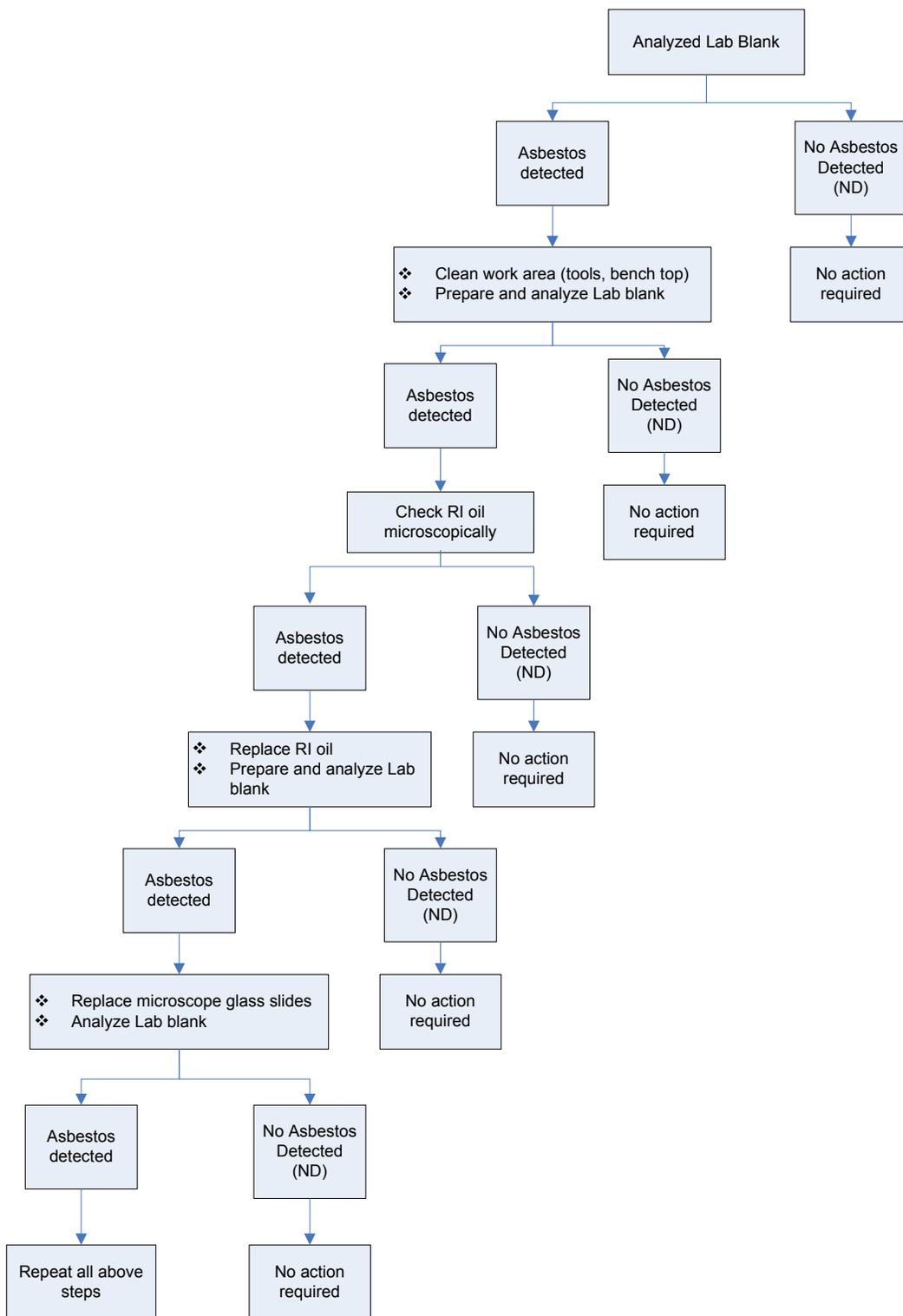
As summarized from (1) EPA 600/R-93/116, (2) NIOSH 9002 and (3) McCrone Asbestos Identification.

CENTRAL STOP DISPERSION STAINING COLORS

MINERAL	RI LIQUID	Reference		⊥
			(parallel)	(perpendicular)
Chrysotile	1.550 ^{HD}	1	magenta to light blue-green	blue-green to pale blue
		2	blue-magenta	blue
		3	magenta	blue
"Amosite"	1.680	1	yellow to magenta	blue-magenta to light blue
		2	gold	blue
		3	golden yellow	blue
"Crocidolite"	1.680	1	yellow to magenta	pale yellow to golden yellow
		2	yellow	pale yellow
		3	golden yellow	pale yellow
	1.700	2	blue-magenta	red-magenta
Anthophyllite	1.605 ^{HD}	1	pale yellow to yellow	golden yellow to light blue-green
		2	gold to golden magenta	blue
		3	yellow	blue-magenta
	1.620 ^{HD}	2	golden yellow	blue-green
Tremolite	1.605 ^{HD}	1	pale yellow to yellow	golden yellow to light blue-green
		2	yellow	pale blue
		3	yellow to pale yellow	blue, magenta
Actinolite	1.605 ^{HD}	1	pale yellow	pale yellow to golden yellow
		2	pale yellow	yellow
		3	pale yellow	yellow to pale yellow
	1.630 ^{HD}	1	yellow to magenta	golden yellow to blue



