Asbestos Analysis of Vermiculite and Vermiculite Containing Materials

New England AIHA
Industrial Hygiene Conference
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Vermiculite

- Hot button issue especially in certain areas of the country and Canada.
- NY State has made it a focal point.
- There is a lot of discussion about the best analytical approach for asbestos in vermiculite and VCMs but little actual data.
Vermiculite Analysis
Vermiculite

LIGHT   MEDIUM   DARK
Vermiculite Ore

Photo courtesy of www.mesotheliomasymptoms.com
Asbestos in Vermiculite - History

Libby, Montana

Vermiculite expands or "pops" when heated. This creates pockets of air that make the material suitable for use as insulation.

Dr. Alley founded the Zonolite Company and developed the mine at Libby.

- The Libby mine was the source of estimated 70-90% of all vermiculite sold in the U.S. from 1919 to 1990.
- W. R. Grace bought the mine and processing facility in 1963 and operated it until 1990.
Vermiculite’s Many Uses

- Block Fill
- Fertilizer
- Vermiculite fire board
Vermiculite Uses

General
- Loose fill
- Carriers
- Density modifiers
- Fire Protection
- Insulation

Industrial
- Absorbent Packing
- Brake Pads / Shoes
- Drilling Muds
- Filtration Beds
- Fireproof safes
- Furnaces
- Paints
- Sealants

Construction
- Floor and Roof Screed
- Gypsum Plaster
- Loft Insulation
- Acoustic finishes
- Sound Deadening
- Spray on Fireproofing

Horticultural
- Hydroponics
- Potting soil Mixes
- Carrier for fertilizers and insecticides
Asbestos in Vermiculite

Libby, Montana

• Unfortunately, veins of asbestos contaminated most, if not all of the material taken from the mine; Libby Amphibole was an “accessory mineral”.

• EPA has been working in Libby since 1999 when an Emergency Response Team was sent to investigate local concern and news articles about asbestos-contaminated vermiculite.
Asbestos in Vermiculite
Libby, Montana
Asbestos in Vermiculite

Libby, Montana

“Libby Amphiboles” is a collective term for the complex mixture of amphiboles that are known to exist at the Libby site.

In order of decreasing abundance:

- **Winchite** (non-regulated amphibole)
- **Richterite** (non-regulated amphibole)
- **Tremolite** (regulated amphibole)

Not as prevalent but identified:

- Magnesioriebeckite (non-regulated amphibole)
- Edenite (non-regulated amphibole)
- Magnesio-arfvedsonite (non-regulated amphibole)
Asbestos in Vermiculite – Health Effects
Libby, Montana

- **Asbestosis** - A serious, chronic, non-cancerous respiratory disease. It is a scarring of lung tissue caused by the presence of asbestos fibers.

- **Mesothelioma** - Mesothelioma is a rare form of cancer which occurs in the thin membrane lining of the lungs, chest (pleural cavity), abdomen (peritoneal cavity)

- **Lung Cancer** - An uncontrolled, malignant growth of cells in the lung tissue. Indistinguishable from lung cancer seen without asbestos exposure.

Latency period (slow response, 15-40 years) for onset of disease from first exposure for Lung Cancer and Meso
Libby Montana

Increased mortality rates are observed not only with the mine workers and their families but even in citizens with no direct connection to the mine.

Mortality rates 40 times higher than the rest of Montana and 60 times higher than rest of the US.

These mortality rates are associated not just with regulated asbestos but other amphiboles as well.

Libby Amphiboles = NRA
NRA ≈ Libby Amphiboles
Major Receiving Points

Over 250 expansion sites
Asbestos in Vermiculite
Libby, Montana
Mine site mid 1900’s
Asbestos in Vermiculite

Libby, Montana

Mine site in 2009
Asbestos in Vermiculite

Libby, Montana

Mine site today
Libby

Contaminated Soil Returned to the Mine
Libby

VAI Removed from Homes
The Scale of the Issue

Production Levels and Ore Content

• 80% of world production for 70 years, 50% of U.S. supply in 1990

• Ore is up to 20% asbestos, airborne particles up to 40% asbestos (Grace insisted ore was <1% asbestos)

• 15.6 billion pounds of asbestos contaminated Zonolite shipped and distributed around North America
Vermiculite Attic Insulation

Between 10-30 Million Homes are Estimated to Contain Vermiculite!

