National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1867a

Uncommon Commercial Asbestos

This standard reference material (SRM) is comprised of three uncommon commercial asbestos materials (tremolite asbestos, actinolite asbestos, and anthophyllite asbestos) intended for use in the identification of these minerals by polarized light microscopy (PLM) [1,2]. The certified values for SRM 1867a are identical to the original certified values provided in the previous issuance of this material, SRM 1867. Additional reference values and information values are provided with this reissue. The materials used in SRM 1867a came from the original lots of material used for SRM 1867 and represent the last of the original supply. Each unit of SRM 1867a consists of a set of three bottles, each containing several grams of one of the three mine-grade asbestos materials, with certified optical properties that can be measured by PLM.

The three materials contained in this SRM are single representatives of their mineral types and cannot represent all the variability inherent to these mineral species. The changes in optical properties due to differences in chemical composition for tremolite and actinolite from the materials contained in this SRM are detailed in Reference 3. The unique morphology of asbestos may alter the optical properties of tremolite, actinolite, and anthophyllite asbestos from those reported for the materials contained in this SRM, as described in Reference 4.

Certified Properties and Uncertainties: Refractive indices were measured in the range of visible wavelengths by using the double variation technique on individual fibers oriented with a spindle stage [5,6] in immersion liquids calibrated by an independent technique (minimum deviation). Refractive indices were fit to a Cauchy equation to calculate the refractive index at 589.3 nm (n_D) (Table 1) and to provide the dispersion constants (Table 2). Expanded uncertainties were calculated as Working-Hotelling confidence intervals [7,8] with a coverage factor 2 corrected for bias from calibration measurements.

Reference Values: Extinction angles (Table 1) were measured from oriented fibers with uncertainties on the means calculated as expanded uncertainties with a coverage factor 2 [8]. The compositions of the materials were determined by electron probe microanalysis (EPMA) on polished specimens and are given in Table 3 in % mass fraction of the oxide and in atoms per formula unit (apfu) calculated on the basis of 23 oxygens. The unit cell dimensions (Table 4) of the materials were determined by X-ray diffraction (XRD) on powdered specimens using an internal standard. Reference values are non-certified values that are the best estimate of the true value; however, the values, which are based on determinations done with a single reliable method, do not meet the NIST requirement for certification and are provided with associated uncertainties that may reflect only measurement precision and may not include all sources of uncertainty.

Expiration of Certification: The certification of this SRM is valid until **31 December 2017**, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given. However, the certification will be nullified if the SRM is damaged, contaminated, or otherwise modified.

The support aspects involved in the certification and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald.

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Gaithersburg, MD 20899
Certificate Issue Date: 12 March 2003

John Rumble, Jr., Chief
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Characterization of the Standard Reference Material was performed in the NIST Surface and Microanalysis Science Division by J.R. Verkouteren.

Data from the original issue (SRM 1867) were collected by J.R. Verkouteren, J.M. Phelps, E.S. Windsor, D.M. Hues, and E.B. Steel of the NIST Surface and Microanalysis Science Division; and by R.L. Perkins, B.W. Harvey, S.S. Doorn, and T.F. Bergin of Research Triangle Institute, Research Triangle Park, NC.

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

Source and Packaging of Materials:[1] The anthophyllite was purchased from Ward's Natural Science Establishment, Incorporated, who report the sample origin as the Rakabedo Mines near Udaipur, India. The actinolite was collected by Eric Steel and John Phelps of NIST at a construction site in Fairfax County, VA. The tremolite was collected by Stephen Bezore of the State of California Department of Conservation from the Conda deposit [9] near Barstow, CA. The materials were prepared and packaged by Research Triangle Institute, Research Triangle Park, NC, under the direction of R.L. Perkins.

INSTRUCTIONS FOR USE

Use: The optical properties of the fibers can be measured by immersing the fibers in liquids of known refractive index, either in random orientation or more precisely with fibers oriented on a spindle stage. There are specific guidelines included with this SRM for the measurement of refractive index from randomly oriented fibers in grain mounts. These guidelines may not be applicable to the measurement of unknown fibers in bulk insulation materials, soils, mined products, etc., because they assume relatively pure materials for which the variation in refractive index is due solely to fiber orientation (see *Measurement Guidelines* section).