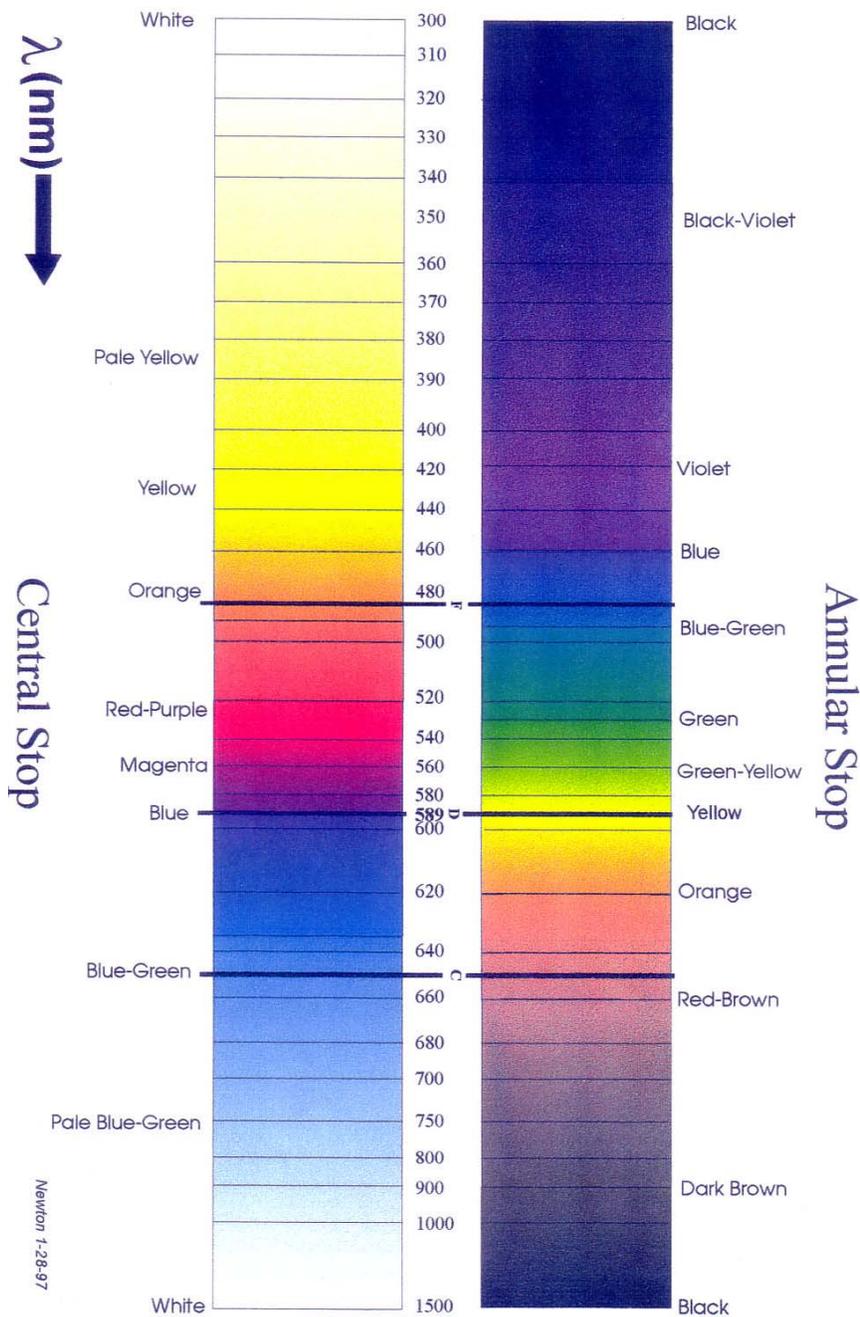
16.5 Contamination Flowchart





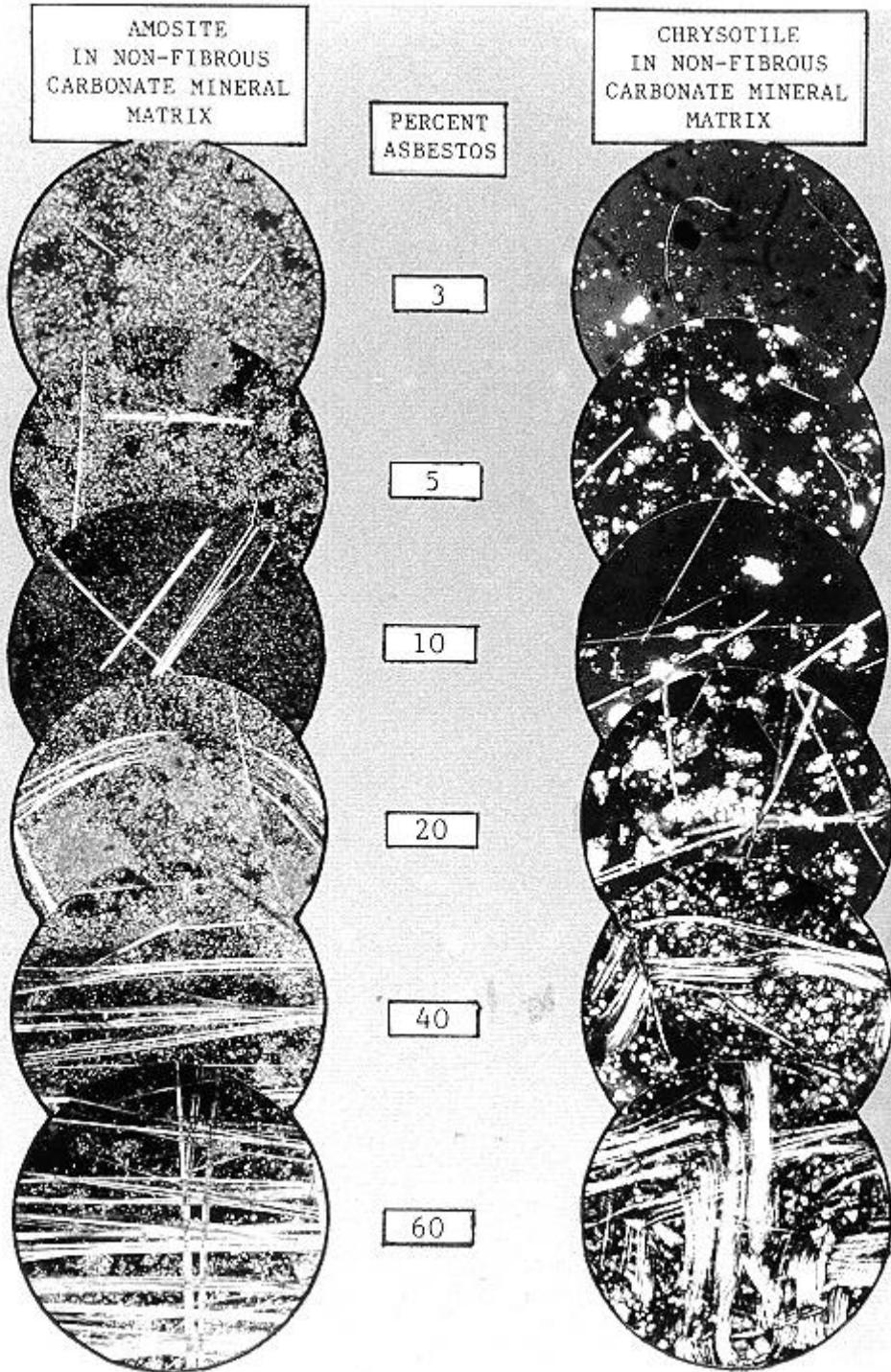
16.6 Dispersion Staining Colors / Wavelength Chart

Note: This chart is presented as an example. Due to variations in the chart scan coloring and monitor to monitor differences, actual color / wavelength determinations should be made using hardcopies of charts available in the lab.





16.7 Asbestos Concentration Chart for NIOSH 9002





17.0 References

- 17.1 EMSL QA Manual Revision 9 April 2007
- 17.2 EMSL Chemical Hygiene Plan Revision 0, September 2004
- 17.3 Less is Better- Guide to Minimizing Waste in Laboratories prepared by the Task Force on Laboratory Environment, Health and Safety- American Chemical Society 2002.
- 17.4 Method for the Determination of Asbestos in Bulk Building Materials EPA 600/R-93/116
- 17.5 Interim Method for the Determination of Asbestos in Bulk Insulation Samples EPA 600/M4/82/020
- 17.6 PLM Methods for Identifying and Quantitating Asbestos in Bulk Samples NYSDOH ELAP 198.1
- 17.7 PLM Methods for Identifying and Quantitating Asbestos in Non-Friable Organically Bound Bulk Samples NYSDOH ELAP 198.6
- 17.8 Asbestos (bulk) by PLM NIOSH 9002
- 17.9 Asbestos Identification McCrone Institute
- 17.10 NVLAP Handbook and Checklist Bulk Asbestos Analysis 150-3 2006 Edition.
- 17.11 Asbestos NESHAP (1990 Point Counting)
- 17.12 Asbestos NESHAP Clarification (40 CFR Part 61 Jan-5-1994 & Dec-19-1995 Analysis of Multi-Layered Systems)
- 17.13 EPA Asbestos Sampling Bulletin (September 30, 1994)
- 17.14 Rapidly and Accurately Determining RI of Asbestos Fibers by Dispersion Staining, Dr. Su (1994 & 2003)



18.0 Revision History

Rev #	Date	Revision	Initials
pre 7	various	Include non-friable organically bound (NOB) materials in Applicable Matrix section. Clarify the term 'trace' and the policy for reporting such occurrences as <1%. Discuss PLM worksheet and include copy in Appendix. Require that at least the first 4 fibers of asbestos in each sample be positively identified by the 6 microscopic parameters (morphology, sign of elongation, refractive index, etc.). Added reporting requirements in compliance with Ontario Regulation 278/05	unknown
7	02/29/08	Appendix B modified to include detailed alignment procedures (Appendix B Section 4.0) coinciding with the Daily PLM Calibration & Contamination Record (Alignment worksheet). Logo updated. Inserted PLM Contamination Flowchart. Added a description in Section 10.0 Recording Results of PLM analytical worksheet usage including recording observations using worksheet codes and recording optical properties for more than one asbestos type. Corrected Point Count calculations in Section 10.1 and 10.2 to divide asbestos points by total non-empty points.	KN
8	09/12/08	Complete conversion to current SOP format. Removal or integration of appendices into SOP. Inclusion of all pertinent methods including NIOSH 9002, NYS ELAP 198.6 and EPA PLM NOB (EPA 600 R-93/116 Section 2.3 Gravimetry). Added a section on Uncertainty Reporting to client (section 11.1.8).	KN
9	01/07/09	Modified section 5.2 to suggest coverslip thickness selection may be microscope specific. Modified section 8.4.3 to include centering of the central stop prior to sub stage condenser alignment and to indicate alignment of substage condenser on the field of view / crosshair when the central stop is able to be centered. Changed suggested ashing time in section 9.5.12 to 12 hours. Added verbiage to indicate that increasing the number of preps used in an extended point count may increase accuracy of the point count in section 9.8.5.10. Added section 8.6 for the monthly documentation of amosite dispersion colors / dispersion color wavelengths. Added verbiage to better describe the analytical sensitivity of None Detected samples using visual estimates in section 9.8.3.4. Changed verbiage in sections 3.2.1.1, 3.2.2, 4.2.1 and 9.5.14 for clarity and typos. Changed step numbers 9.8.7.3.1 - 4 to 9.8.3.2.1 - 4. Report requirements for Section 11.2 have been changed to reference Canadian not just Ontario and also to reference the reporting of None Detected samples following section 9.8.3.4.	KN
9.1	01/27/09	Changed muffle furnace calibration frequency (8.2) to quarterly from monthly. Corrected various typos throughout document.	KN
9.2	02/18/09	Changed required oil calibration frequency. Removed requirement for calibration at ½ and near the end of the bottle in section 8.5.3	KN
9.3	03/03/09	Expanded Asbestos Contamination section (4.1) to include daily procedures. Added note on 16.6 (Dispersion color chart) to indicate use should be from actual hard copies in laboratory.	KN



10	11/13/09	9.9.1 added references to DDE via EMSL's "iLab" and referenced the "Asbestos DDE Manual" for instructions on use. 9.6.7.1.1.4 changed recommended RI Oil for Anthophyllite, Actinolite and Tremolite mounting to 1.625. 16.3 Optical Properties Table: changed acceptable extinction of Tremolite to up to 21°. 6.2 Changed required Calibrated RI Glass Bead set to M-25 to M-7. 9.3.3.2 changed "composting" to "compositing" 8.4.6.1 changed suggested confirmed negative material for contamination checks to fine grained table salt. 5.14.6 changed wavelength of the retardation plate (gypsum) to 530-550nm. 11.1.4 & 11.1.6 included instruction for reporting NOB samples whose final residue of the gravimetric reduction is insufficient to reliably achieve and analytical result.	KN
10.1	11/18/09	Fixed sequencing of section 9.9 (Data Recording).Section 9.8 was listed twice(for Data Recording and Quantitation)	MAC
10.2	11/24/09	Fixed calculation formatting in section 10.1 and section 14. Fixed various typos. 9.5.18 Requires petri dish to cool to room temperature before final weight is read.	MAC/KN
11	11/12/10	11.1 Inserted EMSL's Policy on the use of the term "Trace", this had inadvertently been removed starting with Revision #8. This Policy has not been changed from earlier version of the SOP. 9.8.1 Inserted suggestion to recommend further analysis when problematic samples at or around 1% are encountered and other procedures / technologies would be beneficial to determining the actual asbestos content of a sample.	KN

Authorizing Signatures

Ken Najuch
Author (print)



Author Signature

11/12/2010
Date

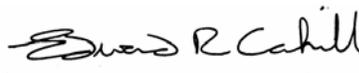
Ed Cahill
Reviewer (print)



Reviewer Signature

11/12/2010
Date

Ed Cahill
Corporate Approval (print)



Corporate Approval Signature

11/12/2010
Date



EMSL Analytical S.O.P.

Asbestos and Other Fibers by PCM

NIOSH 7400 Revision 3 Issue 2 - August 15, 1994

1.0 Method Description

1.1 Applicable Matrix

This procedure is for the determination of fibers in air by phase contrast microscopy. Air samples are collected on 0.45 to 1.2 μm mixed cellulose ester membrane (MCE) filters using 25 cassettes with a conductive cowl, at air flow rates of 0.5 to 16 L/min.

1.2 Scope and Application

This procedure is based on the NIOSH 7400 method and provides a concentration of optically visible airborne fibers. This method is primarily used for air monitoring during asbestos related activities, but since PCM does not differentiate between asbestos and other fibers, results can only be applied to overall fiber concentrations. This method may be used in conjunction with transmission electron microscopy (NIOSH 7402) to estimate the asbestos fiber concentration of a sample.

1.3 Summary of Method

The sample is collected utilizing a MCE air cassette. The filter is cleared and fixed on a microscope slide with acetone vapor, then immersed in triacetin and covered with a glass cover slip. Fibers are counted at a magnification of 400X using a positive phase contrast microscope.

1.4 Detection Limit

The limit of detection for this method is 7.0 fibers/ mm^2 (or 5.5 fibers/100 microscopic fields) as established by the published NIOSH 7400 method.

