



# National Institute of Standards & Technology

## Certificate

### Standard Reference Material 1867

#### Uncommon Commercial Asbestos

Standard Reference Material (SRM) 1867 is a set of three mine-grade asbestos materials. The three asbestos types are anthophyllite, tremolite, and actinolite, which are relatively uncommon as additions to building materials. A unit of SRM 1867 contains one (1) bottle each of anthophyllite, tremolite, and actinolite in quantities of several grams each. 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 in building materials.[1] The three asbestos materials are typical representatives of their mineral types; however, the amphibole group of minerals is complex and exhibits a wide range of possible compositions and optical properties. Differences in composition and heat or solvent treatment may cause the optical properties of the asbestos in bulk insulation samples to differ considerably from the materials in this SRM.

**Source of Materials:** The anthophyllite was purchased from Ward's Natural Science Establishment, Incorporated, who report the sample locality as the Rakabedo Mines near Udaipur, India. The actinolite was collected by E.B. Steel and J.M. Phelps of NIST at a construction site in Fairfax County, VA. The tremolite was collected by S. Bezore of the State of California Department of Conservation from the Conda deposit near Barstow, CA.[2]

**Intended Use:** The three materials are intended to be used as calibration standards for the identification of anthophyllite, tremolite, and actinolite asbestos by polarized light microscopy (PLM). All of the optical properties given for the three asbestos types are measurable by PLM. The refractive indices of the fibers can be measured from grain mount preparations; however, more precise methods which involve fiber orientation are used to produce more accurate results.[3,4] There are specific guidelines for the measurement of refractive index of fibers in grain mounts included with this SRM. These guidelines may not be applicable to the measurement of unknown fibers in bulk insulation materials because they assume relatively pure materials in which the variation in refractive index is due solely to fiber orientation.

The SRMs were prepared and packaged by Research Triangle Institute, Research Triangle Park, NC, under the direction of R.L. Perkins.

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

Statistical analysis of the certification data was performed in the NIST Statistical Engineering Division by S.D. Leigh.

The overall direction and coordination of the technical measurements leading to certification was performed in the NIST Surface and Microanalysis Science Division by E.B. Steel and R.A. Velapoldi.

The technical and support aspects involved in the certification and issuance of this SRM were coordinated through the Standard Reference Materials Program by N.M. Trahey.

Gaithersburg, MD 20899  
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(over)

**Characterization:** The SRM materials were characterized by optical crystallographic analysis, x-ray powder diffraction, electron microprobe analysis, and analytical electron microscopy. The refractive indices of the asbestos materials were measured on oriented asbestos fibers throughout the visible light range using the spindle stage with the double variation technique.[3,4]

Table 1  
Certified Properties

Material	Anthophyllite	Tremolite	Actinolite
<b>Macroscopic Properties</b>			
Color	tan	white, pale green for massive pieces	white, green for massive pieces
Texture	asbestiform <sup>a</sup>	asbestiform <sup>a,b</sup>	asbestiform <sup>a,b</sup>
Distribution of phases	homogeneous	homogeneous	homogeneous
Concentration of asbestos	> 80 %	> 90 %	> 90 %
<b>Microscopic Properties</b>			
Morphology	asbestiform	asbestiform	asbestiform
Color	colorless	colorless	colorless
Pleochroism	none	none	none
Birefringence	0.021	0.028	0.027
Extinction angle <sup>c</sup>	0 (parallel extinction)	16.6 ± 0.3°	15.9 ± 0.2°
Sign of elongation	(+)	(+)	(+)
$\gamma n_D^{d,e}$	1.6362 ± 0.0007	1.6343 ± 0.0007	1.6393 ± 0.0007
$\beta n_D^{d,e}$	1.6273 ± 0.0007	1.6230 ± 0.0007	1.6288 ± 0.0007
$\alpha n_D^{d,e}$	1.6148 ± 0.0007	1.6063 ± 0.0007	1.6126 ± 0.0007

<sup>a</sup>Asbestiform: crystallizes with the habit of asbestos. These asbestos minerals possess properties such as long fiber length and high tensile strength. Under the light microscope, these samples exhibit the asbestiform habit as defined by several of the following characteristics: 1) mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than 5  $\mu\text{m}$ , 2) very thin fibrils, usually less than 0.5  $\mu\text{m}$  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.