Pouring and leveling Zonolite Insulation Fill.
Regulatory Guidance
EPA and ATSDR launch a national consumer awareness campaign for homeowners with vermiculite attic insulation (VAI) which may contain asbestos.

People who have homes with vermiculite attic insulation should become informed, not alarmed,” said Stephen L. Johnson, EPA’s Assistant Administrator for the Office of Prevention, Pesticides, and Toxic Substances.
Key Recommendations for Homeowners

- Do not disturb vermiculite attic insulation.
- Don’t go into the attic.
- Boxes or other items should not be stored in attics if retrieving them will disturb the insulation.
- Homeowners should never attempt to remove the vermiculite insulation.
- If removal is necessary, hire professionals trained and certified to safely remove the material.
New York DOH Guidance on Vermiculite
2. If Item 198.1 indicates that the material contains greater than 10% vermiculite, continue with Item 198.6 to determine asbestos content. Gravimetric reduction is required.

   a. If asbestos is 1% or less, report as non-ACM according to Section 6 of Item 198.6 with the following conspicuous disclaimer:

   “This method does not remove vermiculite and may underestimates the level of asbestos present in a sample containing greater than 10% vermiculite.”

   b. If asbestos is greater than 1%, report as ACM according to Section 6 of Item 198.6 with the following conspicuous disclaimer:

   “This method does not remove vermiculite and may underestimates the level of asbestos present in a sample containing greater than 10% vermiculite.”

• Addison Davies?
• Liquid Separation?
Samples Primarily Affected

- Spray on fireproofing
- Textures, Popcorn Ceilings, etc.
New York Guidance – 06/22/12

Dear Interested Party,

Additional Vermiculite guidance:

<table>
<thead>
<tr>
<th>Material type</th>
<th>Testing Requirement</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vermiculite materials used for thermal systems insulation, surfacing materials and other miscellaneous ACM (including but not limited to: existing or new surfacing material, plaster, pipe lagging, and sprayed-on fireproofing)</td>
<td>NYS ELAP Certification NYS Item 198.1 (PLM Friable) tests used if Vermiculite &lt; 10%</td>
</tr>
<tr>
<td>Vermiculite attic fill, block fill and other loose bulk vermiculite materials</td>
<td>Because there is no currently approved analytical method that can reliably confirm vermiculite materials containing asbestos, these materials must be assumed to be contaminated with asbestos and therefore designated as ACM.</td>
</tr>
</tbody>
</table>

Material Containing Vermiculite

Vermiculite materials used for thermal systems insulation, surfacing materials and other miscellaneous ACM

- Proceed with Item 198.1 (stratified point counting (all 4 cover slips))
  - Vermiculite < 10%
    - Continue w/ Item 198.1
      - Asbestos fibers
        - Yes
          - Report ACM accordingly
        - No
          - Report Non-ACM accordingly
          - Proceed as ACM
  - Vermiculite > 10%
    - Report Non-ACM accordingly

Vermiculite attic fill, block fill and other loose bulk vermiculite materials
New Definitions

- “Asbestos-containing material” or “ACM” means a material or product that contains more than 1% of asbestos, as determined using the method specified in 40 CFR Part 763, Appendix E to Subpart E, Section I, “Polarized Light Microscopy,” and a material meeting the definition of suspect asbestos-containing material.

- “Suspect asbestos containing material” means vermiculite insulation, and any untested material used in or on a building component, except for metal, glass, wood, or fiberglass. Vermiculite insulation must be treated as ACM unless a recommended EPA sampling and analysis protocol specific to vermiculite insulation proves that it does not contain asbestos. (No such recommended protocol has been established to date.)
Date: August 12, 2003

To: Regulated asbestos industry and other interested parties

From: Minnesota Pollution Control Agency
Asbestos Program
651-296-6300

Minnesota Department of Health
Asbestos/Lead Compliance Program
651-215-0900

Subject: Updated information on Vermiculite, Floor Tile, Transite Air Ducts and Mold

VERMICULITE
Attached is a news release from the U.S. Environmental Protection Agency (EPA), dated May 21, 2003, calling attention to their national consumer awareness campaign on vermiculite insulation. This news announcement and related fact sheets can be accessed through the internet at http://www.epa.gov/asbestos/index.html. Of particular importance in the information released is the recommendation to identify vermiculite by visual observation only and assume it is asbestos containing. As stated in the news release,

“Due to scientific uncertainties associated with existing testing techniques, there is no easy way or dependable testing method to differentiate between vermiculite insulation that might have some asbestos fibers and vermiculite that does not. … Therefore, it is best to assume that the material may contain asbestos and take the appropriate precautions.”

