National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1866

Bulk Asbestos - Common

Standard Reference Material (SRM) 1866 is a set of the three common bulk mine-grade asbestos materials and one synthetic glass fiber sample. The three asbestos types are chrysotile, grunerite (Amosite), and riebeckite (crocidolite). The optical properties of each of these materials observed by polarized light microscopy have been characterized so that these samples may serve as primary calibration standards for the identification of asbestos types in building materials [1]. 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. 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 vary considerably from the materials comprising this SRM [2].

The certification of each of the asbestos materials in the set of samples comprising SRM 1866 is primarily qualitative, each material certified to be the asbestos type listed and to exhibit the following properties:

CERTIFIED PROPERTY

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%

Characterization of the Standard Reference Materials was performed in the NIST Gas and Particulate Science Division, by J.R. Verkouteren, J.M. Phelps, C.J. Poston, D.M. Johansen, and E.B. Steel; and by R.L. Perkins and B.W. Harvey of Research Triangle Institute and J. Renton of West Virginia University.

Support for the preparation of these materials was supplied by the U.S. Environmental Protection Agency, Office of Toxic Substances under the guidance of E.A.Dutrow and J.J. Breen.

The materials were mixed and packaged by Research Triangle Institute under the direction of R.L. Perkins.

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

The overall direction and coordination of the technical measurements leading to certification were under the direction of E.B. Steel, Gas and Particulate Science Division, NIST.

The technical and support aspects involved in the preparation, certification and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.L. McKenzie.

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Stanley D. Rasberry, Chief
Office of Standard Reference Materials

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CERTIFIED PROPERTY (Continued)

Microscopic Properties:	Chrysotile	Grunerite (Amosite)	Riebeckite (Crocidolite)
Morphology	asbestiform[a]	asbestiform	asbestiform
Pleochroism	none	very weak	α' dark blue γ' light blue
Birefringence	low (0.007)[b]	medium (0.022)[b]	low anomalous red
Extinction	parallel wavy	mostly parallel[c]	mostly parallel[c]
Sign of elongation	positive	positive	negative

[a]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 samples exhibit the asbestiform habit characterized by the following:

1) mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than 5 µm,
2) very thin fibrils, usually less than 0.5 µm in width,
3) parallel fibers occurring in bundles,
4) fiber bundles displaying splayed ends,
5) fibers in the form of thin needles,
6) matted masses of individual fibers, and
7) fibers showing curvature.

[b]Calculated from the measured refractive indices reported in the following tables.

[c]Thicker fibrils showing oblique extinction are rare, but do occur, and if oriented properly will show extinction angles similar to those reported in texts [3, 4] for the non-asbestiform variety of the mineral.

Refractive Index

	Chrysotile					
	α'			γ'		
wavelength (nm)	lower limit[d]	fitted value[e]	upper limit[d]	lower limit[d]	fitted value[e]	upper limit[d]
460	1.554	1.559	1.563	1.564	1.568	1.573
480	1.552	1.557	1.561	1.561	1.566	1.570
500	1.551	1.555	1.559	1.559	1.563	1.568
520	1.549	1.553	1.557	1.557	1.562	1.566
540	1.548	1.552	1.556	1.555	1.560	1.564
560	1.546	1.550	1.554	1.554	1.558	1.563
589.3	1.544	1.549	1.553	1.552	1.556	1.561
600	1.544	1.548	1.552	1.551	1.556	1.560
620	1.543	1.547	1.551	1.550	1.555	1.559

[d]The reported lower and upper limits are uncertainties which 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 [5]. The uncertainties reported here do not take into account error in the measured wavelengths nor 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.

[e]The refractive index values reported here are the best fit of a two term Cauchy equation to the optical data collected using the double variation method.

Refractive Index	Grunerite (Amosite)[f]					
	α'			γ'		
wavelength (nm)	lower limit[g]	fitted value[h]	upper limit[g]	lower limit[g]	fitted value[h]	upper limit[g]
500	1.687	1.691	1.694	1.709	1.713	1.716
520	1.684	1.688	1.691	1.706	1.710	1.713
540	1.681	1.685	1.688	1.703	1.707	1.710
560	1.679	1.682	1.686	1.701	1.704	1.708
589.3	1.676	1.679	1.683	1.697	1.701	1.704
600	1.675	1.678	1.682	1.696	1.700	1.703
620	1.673	1.676	1.680	1.694	1.698	1.701

[f]The major asbestiform mineral phase present, and the phase for which the refractive indices are listed, is grunerite asbestos. Another asbestiform phase with much lower indices is present at concentrations of less than approximately 5%. This accessory asbestiform phase has not been characterized for this standard.

[g]The reported lower and upper limits are uncertainties which 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 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.

[h]The refractive index values reported here are the best fit of a two term Cauchy equation to the optical data collected using the double variation method.

Because strong absorption in the visible light range results in anomalous dispersion characteristics [6] that would not be useful to the analyst, no certified values of refractive index are reported for riebeckite. Refer to the appendix 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 be free of asbestos mineral(s) when analyzed with the polarized light microscope.

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. A minimum of 10 fibers must be measured to compare with reference values to take 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.

Analysis: The SRM 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 [7] 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 crystallites or 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 asbestos minerals exhibiting parallel extinction is to determine two indices, one parallel to the bundle length, and one perpendicular to the bundle length. The higher value is reported as γ' and the lower value is reported as α' [8]. For chrysotile and grunerite, γ' is parallel to the fiber bundle length, and α' is perpendicular. The reverse is true for riebeckite.

Supplemental Information

The following information is supplied for the convenience of the user of this Standard Reference Material. The information provided is not certified and is supplied for informational purposes only [9].

Size distribution of asbestos phases in Standard Reference Materials:
In general, the Standard Reference Material samples of chrysotile, grunerite, and riebeckite are comprised of long bundles of asbestos fibers surrounded by a finer-grained matrix of asbestos fibers. The grunerite and riebeckite standards 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 Standard Reference Material samples exhibit a range of fiber lengths from micrometers to centimeters.

Accessory phases: The samples of chrysotile, grunerite, and riebeckite contain accessory phases including opaque minerals. Grunerite does contain an asbestiform accessory phase which has much lower refractive indices than the primary grunerite asbestos. The combined total of all accessory phases in any of the asbestos Standard Reference Material samples 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 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 solid and liquid dispersion curves gives the wavelength at which the two match, which 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 [6], no certified values of refractive index are reported for riebeckite. The anomalous dispersion of riebeckite combined with 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. Example information is reported below to provide guidance in this calibration including dispersion colors and Becke line information. A range of central stop dispersion colors and Becke line colors was 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 [8]) 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 immersion methods 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 which are covered non-uniformly 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:

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