Special Handling Requirements: Proper procedures for the safe handling of asbestos must be employed during preparation, analysis, and storage of this SRM. Store this SRM in the original bottles, tightly closed, and within the range of normal room temperature and humidity.

CERTIFIED PROPERTIES

Table 1. Refractive index (n) at the sodium D line (589.3 nm) for principal orientations. The uncertainties of the values of n_D are ± 0.0007. For orientation of principal refractive indices with fiber morphology, see the *Measurement Guidelines* section .

Asbestos type	γ n_D	β n_D	α n_D	Birefringence (γ n_D - α n_D)	Extinction angle[a]
Anthophyllite	1.6362	1.6273	1.6148	0.021	0 (parallel extinction)
Tremolite	1.6343	1.6230	1.6063	0.028	16.6 ± 0.3°
Actinolite	1.6393	1.6288	1.6126	0.027	15.9 ± 0.2°

[a] Provided as reference values only.

Table 2. To calculate the refractive index (n) at any wavelength (λ) in the range 470 nm to 620 nm, substitute

[1]Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose

parameters a and b into the equation: $n(\lambda) = a + b/\lambda^2$. The uncertainties in the calculated refractive indices are not necessarily symmetric and vary with wavelength as follows: + 0.0005 and – 0.0015 @ 434 nm, + 0.0005 and – 0.0010 @ 486.1 nm, ± 0.0007 @ 589.3 nm, ± 0.0007 @ 656.3 nm. Uncertainties at all other wavelengths within the range 470 nm to 620 nm can be interpolated from these values.

	$n(\lambda) = a + b/\lambda^2$		
Asbestos type	γ	β	α
Anthophyllite	a = 1.61762 b = 6442	a = 1.61084 b = 5701	a = 1.59808 b = 5812
Tremolite	a = 1.61713 b = 5953	a = 1.60832 b = 5088	a = 1.59236 b = 4824
Actinolite	a = 1.61973 b = 6802	a = 1.61137 b = 6041	a = 1.59653 b = 5574

NOTE: The optical properties leading to certification were measured from the larger, single crystal fibers present in the samples. Some portion of the fibers in each sample will show anomalous optical properties due to the unique fibrillar structure of asbestos and {100} twinning. The anomalous properties of tremolite asbestos and actinolite asbestos include lower extinction angles or parallel extinction and slightly altered refractive indices, as detailed in [4].

REFERENCE VALUES

Table 3. Elemental compositions determined by EPMA calculated in % mass fraction of oxide (assuming all Fe as Fe^{2+}) and in atoms per formula unit (apfu) based on 23 oxygens. The ideal formulas for the minerals are: $Ca_2(Mg,Fe)_5Si_8O_{22}(OH)_2$ for tremolite and actinolite, $(Mg,Fe)_7Si_8O_{22}(OH)_2$ for anthophyllite. The mass fraction of each oxide with a concentration > 1 % mass fraction has a relative error of ≤ 10 %. The reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor is 2, (95 % confidence level). The concentrations of H_2O, F, and Cl were not determined.

Asbestos Type	% SiO_2	apfu Si	% FeO	apfu Fe	% MgO	apfu Mg	% CaO	apfu Ca	Mg/(Mg+Fe+Mn)
Anthophyllite	58.4	8.01	8.3	0.95	29.2	5.97	0.4	0.06	0.86
Tremolite[a]	56.3	7.88	1.3	0.16	23.6	4.93	13.1	1.97	0.96
Actinolite[b]	55.6	7.92	6.5	0.77	20.3	4.32	12.6	1.90	0.84

[a] Also contains 0.4 % mass fraction Al_2O_3 (0.07 apfu Al), 0.5 % mass fraction MnO (0.06 apfu Mn), and 0.2 % mass fraction Na_2O (0.05 apfu Na).

