# ASBESTOS, CHRYSOTILE by XRD

$Mg_3Si_2O_5(OH)_4$ MW: ca. 283 CAS: 12001-29-5 RTECS: CI6478500

**METHOD:** 9000, Issue 2 **EVALUATION:** FULL **Issue 1:** 15 May 1989 **Issue 2:** 15 August 1994

**EPA Standard (Bulk):** 1% by weight

**PROPERTIES:** solid, fibrous mineral; conversion to Forsterite at 580 °C; attacked by acids; loses water above 300 °C

**SYNONYMS:** Chrysotile

## SAMPLING

**BULK SAMPLE:** 1 to 10 grams

**SHIPMENT:** seal securely to prevent escape of asbestos

**SAMPLE STABILITY:** indefinitely

**BLANKS:** none required

## ACCURACY

**RANGE STUDIED:** 1 to 100% in talc [1]

**BIAS:** negligible if standards and samples are matched in particle size [1]

**OVERALL PRECISION ($\hat{S}_{rT}$):** unknown; depends on matrix and concentration

**ACCURACY:** ± 14% to ± 25%

## MEASUREMENT

**TECHNIQUE:** X-RAY POWDER DIFFRACTION

**ANALYTE:** chrysotile

**PREPARATION:** grind under liquid $N_2$; wet-sieve through 10-$\mu$m sieve

**DEPOSIT:** 5 mg dust on 0.45-$\mu$m Ag membrane filter

**XRD:** Cu target X-ray tube; Optimize for intensity; 1° slit; integrated intensity with background subtraction

**CALIBRATION:** suspensions of asbestos in 2-propanol

**RANGE:** 1 to 100% (w/w) asbestos

**ESTIMATED LOD:** 0.2% asbestos in talc and calcite; 0.4% in heavy X-ray absorbers such as $Fe_2O_3$

**PRECISION ($\bar{S}_r$):** 0.07 (5 to 100%); 0.10 (@ 3%); 0.125 (@ 1%)

**APPLICABILITY:** Analysis of percent chrysotile asbestos in bulk samples.

**INTERFERENCES:** Antigorite (massive serpentine), Chlorite, Kaolinite, Bementite, and Brushite interfere. X-ray fluorescence and absorption is a problem with some elements; fluorescence can be circumvented with a diffracted beam monochromator, and absorption is corrected for in this method.

**OTHER METHODS:** This is P&CAM 309 [2] applied to bulk samples only, since the sensitivity is not adequate for personal air samples. The EPA Test Method for the determination of asbestos in bulk insulation samples is similar to this one [3]. Method 7400 is an optical counting procedure for airborne fibers in personal samples. Methods 7402 (Asbestos by Transmission Electron Microscopy) and 9002 (Asbestos by Polarized Light Microscopy) are also useful for positive identification of asbestos.

**REAGENTS:**

1. Chrysotile*, available from: Analytical Reference Minerals, Measurements Research Branch, DPSE, NIOSH, 4676 Columbia Parkway, Cincinnati, OH 45226; or UICC Asbestos Reference Sample Set, UICC MRC Pneumoconiosis Unit Llandough Hospital, Penarth, Glamorgan, CF6 1XW, UK.2.
2. 2-Propanol.*
3. Desiccant.
4. Glue or tape for securing Ag filters to XRD holders.

* See SPECIAL PRECAUTIONS.

**EQUIPMENT:**

1. Vials, plastic (for bulk sample).
2. Freezer mill, liquid $N_2$-cooled, (Spex Model 6700 or equivalent), grinding vials (Spex 6701), extractor (Spex 6704).
3. Ultrasonic bath.
4. Sieve, 10-$\mu$m, for wet-sieving.
5. Filters, polycarbonate, 1.0-$\mu$m, 37-mm (Nuclepore or equivalent).
6. Filtration apparatus and side-arm vacuum flask with 25- and 37-mm filter holders.
7. Oven, drying, 110 °C.
8. Analytical balance, readable to 0.01 mg.
9. Beaker, Griffin, 50-mL, with watchglass cover.
10. Filters, silver membrane, 25-mm diameter, 0.45-$\mu$m pore size (Millipore Corp., Poretics Corp., or equivalent).
11. Desiccator.
12. Bottles, glass, 1-L, with ground glass stoppers.
13. Wash bottle, polyethylene.
14. Magnetic stirrer.
15. X-ray powder diffractometer with copper target x-ray tube and scintillation detector.
16. Reference specimen (mica, Arkansas stone or other stable standard) for data normalization.
17. Volumetric pipettes and flasks.

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**SPECIAL PRECAUTIONS:** Asbestos, a human carcinogen, should be handled in a hood [4].

2-Propanol is flammable.

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**SAMPLING:**

1. Place several grams of the dust to be analyzed in a plastic vial, seal the vial securely and ship in a padded carton.

**SAMPLE PREPARATION:**

2. Place ca. 0.5 g of sample dust in a grinding vial and grind in a liquid nitrogen-cooled mill for 2 to 10 min.
3. Wet sieve the ground dust using a 10-$\mu$m sieve and 2-propanol. Place the dust on the sieve and place the sieve directly in an ultrasonic bath or in a wide dish in the bath. Use enough 2-propanol to cover the dust (put water in the bath if a dish is used to contain the 2-propanol). Apply ultrasonic power to sieve the dust.
   NOTE: It may take some time to obtain several mg of dust. Heating of the 2-propanol is likely and cooling periods may be required.
4. Recover the sieved sample dust from the 2-propanol by filtering the suspension through a non-fibrous filter (polycarbonate) or by driving off the 2-propanol on a hot plate. Dry the sieved sample in 110 °C oven for 4 h or more.