MDH and MPCA strongly recommend assuming vermiculite contains asbestos. There is no dependable way of determining the asbestos content of vermiculite. MDH and MPCA have determined that removal of vermiculite insulation assumed to be asbestos containing falls under all applicable state and federal requirements.

In the event sampling of vermiculite occurs, the MPCA will be presenting information and developing a fact sheet related to proper sampling of vermiculite later this Fall. In the meantime, either assume vermiculite contains asbestos or for specific questions or guidance about vermiculite sampling procedures contact MPCA at 651-296-6300.
Wisconsin
Minnesota
New York
Rhode Island?
In Canada
British Columbia
Just a Libby Issue?

Figure 4. SEM photograph of vermiculite-rich material collected from Gem Park Complex, Colorado. Platy minerals visible are vermiculite and hydrobiotite. Fibrous particles are amphibole phases with compositions identified as winchite, richterite, and riebeckite (table 1).
USGS Study Conclusions

• The results of this mineralogical survey are preliminary
• The results of this preliminary survey are consistent and suggest that fibrous amphiboles, in more than trace amounts, may not be common in the ore zones of some types of vermiculite deposits.
• However, the asbestiform amphibole mineralogy of the Libby deposit is not unique.
Vermiculite Analysis
Analysis of Vermiculite

The problems with Vermiculite Analysis

• Particle size prohibits making a proper slide mount
• Asbestos not always homogenous within the sample
• Asbestos can be locked between plates and therefore not easily detected
• Non-regulated Libby Amphiboles present
Libby Vermiculite

- Rice grain sized Asbestos chunks can sometimes be detected visually

Heterogeneity!
Libby Vermiculite

Hand picking large amphibole chunks is straightforward.

Their absence does not mean the sample is asbestos free.
Looking for Smaller Fibers
Identification Strong

Amosite Dispersion staining mode (blue/gold)

Chrysotile, crossed polars, gypsum plate in (pos. SOE)

Chrysotile kink bands
Not true RI colors
### Quantitation Weak

Limitations of the PLM Method

**TABLE 2-1. SUGGESTED ACCEPTABLE ERRORS FOR PLM ANALYSIS**

(Based on 400 point counts of a reasonably homogeneous sample or 100 fields of view for visual estimate)

<table>
<thead>
<tr>
<th>% Area Asbestos</th>
<th>Acceptable Mean Result</th>
<th>% Area Asbestos</th>
<th>Acceptable Mean Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>&gt; 0-3%</td>
<td>50</td>
<td>40-60%</td>
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<tr>
<td>5</td>
<td>&gt; 1-9%</td>
<td>60</td>
<td>50-70%</td>
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<td>10</td>
<td>5-15%</td>
<td>70</td>
<td>60-80%</td>
</tr>
<tr>
<td>20</td>
<td>10-30%</td>
<td>80</td>
<td>70-90%</td>
</tr>
<tr>
<td>30</td>
<td>20-40%</td>
<td>90</td>
<td>80-100%</td>
</tr>
<tr>
<td>40</td>
<td>30-50%</td>
<td>100</td>
<td>90-100%</td>
</tr>
</tbody>
</table>
What’s the Percentage?

5%
Quantitation Weak

• Calibrated Visual Estimation (CVE) of Asbestos Percentage from a combination of stereo and PLM
• Point Count using a single cross hair graticule or a 25 point Chalkley Array graticule.
The problems with Vermiculite Analysis

• Particle size prohibits making a proper slide mount
• Asbestos not always homogeneously distributed within the sample
• Non-regulated Libby Amphiboles may be present
Analysis Only Good as the Prep

Especially for Point Count
• Uniform
• Random
• Monolayer
Analysis Only Good as the Prep

Especially for Point Count
- Uniform
- Random
- Monolayer
Quantification Issues

- Particle size (height) prevents making a nice monolayer prep
- Particle size (L and W) makes for large variability in percentage by point counting
Typical VCMs

Textured Ceiling

Exfoliated Books of Vermiculite

Fireproof Insulation

No Intact Exfoliated Vermiculite Books
Only Thin Sheets/Flakes
Fireproof Insulation - Stereo