[b] Also contains 0.5 % mass fraction Al_2O_3 (0.09 apfu Al), 0.3 % mass fraction MnO (0.03 apfu Mn), and 0.1 % mass fraction Na_2O (0.02 apfu Na).

Table 4. Unit cell dimensions determined by XRD. The reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor is 2, (95 % confidence level).

Asbestos type	a, Å	b, Å	c, Å	β °
Anthophyllite	18.543 ± 0.004	18.010 ± 0.005	5.285 ± 0.005	---------
Tremolite	9.843 ± 0.002	18.063 ± 0.002	5.278 ± 0.004	104.75 ± 0.1
Actinolite	9.852 ± 0.002	18.088 ± 0.002	5.283 ± 0.002	104.69 ± 0.1

SUPPLEMENTAL INFORMATION

Table 5. Descriptive information from observations with low-power microscopy and polarized light microscopy and approximate concentrations of accessory phases determined by XRD analysis are provided for information purposes only in Table 5.

Anthophyllite

Texture: asbestiform[a]

Color: tan in hand specimen, colorless in plane polarized light

Concentration of anthophyllite: > 80 % mass fraction Talc is estimated to be present at concentrations of approximately 5 % mf to 15 % mf.

Tremolite

Texture: asbestiform[a] Some of the fibers are loose and others are more tightly bound together. A small amount of material may be massive.

Color: white to pale green in hand specimen, colorless in plane polarized light

Concentration of tremolite: > 90 % mass fraction Talc is estimated to be present at concentrations less than 5 % mf.

Actinolite

Texture: asbestiform[a] Some of the fibers are loose and others are more tightly bound together. A considerable amount of material may be massive. The massive material may contain significant clinochlore (chlorite).

Color: white to green in hand specimen, colorless in plane polarized light

Concentration of actinolite: > 80 % mass fraction of the fibrous material Clinochlore (chlorite) is estimated to be present at concentrations that can exceed 20 % mf if a considerable amount of massive material is present. The massive material can be easily segregated, leaving primarily actinolite in the fibrous portion. A small amount (< 5 % mass fraction) of talc may also be present in the fibrous component.

[a] 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, **some portion** of 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 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.

MEASUREMENT GUIDELINES

Anthophyllite, actinolite, and tremolite are biaxial, and therefore, have three principal vibration directions that correspond to three principal refractive indices: α, β, and γ. Amphibole asbestos fibers are elongated along the *c* crystallographic axis, and tend to lay on surfaces parallel to *c* (*hk*0) in immersion slide mounts. The principal refractive indices are found only on the (100) and (010) surfaces, as shown in Figure 1. Since the largest refractive index, γ, is closest to the fiber axis, anthophyllite, tremolite, and actinolite are all optically length slow (positive sign of elongation).

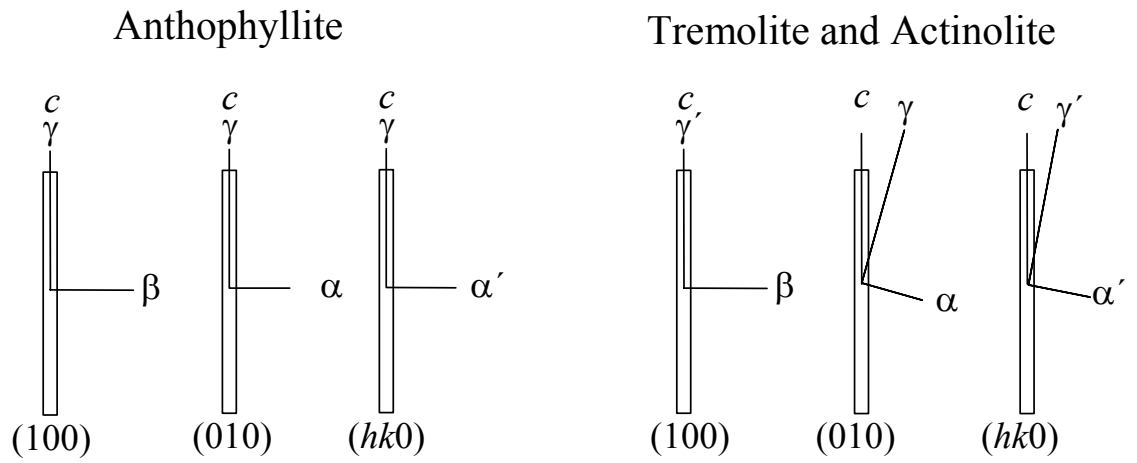

Figure 1. Possible orientations of amphibole asbestos fibers in immersion slide mounts.

