National Institute of Standards & Technology

Certificate

Standard Reference Material 1866a

Common Commercial Asbestos

Standard Reference Material (SRM) 1866a is a set of three common bulk mine-grade asbestos materials and one synthetic glass fiber material. The three asbestos types are chrysotile, grunerite (Amosite), and riebeckite (crocidolite). The optical properties of each of these materials have been characterized by polarized light microscopy so that these samples may serve as primary calibration standards in the identification of asbestos types in building materials [1]. These asbestos materials are typical of the asbestos found in bulk samples during routine asbestos inspections of building materials. However, various conditions, such as geographic origin or acid/heat treatment of the asbestos, could cause the optical properties of the asbestos in bulk insulation samples to differ considerably from the materials comprising this SRM [2]. The glass fiber sample serves as a non-asbestos-containing material (or blank) to check for contamination that would affect the accuracy and limits of detection of asbestos analysis.

The materials were prepared and packaged by Research Triangle Institute, Research Triangle Park, N.C., under the direction of R.L. Perkins.

Characterization of the Standard Reference Materials was performed in the NIST Surface and Microanalysis Science Division, by J.R. Verkouteren, J.M. Phelps, E.S. Windsor, D.M. Hues, and E.B. Steel; and by R.L. Perkins and B.W. Harvey of Research Triangle Institute.

Statistical analysis of the certification data was provided by S.D. Leigh and K.R. Eberhardt of the Statistical Engineering Division, NIST.

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The overall direction and coordination of the technical measurements leading to certification were under the direction of E.B. Steel and R.A. Velapoldi, Chief of the Surface and Microanalysis Science Division, NIST.

The technical and support aspects involved in the preparation, certification and issuance of this Standard Reference Material were coordinated through the Standard Reference Materials Program by T.E. Gills.

Gaithersburg, MD 20899
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William P. Reed, Chief
Standard Reference Materials Program

(over)

The certification of each of the asbestos materials in this set comprising SRM 1866a is primarily qualitative with each material certified to be the asbestos type identified and to exhibit the properties given on pages 3 and 4 of this certificate.

Characterization: The SRM asbestos materials were characterized by optical crystallographic analysis, x-ray powder diffraction, electron microprobe analysis, and analytical electron microscopy. The variation of refractive index as a function of wavelength was characterized using oblique illumination coupled with the double variation method to yield accuracies of ± 0.001 refractive index units or better as measured on standard materials. Becke-line and central stop dispersion techniques were used as supporting refractive index measurement techniques.

Individual asbestos fibrils cannot generally be resolved using polarized light microscopy because they are small compared to the wavelength of light in the visible region. For this reason, the refractive indices are measured on fiber bundles (comprised of many fibrils). Because the asbestos minerals are all biaxial, there should be an α, β, and γ index of refraction associated with each (for chrysotile α and β should be equivalent). However, because the fibrils are oriented randomly about the c-axis of the fiber bundle (long direction), and all the fibrils in the bundle may not be parallel to one another, the true α, β, and γ cannot be determined. The convention for reporting the refractive indices of chrysotile, grunerite, and riebeckite asbestos when they exhibit parallel extinction is to determine two indices, one parallel to the bundle length, and one perpendicular to the bundle length [3]. The higher value is reported as γ' and the lower value is reported as α'. For chrysotile and grunerite, γ' is parallel to the fiber bundle length, and α' is perpendicular to the fiber bundle length. The reverse is true for riebeckite.

Use: The bulk mine-grade asbestos samples are each comprised of approximately 4 g of material. This should provide enough material for hundreds of microscope slide immersion preparations for training and quality assurance activities associated with asbestos analysis by polarized light microscopy. The mean refractive index value determined from a minimum of 10 fibers should be compared with the certified fitted values to determine the correspondence between measured values and reference values. The measurement of at least 10 fibers takes into account the variability in the refractive index of individual fibers. The glass fiber sample serves as a reference blank containing no asbestos and care should be taken not to contaminate it.

CERTIFIED PROPERTIES

Macroscopic Properties:	Chrysotile	Grunerite (Amosite)	Riebeckite (Crocidolite)
Distribution of phases	homogeneous	homogeneous	homogeneous
Texture	asbestiform	asbestiform	asbestiform
Color	white	gray-brown	blue
Concentration of asbestos (by wt. or vol.)	>90%	>90%	>90%

Microscopic Properties:	Chrysotile	Grunerite (Amosite)	Riebeckite (Crocidolite)
Morphology	asbestiform[1]	asbestiform	asbestiform
Pleochroism	none	very weak	α' dark blue γ' light blue
Birefringence	low (0.007)[2]	medium (0.023)[2]	low (anomalous red)
Extinction	parallel or wavy	mostly parallel[3]	mostly parallel[3]
Sign of elongation	positive	positive	negative