2.0 Interferences

Interferences for this method include but are not limited to:

- 2.1 High levels of non-fibrous dust particles may obscure fibers in the sample and indirectly increase the detection limit of a sample.
- 2.2 Some types of airborne spores may appear fiber like and cause artificially high readings.
- 2.3 Chain like particles may be mistaken for fibers.
- 2.4 When this method is used for monitoring a specific fiber type, the presence of any other airborne fibers may interfere since all particles meeting the counting criteria are included in the result.

3.0 Definitions

- 3.1 Aspect Ratio - the ratio of length to width of an object.
- 3.2 Coefficient of Variance - also known as Relative Standard Deviation, this is a measure of the expected difference between a number of analyses.
- 3.3 Fiber - an elongated particle, that must have an aspect ratio of at least 3:1 and a minimum length of $> 5\mu\text{m}$ to be counted.
- 3.4 MCE filter- Mixed Cellulose Ester is a type of filter typically used for PCM analysis.
- 3.5 NIOSH - National Institute of Occupational Safety and Health.



- 3.6 TWA - Time Weighted Average is the effective fiber concentration a person would be exposed to if all exposure were distributed evenly over 8 hours (a normal work day).
- 3.7 PCM - Phase Contrast Microscopy is a form of microscopy that enhances differences in the refractive index of particles, adding to their contrast in the microscope's field of view.
- 3.7 STEL - Short Term Exposure Limit.
- 3.8 TWA - Time Weighted Average.
- 3.9 Walton-Beckett Graticle - an inscribed circle in the eyepiece of the microscope ocular. The graticle is a circle of known area that provides various measuring aids to evaluate fibers for aspect ratio and size.

4.0 Safety

All personnel performing preparation and/or analysis of samples must be familiar with the EMSL Chemical Hygiene Plan (EMSLChemHygiene 200.0).

4.1 Asbestos

- 4.1.1 While there is no guarantee that asbestos is present in the samples, prudent measures must be taken to prevent any possible airborne asbestos fiber release from occurring during sample handling.
- 4.1.2 Any filter handling performed prior to the filter clearing step should be performed under the safety hood.
- 4.1.3 All safety hoods should be capable of flow rates ≥ 75 lfm.

4.2 Acetone

- 4.2.1 Keep away from heat, sparks, and flame.
- 4.2.2 Avoid breathing vapors - use with adequate ventilation.
- 4.2.3 Avoid contact with eyes.
- 4.2.4 Prevent prolonged or repeated contact with skin.

5.0 Equipment and Supplies

- 5.1 Acetone Vaporizer or Hot Block
- 5.2 Coverslips, 1.5 thickness
- 5.3 Forceps
- 5.4 Kimwipes
- 5.5 Mechanical counter / tally counter
- 5.6 Micropipette(s) capable of 5 μ l and 100-500 μ l
- 5.7 Microscope slides 3x1
- 5.8 Phase contrast microscope w/ green or blue filter
- 5.9 Scalpel handle and blades
- 5.10 Syringe
- 5.11 Telescoping ocular or Bertrand lens
- 5.12 Walton Beckett graticle, type G-22

6.0 Reagents and Standards

All reagents should be of recognized analytical grade or better:

- 6.1 Acetone
- 6.2 Triacetin
- 6.3 HSE/NPL (preferred) or HSE/ULO red, green or yellow test slide
- 6.4 Past proficiency samples
- 6.5 Consensus standards



7.0 Sample Collection, Preservation, Shipment and Storage

- 7.1 Samples are collected on 0.45 to 1.2 μm pore size 25mm MCE filters in sample cassettes with a conductive cowl.
- 7.2 Air flow should be between 0.5 and 16 LPM (liters per minute). It is recommended that the sampling flow rate not exceed 10 LPM.
- 7.3 Sample volume should be adjusted to give 100 to 1300 fibers/ mm^2 where possible.
- 7.4 No sample preservation is needed and samples can be stored indefinitely prior to analysis.
- 7.5 Samples are best transported to the lab by hand. When mailing, try to package samples carefully to minimize disturbance and possible dislocation of particulate from the filter surface. Use packing materials that will minimize static charge, it is recommended to avoid the use of "foam peanut" packing material.
- 7.6 All air cassettes must be retained in an easily retrievable manner for a minimum of 60 days.
- 7.7 PCM slide preps may be discarded only after PCM QC is complete and evaluated satisfactorily.

8.0 Calibration and Standardization

Each major component of the method is calibrated and/or standardized including the analyst. Examples follow:

- 8.1 Sample collection vacuum pumps are calibrated at the beginning and end of a sampling event (with the sample cassette in line) using a rotometer.
- 8.2 The rotometer in turn needs to be calibrated to a primary standard periodically. (Rotometer use and calibration is the responsibility of the sample collection entity).
- 8.3 All PCM microscope calibrations based on frequency and are considered per analyst / per microscope. The PCM microscope is calibrated to ensure:
 - 8.3.1 If possible adjust the light source for even illumination across the field of view. Frequency is daily or on next use per analyst / microscope combination.
 - 8.3.2 Center and focus the field iris (if possible) so that it is open only enough to fully illuminate the field of view. Adjust the height of the substage condenser to sharpen the image of the field iris. Frequency is daily or on next use per analyst / microscope combination.
 - 8.3.2 Using a telescoping ocular or Bertrand lens, focus the image of the rings (sub stage and in the 40x phase shift objective). If the rings do not form concentric circles, adjust the condenser adjustments screws to bring the rings into alignment. Frequency is daily or on next use per analyst / microscope combination.
 - 8.3.3 Check the shift detection limit using the HSE/NPL (**Mark II**) test slide. Alternately, because the HSE/NPL test slide is no longer available in production, an HSE/ULO test slide may need to be used. The HSE/ULO (**Mark III**) test slide has three different grades of quality, denoted as; i) red, ii) green or iii) yellow. Each of the slides consists of 7 sets of increasingly narrowing diameter inscribed lines. Each class of slide has different resolution of the lines, the lines that are able to be seen in each are listed as follows. Frequency is weekly per analyst / microscope combination.
 - 8.3.3.1 HSE/NPL (Mark II) - The microscope should be able to completely resolve the first 3 sets of lines (1-3), the next two sets of lines (4-5) should be partially visible. The microscope should not be able to see the last two sets (6-7) of lines at all, they should be invisible.



- 8.3.3.2 HSE/ULO Red (Mark III Red) - The microscope should be able to completely resolve the first 4 sets of lines (1- 4), the set of lines (5) should be partially visible. The microscope should not be able to see the last two sets (6-7) of lines at all, they should be invisible.
- 8.3.3.3 HSE/ULO Green (Mark III Green) - The microscope should be able to completely resolve the first 5 sets of lines (1-5), the set of lines (6) should be partially visible. The microscope should not be able to see the last set (7) of lines at all, they should be invisible.
- 8.3.3.4 HSE/ULO Yellow (Mark III Yellow) - This slide is not acceptable for this calibration and should not be used.
- 8.3.4 The area of the Walton Beckett graticle should be checked to ensure the correct field area is used for analytical calculations. The field area is measured using the stage micrometer and then calculated using the formula for the area of a circle (πr^2) where r is the radius of the graticle field. The diameter of the graticle field should be $100\mu\text{m} \pm 2\%$ (or graticle areas of 0.00754 to 0.00817 mm^2). Record the field diameter and use the actual calibrated graticule area in mm^2 . Frequency is monthly per analyst / microscope combination, for labs meeting the TNI Standard (NELAC) the frequency should be daily per analyst / microscope combination.
- 8.4 Mechanical counter accuracy is documented by counting to 100 while clicking the counter with each count. The clicker should read 100 on the 100th count. Frequency is monthly.
- 8.5 Analyst precision and accuracy. Frequency is detailed in Quality Control Section 13.0.
- 8.5.1 A reference slide is submitted to each analyst on a daily basis. This slide should be submitted blindly so the analyst does not know the identity of the sample. The reference slides library should consist of old proficiency, round robin and real time samples where the target concentration and acceptance limits have been established and are known.
- 8.5.2 Measuring Accuracy - To measure and judge accuracy, the analysis must fall within the acceptable limits before analysis may proceed. If the sample falls out of the limits;
- 8.5.2.1 A Corrective Action Response (CAR) must be initiated.
- 8.5.2.2 Another reference slide in the same fiber concentration range must be analyzed acceptably before analysis may proceed.
- 8.5.2.3 The CAR does not need to be closed before analysis may proceed.
- 8.5.3 Measuring Precision - Precision is measured by maintaining a spreadsheet log of all analyses of each reference slide per analyst.
- 8.5.3.1 The most recent 20 analyses of each slide are considered for this measurement and the pooled SD and Mean are calculated and the Coefficient of Variance is then determined. This is detailed in the Asbestos QC SOP.
- 8.5.3.2 Coefficient of Variance results of >0.45 should be considered suspect and an CAR initiated.

9.0 Procedure

9.1 Sample Receipt

- 9.1.1 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.



- 9.1.2 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.
- 9.1.3 Information required on the Chain of Custody includes:
 - 9.1.3.1 Client name, address, telephone number, contact person, fax number
 - 9.1.3.2 Project number/ name, state where samples were taken
 - 9.1.3.3 Number of samples sent and sample ID's
 - 9.1.3.4 Type of analysis requested
 - 9.1.3.5 Sample volumes or areas if applicable
 - 9.1.3.6 Turn around time. "RUSH" is not acceptable
 - 9.1.3.7 A date and signature of the person relinquishing the samples
 - 9.1.3.8 All samples MUST be accounted for with the proper sample ID's
 - 9.1.3.9 All samples MUST be sealed, properly bagged and undamaged.
- 9.1.4 All samples must be clocked in at the time of receipt and signed and dated by an EMSL employee. If the lab does not have clock for sample receipt the receiving employee should record the time of receipt also.
- 9.1.5 Check to see if the samples match the COC and if the cassettes are open, damaged, or contaminated. If the samples are damaged or if the COC does not match, notify the client.

9.2 Sample Log In

If all of the above criteria for sample receiving are met then the sample can be logged in to Sample Master (LIMS) as per the Sample Master SOP.

