

PARTICULATE ANALYSES

- Analysis for Asbestos and other Fibrous Particulates
- Spectrophotometric Analyses for Inorganic Particulate Matter
- Other Analyses for Inorganic Particulates (and Vapors)

Analysis for Asbestos and other Fibrous Particulates

For Industrial Hygiene Purposes

The Asbestos Problem

- Introduction to asbestos
- Industrial exposures--asbestos
- Six different crystalline forms of asbestos: chrysotile and amosite most common

The Asbestos Problem (cont'd.)

- Respiratory disease
 - Asbestosis
 - Mesothelioma
- Sampling (bulk vs. airborne)
- Analysis--microscopy(light &electron)

Crystalline and Glassy Materials

- Physical properties
- Molecular Structure
 - Ordered vs. Random
- Crystalline
 - Fibrous
 - Non-fibrous
- Most minerals are not pure, but a mixture of several compounds with different properties

Microscopy

- Physical observations
- Phase contrast light microscopy (PCMA)
(NIOSH Procedure 7400)
- Polarized light microscopy (P
9002)



Microscopes



C. NIOSH Procedure 7400 for Air samples on Filters

- **Sampling on membrane filters**
- **Mounting and clarification of filters**
- **Phase contrast light microscopy (PCM)**
- **Counting rules and protocol**
- **Calculations**
- **Statistical evaluation**

Determination of Concentrations of Asbestos in Air

NIOSH Method 7400

1. Sampling

- Cellulose ester membrane filters, 25mm., diameter, 0.8 μm pore size for personal sampling, <5 fibers/100 fields background
- Conductive cassettes (carbon impregnated)
- Flow rate: 0.5-16 liters per minute
- Optimum volume considerations
- Filter overloading
- Filter must have less than

2. Essentials for the Phase Contrast Microscopy

- Need to make filter transparent
- 400x
- Need to calibrate size of viewing area
- Need to adjust the microscope to focus on fibers in various planes of the filter

3. Counting Rules and Protocol

- **Count any fiber longer than 5 μm which lies entirely within the graticule area.**
 - **Count only fibers with L/W ratio $>3:1$**
- **Special rules for fibers crossing the graticule boundary**
- **Count all fibers which meet these criteria--may include non-asbestiform materials**

3. Counting Rules and Protocol (Continued)

- Count bundles of fibers as one fiber, unless individual fibers can be identified.
- “Stop” Counting Rules
 - Count enough graticule fields to yield 100 fibers
 - Count a minimum of 20 fields
- Move stage randomly through transparent part of filter (I.e. without prejudice)
- Estimated Limit of Detection is 7 fibers/mm²

4. Fiber Calculations

- Fiber Density:

$$E = (R/n_f - B/n_b) / A_f \text{ fibers/mm}^2$$

- Concentration:

$$C = E A_c / V \cdot 10^3 \text{ fibers/mL.}$$

Example Calculation

- (R) No. Fibers = 100
- (n_f) No. Fields = 46
- (A_f) Graticule Area = 0.00785mm^2
- (A_c) Filter Area = 365mm^2
- (F) Flow Rate = 2 LPM.
- (t) Time = 60 minutes
- (V) Air Volume = $F \times t$
- Assume $B/n_b = 0$
- $E = 100/46/0.00785$
- $= 277$
- $C = E \times 365 / 2.0 \times 60 \times 1000$
- $= 0.84$ fibers per cc.

Identification of Asbestos in Bulk

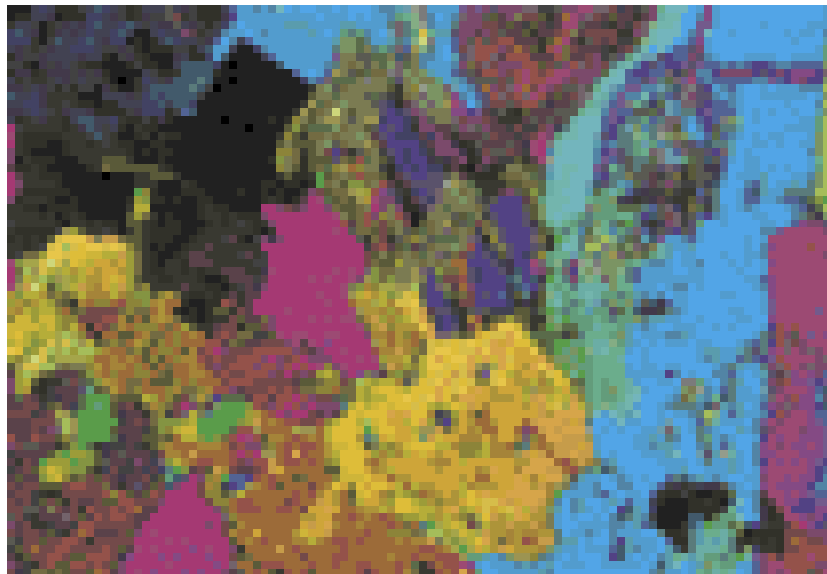
By Polarized Light Microscopy
(PLM) (NIOSH 9002)

Tools of Polarized Light Microscopy (PLM)

- Morphology, appearance, visual characteristics
- Pleochroism-the variation in light absorption with direction in colored, anisotropic crystals
- Birefringence: the property of having more than one refractive index (anisotropic)
- Angle of extinction as rotated between crossed polars
- Dispersion staining
- Refractive index matching-the greater the difference in R.I.'s, the greater the contrast

Polarized View of Fibers

- Pass around the asbestos particle atlas



Refractive Indices of the Types of Asbestos

■ Asbestos	■ Refractive Index
■ Chrysotile	■ 1.54-1.56
■ Amosite	■ 1.68
■ Crocidolite	■ 1.70
■ Anthophyllite	■ 1.60

EPA Procedure 600/M4-82-020 & NIOSH 9002

- Bulk sampling--representative samples.
- Bulk analysis (selecting typical fiber areas)
- Sample handling and preparation (refractive index immersions)

Other Methods of Mineral Analysis

- Electron Microscopy (EM)
- X-ray Diffraction (XRD)
- Infra Red Spectroscopy (IR)
- Differential Thermal Analysis (DTA)
- Screening Tests (Chemical Detection)

Other Methods of Particulate Analysis (cont'd.)

- Gravimetric Analysis (non-specific)
 - sensitivity
 - appropriate filter
 - moisture issues

Textbook References

Microscopy	380-385
X-ray Diffraction	385
Electrochemical Methods	387
Ion Chromatography	376
Electrostatic Charge Effects	525
Effects of Water Vapor	515
Atomic Spectroscopy	376-378
Gravimetry	378