9 5. Weigh out ca. 5 mg of the sieved material onto a small square of tared weighing paper. Record the actual weight, W, to the nearest 0.01 mg. Transfer the dust to a 50-mL beaker, washing the weighing paper with several mL of 2-propanol. Add 10 to 15 mL 2-propanol to the beaker.

6. Cover the beaker with a watchglass. Agitate in an ultrasonic bath at least 3 min until all agglomerated particles are dispersed. Wash the underside of the watchglass with 2-propanol, collecting the washings in the beaker.

7. Place a silver filter in the filtration apparatus. Attach the funnel securely over the entire filter circumference. With no vacuum, pour 2 to 3 mL 2-propanol onto the filter. Pour the sample suspension from the beaker into the funnel and apply vacuum. During filtration, rinse the beaker several times and add rinsings to the funnel.

   NOTE: Control the filtration rate to keep the liquid level in the funnel near the top during rinsing. Do not wash the walls or add 2-propanol to the funnel when the liquid level is lower than 4 cm above the filter. Leave the vacuum on after filtration for sufficient time to produce a dry filter.

8. Remove the filter with forceps and attach it to the sample holder for XRD analysis.

**CALIBRATION AND QUALITY CONTROL:**

9. Prepare and analyze working standard filters:

   a. Prepare two suspensions of chrysotile asbestos in 2-propanol by weighing 10 and 100 mg of the dry powder to the nearest 0.01 mg. Quantitatively transfer each to a 1-L glass-stoppered bottle using 1.00 L 2-propanol.

      NOTE: Depending on the particle size of the standard, it may need to be ground and wet sieved (step 3). Dry the standards in a 110 °C oven for 4 h or more. Store in a desiccator.

   b. Suspend the powder in the 2-propanol with an ultrasonic probe or bath for 20 min. Immediately move the flask to a magnetic stirrer with thermally-insulated top and add a stirring bar to the suspension. Cool the solution to room temperature before withdrawing aliquots.

   c. Mount a filter on the filtration apparatus. Place several mL 2-propanol on the filter surface. Turn off the stirrer and shake vigorously by hand. Within a few seconds of setting the bottle down, remove the lid and withdraw an aliquot from the center of the 10 or 100 mg/L suspension. Do not adjust the volume in the pipet by expelling part of the suspension. If more than the desired aliquot is withdrawn, return all of the suspension to the bottle, rinse and dry the pipet, and take a new aliquot. Transfer the aliquot from the pipet to the filter. Keep the tip of the pipet near the surface but not submerged in the delivered suspension.

   d. Rinse the pipet with several mL 2-propanol, draining the rinse into the funnel. Repeat the rinse several more times. Prepare working standard filters, in triplicate, by this technique, at e.g., 0, 20, 30, 50 100, 200 and 500 $\mu$g.

   e. Apply vacuum and rapidly filter the suspension. Leave vacuum on until filter is dry. Do not wash down the sides of the funnel after the deposit is in place since this will rearrange the material on the filter. Transfer the filter to the sample holder.

   f. Analyze by XRD (step 12). The XRD intensities (12.d.) are designated $I_x^\circ$ and are then normalized (12.e.) to obtain $\hat{I}_x^\circ$. The intensities for standards greater than 200 mg should be corrected for matrix absorption (12.f. and 13).

   g. Prepare a calibration graph by plotting $\hat{I}_x^\circ$, as a function of $\mu$g of each standard.

      NOTE: Poor repeatability (greater than 10% above 0.04 mg chrysotile) indicates that new standards should be made. The data should lie along a straight line. It is preferable to use a weighted least squares with $1/\sigma^2$ weighing, where $\sigma^2$ is the variance of the data at a given loading.

   h. Determine the slope, m, of the calibration curve in counts/$\mu$g. The intercept on the abscissa should be within $\pm 5$ $\mu$g of zero.

NOTE: A large intercept indicates an error in determining the background, i.e., an incorrect baseline has been calculated or interference by another phase.

10. Select six silver membrane filters as media blanks (for determination of sample self-absorption, step 13) randomly from the same box of filters to be used for depositing the samples. Mount each of the media blanks on the filtration apparatus and apply vacuum to draw 5 to 10 mL of 2-propanol through the filter. Remove, let dry and mount on sample holders. Determine the net normalized count for the silver peak, $\hat{I}^{\circ}_{Ag}$, for each media blank (step 12). Obtain an average value for the six media blanks.

