Mineral Analysis

Asbestos Sampling and Analysis

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Sampling and Analysis for Minerals (Asbestos)

For Industrial Hygiene Purposes

I. The Asbestos Problem

- Minerals, Introduction to Asbestos (Example: Techniques apply most minerals)
- Industrial Exposures--Asbestos, Silica (Examples)
- Respiratory Disease
- Sampling
- Analysis--Microscopy (Light & EM), XRD, DTA, IR, Chemical

II. Properties of Minerals

- Physical Properties: MP, D, Color, Hardness, Malleability
- Crystal Form
- Symmetry
- Crystal Systems
- Habits, Form, Size, Shape

III. Microscopy

- Observations
- Physical Measurements
- Phase Contrast Light Microscopy (PCM) (NIOSH Procedure 7400)
- Polarized Light Microscopy (PLM) (NIOSH 9002)

A. Observations

- Observing Small Objects
- Shape, form, size, homogeneity
- Crystal Growth and Habit
- Chemical Microscopy
- Physical Measurements

B. Physical Measurements

- Density,
- Refractive Indexes,
- Axis of Elongation &
- Retardation
- Angles and cleavages

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

Identification of Asbestos in Bulk

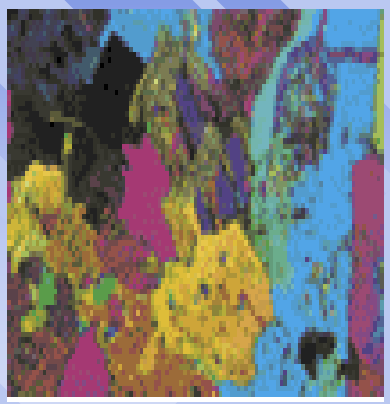
By Polarized Light Microscopy
(PLM) (NIOSH 9002)

Tools of Polarized Light Microscopy (PLM)

- Morphology, Appearance, Visual Characteristics.
- Birefringence (Anisotropic) vs. Isotropic
- Angle of Extinction as Rotated between Crossed Polars
- Dispersion Staining--Refractive Index Matching

Polarized Light Microscopy

- Polarized light
- Dispersion staining lens
- Colors depend on refractive index of fibers in HD Liquid.



Features for the PLM Microscope

- Critical Illumination,
- Rotating Stage,
- Dispersion Staining Objective Lens,
- Polarizer and Analyzer,
- Retardation Plates,
- Ocular with Cross-Hair

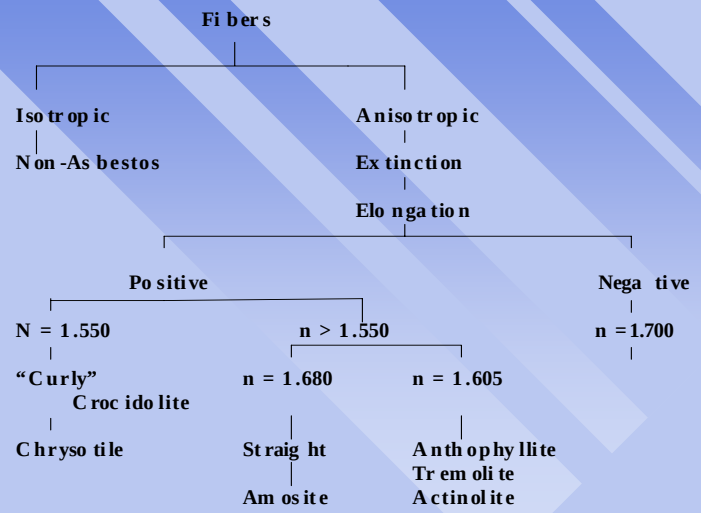
Polarized View of Fibers

Refractive Indixes of the Types of Asbestos

- Chrysotile
- Amosite
- Crocidolite
- Anthophylite

- 1.56
- 1.68
- 1.70
- 1.60

Sequence of PLM Analysis



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

- Bulk Sampling--REPRESENTATIVE SAMPLES.
- Bulk analysis (Selecting Typical Fiber Areas)
- Sample Handling and Preparation (RI Immersions)
- Schematic for Analysis--EPA and others

Determination of Concentrations of Asbestos in Air

NIOSH 7400 Method

1. Sampling

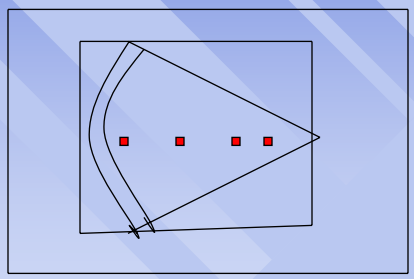
- Membrane Filters,
- Conductive Cowl Cassettes
- Flow Rate: 0.5-16 Liters per minute
- Optimum Volume Considerations

2. Essentials for the Phase Contrast Microscopy

- Kohler Illumination,
- Phase Contrast Lenses and Condenser
- 400x
- Walton-Becket Graticule
- Stage Micrometer for Calibration
- Phase Contrast Test Slide