Chrysotile Refractive Indices

Wavelength nm	α' lower limit[4]	α' fitted value[5]	α' upper limit[4]	γ' lower limit[4]	γ' fitted value[5]	γ' upper limit[4]
460	1.554	1.558	1.563	1.563	1.568	1.572
480	1.552	1.557	1.561	1.561	1.565	1.569
500	1.551	1.555	1.559	1.559	1.563	1.567
520	1.549	1.553	1.557	1.557	1.561	1.565
540	1.548	1.552	1.556	1.556	1.560	1.564
560	1.547	1.551	1.555	1.554	1.558	1.562
589.3	1.545	1.549	1.553	1.552	1.556	1.560
600	1.545	1.549	1.553	1.551	1.556	1.560
620	1.544	1.548	1.552	1.550	1.554	1.559
640	1.543	1.547	1.551	1.549	1.553	1.558

[1] Asbestiform: crystallizes with the habit of asbestos. These commercial asbestos minerals possess properties such as long fiber length and high tensile strength. Under the light microscope, these materials exhibit the asbestiform habit characterized by the following:

a) mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than 5 μm,
b) very thin fibrils, usually less than 0.5 μm in width,
c) parallel fibers occuring in bundles,
d) fiber bundles displaying splayed ends,
e) fibers in the form of thin needles,
f) matted masses of individual fibers, and
g) fibers showing curvature.

[2] Calculated from the measured refractive indices.

[3] Thicker fibrils showing oblique extinction are rare, but do occur, and if oriented properly will show extinction angles similar to those reported in texts [5] for the non-asbestiform variety of the mineral.

[4] The reported lower and upper limits are uncertainties that were computed as simultaneous tolerance interval values designed to cover 95% of the refractive index values measured at the nominal wavelength at a 95% confidence level [6]. The uncertainties reported here do not take into account error in the measured wavelengths or the internal correlation associated with the measurements on each individual fiber that is characteristic of these particular dispersion curves. The uncertainties reflect the composite measurement variation introduced by the double variation method and by multiple analysts, fibers, and refractive index liquids.

[5] The refractive index values reported here are the least squares fit of a two-term Cauchy equation to the optical data collected using the double variation method [7].

Grunerite (Amosite)[6] Refractive Indices

Wavelength nm	α' lower limit[7]	α' fitted value[8]	α' upper limit[7]	γ' lower limit[7]	γ' fitted value[8]	γ' upper limit[7]
500	1.685	1.688	1.692	1.707	1.711	1.714
520	1.682	1.686	1.689	1.704	1.708	1.711
540	1.680	1.683	1.686	1.702	1.706	1.709
560	1.678	1.681	1.684	1.700	1.704	1.707
589.3	1.675	1.678	1.681	1.697	1.701	1.704
600	1.674	1.677	1.681	1.696	1.701	1.704
620	1.673	1.676	1.679	1.695	1.699	1.702
640	1.671	1.674	1.678	1.693	1.698	1.701

Riebeckite (Crocidolite)

Because strong absorption in the visible light range results in anomalous dispersion characteristics [8] that would not be useful to the analyst, no certified values of refractive index are reported for riebeckite. Refer to the supplemental information where descriptions are given of the Becke lines and central stop dispersion colors observed for riebeckite.

Glass Fiber Sample

The glass fiber sample is certified to have no asbestos at or above the detection limit of the polarized light microscope technique.

[6] The major asbestiform mineral phase present, and the phase for which the refractive indices are listed, is grunerite (Amosite) asbestos. Another asbestiform phase with much lower indices is present at concentrations of ≤ 5%. This accessory asbestiform phase has not been characterized for this SRM material.

[7] The reported lower and upper limits are uncertainties that were computed as simultaneous tolerance interval values designed to cover 95% of the refractive index values measured at the nominal wavelength at a 95% confidence level. The grunerite (Amosite) γ' lower tolerance limits have been conservatively adjusted for special dispersive effects at the lower range of wavelengths. The uncertainties reported here do not take into account error in the measured wavelengths nor the internal correlation associated with the measurements on each fiber that is characteristic of these particular dispersion curves. The uncertainties reflect the composite measurement variation introduced by the double variation method and by multiple analysts, fibers, and refractive index liquids.

[8] The refractive index values reported here are the least squares fit of a two-term Cauchy equation to the optical data collected using the double variation method [7].

Supplemental Information

The following non-certified technical information is supplied for the convenience of the user of this SRM [9].