**MEASUREMENT:**

11. Obtain a qualitative X-ray diffraction scan (e.g., 10 to 80 degrees 2-theta) of the sample to determine the presence of chrysotile and interferences. The expected diffraction peaks are as follows:

| | Peak (2-Theta Degrees) | |
|---|---|---|
| Mineral | Primary | Secondary |
| Chrysotile | 12.08 | 24.38 |
| Silver | 38.12 | 44.28 |

12. Mount the filter (sample, standard or blank) in the XRD instrument and:
    a. Determine the net intensity, $I_r$, of the reference specimen before each filter is scanned. Select a convenient normalization scale factor, N, which is approximately equivalent to the net count for the reference specimen peak, and use this value of N for all analyses.
    b. Measure the diffraction peak area of a chrysotile peak that is free of interference. Scan times should be long, e.g., 15 min.
    c. Measure the background on each side of the peak for one-half the time used for peak scanning. The sum of these two counts is the average background. Determine the position of the background for each sample.
    d. Calculate the net intensity, $I_x$ (the difference between the peak integrated count and the total background count).
    e. Calculate and record the normalized intensity, $\hat{I}_x$, for the sample peak on each sample and standard:

$$\hat{I}_x = \frac{I_x}{I_r} \cdot N.$$

NOTE: Normalizing to the reference specimen intensity compensates for long-term drift in X-ray tube intensity. If intensity measurements are stable, the reference specimen may be run less frequently; net intensities should be normalized to the most recently measured reference intensity.

    f. Determine the net count, $I_{Ag}$, of an interference-free silver peak on the sample filter following the same procedure. Use a short scan time for the silver peak (e.g., 5% of scan time for analyte peaks) throughout the method.
    g. Scan each field blank over the same 2-theta range used for the analyte and silver peaks. These analyses serve only to verify that contamination of the filters has not occurred. The analyte peak should be absent. The normalized intensity of the silver peak should match that of the media blanks.

**CALCULATIONS:**

13. Calculate the percentage of chrysotile in the bulk dust sample:

$$C = \frac{(\hat{I}_x \cdot f(T) - b) \cdot 100}{m \cdot W}, \%$$

where: $\hat{I}_x$ = normalized intensity for sample peak

b = intercept of calibration graph ($\hat{I}_x^\circ$ vs. W)

m = slope of calibration graph (counts/$\mu$g)

$f(T) = \frac{-R \ln T}{1 - T^R}$ = absorption correction factor (Table 1)

$R = \sin(\theta_{Ag})/\sin(\theta_x)$

$T = \hat{I}_{Ag}/(\text{average } \hat{I}_{Ag}^\circ)$ = transmittance of sample

$\hat{I}_{Ag}$ = normalized silver peak intensity from sample

average $\hat{I}_{Ag}^\circ$ = normalized silver peak intensity from media blanks (average of six values)

W = mass of deposited sample in $\mu$g.

NOTE: For a more detailed discussion of the absorption correction procedure, see references [5] to [8].

**EVALUATION OF METHOD:**

This method is based on the work of B.A. Lange in developing P&CAM 309 [1,2]. Samples in the range of 1 to 100% chrysotile in talc were studied to establish the feasibility of an XRD method for airborne asbestos. Analytical precision was as follows:

| % Chrysotile in Talc | $\bar{S}_r$ (%) |
|---|---|
| 100 | 6.9 |
| 10 | 4.7 |
| 7 | 9.8 |
| 5 | 8.2 |
| 3 | 10.1 |
| 1 | 12.5 |

This work also showed that bias of results after absorption corrections are made is negligible.

**REFERENCES:**

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[2] NIOSH Manual of Analytical Methods, 2nd ed., V. 5, P&CAM 309, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 79-141 (1979).

[3] Perkins, R.L. and B.W. Harvey. U.S. Environmental Protection Agency Test Method for the Determination of Asbestos in Bulk Building Materials, EPA/600/R-93/116 (June, 1993).

[4] Criteria for a Recommended Standard...Occupational Exposure to Asbestos (Revised), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-169 (1976).

[5] Leroux, J. and C. Powers. Direct X-Ray Diffraction Quantitative Analysis of Quartz in Industrial Dust Films Deposited on Silver Membrane Filters, Occup. Health Rev., 21:26 (1970).

[6] Williams, D. D. Direct Quantitative Diffractometric Analysis, Anal. Chem., 31:1841 (1959).

[7] Abell, M. T., D. D. Dollberg, B. A. Lange, R. W. Hornung and J. C. Haartz. Absorption Corrections in X-ray Diffraction Dust Analyses: Procedures Employing Silver Filters, Electron Microscopy and X-ray Applications, V. 2, 115, Ann Arbor Science Publishers, Inc. (1981).

[8] Dollberg, D. D., M. T. Abell, and B. A. Lange. Occupational Health Analytical Chemistry: Quantitation Using X-Ray Powder Diffraction, ACS Symposium Series, No. 120, 43 (1980).

**METHOD REVISED BY:**

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**TABLE 1. ABSORPTION CORRECTION FACTOR AS A FUNCTION OF TRANSMITTANCE FOR SOME CHRYSOTILE-SILVER PEAK COMBINATIONS.**