- 9.2.1 This process will assign a unique EMSL order number for the project as well as unique lab sample ID's.
- 9.2.2 Sample Master will create the appropriate analytical worksheets and an internal chain of custody.
- 9.2.3 Care should be taken for blank correction purposes to identify samples and field blanks from the same batch of samples accordingly. SMXP allows for a Sample Type field to be completed during log in that "links" certain Field Blank samples to associated Sample IDs.
- 9.2.4 All samples in a given "set" should be logged in with "Customer Set X" where the x is a set of samples from 1 to 7.
- 9.2.5 Associated Field blanks for each set should be logged in as "Field Blank X" where x is a set of Field Blanks from 1 to 7 that corresponds to the "Customer Set" of samples in step 9.2.4 above.
- 9.2.6 SMXP will now link the samples and blanks and perform a blank correction (following the procedures outlined in sections 9.4.15 or 9.5.15 for A or B rules respectively) automatically on the associated filters.

9.3 Sample Preparation

- 9.3.1 In a safety hood, place the samples in order corresponding to the COC. Cut the outer band (if present) on the cassettes with a scalpel or strait razor.
- 9.3.2 Lay out in the hood, enough clean 3x1 microscope slides to accommodate all samples in the batch. Each slide can accommodate 2 or 3 sample wedges depending on the person prepping. Lay the slides out parallel to each other either horizontally or vertically on the bottom of the hood.
- 9.3.3 Cut a wedge from the first sample and place it on the first slide. The first slide will be either the left most (if laid out vertically) or bottom most slide (if laid out horizontally) in the set of slides.



- 9.3.4 Place all cut wedges in the corresponding sample order, starting at the top (if laid out vertically) or the left side (if laid out horizontally) of each slide. Place 2-3 wedges on each slide.
- 9.3.5 When all samples have been cut and their wedges placed on the slides, collapse the filters using the acetone vapor generator. This can be done *one of two ways*; the first requires the samples to be heated before analysis.
- Procedure requiring sample heating:
- 9.3.5.1 Draw fresh acetone into a syringe; insert the syringe into the top of the acetone vaporizer or hot block.
- 9.3.5.2 Insert the first slide into the area where the acetone vapors are exhausted, centered on the first wedge to be cleared. Slightly depress the plunger of the syringe; the amount of acetone should be just enough to clear the first wedge on the slide, approximately 250 μ l. Move the slide to the next wedge and continue the process until all wedges on the slide are clear.
- 9.3.5.3 Using a 5 μ l pipette, place one drop of approximately 3.0 - 3.5 μ l of triacetin on each cleared filter wedge, being careful not to touch the sample with the pipette tip.
- 9.3.5.4 Carefully place a clean coverslip on each sample, do not press down on the coverslip, this will foster fiber migration on the filter.
- 9.3.5.5 Place the prepped slide on a slide warmer or hot surface (approximately 50°C) of the acetone vaporizer to complete clearing. When the triacetin is applied to samples after the acetone cleared sample has been allowed to sit momentarily, the slide preparation will become cloudy and this step is needed to ensure a clear prep. Continue until all samples are prepped.
- 9.3.5.6 Optional - outline the cleared filter from underneath the slide.
- Procedure not requiring sample heating:
- 9.3.5.7 Lay cover slips (one per sample wedge) on the bottom of the hood. Arrange the coverslips in the same order as the samples on the slides.
- 9.3.5.8 On each coverslip, place a drop approximately 3.0 - 3.5 μ l of triacetin using a 5 μ l pipette; be careful not to touch the pipette tip to the coverslip.
- 9.3.5.9 Draw fresh acetone into a syringe; insert the syringe into the top of the acetone vaporizer or hot block.
- 9.3.5.10 Insert the first slide into the area where the acetone vapors are exhausted, centered on the first wedge to be cleared. Slightly depress the plunger of the syringe; the amount of acetone should be just enough to clear the first wedge on the slide, approximately 250 μ l. Move the slide to the next wedge and continue the process until all wedges on the slide are clear.
- 9.3.5.11 Immediately, place the slide filter side down, on the coverslips corresponding to the slide in question. Continue until all slides / samples are prepped.
- 9.3.5.12 Optional - outline the cleared filter from underneath the slide.
- 9.3.6 With a permanent marker (such as a 'Sharpie'), write the sample numbers on the slide. This may be done prior to sample prep, when the slides are being laid out in the hood. Care is needed to not "wash" the marking away during the



sample clearing process with acetone vapors. In practice this is possible, but difficult.

- 9.3.7 If the samples cannot be read within 24 hours of prep, each sample coverslip must be sealed with clear nail polish to preserve the prep.

9.4 Sample Analysis "A" Rules

Under normal circumstances analysis of samples via NIOSH 7400 is performed by the "A" counting rules. At the clients discretion however, are alternate "B" counting rules (section 9.5 of this procedure). In the absence of any contrary direction by the client, the "A" rules will be used for analysis of PCM samples via NIOSH 7400.

- 9.4.1 Place the first slide of the sample set to be analyzed on the microscope stage. Adjust the sample translators to bring the first sample to be analyzed into the microscope's field of view. It may be helpful to view the slide at a low magnification (40x) to find and center the samples.
- 9.4.2 With the first sample in the field of view, and at a magnification of 400x, focus the sample taking care not to allow the 40x objective to touch the sample coverslip.
- 9.4.3 Once the filter is focused, observe the filter.
- 9.4.3.1 If the filter is judged to be loaded with > 50 particulate, reject the filter as being too heavily loaded and report as "Overloaded".
- 9.4.3.2 If the filter contains a sample loading that is uneven, reject the filter and report it as "Non-Uniform Distribution".
- 9.4.4 If the filter is acceptable for analysis, move to the cut tip of the filter. Using one stage adjustment control, move the slide to a random field of view, in a direction away from the cut tip and toward the outer edge of the filter.
- 9.4.5 Focus on the field of view, including over and under focusing to see all fibers in the field. If the field of view contains > 1/6 particulate, reject the field and move on. Fibers as defined by this method are:
- 9.4.5.1 Fibers > 5 μ m in length and
- 9.4.5.2 having an aspect ratio \geq 3:1.
- 9.4.6 Count fibers that are completely within the graticle field as one (1) fiber.
- 9.4.7 Count fibers that cross the graticle boundary as follows:
- 9.4.7.1 Count as one half (1/2) fiber any fiber with only one end lying within the graticle field.
- 9.4.7.2 Do not count any fiber that crosses the boundary more than once.
- 9.4.8 Do not count fibers that lie completely outside the graticle field.
- 9.4.9 Count bundles as one (1) fiber unless both ends of individual fibers can be observed, then count the fibers separately.
- 9.4.10 Count fibers protruding from matrix material providing that at least 5 μ m of the fiber is visible and the protrusion has at least a 3:1 aspect ration.
- 9.4.11 Once all fibers within the graticle have been counted, move to the next field of view in the same direction. The analyst should move along a radial line toward the outer edge of the filter when selecting fields of view. Each new field of view should be sufficiently far away from the previous field of view so that the Walton Beckett graticle is not superimposed on areas that in the previous filed of view.
- 9.4.12 Once the edge of the filter has been reach, shift up or over and continue counting in the opposite direction.
- 9.4.13 Continue counting until:
- 9.4.13.1 At least 100 fields have been counted
- 9.4.13.2 At least 100 fibers have been counted in a minimum of 20 fields.



- 9.4.13.3 Always complete counting the final field of the analysis, never stop counting mid-field.
- 9.4.14 Information is recorded on EMSL's PCM worksheet, or in EMSL's Direct Data entry database interface "iLab". For information on the use of "iLab" please see EMSL's Asbestos DDE Manual. Regardless of the method of recording used, all information detailed below is required to be recorded.:
 - 9.4.14.1 The number of fibers counted
 - 9.4.14.2 The number of fields analyzed
 - 9.4.14.3 A check in the "Overloaded" column if the filter was overloaded
 - 9.4.14.4 Any comments that may be pertinent for the sample. This could be if the sample had an un-even loading, if the filter was wet and required drying, the sample volume or any other comment the analyst believes is important enough to record.
- 9.4.15 Repeat the previous steps in this section until all samples have been analyzed. Once all samples and field blanks in the set have been completed, the samples should be blank corrected. To blank correct:
 - 9.4.15.1 Total the number of fibers counted on the field blanks for each batch of samples. It is possible that one EMSL Order # may contain more than one batch of samples and multiple sets of field blanks. It is important that only the field blanks for each batch be averaged and applied to the batch of samples they apply to as described below.
 - 9.4.15.2 If a field blank has been noted for "Possible Contamination" (see 14.1.4 below), it should not be included in the average field blank count.
 - 9.4.15.3 Divide the total fibers on the field blanks by the number of field blanks in the batch of samples of interest. Record this on the bottom of the analytical sheet in the spot for "Average fibers per 100 fields for the Field Blanks"
 - 9.4.15.4 Subtract the average number of fibers for the field blanks from each sample that the field blanks represent. If the sample that will be blank corrected consisted of less than 100 fields, the field blank fiber average must be extrapolated before subtraction. This process is described in Section 10.3 of Calculations.
 - 9.4.15.5 Record this number in the "# Blank Corrected Fibers" column for each sample. This is not necessary for samples processed using "iLab", "iLab" internally handles blank correction.
 - 9.4.14.5.1 Do not blank correct field blanks.
 - 9.4.14.5.2 If the results of this calculation would produce a negative number, record zero (0).
- 9.4.16 After all blank correction has be completed, complete the bottom of the analytical bench sheet by (handled internally if using "iLab"):
 - 9.4.16.1 Signing the analyst's signature
 - 9.4.16.2 Filling in the date of analysis
 - 9.4.16.3 Filling in the Scope ID

9.5 Sample Analysis "B" Rules

At the client's discretion and request, EMSL will analyze PCM samples by NIOSH 7400 "B" rules. This alternate set of counting rules has different criteria for fibers, the "B" rule's fiber criteria is listed in item 9.5.5 below. If a client requests "B" rules be used for analysis, the entire sample must be analyzed by "B" rules, it is not permissible to produce



the analysis using both, or a hybrid of both, sets of rules. Unless the client specifies this alternate counting protocol, EMSL will use the "A" rules for routine analysis of samples via NIOSH 7400.