Mounting and Clarification of Filters

- Cut Filter Wedge
- Place on Clean Slide
- Clarify with Acetone Vapor
- Add Triacitin Liquid
- Place Cover Glass
- Seal Cover Glass



Kohler Illumination

- Focus on Sample
- Adjust Field Iris
- Center Phase Rings
- “Phase Contrast”

Counting Rules and Protocol

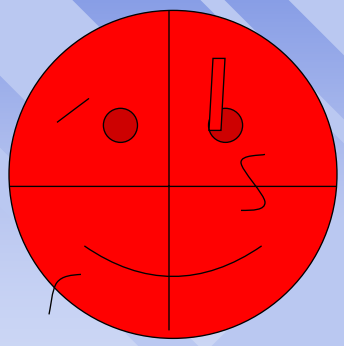
- Count any Fiber longer than 5 µm which lies entirely within the graticule area.
  - Count only fibers longer the 5µm. (along curve)
  - Count only fibers with L/W ratio >3:1
- For Fibers Crossing Graticule Boundary
  - Count as 1/2, fibers with one end inside
  - Do not count fibers crossing more than once
  - Reject and do not count all other fibers

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
  - Stop at 100 Graticule Fields regardless of count

Example: Counting Field

- Examine Quandrants
- Count fibers in each
- Examine Border & Count half Fibers



Fiber Calculations

- Fiber Density = E = $(R/n_f - B/n_b)/A_f$, fibers/mm2
- Concentration = C = E A_c/ V 10^3, fibers/ml.

Example Calculation

- No. Fibers = 100
- No. Fields = 46
- Field Area = 0.00785mm^2
- Filter Area = 365 mm^2
- Flow = 2 LPM.
- Time = 60 minutes

- E = (100/46)/(0.00785)
- C = $\frac{E\ (365)}{(2)(60)(1000)}$
- F/cc = $\frac{(f/fld)(mm^2)}{(mm^2/fld)(L/M)(M)(cc/L)}$

  =0.84 f/cc.

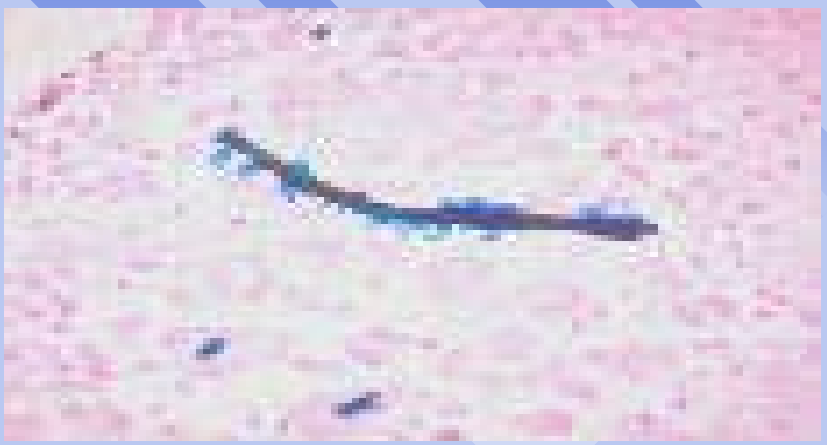
Other Methods of Mineral (Asbestos) Analysis

- IV. Electron Microscopy (EM)
- V. X-ray Diffraction (XRD)
- VI. Infra Red Spectroscopy (IRS)
- VII.Differential Thermal Analysis (DTA)
- VIII.Screening Tests (Chemical Detection)
- 1X. Fibrous Aerosol Monitior (FAM)

IV. Electron Microscopy (EM)

- Mount Samples
- Distinguish Shapes
- Identify by Electron Diffraction
- Count
- X-ray Fluorescence

Ferrugenous body in lung tissue



V. X-ray Diffraction (XRD)

- Mount Samples
- Crystal Lattice Spacing
- X-Ray Diffraction Patterns

VI. Infra Red Spectroscopy (IRS)

- Absorption of Infra-red radiation by molecular bonding
- Characteristic absorbance's at specific energies
- Unique spectral bands
- Regression of standards for quantization

VII.Differential Thermal Analysis (DTA)

- Temperature changes produce:
  - Phase changes and
  - Crystal Lattice Changes
  - Exo- and Endo-thermic interaction
- Thermo grams (DTA Curves)
- Apparatus = Differential Temperature Measurement
- Characteristic Peak Changes

VIII.Screening Tests (Chemical Detection)

- Appearance
- Flame Tests
- K-2 Chemical Test Kit
  - False Positives and Negatives

1X. Fibrous Aerosol Monitor (FAM)

- Direct Aerosol Type Monitor
- Detects fibrous shapes suspended or flowing through a beam
- Utilizes Light Scattering
- Scattered Light Proportional to Fiber Concentration