| Transmittance | Chrysotile / Silver | f(T) 12.08 / 38.12 | f(T) 24.38 / 38.12 | Transmittance | f(T) 12.08 / 38.12 | f(T) 24.38 / 38.12 |
|---|---|---|---|---|---|---|
| *T* | | | | *T* | | |
| 1.00 | | 1.0000 | 1.0000 | 0.69 | 1.6839 | 1.3142 |
| 0.99 | | 1.0157 | 1.0078 | 0.68 | 1.7151 | 1.3277 |
| 0.98 | | 1.0317 | 1.0157 | 0.67 | 1.7470 | 1.3414 |
| 0.97 | | 1.0480 | 1.0237 | 0.66 | 1.7797 | 1.3555 |
| 0.96 | | 1.0647 | 1.0319 | 0.65 | 1.8132 | 1.3698 |
| 0.95 | | 1.0817 | 1.0402 | 0.64 | 1.8475 | 1.3845 |
| 0.94 | | 1.0991 | 1.0486 | 0.63 | 1.8827 | 1.3995 |
| 0.93 | | 1.1168 | 1.0572 | 0.62 | 1.9188 | 1.4148 |
| 0.92 | | 1.1350 | 1.0659 | 0.61 | 1.9558 | 1.4305 |
| 0.91 | | 1.1535 | 1.0747 | 0.60 | 1.9938 | 1.4465 |
| 0.90 | | 1.1724 | 1.0837 | 0.59 | 2.0328 | 1.4629 |
| 0.89 | | 1.1917 | 1.0928 | 0.58 | 2.0728 | 1.4797 |
| 0.88 | | 1.2114 | 1.1021 | 0.57 | 2.1139 | 1.4969 |
| 0.87 | | 1.2316 | 1.1115 | 0.56 | 2.1560 | 1.5145 |
| 0.86 | | 1.2522 | 1.1212 | 0.55 | 2.1993 | 1.5325 |
| 0.85 | | 1.2733 | 1.1309 | 0.54 | 2.2438 | 1.5510 |
| 0.84 | | 1.2948 | 1.1409 | 0.53 | 2.2895 | 1.5700 |
| 0.83 | | 1.3168 | 1.1510 | 0.52 | 2.3365 | 1.5895 |
| 0.82 | | 1.3394 | 1.1613 | 0.51 | 2.3848 | 1.6095 |
| 0.81 | | 1.3624 | 1.1718 | 0.50 | 2.4344 | 1.6300 |
| 0.80 | | 1.3859 | 1.1825 | 0.49 | 2.4855 | 1.6510 |
| 0.79 | | 1.4100 | 1.1933 | 0.48 | 2.5380 | 1.6727 |
| 0.78 | | 1.4346 | 1.2044 | 0.47 | 2.5921 | 1.6950 |
| 0.77 | | 1.4598 | 1.2157 | 0.46 | 2.6478 | 1.7179 |
| 0.76 | | 1.4856 | 1.2272 | 0.45 | 2.7051 | 1.7414 |
| 0.75 | | 1.5120 | 1.2389 | 0.44 | 2.7642 | 1.7657 |
| 0.74 | | 1.5390 | 1.2508 | 0.43 | 2.8251 | 1.7907 |
| 0.73 | | 1.5666 | 1.2630 | 0.42 | 2.8879 | 1.8165 |
| 0.72 | | 1.5949 | 1.2754 | 0.41 | 2.9526 | 1.8431 |
| 0.71 | | 1.6239 | 1.2881 | 0.40 | 3.0195 | 1.8705 |
| 0.70 | | 1.6536 | 1.3010 | | | |

# ASBESTOS (bulk) by PLM

various MW: various CAS: 1332-21-4 RTECS: CI6475000

**METHOD: 9002, Issue 2** **EVALUATION: PARTIAL** **Issue 1: 15 May 1989**
**Issue 2: 15 August 1994**

**EPA Standard (Bulk): 1%** **PROPERTIES:** solid, fibrous, crystalline, anisotropic

**SYNONYMS [CAS #]:** actinolite [77536-66-4], or ferroactinolite [15669-07-5]; amosite [12172-73-5]; anthophyllite [77536-67-5]; chrysotile [12001-29-5]; serpentine [18786-24-8]; crocidolite [12001-28-4]; tremolite [77536-68-6]; amphibole asbestos [1332-21-4].

## SAMPLING

**BULK SAMPLE:** 1 to 10 grams

**SHIPMENT:** seal securely to prevent escape of asbestos

**SAMPLE STABILITY:** stable

**BLANKS:** none required

## ACCURACY

**RANGE STUDIED:** <1% to 100% asbestos

**BIAS:** not determined

**PRECISION:** not determined

**ACCURACY:** not determined

## MEASUREMENT

**TECHNIQUE:** MICROSCOPY, STEREO AND POLARIZED LIGHT, WITH DISPERSION STAINING

**ANALYTE:** actinolite asbestos, amosite, anthophyllite asbestos, chrysotile, crocidolite, tremolite asbestos

**EQUIPMENT:** microscope, polarized light; 100-400X dispersion staining objective, stereo microscope: 10-45X

**RANGE:** 1% to 100% asbestos

**ESTIMATED LOD:** <1% asbestos [1]

**PRECISION:** not determined

**APPLICABILITY:** this method is useful for the qualitative identification of asbestos and the semi-quantitative determination of asbestos content of bulk samples. The method measures percent asbestos as perceived by the analyst in comparison to standard area projections, photos, and drawings, or trained experience. The method is not applicable to samples containing large amounts of fine fibers below the resolution of the light microscope.

**INTERFERENCES:** Other fibers with optical properties similar to the asbestos minerals may give positive interferences. Optical properties of asbestos may be obscured by coating on the fibers. Fibers finer than the resolving power of the microscope (ca. 0.3 $\mu$m) will not be detected. Heat and acid treatment may alter the index of refraction of asbestos and change its color.