- 9.5.1 Place the first slide of the sample set to be analyzed on the microscope stage. Adjust the sample translators to bring the first sample to be analyzed into the microscope's field of view. It may be helpful to view the slide at a low magnification (40x) to find and center the samples.
- 9.5.2 With the first sample in the field of view, and at a magnification of 400x, focus the sample taking care not to allow the 40x objective to touch the sample coverslip.
- 9.5.3 Once the filter is focused, observe the filter.
 - 9.5.3.1 If the filter is judged to be loaded with > 50 particulate, reject the filter as being too heavily loaded and report as "Overloaded".
 - 9.5.3.2 If the filter contains a sample loading that is uneven, reject the filter and report it as "Non-Uniform Distribution".
- 9.5.4 If the filter is acceptable for analysis, move to the cut tip of the filter. Using one stage adjustment control, move the slide to a random field of view, in a direction away from the cut tip and toward the outer edge of the filter.
- 9.5.5 Focus on the field of view, including over and under focusing to see all fibers in the field. If the field of view contains > 1/6 particulate, reject the field and move on. Fibers as defined by this part of the method are:
 - 9.5.5.1 Fibers > 5 μ m in length and
 - 9.5.5.2 fibers < 3 μ m in width and
 - 9.5.5.2 having an aspect ratio \geq 5:1.
- 9.5.6 Count each fiber end that falls within the graticle field as one end.
- 9.5.7 Count split ends as long as the split fiber segment meets the criteria in 9.5.5.
- 9.5.8 For fibers attached to particulate, count as follows:
 - 9.5.7.8 Count visibly free ends that meet the criteria in 9.5.5 when they are attached to particulate regardless of the size of the particle they are attached to.
 - 9.5.7.8 Count the end of a fiber obscured by another particle if the particle covering the fiber is < 3 μ m in diameter.
 - 9.5.7.8 Count free ends of fibers attached to or emanating from to large clumps or bundles, up to a maximum of 10 ends, provided each fiber segments counted meets the criteria in 9.5.5.
- 9.5.9 Do not count fibers that lie completely outside the graticle field.
- 9.5.10 Once all fibers within the graticle have been counted, move to the next field of view in the same direction. The analyst should move along a radial line toward the outer edge of the filter when selecting fields of view. Each new field of view should be sufficiently far away from the previous field of view so that the Walton Beckett graticle is not superimposed on areas that in the previous field of view.
- 9.5.11 Once the edge of the filter has been reached, shift up or over and continue counting in the opposite direction.
- 9.5.12 Continue counting until:
 - 9.5.12.1 At least 100 fields have been counted or
 - 9.5.12.2 At least 200 ends have been counted in a minimum of 20 fields.
 - 9.5.12.3 Always complete counting the final field of the analysis, never stop counting mid-field.
- 9.5.13 Divide the number of ends by two (2) to calculate total fibers.



- 9.5.14 Record on the analytical bench sheet in the appropriate line corresponding to the sample that was analyzed:
- 9.5.14.1 The number of fibers counted
 - 9.5.14.2 The number of fields analyzed
 - 9.5.14.3 A check in the "Overloaded" column if the filter was overloaded
 - 9.5.14.4 Any comments that may be pertinent for the sample. This could be if the sample had an un-even loading, if the filter was wet and required drying, the sample volume or any other comment the analyst believes is important enough to record.
- 9.5.15 Repeat the previous steps in this section until all samples have been analyzed. Once all samples and field blanks in the set have been completed, the samples should be blank corrected. To blank correct:
- 9.5.15.1 Total the number of fibers counted on the field blanks for each batch of samples. It is possible that one EMSL Order # may contain more than one batch of samples and multiple sets of field blanks. It is important that only the field blanks for each batch be averaged and applied to the batch of samples they apply to as described below.
 - 9.5.15.2 If a field blank has been noted for "Possible Contamination" (see 14.1.4 below), it should not be included in the average field blank count
 - 9.5.15.3 Divide by the number of field blanks in the batch of samples of interest. Record this on the bottom of the analytical sheet in the spot for "Average fibers per 100 fields for the Field Blanks"
 - 9.5.15.4 Subtract the average number of fibers for the field blanks from each sample that the field blanks represent. If the sample that will be blank corrected consisted of less than 100 fields, the field blank fiber average must be extrapolated before subtraction. This process is described in Section 10.3 of Calculations.
 - 9.5.15.5 Record this number in the "# Blank Corrected Fibers" column for each sample.
 - 9.5.14.5.1 Do not blank correct field blanks.
 - 9.5.14.5.2 If the results of this calculation would produce a negative number, record zero (0).
- 9.5.16 After all blank correction has been completed, complete the bottom of the analytical benchsheet by:
- 9.5.16.1 Signing the analyst's signature
 - 9.5.16.2 Filling in the date of analysis
 - 9.5.16.3 Filling in the Scope ID.

9.6 Procedures for use of TWAs

- 9.6.1 TWA samples are logged into Sample Master using the test "PCM w/TWA".
- 9.6.2 Ensure that the client understands that the calculation is based on an 8 hour exposure period. The final report is titled "...with 8 Hour Time Weighted Average"
- 9.6.3 Samples that are overloaded do not provide a viable analytical result and are not included in a TWA.
- 9.6.4 Sample Master calculates the TWA using the following criteria:
 - 9.6.4.1 STEL (Short Term Exposure Limit) samples that overlap the sampling times of the associated personal samples are not included in the TWA.
 - 9.6.4.2 For the TWA calculation, use the detection limit in f/cc for samples that are reported below the analytical detection limit.



- 9.6.4.3 If all samples are below their associated detection limits, report the final TWA as less than the calculated result.
- 9.6.4.4 Use the TWA calculation as found in section 10.7 of Calculations below.
- 9.6.5 Interpretation of these results is the responsibility of the Industrial Hygienist. TWA can be calculated using various exposure times as directed by the client. For times other than 8 hour, calculate the total exposure time in minutes (denominator). The final report must indicate what total time was used for the calculated result.

9.7 QC Recount Selection, Resubmission and Analysis

- 9.7.1 Recount QC is selected randomly at a rate of 10% of real time analyses.
- 9.7.2 These recounts should be resubmitted blindly to the original analyst, so they do not know the original sample results and preferably sample identity. Optimally this will entail the selection and relabeling of QC recount samples by a second analyst or other lab personal (as per the method).
- 9.7.3 QC samples to be analyzed are listed on a new blank PCM QC bench sheet and resubmitted to the original analyst with the QC slides for analysis.
- 9.7.3 Recounts are analyzed according to the procedures outlined in the analysis section of this SOP (9.4 for A rules, 9.5 for B rules),
- 9.7.4 Recounts are recorded on an appropriate Blank QC Benchsheet;
 - 9.7.4.1 Bench Sheets for non-NYS samples

Fiber Count by Phase Contrast Microscopy (PCM), NIOSH 7400 Method, Revision 3, Issue 2, 8/15/94

Client: Address: Phone: Fax: Project:	Logged: Date/Time Due:	TAT:	QC																								
		Special Instructions	Order ID																								
<table border="1" style="width: 100%; border-collapse: collapse; text-align: center;"> <thead> <tr> <th style="width: 12.5%;">Sample ID</th> <th style="width: 12.5%;">Location</th> <th style="width: 12.5%;">Sample Date</th> <th style="width: 12.5%;"># Fibers</th> <th style="width: 12.5%;"># Fields</th> <th style="width: 12.5%;"># Blank Corrected Fibers</th> <th style="width: 12.5%;">Overloaded</th> <th style="width: 12.5%;">Comment</th> </tr> </thead> <tbody> <tr> <td> </td> </tr> <tr> <td> </td> </tr> </tbody> </table>				Sample ID	Location	Sample Date	# Fibers	# Fields	# Blank Corrected Fibers	Overloaded	Comment																
Sample ID	Location	Sample Date	# Fibers	# Fields	# Blank Corrected Fibers	Overloaded	Comment																				

Fiber Analysis of Air Samples via NIOSH 7400, Revision 3, Issue 2, 8/15/94 (with 8 Hour Time Weighted Average)

Includes OSHA Time-Weighted Average

Client: Address: Phone: Fax: Project:	Logged: Date/Time Due:	TAT:	QC																														
		Special Instructions	Order ID																														
<table border="1" style="width: 100%; border-collapse: collapse; text-align: center;"> <thead> <tr> <th style="width: 12.5%;">Sample ID</th> <th style="width: 12.5%;">Location</th> <th style="width: 12.5%;">Sample Date</th> <th style="width: 12.5%;"># Fibers</th> <th style="width: 12.5%;"># Fields</th> <th style="width: 12.5%;">Start Rate</th> <th style="width: 12.5%;">Stop Rate</th> <th style="width: 12.5%;"># Blank Corrected Fibers</th> <th style="width: 12.5%;">Overloaded</th> <th style="width: 12.5%;">Comment</th> </tr> </thead> <tbody> <tr> <td> </td> </tr> <tr> <td> </td> </tr> </tbody> </table>				Sample ID	Location	Sample Date	# Fibers	# Fields	Start Rate	Stop Rate	# Blank Corrected Fibers	Overloaded	Comment																				
Sample ID	Location	Sample Date	# Fibers	# Fields	Start Rate	Stop Rate	# Blank Corrected Fibers	Overloaded	Comment																								



9.7.4.2 In the case of NYSDOH ELAP certified labs, on a QC Benchsheet that allows for recording the number of fibers in each field counted.

NYS PCM QC Sheet

EMSL NYS PCM QC
 Revision 3
 May 2007

Analyst: _____

Scope : _____

Date: _____

Date: _____

Lab #:		QC #:							
Orig	F/F	F/mm2							
QC:	F/F	F/mm2							
1	2	3	4	5	6	7	8	9	10
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									

Lab #:		QC #:							
Orig	F/F	F/mm2							
QC:	F/F	F/mm2							
1	2	3	4	5	6	7	8	9	10
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									



9.7.4.3 Older style PCM QC recording tables should not longer be used for PCM QC.

EMSL PCM QC Analysis Benchsheet
 Revision 1
 Effective Date: October 26, 2007



PCM QC ANALYSIS SHEET

Month/Year: _____ Scope: _____

Date	Billing Number	Sample Number	Analyst	First Count fibers /fields	Second Count fibers/fields

9.7.6 After QC analysis is complete both original and QC results are entered into the PCM QC spreadsheet, as detailed in the Asbestos QC SOP.

10.0 Calculations

When calculating fiber density and concentrations, and the fiber count is less than 5.5 fibers (the detection limit) use 5.5 fibers in place of the actual fiber count.