No Intact Exfoliated Vermiculite Books
Only Thin Sheets/Flakes
Textured Ceiling - Stereo

A Few Exfoliated Books of Vermiculite
Vermiculite Ore

Photo courtesy of www.mesotheliomasymptoms.com
Analysis Options

Loose Fill

• Block fill
• VAI
• Packing Material, etc.
Analysis Options for Loose Fill

**ASTM TEM Qualitative Method**
Excellent fiber ID but no quantification

**EPA 600 Milling followed by PLM** *(CARB 435 Method)*
Sample is milled, followed by a PLM point count

**EPA 600 Milling followed by TEM Analysis**
Mass % with/without the Non Regulated Amphiboles

**Cincinnati Method**
Floats, sinks and suspended fractions. PLM / TEM

**Heavy Liquid Separation**

**Addison Davies Method**
Remove vermiculite, and Chrysotile °o( prior to analysis
Analysis Options for Loose Fill

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Remove vermiculite, and Chrysotile :o( prior to analysis

**Heavy Liquid Separation**
NY: These Interim guidelines are effective “until new testing methods are established that effectively remove vermiculite from test samples and accurately identify asbestos.”
Addison Davies

Boiling Acid  Sample Recovery  Boiling Base
NY Heavy Liquid Separation
The “Cincinnati Method”

Research Method for Sampling and Analysis of Fibrous Amphibole in Vermiculite Attic Insulation

EPA/600/R-04/004 (January 2004)
EPA 600 Method with Milling

Fine fibers like these can be missed by the Cincinnati method.

As an alternative to the Cincinnati method, a CARB style milling prior to analysis can be used.
After milling, a PLM CVE, 400 or 1000 point count
After milling the sample is conducive to a TEM analysis as well!
ASTM TEM Qualitative

Ignore floats
Ignore sinks
Analyze the suspension
ASTM TEM Qualitative

Drop Mount (quick and cheaper) or Filtration ($uperior)
ASTM TEM Qualitative

• Magnification at 20,000X so can see even individual asbestos fibers / fibrils
• ID by EDXA and SAED
• Positive / Negative only, no quantitation
TEM is Good for ID of Libby Amphiboles

Libby Amphibole Standard
Like actinolite / tremolite, with addition of Na & K
Analysis Options

Vermiculite Containing Materials

• Spray On Fireproofing
• Textures
• Plasters
• Popcorn Ceilings
• etc.
Analysis Options for **VCM**

**EPA 600 R-93-116** (knowing limitations)

**Gravimetric Reduction** Prep followed by PLM/TEM

**EPA 600 Milling** followed by PLM (**CARB 435**)

**EPA 600 Milling** followed by **TEM** Analysis
Can break out a mass percent with and without the contribution of Libby Amphiboles

**Combo Approach?**
Light milling followed by Grav prior to PLM
Spray-On can have quite a bit of removable matrix
2g of VCM (spray on)
Spray On Residue After Grav
Method Performance

- Negative Vermiculite Spiked (10 Labs)
  - Cincinnati
  - EPA 600
  - EPA 600 with Milling
  - ASTM D5721
  - ASTM TEM Qualitative

- Zonolite

- Negative Spray On Fire Proofing Spiked
  - EPA 600
  - EPA 600 with Milling
  - EPA 600 with Grav Reduction
  - EPA 600 with Milling and Grav Reduction
  - ASTM D5721
  - ASTM TEM Qualitative

- Positive Monokote
Vermiculite “Therm-O-Rock” from Chandler AZ
270 Spiked Samples

- Each sample individually spiked dry - guaranteed %age but fiber integration issues
- Next round (samples already made) slurry used to aid mixing
Method Performance - Loose Fill

**Percent Recoveries at 1%**

- **Cincinnati**
- **ASTM D7521**
- **EPA 600 with Milling**
- **EPA 600**

First Place – Cincinnati
Second Place – EPA 600 with Milling

**Standard Deviations at 1%**

- **Cincinnati**
- **ASTM D7521**
- **EPA 600 with Milling**
- **EPA 600**

First Place – EPA 600 with Milling
Second Place – Plain Vanilla EPA 600
### Forget About Quantification?