**OTHER METHODS:** This method (originally designated as method 7403) is designed for use with NIOSH Methods 7400 (phase contrast microscopy) and 7402 (electron microscopy/EDS). The method is similar to the EPA bulk asbestos method [1].

**REAGENTS:**

1. Refractive index (RI) liquids for Dispersion Staining: high-dispersion (HD) series, 1.550, 1.605, 1.620.
2. Refractive index liquids: 1.670, 1.680, and 1.700.
3. Asbestos reference samples such as SRM #1866, available from the National Institute of Standards and Technology.*
4. Distilled Water (optional).
5. Concentrated HCl: ACS reagent grade.

* See SPECIAL PRECAUTIONS.

**EQUIPMENT:**

1. Sample containers: screw-top plastic vials of 10- to 50-mL capacity.
2. Microscope, polarized light, with polarizer, analyzer, port for retardation plate, 360° graduated rotating stage, substage condenser with iris, lamp, lamp iris, and:
   a. Objective lenses: 10X, 20X, and 40X or near equivalent.
   b. Ocular lense: 10X minimum.
   c. Eyepiece reticle: crosshair.
   d. Dispersion staining objective lens or equivalent.
   e. Compensator plate: ca. 550 nm ± 20 nm, retardation: "first order red" compensator.
3. Microscope slides: 75 mm x 25 mm.
4. Cover slips.
5. Ventilated hood or negative-pressure glove box.
6. Mortar and pestle: agate or porcelain.
7. Stereomicroscope, ca. 10 to 45X.
8. Light source: incandescent or fluorescent.
9. Tweezers, dissecting needles, spatulas, probes, and scalpels.
10. Glassine paper or clean glass plate.
11. Low-speed hand drill with coarse burr bit (optional).

**SPECIAL PRECAUTIONS:** Asbestos, a human carcinogen, should be handled only in an exhaust hood (equipped with a HEPA filter) [2]. Precautions should be taken when collecting unknown samples, which may be asbestos, to preclude exposure to the person collecting the sample and minimize the disruption to the parent material [3]. Disposal of asbestos-containing materials should follow EPA Guidelines [4].

**SAMPLING:**

1. Place 1 to 10 g of the material to be analyzed in a sample container.
   NOTE: For large samples (i.e., whole ceiling tiles) that are fairly homogenous, a representative small portion should be submitted for analysis. Sample size should be adjusted to ensure that it is representative of the parent material.
2. Make sure that sample containers are taped so they will not open in transit.
3. Ship the samples in a rigid container with sufficient packing material to prevent damage or sample loss.

**SAMPLE PREPARATION:**

4. Visually examine samples in the container and with a low-magnification stereomicroscope in a hood. (If necessary, a sample may be carefully removed from the container and placed on glassine transfer paper or clean glass plate for examination). Break off a portion of the sample and examine the edges for emergent fibers. Note the homogeneity of the sample. Some hard tiles can be broken, and the edges examined for emergent fibers. If fibers are found, make an estimate of the amount and type of fibers present, confirm fiber type (step 14) and quantify (step 15).

5. In a hood, open sample container and with tweezers remove small, representative portions of the sample.
   a. If there are obvious separable layers, sample and analyze each layer separately.
   b. If the sample appears to be slightly inhomogeneous, mix it in the sample container with tweezers or a spatula before taking the portion of analysis. Alternatively, take small representative portions of each type of material and place on a glass slide.
   c. On hard tiles that may have thin, inseparable layers, use a scalpel to cut through all the layers for a representative sample. Then cut it into smaller pieces after placing RI liquid on it before trying to reduce the thickness. Alternatively, use a low-speed hand drill equipped with a burr bit to remove material from hard tiles. Avoid excessive heating of the sample which may alter the optical properties of the material.
      NOTE: This type of sample often requires ashing or other specialized preparation, and may require transmission electron microscopy for detection of the short asbestos fibers which are characteristic of floor tiles.
   d. If the sample has large, hard particles, grind it in a mortar. Do not grind so fine that fiber characteristics are destroyed.
   e. If necessary, treat a portion of the sample in a hood with an appropriate solvent to remove binders, tars, and other interfering materials which may be present in the sample. Make corrections for the non-asbestos material removed by this process.
      NOTE: Other methods of sample preparation such as acid washing and sodium metaphosphate treatment and ashing may be necessary, especially to detect low concentrations of asbestos. If needed, use as described in Reference [1].
6. After placing a few drops of RI liquid on the slide, put a small portion of sample in the liquid. Tease apart with a needle or smash small clumps with the flat end of a spatula or probe, producing a uniform thickness or particles so that better estimates of projected area percentages can be made. Mix the fibers and particles on the slide so that they are as homogeneous as possible.
   NOTE: An even dispersion of sample should cover the entire area under the cover slip. some practice will be necessary to judge the right amount of material to place on the slide. Too little sample may not give sufficient information and too much sample cannot be easily analyzed.