For all calculations:

E - fiber density of filter in fibers / mm²

C - fiber concentration of air sampled fibers / cc

F - fibers counted during analysis

B - average fiber count per field blank

B_{corr} - average fiber count per field blank corrected for samples where < 100 field are counted

F_{bc} - blank corrected fiber count

UCL- upper 95% confidence limit of fiber count

A_f - Area of one (1) Walton Beckett graticle field (0.00785mm²)

- number of graticle fields analyzed in sample

EFA - effective filter area of sample filter

Vol - volume of air sampled in liters (L)

CV - coefficient of variance

10.1 Filter Density

$$E = \frac{F_{bc}}{A_f \cdot \#} \quad E = \frac{12.5}{0.00785 \cdot 100} \quad E = 15.9 \text{ f/mm}^2$$



10.2 Sample (Air) Concentration

$$C = \frac{EFA \cdot F_{bc}}{A_f \cdot \# \cdot 1000 \cdot Vol} \quad C = \frac{385 \cdot 12.5}{0.00785 \cdot 100 \cdot 1000 \cdot 1200} \quad C = 0.0051 \text{ f/cc}$$

10.3 Fiber Count Blank Correction

10.3.1 For fibers counts that end with 100 graticle fields analyzed.

$$F_{bc} = F - B \quad F_{bc} = 13 - 0.5 \quad F_{bc} = 12.5 \text{ fibers}$$

10.3.2 For fiber counts that end with fewer than 100 graticle fields.

$$B_{corr} = \frac{\#}{100} \cdot B \quad B_{corr} = \frac{57}{100} \cdot 1.5 \quad B_{corr} = 0.855$$

then

$$F_{bc} = F - B_{corr} \quad F_{bc} = 101 - 0.855 \quad F_{bc} = 100.15 \text{ fibers}$$

10.4 Upper Confidence Limit

The upper confidence limit is calculated for the fiber count. This result can then be substituted into the formulas for fiber density and/or sample concentration to calculate the UCL in either f/mm² or f/cc.

$$UCL = \frac{2 \cdot F_{bc} + 2.25 + \left[(2.25 + 2 \cdot F_{bc})^2 - 4(1 - 2.25 \cdot CV^2) F_{bc}^2 \right]^{1/2}}{2(1 - 2.25 \cdot CV^2)}$$

$$UCL = \frac{2 \cdot 12.5 + 2.25 + \left[(2.25 + 2 \cdot 12.5)^2 - 4(1 - 2.25 \cdot 0.25^2) 12.5^2 \right]^{1/2}}{2(1 - 2.25 \cdot 0.25^2)}$$

$$UCL = 24.19 \text{ fibers}$$

10.5 QC Recount Acceptance / Rejectance:

Where E1 is the first or original result (in f/mm²) and E2 is the second or QC result. The test fails when the equation is true.

$$\left| \sqrt{E_1} - \sqrt{E_2} \right| > 2.8 \cdot \left(\frac{\sqrt{E_1} + \sqrt{E_2}}{2} \right) \cdot \frac{CV}{2}$$

$$\left| \sqrt{15.9} - \sqrt{25.5} \right| > 2.8 \cdot \left(\frac{\sqrt{15.9} + \sqrt{25.5}}{2} \right) \cdot \frac{0.18}{2}$$

1.06 > 1.14 is false so the analysis passes.



10.6 TWA (Time Weighted Average)

Where C_i and T_i are the concentrations (in f/cc) and time (in minutes) of a particular sample up to and including the n^{th} sample:

$$TWA = \frac{(C_1 \cdot T_1) + (C_2 \cdot T_2) + \dots + (C_n \cdot T_n)}{480} \quad TWA = \frac{(0.1 \cdot 60) + (0.02 \cdot 210)}{480}$$

$$TWA = 0.021 \text{ f/cc}$$

11.0 Reporting

The following items are included in the final report (on EMSL letterhead) to the client:

- 11.1 Fiber concentration in fibers/mm²
- 11.2 Fiber concentration in fibers/cc (if volume is supplied)
- 11.3 Detection limit of the analysis in fibers/cc
- 11.4 Number of fibers counted
- 11.5 Number of graticle fields analyzed.
- 11.6 The date the sample was taken (if supplied)
- 11.7 Any notes pertaining to the sample
- 11.8 Volume of air collected by the client (if supplied)
- 11.9 Location of the sample in the field (if supplied)
- 11.10 Client and Lab sample numbers
- 11.11 Client identification and contact information
- 11.12 EMSL Order ID
- 11.13 Client Project information (if supplied)
- 11.14 Sampling (if supplied), analysis and report date
- 11.15 Signature of Lab Manager
- 11.16 Report comments
- 11.17 Lab accreditations
- 11.18 The following disclaimers:
 - 11.18.1 "The laboratory is not responsible for the data in fibers/cc, which is dependent on volume collected by non-laboratory personnel."
 - 11.18.2 "This report relates only to the samples reported above. The report may not be reproduced, except in full, without written approval by EMSL."
 - 11.18.3 If results exceed the recommended upper density range of 1300 f/mm² for the method, the results must be noted with a sample comment as "Possibly Biased"
 - 11.18.4 If the sample contains a particulate loading of ≥50%, but the client requests the sample be analyzed anyway (if possible), the sample result should be reported with a comment stating "Sample Overloaded, analyzed at client's request. Possibly Biased"
- 11.19 For reports that determine TWA (time weighted average) the following are also reported:
 - 11.19.1 Activity instead of sample location
 - 11.19.2 Start and Stop Time of samples
 - 11.19.3 Flow rate of samples
 - 11.19.4 Notes/Comments relating to whether the sample is included in the TWA or not.
 - 11.19.5 TWAs broken down by collector and day collected.
- 11.20 Reporting Uncertainty to clients
 - 11.20.1 Refer to the excel spreadsheet: **Uncertainty Worksheet – Asbestos.**



- 11.20.2 The concentration at the upper and lower confidence limits will be calculated using the calculations that are in the NIOSH 7400 method (page11).

$$UCL = \frac{2 X + 2.25 + [(2.25 + 2 X)^2 - 4 (1 - 2.25 S_r^2) X^2]^{1/2}}{2 (1 - 2.25 S_r^2)}$$

$$LCL = \frac{2 X + 4 - [(4 + 2 X)^2 - 4 (1 - 4 S_r^2) X^2]^{1/2}}{2 (1 - 4 S_r^2)}$$

Where S_r = subjective interlaboratory relative standard deviation, which is close to the total Inter-laboratory S_r when approximately 100 fibers are counted.

X = total fiber counted on sample

LCL = lower 95% confidence limit.

UCL = upper 95% confidence limit.

Note that the range between these two limits represents 90% of the total range.

- 11.20.3 Results can be reported with a stated value of uncertainty where requested by the customer or accreditation agency. This uncertainty is expressed as the lab's relative standard deviation (S_r) for the method. A S_r is generated by each laboratory for each analyst in 3 ranges and then pooled to determine the laboratory S_r . This value is also used in the calculation for the acceptance criteria of the QC recount (see above section QC Recount Acceptance / Rejectance).
- 11.20.4 If requested by the client, this uncertainty is reported as the (+/-) % value without application to a specific result.

12.0 Method Performance

Method performance data can be found in the NIOSH method 7400 - Asbestos and Other Fibers by PCM.

12.1 MDL

The method detection limit is stated as 7 fibers / mm^2 . This translates to 5.5 fibers per 100 fields using a graticle (field) area of $0.00785mm^2$.

12.2 DOC's

Demonstrations of Capability (DOC) are required for each analytical method. For compliance w/ NYS ELAP when analyst sample loads exceed 2 samples per hour, and initial DOC (iDOC) consisting of a timed analysis of 25 PT samples must be conducted. This DOC must be challenged quarterly, documentation of this review must be maintained.

12.3 PT's

Proficiency tests for this method exist from AHIA in the form of PAT and AAR (Asbestos Analyst Registry) samples and also from NY State ELAP.



12.4 Accuracy

There is no independent means of assessing the overall accuracy of this method although it may be possible to do so utilizing large numbers of inter-laboratory analyses of a single sample. EMSL attempts this by reanalysis of reference slides on a daily basis. Regardless it should be noted that analysis of lightly loaded samples $< 100 \text{ f/mm}^2$ is not as accurate as analysis of samples in the recommended loading range (100 - 1300 f/mm²) and are most likely an overestimate of the actual density.

12.5 Precision

Precision is determined per analyst over loading ranges 5-20, 21-50 and >51 fibers per 100 fields as the coefficient of variance (also called relative standard deviation). An overall laboratory precision is also calculated.

13.0 Quality Control

- 13.1 All QC data must be maintained and available for easy reference and inspection.
- 13.2 QC that is part of new analyst's training cannot be used as QC for real time samples.
- 13.3 Field blanks, when provided, are analyzed and sample results are blank corrected. Field blanks should be submitted at a rate of 10% or a minimum of 2 per sample set.
- 13.4 Reference samples are analyzed daily per analyst.
- 13.5 Intra-Analyst QC is done at a rate of 10% (excluding blanks).
- 13.6 Inter-Analyst QC is not performed with this method.
- 13.7 Inter-laboratory QC in the form of Round Robins should be conducted.

14.0 Data Assessment

14.1 Acceptance criteria for QC measures

These are addressed in the EMSL's QA Manual Module A section A.11.3

- 14.1.1 Recounts are compared using the analyst's CV for the fiber range in question and formula 10.6 in the calculations section.
- 14.1.2 If a real time sample falls outside the acceptable limits:
 - 14.1.2.1 It needs to be reconciled with the analyst and/or a 2nd analyst when necessary.
 - 14.1.2.2 If a non-analytical cause was determined (ie. analysis of the wrong sample, incorrect data entry, etc.) the data is corrected and the sample results are re-evaluated by the PCM recount QC evaluation criteria. If sample Passes this test, report the results as normal.
 - 14.1.2.3 If a non-analytical cause for the failure cannot be found, all samples in the sample set are to be reanalyzed and tested for using the PCM recount QC evaluation formula for Pass/Failure.
 - 14.1.2.4 For samples that Passed this QC test, report the original results.
 - 14.1.2.5 For samples that Fail this QC test, re-prepare and recount all samples that failed QC. Perform recounts on all re-prepped samples and report all Passing samples as usual.
 - 14.1.2.6 Any sample results failing this second QC test, if reported, must include a sample comment noting the QC Failure.
- 14.1.3 Reference slide analysis acceptance is determined:
 - 14.1.3.1 For past proficiency samples by comparing the daily analysis to the published acceptance limits for the sample.