**(Draft) ASTM TEM Qualitative**

<table>
<thead>
<tr>
<th>Sample</th>
<th>% Asbestos</th>
<th>Lab ID</th>
<th>Asbestos Type</th>
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<tbody>
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<td>1</td>
<td>Amosite</td>
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<tr>
<td>122</td>
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<tr>
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<td>1.00%</td>
<td>3</td>
<td>Amosite</td>
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<tr>
<td>124</td>
<td>1.00%</td>
<td>4</td>
<td>Amosite</td>
</tr>
<tr>
<td>125</td>
<td>1.00%</td>
<td>5</td>
<td>Amosite</td>
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<tr>
<td>126</td>
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**Percent True Positives**: 100%

### Loose Fill Spiked

<table>
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<td>0.106%</td>
<td>4</td>
<td>Amosite</td>
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<td>140</td>
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**Percent True Positives**: 100%

### Loose Fill Spiked

<table>
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<tr>
<th>Sample</th>
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<th>Lab ID</th>
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</table>

**Percent False Positives**: 0%
Method Performance for VCM

First Place - EPA 600 with Grav Reduction
Second Place - ASTM D7521

First Place - EPA 600 with Grav Reduction
Second Place - ASTM D7521
## ASTM Draft TEM Qualitative Analysis

### Spiked Monokote TEM Qualitative

<table>
<thead>
<tr>
<th>Sample</th>
<th>% Asbestos</th>
<th>Lab ID</th>
<th>Asbestos Type</th>
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<tr>
<td>250</td>
<td>0.99%</td>
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### Spiked Monokote TEM Qualitative

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<td>0.09%</td>
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<tr>
<td>253</td>
<td>0.10%</td>
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<td>Amosite</td>
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<tr>
<td>254</td>
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<tr>
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<tr>
<td>259</td>
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<td>ND</td>
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<tr>
<td>260</td>
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<th>Asbestos Type</th>
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</table>

**Percent True Positives**

- Spiked Monokote: 100
- Spiked Monokote: 80
- Spiked Monokote: 0

Re-analysis by both labs found asbestos in trace amounts.

- Trace element distributions of Ba, Cr & V can be used to distinguish Libby vermiculite samples from other mines

- Major sources of vermiculite used in the US:
  - Libby, MT
  - Enoree, SC
  - Palabora, South Africa
  - Xinjiang Province, China
Potential New Approach for VAI...

- Not an asbestos analytical method, but trace metals via traditional environmental chemistry methods using ICP SW846-3050B/6010C

- Study showed Ba concentration in the Libby samples were consistently over 2,000 ppm

- Libby samples varied less in composition than the other mines tested

- South Carolina Mine showed the most compositional variation – 1 sample found with Ba content overlap, considered an outlier
Potential New Approach for VAI...

- Zonolite Attic Insulation (ZAI) Trust - Settlement

- WR Grace to pay:
  - $30MM immediately
  - $30MM in 3 years
  - Potential for additional $80MM
  - $8MM per year for 10 years
  - $2MM education budget
  - Reimbursement of abatement cost only
  - Caps on amount paid - 55% of max cost of $7,500=$4,125

www.zonoliteatticinsulation.com
Potential New Approach for VAI...

- ZAI Trust will reimburse for homeowner VAI abatement if the Ba results are ≥ 1,500 ppm
- EMSL selected as testing lab, to date ~80% of sample results have exceeded the action level, however ~10% of the samples are not valid VAI samples, i.e., pink fiberglass, therefore ~90% positive

Known Libby Vermiculite Sample from EMSL’s archive:

![Analytical Results](image-url)
EMSL ANALYTICAL, INC.
200 Route 130 North
Cinnaminson, NJ 08077
Phone (856) 303-2565
Cell (845) 238-4559
ecahill@emsl.com
www.emsl.com

Ed Cahill
Vice President, Asbestos Division

Quality Laboratory Testing Since 1981
Acknowledgements

- Rob DeMalo, EMSL Analytical, Inc.
- **Owen S. Crankshaw**, *Research Environmental Scientist RTI*
- US EPA, [http://www2.epa.gov/asbestos/protect-your-family-asbestos-contaminated-vermiculite-insulation](http://www2.epa.gov/asbestos/protect-your-family-asbestos-contaminated-vermiculite-insulation)
- NYS DOH ELAP, [http://www.wadsworth.org/labcert/elap/asbestos.html](http://www.wadsworth.org/labcert/elap/asbestos.html)
- NYS DOL, [http://www.labor.state.ny.us/formsdocs/wp/CR56.pdf](http://www.labor.state.ny.us/formsdocs/wp/CR56.pdf)