**CALIBRATION AND QUALITY CONTROL:**

7. Check for contamination each day of operation. Wipe microscope slides and cover slips with lens paper before using. Check refractive index liquids. Record results in a separate logbook.
8. Verify the refractive indices of the refractive index liquids used once per week of operation. Record these checks in a separate logbook.
9. Follow the manufacturer's instructions for illumination, condenser alignment and other microscope adjustments. Perform these adjustments prior to each sample set.
10. Determine percent of each identified asbestos species by comparison to standard projections (Figure 1) [1]. If no fibers are detected in a homogeneous sample, examine at least two additional preparations before concluding that no asbestos is present.
11. If it appears that the preparation technique might not be able to produce a homogeneous or representative sample on the slide, prepare a duplicate slide and average the results. Occasionally, when the duplicate results vary greatly, it will be necessary to prepare additional replicate slides and average all the replicate results. Prepare duplicate slides of at least 10% of the samples analyzed. Average the results for reporting.
12. Analyze about 5% blind samples of known asbestos content.
13. Laboratories performing this analytical method should participate in the National Voluntary Laboratory Accreditation Program [5] or a similar interlaboratory quality control program. Each analyst should have complete formal training in polarized light microscopy and its application to crystalline materials. In lieu of formal training, laboratory training in asbestos bulk analysis under the direction of a trained asbestos bulk analyst may be substituted. Owing to the subjective nature of the method, frequent practice is essential in order to remain proficient in estimating projected area percentages.

**QUALITATIVE ASSESSMENT:**

14. Scan the slide to identify any asbestos minerals using the optical properties of morphology, refractive indices, color, pleochroism, birefringence, extinction characteristics, sign of elongation, and dispersion staining characteristics.
    NOTE: Identification of asbestos using polarized light microscopy is unlike most other analytical methods. The quality of the results is dependent on the skill and judgment of the analyst. This method does not lend itself easily to a step-wise approach. Various procedures devised by different analysts may yield equivalent results. The following step-wise procedure repeatedly utilizes the sample preparation procedure previously outlined.
    a. Prepare a slide using 1.550 HD RI liquid. Adjust the polarizing filter such that the polars are partially crossed, with ca. 15° offset. Scan the preparation, examining the morphology for the presence of fibers. If no fibers are found, scan the additional preparations. If no fibers are found in any of the preparations, report that the sample does not contain asbestos, and stop the analysis at this point.
    b. If fibers are found, adjust the polarizing filter such that the polars are fully crossed. If all of the fibers are isotropic (disappear at all angles of rotation) then those fibers are not asbestos. Fibrous glass and mineral wool, which are common components of suspect samples, are isotropic. If only isotropic fibers are found in the additional preparations, report no asbestos fibers detected, and stop the analysis.
    c. If anisotropic fibers are found, rotate the stage to determine the angle of extinction. Except for tremolite-actinolite asbestos which has oblique extinction at 10-20°, the other forms of asbestos exhibit parallel extinction (Table 1). Tremolite may show both parallel and oblique extinction.
    d. Insert the first order red compensator plate in the microscope and determine the sign of elongation. All forms of asbestos have a positive sign of elongation except for crocidolite. If the sign of elongation observed is negative, go to step "g."
       NOTE: To determine the direction of the sign of elongation on a particular microscope configuration, examine a known chrysotile sample and note the direction (NE-SW or NW-SE) of the blue coloration. Chrysotile has a positive sign of elongation.
    e. Remove the first-order red compensator and uncross the polarizer. Examine under plane polarized light for blue and gold-brown Becke colors at the fiber-oil interface (i.e., index of refraction match). Becke colors are not always evident. Examine fiber morphology for twisted, wavy bundles of fibers which are characteristic of chrysotile. Twisted, ribbon-like morphology with cellular internal features may indicate cellulose fibers. It may be necessary to cross the polars partially in order to see the fibers if the index of refraction is an exact match at 1.550. If the fibers appear to have higher index of refraction, go to step "h," otherwise continue.
    f. Identification of chrysotile. Insert the dispersion staining objective. Observation of dispersion staining colors of blue and blue-magenta confirms chrysotile. Cellulose, which is a common interfering fiber at the 1.550 index of refraction, will not exhibit these dispersion staining colors. If chrysotile is found, go to step 15 for quantitative estimation.
    g. Identification of crocidolite. Prepare a slide in 1.700 RI liquid. Examine under plane-polarized light (uncrossed polars); check for morphology of crocidolite. Fibers will be straight, with rigid appearance, and may appear blue or purple-blue. Crocidolite is pleochroic, i.e., it will appear to change its color (blue or gray) as it is rotated through plane polarized light. Insert the dispersion staining objective. The central stop dispersion staining color are red magenta and blue magenta, however, these colors are sometimes difficult to impossible to see because of the opacity of the dark blue fibers. If observations above indicate crocidolite, go to step 15 for quantitative estimation.
    h. Identification of amosite. Prepare a slide in 1.680 RI liquid. Observed the fiber morphology for amosite characteristics: straight fibers and fiber bundles with broom-like or splayed ends. If the morphology matches amosite, examine the fibers using the dispersion staining objective. Blue and pale blue colors indicate the cummingtonite form of amosite, and gold and blue colors indicate the grunerite form of amosite. If amosite is confirmed by this test,

go to step 15 for quantitative estimation, otherwise continue.

i. Identification of anthophyllite-tremolite-actinolite. Prepare a slide in 1.605 HD RI liquid. Examine morphology for comparison to anthophyllite-tremolite-actinolite asbestos. The refractive indices for these forms of asbestos vary naturally within the species. Anthophyllite can be distinguished from actinolite and tremolite by its nearly parallel extinction. Actinolite has a light to dark green color under plane-polarized light and exhibits some pleochroism. For all three, fibers will be straight, single fibers possibly with some larger composite fibers. Cleavage fragments may also be present. Examine using the central stop dispersion staining objective. Anthophyllite will exhibit central stop colors of blue and gold/gold-magenta; tremolite will exhibit pale blue and yellow; and actinolite will exhibit magenta and golden-yellow colors.