- 14.1.3.2 For past real time samples by comparing the daily analysis to the calculated limits for the sample, after multiple analyses to determine mean, standard deviation and limits.
- 14.1.3.3 Analysis of samples may not start until a satisfactory reference sample analysis is completed on a daily basis.
- 14.1.3.4 Failed analyses are not removed from the data pool.
- 14.1.4 Field blanks containing more than 7 fibers per 100 graticule fields are noted with a report comment as being possibly contaminated and are not used for blank correction.

14.2 Corrective Actions

These policies are addressed fully in the EMSL's QA manual section 13.

14.2.1 All corrective actions should look for the root cause of the error.

14.2.2 All out of control or unacceptable data must be brought to the attention of the Laboratory Manager.

14.2.3 The Laboratory manager is responsible for generating a corrective action including an investigation of calibration procedures, a review of analytical technique and investigation of training policies and compliance.

14.2.4 Corrective actions will be reported to the QA Department by means of the Quarterly Management Report or sooner when appropriate.

14.3 Contingencies for handling out-of control or unacceptable data.

Any quality control requirements not met must have an explanation to their nonconformance.

15.0 Pollution Prevention / Waste Management

15.1 Pollution Prevention

EMSL Analytical makes all efforts to reduce the volume and toxicity of the waste generated by the laboratory. An effort to manage procurement of hazardous materials has been implemented in order to avoid over ordering. Hazardous waste is classified for proper disposal.

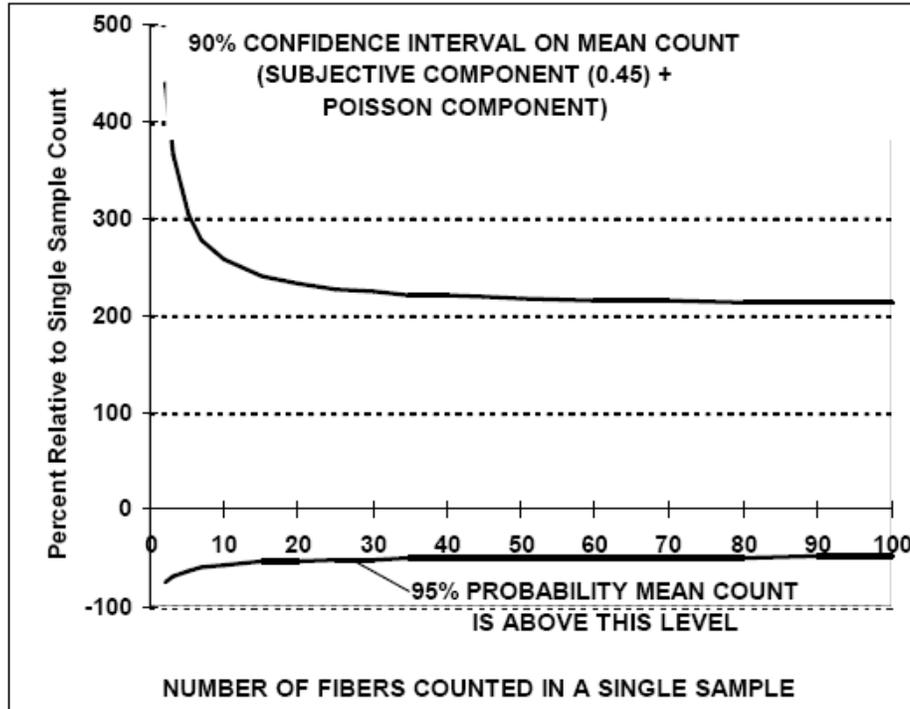
15.2 Waste Management

The waste generated during prep and analysis will be disposed of following safety procedures outlined in the chemical hygiene plan (EMSLChemHygiene 200.0).



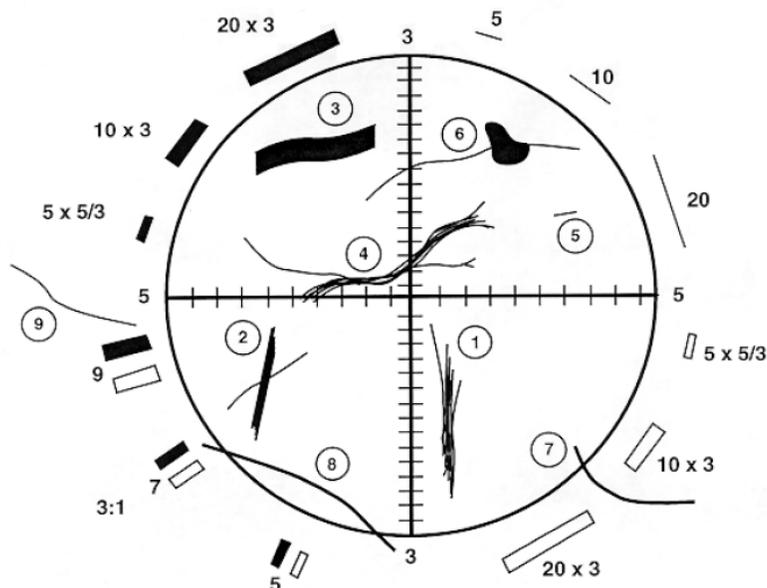
16.0 Tables, Diagrams, Flowcharts, and Validation Data

16.1 Example confidence limits for NIOSH 7400



16.2 Example fibers and counts.

Discussion of the fiber counts are continued on the next page.





<u>FIBER COUNT</u>		
<u>Object</u>	<u>Count</u>	<u>DISCUSSION</u>
1	1 fiber	Optically observable asbestos fibers are actually bundles of fine fibrils. If the fibrils seem to be from the same bundle the object is counted as a single fiber. Note, however, that all objects meeting length and aspect ratio criteria are counted whether or not they appear to be asbestos.
2	2 fiber	If fibers meeting the length and aspect ratio criteria (length >5 • m and length-to-width ratio >3 to 1) overlap, but do not seem to be part of the same bundle, they are counted as separate fibers.
3	1 fiber	Although the object has a relatively large diameter (>3 • m), it is counted as fiber under the rules. There is no upper limit on the fiber diameter in the counting rules. Note that fiber width is measured at the widest compact section of the object.
4	1 fiber	Although long fine fibrils may extend from the body of a fiber, these fibrils are considered part of the fiber if they seem to have originally been part of the bundle.
5	Do not count	If the object is • 5 • m long, it is not counted.
6	1 fiber	A fiber partially obscured by a particle is counted as one fiber. If the fiber ends emanating from a particle do not seem to be from the same fiber and each end meets the length and aspect ratio criteria, they are counted as separate fibers.
7	1/2 fiber	A fiber which crosses into the graticule area one time is counted as 1/2 fiber.
8	Do not count	Ignore fibers that cross the graticulate boundary more than once.
9	Do not count	Ignore fibers that lie outside the graticule boundary.

17.0 References

- 17.1 NIOSH 7400 - Asbestos and Other Fibers by PCM Revision 3 Issue 2 - August 15, 1994.
- 17.2 Less is Better- Guide to Minimizing Waste in Laboratories prepared by the Task Force on Laboratory Environment, Health and Safety- American Chemical Society 2002.
- 17.3 EMSL QA 101.7 Revision 7, September 2004– EMSL Quality Assurance Manual
- 17.4 EMSL QA Manual Revision 9 April 2007
- 17.5 EMSL Chemical Hygiene Plan Revision 0, September 2004
- 17.6 ISO/IEC 17025:2005 General requirements for the competence of testing and calibration laboratories.
- 17.7 American Hygiene Association: *Quality Assurance manual for Industrial Hygiene Chemistry*. Fairfax, VA. American Industrial Hygiene Association 1995.



18.0 Revision History

Formatted to comply with NELAC and ISO standard requirements
 Included counting criteria following NIOSH 7400 B rules

<u>Revision</u>	<u>Revision Date</u>	<u>Notes</u>	<u>Initials</u>
10	1/22/08	Added Corporate Review Section	
11	2/22/08	Reformatted for more cohesiveness with NELAC and ISO standard format. Modified procedures to accommodate multiple accrediting agencies.	
12	9/12/08	Added Reporting of Uncertainty to client in section 11.2. Section 13.6 should read: Inter-Analyst QC is not performed with this method.	
13	6/4/09	Added section 11.20.3 (Pooled Sr Calculations). Replaced Signature Page with Acknowledgement Form Added Authorizing Signature Section Fixed numbering in section 10. It skipped from 10.4 to 10.6	PK/MAC
13.1	9/8/2009	Changed Section 14.1.2.4 to not remove failed reference sample data. Changed Blank Correction procedure (9.4.15 & 9.5.15) to exclude blank reported as possibly contaminated as detailed in section 14.1.4.	KN
13.2	11/13/2009	9.4.14 added references to DDE via EMSL's "iLab" and referenced the "Asbestos DDE Manual" for instructions on use. 9.4.15.5 & 9.4.16 referenced internal handling of data recording if using "iLab"	KN
14	11/12/2010	Added suggested quantities for acetone, triacetin and temperature in sections 9.3.5.2/9.3.5.10, 9.3.5.3/9.3.5.7 and 9.3.5.5 respectively. Added verbiage in 14.1.4 indicating that a "Report" comment noting possible contamination is added when a field blank is found to exceed 7 fibers per 100 graticule field. Modified definition of a fiber in 3.3. Added required cover glass thickness of 1.5 in 5.2 Added calibration criteria for additionally available HSE/ULO test slides in 8.3.3 Added details for requirements of passing reference slide analysis on a daily basis and required CAR initiation in 8.5 Added SMXP log in requirements of Sample Type field so Customer Sets and Field Blank Sets match to ensure correct blank correction calculations in 9.2 Corrected step numbering issues in section 14.1. Added requirement and frequency for all PCM Scope calibrations to be specific to per analyst / per microscope in section 8.3 9.7 Added requirement that all PCM QC be resubmitted blindly to the individual analyst, and to be recorded on PCM QC Benchsheets that do not maintain both original and QC results as available on EMSL's elink repository. Section 7.7 details that the PCM sample preps may be discarded only after sample QC is complete and passing. Added sections 18.11.3 & 4 to address report samples results when the fibers loading exceeds 1300 f/mm ² and when overloaded samples are analyzed at the client's request respectively. 14.1.2 Modified procedure for handling samples set where Recount QC Fails.	KN
14.1	11/15/2010	3.3 Changed definition of Fiber to be >5µm instead of ≥5µm.	KN
14.2	10/21/2011	Updated acceptance criteria for Phase Shift Detection Calibration in section 8.3.3. Updated required frequency (Daily per analyst / scope combination) for measurement of Walton Becket graticle when compliance with TNI Standard (NELAC) is required section 8.3.4. Updated section 12.2 for compliance with NYS DOH ELAP's iDOC utilizing a timed speed test on PT samples with the DOC being challenged quarterly.	KN



Authorizing Signatures

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APPENDIX C

ASTM E1908-10: Standard Guide for Sample Selection of Debris Waste from a Building Renovation or Lead Abatement Project for Toxicity Characteristic Leaching Procedure (TCLP) Testing for Leachable Lead (Pb)



Standard Guide for Sample Selection of Debris Waste from a Building Renovation or Lead Abatement Project for Toxicity Characteristic Leaching Procedure (TCLP) Testing for Leachable Lead (Pb)¹

This standard is issued under the fixed designation E1908; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide describes a method for selecting samples of building components coated with paints suspected of containing lead. The samples are collected from the debris waste stream created during demolition, renovation, lead hazard control, or abatement projects. The samples are subsequently analyzed in the laboratory for lead.