<sup>b</sup>A small amount of the material is massive in texture.

<sup>c</sup>The uncertainties given for extinction angle are 95% confidence intervals.

<sup>d</sup>The refractive index (n) at the sodium D line (doublet at 589.3 nm).

<sup>e</sup>The uncertainties for the  $n_D$  values are 95% Working-Hotelling confidence intervals corrected for bias from calibration measurements.[5]

**Dispersion of refractive indices:** The dispersion of each principal index,  $\gamma$ ,  $\beta$ , and  $\alpha$ , has been determined for the wavelength range 470-620 nm. To calculate the refractive index at any wavelength in that range, substitute parameters a and b, given in Table 1, into the equation

$$n(\lambda) = a + b/\lambda^2$$

where n is refractive index, and  $\lambda$  is wavelength in nanometers (nm).

Table 2  
Dispersion Constants

Principal Index	Anthophyllite	Tremolite	Actinolite
$\gamma^a$	a = 1.61762 (0.00045) b = 6442 (122)	a = 1.61713 (0.00024) b = 5953 (69)	a = 1.61973 (0.00040) b = 6802 (107)
$\beta^a$	a = 1.61084 (0.00030) b = 5701 (83)	a = 1.60832 (0.00049) b = 5088 (142)	a = 1.61137 (0.00053) b = 6041 (147)
$\alpha^a$	a = 1.59808 (0.00046) b = 5812 (133)	a = 1.59236 (0.00035) b = 4824 (104)	a = 1.59653 (0.00042) b = 5574 (120)

<sup>a</sup>The value in parentheses following each parameter is the estimated standard deviation ( $1\sigma$ ) of the parameter.

There is a bias associated with wavelength in the measurement technique used that results in positive errors at short wavelengths. The 95% confidence intervals given in the following table have been corrected for this bias. As a result, the errors are not equivalent for each wavelength nor necessarily symmetric. The uncertainties associated with any calculated refractive index can be estimated by interpolation from the errors given for the 4 wavelengths in Table 3.

Table 3  
Uncertainties for Calculated Refractive Indices

$\lambda$ , nm	Uncertainties
434	+0.0005, -0.0015
486.1	+0.0005, -0.0010
589.3	$\pm 0.0007$
656.3	$\pm 0.0007$

## INFORMATION VALUES

The elemental compositions for the three asbestos minerals were determined by electron probe microanalysis (EPMA) and are given as information values only. The weight percent (Wt %) of each oxide has a relative error of 10 % or less. The relative error of the chemical composition is defined by 2 times the standard deviation of the EPMA analyses and therefore takes into account the precision of the analytical method, but does not include any potential for bias. The water content of the three asbestos types was not determined.

Asbestos type	SiO <sub>2</sub>		FeO <sup>a</sup>		MgO		CaO		Mg/(Mg+Fe)
	Wt %	cations <sup>b</sup>	Wt %	cations <sup>b</sup>	Wt %	cations <sup>b</sup>	Wt %	cations <sup>b</sup>	
Anthophyllite	58	8.0	8.9	1.0	29	5.9	—	—	0.86
Tremolite	56	8.0	2.2	0.3	22	4.7	13	2.0	0.94
Actinolite	57	8.0	6.5	0.8	20	4.3	13	1.9	0.84

<sup>a</sup>All Fe calculated as Fe<sup>2+</sup>.

<sup>b</sup>Calculated on a water-free basis to 23 oxygens and 2 (OH,F,Cl)<sub>2</sub> as given in reference [6].