NOTE: In this refractive index range, wollastonite is a common interfering mineral with similar morphology including the presence of cleavage fragments. It has both positive and negative sign of elongation, parallel extinction, and central stop dispersion staining colors of pale yellow and pale yellow to magenta. If further confirmation of wollastonite versus anthophyllite is needed, go to step "j". If any of the above forms of asbestos were confirmed above, go to step 15 for quantitative estimation. If none of the tests above confirmed asbestos fibers, examine the additional preparations and if the same result occurs, report the absence of asbestos in this sample.

j. Wash a small portion of the sample in a drop of concentrated hydrochloric acid on a slide. Place the slide, with cover slip in place, on a warm hot plate until dry. By capillary action, place 1.620 RI liquid under the cover clip and examine the slide. Wollastonite fibers will have a "cross-hatched" appearance across the length of the fibers and will not show central stop dispersion colors. Anthophyllite and tremolite will still show their original dispersion colors.

NOTE: There are alternative analysis procedures to the step-wise approach outlined above which will yield equivalent results. Some of these alternatives are:

i. Perform the initial scan for the presence of asbestos using crossed polars as well as the first-order red compensator. This allows for simultaneous viewing of birefringent and amorphous materials as well as determine their sign of elongation. Some fibers which are covered with mortar may best be observed using this configuration.

ii. Some analysts prefer to mount their first preparation in a RI liquid different than any asbestos materials and conduct their initial examination under plane-polarized light.

iii. If alternative RI liquids are used from those specified, dispersion staining colors observed will also change. Refer to an appropriate reference for the specific colors associated with asbestos in the RI liquids actually used.

**QUANTITATIVE ASSESSMENT:**

15. Estimate the content of the asbestos type present in the sample using the 1.550 RI preparation. Express the estimate as an area percent of all material present, taking into account the loading and distribution of all sample material on the slide. Use Figure 1 as an aid in arriving at your estimate. If additional unidentified fibers are present in the sample, continue with the qualitative measurement (step 14).

NOTE: Point-counting techniques to determine percentages of the asbestos minerals are not generally recommended. The point-counting method only produces accurate quantitative data when the material on the slide is homogeneous and has a uniform thickness, which is difficult to obtain [6]. The point-counting technique is, recommended by the EPA to determine the amount of asbestos in bulk [1]; however, in the more recent Asbestos Hazard Emergency Response Act (AHERA) regulations, asbestos quantification may be performed by a point-counting or equivalent estimation method [7].

16. Make a quantitative estimate of the asbestos content of the sample from the appropriate combination of the estimates from both the gross and microscopic examinations. If asbestos fibers are identified, report the material as "asbestos-containing". Asbestos content should be reported as a range of percent content. The range reported should be indicative of the analyst's precision in estimating asbestos content. For greater quantities use Figure 1 in arriving at your estimate.

**EVALUATION OF METHOD:**

The method is compiled from standard techniques used in mineralogy [8-13], and from standard laboratory procedures for bulk asbestos analysis which have been utilized for several years. These techniques have been successfully applied to the analysis of EPA Bulk Sample Analysis Quality Assurance Program samples since 1982 [1,5]. However, no formal evaluation of this method, as written, has been performed.

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**METHOD WRITTEN BY:**

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Figure 1. Percent estimate comparator.