1.1.1 The debris waste stream is assumed to have more than one painted component, for example, metal doors, wood doors, and wood window trim.

1.2 This guide is intended for use when sampling to test for lead only and does not include sampling considerations for other metals or for organic compounds. This guide also does not include consideration of sampling for determination of other possible hazardous characteristics of the waste.

1.3 This guide assumes that the individual component types comprising the debris waste stream are at least partially segregated and that the volume of each type of component in the debris waste stream may be estimated.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

D4840 Guide for Sample Chain-of-Custody Procedures

E105 Practice for Probability Sampling of Materials

E2239 Practice for Record Keeping and Record Preservation for Lead Hazard Activities

2.2 Federal Documents:³

40 CFR 261 Appendix II-Method 1311, Toxicity Characteristic Leaching Procedure (TCLP)

2.3 OSHA Standard:⁴

29 CFR 1926.62 OSHA Lead in Construction Standard

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *component (of the waste), n*—each of those different and distinguishable materials that comprise the waste.

3.1.2 *sample (of the waste), n*—a collection of the components of the waste assembled in proportion to their contribution to the total volume of the waste.

3.1.3 *waste, n*—material resulting from conduct of a demolition, renovation, or lead abatement project that is or will be directed for disposal.

3.1.4 *waste stream, n*—the total flow of waste from a demolition, renovation, lead hazard control or abatement project.

NOTE 1—Regulations promulgated by authorities having jurisdiction may define terms in 3.1.1-3.1.4 differently than defined above.

4. Summary of Practice

4.1 The entirety of the debris waste stream created by demolition, renovation, lead hazard control or abatement projects in and around buildings and related structures is examined visually, and estimates made of the total volume of the waste and of the relative volume proportions of the various components of the waste. A sample of the waste is selected and assembled that contains the various waste components in the same relative volume proportions as these estimates. The

¹ This guide is under the jurisdiction of ASTM Committee E06 on Performance of Buildings and is the direct responsibility of E06.23 on Lead Hazards Associated with Buildings

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, <http://www.access.gpo.gov>.

⁴ Available from Occupational Safety and Health Administration (OSHA), 200 Constitution Ave., NW, Washington, DC 20210, <http://www.osha.gov>.

sample is then submitted to a laboratory for conduct of the Toxicity Characteristic Leaching Procedure (TCLP) for lead in accordance with **40 CFR 261**.

5. Significance and Use

5.1 Waste samples collected using this guide provide representative samples for analysis in a laboratory using the TCLP.

5.2 The TCLP is used to simulate the transfer of lead from buried lead-containing waste into the ground water system upon codisposal of the lead-containing waste and municipal solid waste in unlined solid-waste landfills. The TCLP attempts to simulate rain or ground water leaching, or both. For the procedure to yield a predictor of the subsurface (in-ground) leaching process, a representative sample of the volume of the waste must be selected and submitted for leaching and analysis. The result of the sampling, leaching, and analysis process is used to determine the waste handling and disposal protocols to be followed and to document compliance with applicable laws, regulations, and requirements. The guide addresses the sampling process by defining a component-volume-based method to collect and assemble a representative sample of a solid waste stream that may contain heterogeneous components.

5.3 The collection of a volume-based sample of the waste stream is based on the fact that the TCLP leachate lead concentration limit, like other such TCLP limits, was developed based on the spatial dimensions of landfills.

5.4 Individuals who use this guide are expected to be trained in the proper and safe conduct of sampling of lead-containing wastes, qualified/certified/licensed as required by those authorities having jurisdiction over such activities, and properly utilize tools and safety equipment when conducting these procedures.

5.5 This guide may involve use of various hand and power tools for sampling the components of the waste. It is intended that such tools should be properly and safely used by persons trained and familiar with their performance and use.

5.6 In general terms, building components are drilled to collect samples of the various components in proportion to the volume of those components in the entire building. The component samples are assembled, and the resulting assembled sample is analyzed according to the TCLP protocol.

6. Sampling Supplies

6.1 *Hand and Power Tools*, as needed for the sampling and handling of the various components of the waste (for example, saws, metal snips).

6.2 *Personal Protective Equipment (PPE) and Systems*, as appropriate for the safe collection and handling of the waste.

NOTE 2—Those requirements contained in **29 CFR 1926.62**, for exposure to lead, may be considered. The presence of other chemical hazards in the waste may necessitate the application of other such standards.

6.3 *Containers*, of construction, size, and number to fully hold the waste sample assembled from the various compo-

nents. These containers may be available from the laboratory to which the assembled waste sample is to be sent for analysis.

6.4 *Markers, pens, self-adhesive labels*, for use in uniquely identifying samples of waste collected.

6.5 *Chain of Custody Forms*, similar to those described in Practice **D4840**. These forms may be available from the laboratory to which the assembled waste sample is to be sent for analysis.

7. Procedure

7.1 Determine the Volume Proportions of the Sample

7.1.1 Estimate the volume of each pile of painted waste that has been segregated according to component type, for example metal doors, wood doors, and wood window trim.

7.1.2 Calculate the volume proportion of each component type as a percent of the total volume of the waste.

NOTE 3—If the volume estimate was made in units of cubic feet (ft³), convert to cubic metres (m³) by multiplying the value in cubic feet (ft³) by 0.0283 as follows:

$$0.0283 \times (\text{volume in cubic feet}) = (\text{volume in cubic metres})$$

NOTE 4—If the volume estimate was made in units of cubic yards (yd³), convert to cubic metres (m³) by multiplying the value in cubic yards (yd³) by 0.765 as follows:

$$0.765 (\text{volume in cubic yards}) = (\text{volume in cubic metres})$$

7.2 Collect a Volume Proportional Sample

7.2.1 Collect randomly a waste sample having a mass between 0.25 kg and 1 kg. Follow Practice **E105** to assure that the material collected is representative (NOTE 5).

NOTE 5—After preanalysis processing by the laboratory, this amount of the waste should be enough to yield a minimum of two 100-g TCLP specimens.

7.2.2 The amount collected from each pile shall be taken according to the volume proportion of each pile (NOTE 6).

NOTE 6—For example, assume that the project involved disposal of painted doors, door frames, windows, window frames or trim molding, or both, from several rooms. Assume further that glass is removed to be recycled. Segregated waste piles might then contain metal doors, wood doors (solid and hollow separately), metal door and window frames (no glass), wood door and window frames (no glass), and wood molding. As in 7.1.1, estimate the volume of the waste in each pile and calculate the volume proportions. See Table 1. As in 7.2.2, use the volume proportions to collect the appropriate amount of material from each pile. In this example, the sample would be 17 % wood by volume from window frames.

TABLE 1 Example of Determining Volume Proportions

Painted Component	Estimated Volume	Volume Proportion
wood doors, solid	0.081 m ³	50 % (= 0.081/0.163)
door frames, wood	0.020 m ³	12 %
windows, wood	0.031 m ³	19 %
window frames, steel	0.003 m ³	2 %
window frames, wood	0.028 m ³	17 %
total	0.163 m ³	100 %

8. Sample Handling and Analysis

8.1 Preparation for Shipment

8.1.1 Prepare the total amount of waste collected as the sample from each pile for shipment (7.2.2) to a laboratory for analysis (Note 7).

NOTE 7—All the material sent to the laboratory comprises a single sample of the debris waste stream that is broken, cracked, crushed, cut, ground, etc., as a whole and is homogenized before a subsample is taken for TCLP analysis.

8.1.1.1 Place the total amount of waste collected as the sample into one or more containers. Assure that each container is securely closed to prevent sample loss or contamination during handling and transportation.

8.2 Label each sample container with a unique identifier.

8.3 Complete the request-for-analysis paperwork as required by the laboratory for analysis of the waste sample. Advise the laboratory of the number and types of containers sent and the means and methods of delivery, and that TCLP for lead is to be performed (Note 8).

NOTE 8—It is prudent to contact the analytical laboratory to determine how laboratory personnel intend to or should process the sample prior to subsampling for conduct of the TCLP.

8.4 Complete a Chain of Custody form such as described in Practice D4840.

8.4.1 Record the sample container identifiers on the Chain of Custody form.

8.5 Package the sample container(s) for shipment and label the over-pack, if one is used, according to applicable transportation laws and regulations.

8.5.1 Enclose the completed Chain of Custody form in the shipment package.

8.6 Ship the packaged waste sample to a laboratory for analysis

9. Record Keeping

9.1 Records shall be maintained in accordance with Practice E2239 and shall include, at a minimum, a copy of the report.

10. Report

10.1 A report shall be prepared and include at a minimum the following:

10.1.1 A description of the project and site from which the waste was sampled including names, addresses, locations, waste-producing processes, and dates.

10.1.2 A list and description of the components sampled from the waste stream.

10.1.3 The estimated volumes of each component waste pile.

10.1.4 The calculated volume proportion of each component comprising the entire waste sample.

10.1.5 A copy of the Completed Chain of Custody Form.

10.1.6 A copy of paperwork that was prepared as a record of the laboratory submission and analysis request.

10.1.7 A copy of any packing list(s) or shipping papers, or both, used in shipment of the waste sample to the laboratory.

11. Keywords

11.1 abatement; lead; TCLP; waste

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