Size distribution of asbestos phases in SRM 1866a: In general, the SRM materials of chrysotile, grunerite (Amosite), and riebeckite (crocidolite) are comprised of long bundles of asbestos fibers surrounded by a finer-grained matrix of asbestos fibers. The grunerite and riebeckite materials commonly include bundles approaching 6 cm in length, whereas in chrysotile the bundles typically do not exceed 2 cm in length. All of the asbestos SRM materials exhibit a range of fiber lengths from micrometers to centimeters.

Accessory phases: The SRM materials of chrysotile, grunerite (Amosite), and riebeckite (crocidolite) contain accessory phases including opaque minerals. The grunerite material contains an additional asbestiform phase which has much lower refractive indices than the primary grunerite asbestos. The combined total of all accessory phases in any of the asbestos SRM materials does not exceed 10% by weight or volume.

Central Stop Dispersion Colors and Becke-Line Colors: The central stop dispersion (focal masking) colors for chrysotile and grunerite (Amosite) are not given in this document. They can be determined, however, by plotting the refractive index *vs* wavelength pairs given for chrysotile and grunerite to generate dispersion curves, which can then be compared with the dispersion curve of the immersion liquid used. The intersection of the solid and liquid dispersion curves gives the wavelength at which the two match. The matching wavelength can then be converted to either central or annular stop dispersion colors by consulting texts such as references 2 or 7.

Because strong absorption in the visible light range results in anomalous dispersion characteristics [8], no certified values of refractive index are reported for riebeckite (crocidolite). The anomalous dispersion of riebeckite and its strong pleochroism cause interpretation of Becke-line and dispersion colors to have very low reliability and accuracy in the determination of refractive index. However, the observed optical properties including Becke-line and dispersion colors, are characteristic and can be used to positively identify riebeckite asbestos. Analysts may calibrate themselves by measuring the optical properties on this standard. The information below provides guidance for this calibration. A range of central stop dispersion colors and Becke-line colors can be seen in each mount.

High dispersion liquid n_D	In-focus central stop dispersion color	Corresponding nominal wavelength match, nm
1.680	golden yellow to red-magenta	455 - 520
1.690	magenta to light blue-green	560 - 625
1.700	light blue-green to very pale blue-green	625 - >700

It is difficult to observe Becke-line colors indicative of a refractive index match (sky blue and orange yellow [10]) due to riebeckite's strong absorption and resultant blue color. However, colors indicating matches can be observed on some fibers in the range of immersion liquids from n_D = 1.684 to 1.696. Another indication of a match in this range of liquids is the very low relief exhibited by some fibers. Most of the riebeckite fibers have a low birefringence, and in some cases there is no measurable difference by Becke-line or dispersion colors between the refractive indices of the parallel and perpendicular directions. However, when a measurable difference exists, the higher refractive index (γ') is always perpendicular to the fiber length.

Glass Fiber Sample: The glass fibers are colorless, isotropic fibers that are covered nonuniformly with a pink, phenolic resin. The uncoated fibers have an approximate refractive index of 1.52. The phenolic resin has a higher refractive index than the fibers. Because the phenolic resin does not cover all fibers uniformly, the refractive index of any fiber will be some combination of the refractive indices of the glass fiber and the resin. No asbestos was found in this material when analyzed by polarized light microscopy, x-ray powder diffraction, and analytical transmission electron microscopy.

References:

1. U.S. Environmental Protection Agency Interim Method for the Determination of Asbestos in Bulk Insulation Samples, 40 CFR Pt. 763, Subpt. F, App. A., 1987.

2. McCrone, W.C., Asbestos Identification, McCrone Research Institute, Chicago, Illinois, 1987.

3. Wylie, A., "Optical Properties of the Fibrous Amphiboles", Annals of the New York Academie of Sciences, 1979, pp. 611-619.

4. Kerr, P.F., Optical Mineralogy, McGraw-Hill, Inc., New York, 1977.

5. Deer, W.A., Howie, R.A., and Zussman, J., Rock-Forming Minerals, Vols. 2 and 3, Longman Group Limited, London, 1962.

6. Miller, R.G., Simultaneous Statistical Inference, pp. 123-125 (Sect. 4.2, eq. 30), McGraw-Hill Inc., New York, 1966.

7. Bloss, F.D., The Spindle Stage: Principles and Practice, Cambridge University Press, Cambridge, Massachusetts, 1981.

8. Wahlstrom, E.E., Optical Crystallography, John Wiley & Sons, Inc., New York, 1969.

9. Taylor, J.K., Handbook for SRM Users, NIST SP 260-100 (1985). A copy of this publication is available upon request from the NIST Standard Reference Materials Program, Gaithersburg, Maryland 20899, USA.

10. Bloss, F.D., An Introduction to the Methods of Optical Crystallography, Holt, Rinehart and Winston, Inc., New York, 1961.