**Specific guidelines for refractive index measurement of SRM 1867 from grain mounts:** Anthophyllite, tremolite, and actinolite are biaxial and therefore, have three principal vibration directions that correspond to three principal refractive indices;  $\alpha$ ,  $\beta$ , and  $\gamma$ . The principal refractive indices are only observed for specific crystallographic orientations of the asbestos fibers; all other orientations will yield two intermediate refractive indices, one between  $\alpha$  and  $\beta$  ( $\alpha'$ ) and a second between  $\beta$  and  $\gamma$  ( $\gamma'$ ). Measurements of  $\alpha'$  and  $\gamma'$  from randomly selected fibers in grain mounts are inconclusive for mineral identification purposes because of the large range of possible values. The procedures in this section are meant to provide criteria to select fibers from grain mounts that display refractive indices as close to the principal values as possible.

A classical method of measuring principal refractive indices from a population of randomly oriented grains is to use interference figures to determine optical orientation.[7-9] Unfortunately, asbestos fibers are typically too thin to produce observable interference figures. Extinction angle cannot be used as an indicator of orientation for tremolite and actinolite because the maximum extinction angle is exhibited for a large range of orientations.[10] Birefringence is also an indicator of orientation given particles of equivalent thickness, which is not the case for the asbestos fibers. Since there is no independent indicator of fiber orientation, the only way to measure the principal refractive indices of the asbestos minerals from grain mounts is to observe a large number of fibers to find the maximum ( $\gamma$ ) and minimum ( $\alpha$ ) refractive indices. Because of the inability to determine the optical orientation of the fibers there will be some error in the measurement of the refractive indices;  $\gamma$  will tend to be biased low (all  $\gamma'$  are less than  $\gamma$ ) and  $\alpha$  will tend to be biased high (all  $\alpha'$  are greater than  $\alpha$ ). Errors of this nature should be relatively small and should not limit the usefulness of the measurement. Details of the measurement process are given below for anthophyllite and tremolite/actinolite.

**Anthophyllite:** Anthophyllite has parallel extinction and a positive elongation; fibers lying flat in grain mounts will always exhibit  $\gamma$  parallel to fiber elongation, and either  $\alpha$ ,  $\beta$ , or  $\alpha'$  perpendicular to fiber elongation (Figure 1). The smallest refractive index observed perpendicular to elongation is  $\alpha$ , the largest refractive index perpendicular to elongation is  $\beta$ .

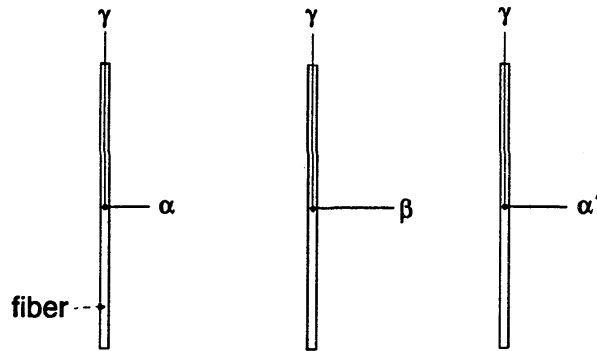


Figure 1. Possible orientations of anthophyllite fibers; the general case shown on the right yields  $\alpha'$ , which can be any value between  $\alpha$  (1.615) and  $\beta$  (1.627).

**Preparation:** Prepare pinch mounts using appropriate refractive index liquids to determine  $\gamma$ , the refractive index parallel to elongation, first. There should be very little difference in the  $\gamma$  refractive index from fiber to fiber. Prepare the next mount in a liquid 0.02 (the birefringence) lower than the measured  $\gamma$  as a starting point to measure  $\alpha$ . There should be a wide range in the refractive indices observed perpendicular to elongation as there is a difference of 0.012 between  $\alpha$  and  $\beta$ . Determine whether any of the fibers have a refractive index perpendicular to elongation that is lower than the liquid's refractive index, observing approximately 50 fibers. Change refractive index liquids as necessary to determine the minimum refractive index ( $\alpha$ ). If desired,  $\beta$  can be measured by determining the maximum refractive index perpendicular to elongation.