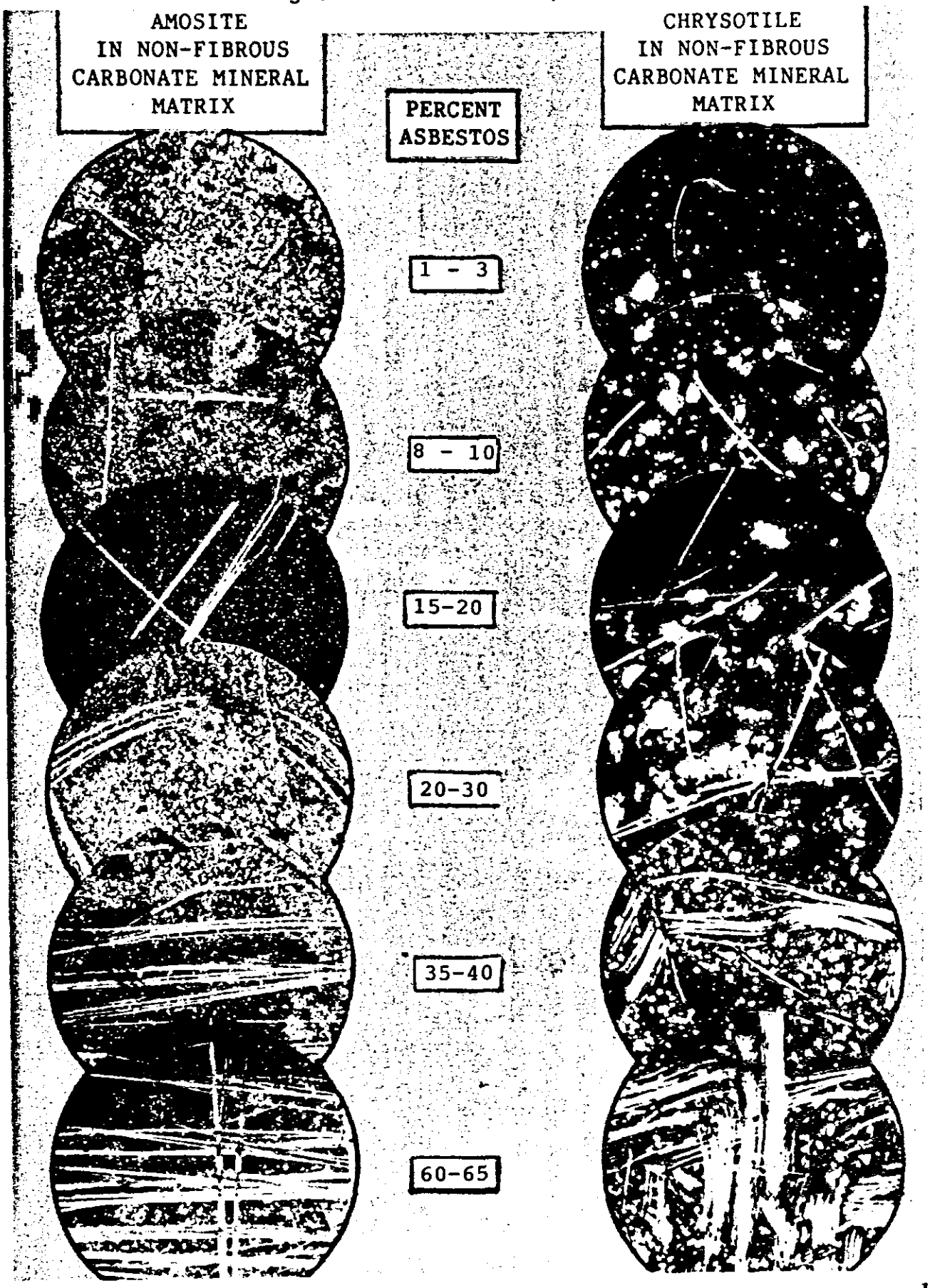

Table 1. Optical Properties of Asbestos Fibers

| | | Refractive Index (Approximate Values) | | |
|---|---|---|---|---|
| Mineral | Morphology and Color | ⊥ to Elongation | ∥ to Elongation | Birefringence |
| Chrysotile | Wavy fibers with kinks. Splayed ends on larger bundles. Colorless to light brown upon being heated. Nonpleochroic. Aspect ratio typically >10:1. | 1.54 | 1.55 | 0.002 - 0.014 |
| Cummingtonite-Grunerite (Amosite) | Straight fibers and fiber bundles. Bundle ends appear broom-like or splayed. Colorless to brown upon heating. May be weakly pleochroic. Aspect ratio typically >10:1. | 1.67 | 1.70 | 0.02 - 0.03 |
| Crocidolite (Riebeckite) | Straight fibers and fiber bundles. Longer fibers show curvature. Splayed ends on bundles. Characteristic blue color. Pleochroic. Aspect ratio typically >10:1. | 1.71 | 1.70 | 0.014 - 0.016 Interference colors may be masked by blue color. |
| Anthophyllite | Straight fibers and fiber bundles. Cleavage fragments may be present. Colorless to light brown. Nonpleochroic to weakly pleochroic. Aspect ratio generally <10:1. | 1.61 | 1.63 | 0.019 - 0.024 |
| Tremolite-Actinolite | Straight and curved fibers. Cleavage fragments common. Large fiber bundles show splayed ends. Tremolite is colorless. Actinolite is green and weakly to moderately pleochroic. Aspect ratio generally <10:1. | 1.60 - 1.62 (tremolite)<br>1.62 - 1.67 (actinolite) | 1.62 - 1.64 (tremolite)<br>1.64 - 1.68 (actinolite) | 0.02 - 0.03 |

Table 1. Optical Properties of Asbestos Fibers (Continued)

| | | | Central Stop Dispersion Staining Colors | | |
|---|---|---|---|---|---|
| Mineral | Extinction | Sign of Elongation | RI Liquid | ⊥ to Vibration | ∥ to Vibration |
| Chrysotile | Parallel to fiber length | + (length slow) | 1.550 HD | Blue | Blue-magenta |
| Cummingtonite-Grunerite (Amosite) | Parallel to fiber length | + (length slow) | 1.670<br>Fibers subjected to high temperatures will not dispersion-stain. | Red magenta to blue | Yellow |
| Cummingtonite | | | 1.680 | pale blue | blue |
| Grunerite | | | 1.680 | blue | gold |
| Crocidolite (Riebeckite) | Parallel to fiber length | - (length fast) | 1.700 | Red magenta | Blue-magenta |
| | | | 1.680 | yellow | pale yellow |
| Anthophyllite | Parallel to fiber length | + (length slow) | 1.605 HD | Blue | Gold to gold-magenta |
| | | | 1.620 HD | Blue-green | Golden-yellow |
| Tremolite-Actinolite | Oblique - 10 to 20° for fragments. Some composite fibers show ∥ extinction. | + (length slow) | 1.605 HD | Pale blue (tremolite) | Yellow (tremolite) |
| | | | | Yellow (actinolite) | Pale yellow (actinolite) |

HD = high-dispersion RI liquid series.