**Tremolite and actinolite:** Tremolite and actinolite have inclined extinction; only one of the three vibration directions ( $\beta$ ) coincides with the fiber axes. Both asbestos types have a positive elongation; the larger refractive index ( $\gamma$  or  $\gamma'$ ) is always closer to the fiber elongation (Figure 2). Fibers in grain mounts will be randomly oriented about the fiber axis to produce many possible orientations, as shown in Figure 2. The general case is shown on the right with a  $\gamma'$  that is always smaller than true  $\gamma$ , and an  $\alpha'$  between  $\alpha$  and  $\beta$ . [The range of possible  $\gamma'$  does not extend all the way down to  $\beta$  because the range of possible orientations is restricted as the fibers tend to lie close to flat on the microscope slide.] There is only one orientation for which  $\gamma$ ,  $\alpha$  and the true extinction angle ( $c \wedge Z$ ) can be measured.

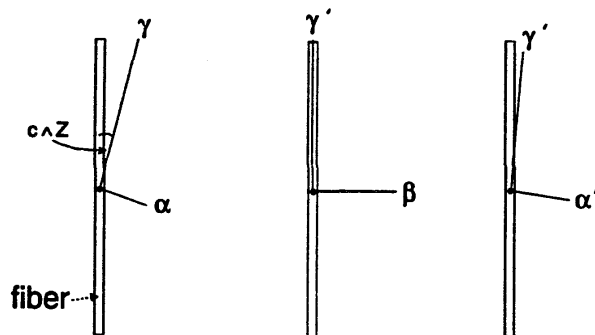


Figure 2. The possible orientations of tremolite and actinolite. The general case on the right exhibits  $\gamma'$  and  $\alpha'$ ;  $\alpha'$  is any value between  $\alpha$  and  $\beta$  (a difference of 0.016-0.017).

**Preparation:** One approach for measuring refractive indices for tremolite and actinolite is given here, but there are many others. Prepare pinch mounts of material in appropriate refractive index liquids to measure  $\gamma$  first. Determine the maximum refractive index parallel to elongation by observing approximately 50 fibers; there should be a range in values of about 0.005. (For more accurate measurements, the fiber should be placed at the extinction position to measure both  $\gamma$  and  $\alpha$ ; measuring the refractive indices when the fibers are parallel or perpendicular to the polarizer will result in refractive index errors of approximately 0.002.) The number of fibers that display the maximum value should be a minority of the total number of fibers observed, probably 20% or less. Prepare the next mount in a liquid 0.027-0.028 (the birefringence) lower than the measured  $\gamma$  as a starting point to measure  $\alpha$ . There should be a wide range in the refractive indices observed perpendicular to elongation as there is a difference of 0.016-0.017 between  $\alpha$  and  $\beta$ . Determine whether any of the fibers have a refractive index perpendicular to elongation that is lower than the liquid's refractive index, observing at least 50 fibers. Change refractive index liquids as necessary to determine the minimum refractive index ( $\alpha$ ). As with the measurement of  $\gamma$ , the number of fibers that display the minimum value should be a minority of the total number of fibers observed, probably 20% or less. If desired,  $\beta$  can be measured on individual fibers exhibiting parallel extinction (see Figure 2).

**Extinction angle:** As shown in Figure 2, the true extinction angle is only exhibited by the orientation that contains true  $\gamma$  and  $\alpha$ . All other orientations will yield extinction angles between  $0^\circ$  (for the orientation that contains  $\beta$ ) and approximately  $1^\circ$  higher than the true extinction angle. Extinction angles should be measured on fibers that have been determined to display the maximum ( $\gamma$ ) and/or minimum ( $\alpha$ ) refractive indices.

#### REFERENCES

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