The most common orientation that fibers will express in immersion slides mounts is shown by the (*hk*0) orientation. For anthophyllite, this means that γ can be measured from any fiber, but that the index perpendicular to fiber elongation for most fibers will have a value between α and β, represented by α'. For tremolite and actinolite, an (*hk*0) orientation will yield γ' (a value smaller than γ), α' (a value between α and β), and an extinction angle that may not be the true extinction angle (see Reference 4 for more detail).

To measure principal refractive indices for anthophyllite:

From slide immersion mounts, measure γ from any fiber by proper orientation of the fiber with respect to the polarizer. Observe a large number of fibers to determine the minimum refractive index perpendicular to elongation, which will correspond to α. Use the certified value of birefringence to estimate the immersion liquids that should be used to measure α. A relatively small number of fibers may display α, as an (010) orientation may not be particularly common. The maximum refractive index perpendicular to elongation will correspond to β. The (100) orientation in asbestos fibers may be more common than the (010) orientation.

To measure principal refractive indices for tremolite and actinolite:

From slide immersion mounts, determine γ by measurement of the index closest to the fiber elongation axis for approximately 50 fibers. Always place the fiber at the extinction position to measure refractive index. There should be a range in values of about 0.005 and the maximum value should correspond to the best estimate for γ. The number of fibers that display the maximum value should be a minority of the total number of fibers observed. Record the extinction angle for each fiber; use the extinction angle from the fibers that exhibit the maximum refractive index (γ) as the best estimate of the true value of extinction angle. As for anthophyllite, α and β are measured by determining the minimum and maximum value perpendicular (or near perpendicular) to elongation, respectively, by observation of a large number of fibers. Use the certified value of birefringence to estimate the immersion liquids that should be used to

measure α. Always place the fiber at the extinction position to measure refractive index. A relatively small number of fibers may display α, as an (010) orientation may not be particularly common. The maximum refractive index perpendicular (or near perpendicular) to elongation will correspond to β, and those fibers will show parallel extinction. The (100) orientation in asbestos fibers may be more common than the (010) orientation.

NOTE: The fibrillar structure and {100} twinning common to asbestos can produce anomalous optical properties in monoclinic amphiboles, such that some portion of the tremolite and actinolite fibers in this SRM will show parallel extinction for orientations other than (100) and/or will not exhibit the true extinction angle at any orientation. The fibrillar structure can alter the refractive indices observed perpendicular (or near perpendicular) to elongation such that a single value that is intermediate between α and β is observed. These, and other anomalous optical properties, are detailed in Reference 4. The anomalous effects of the fibrillar structure are expected to be more prevalent in the very thin fibers.

REFERENCES

[1] *U.S. Environmental Protection Agency Interim Method of the Determination of Asbestos in Bulk Insulation Samples: Polarized Light Microscopy*; 40 CFR Ch. 1, Pt. 763, Subpt. F, Appendix A, 7/1/87 Ed.

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[3] Verkouteren, J.R.; Wylie, A.G.; *The Tremolite-Actinolite-Ferro-Actinolite Series: Systematic Relationships Among Cell Parameters, Composition, Optical Properties and Habit, and Evidence of Discontinuities*; American Mineralogist, Vol. 85, pp. 1239-1354 (2000).

[4] Verkouteren, J.R.; Wylie, A.G.; *Anomalous Optical Properties of Fibrous Tremolite, Actinolite, and Ferro-actinolite. American Mineralogist*; Vol. 87, pp. 1090-1095 (2002).

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[6] Verkouteren, J.R.; Steel, E.B.; Windsor, E.S.; Phelps, J.M.; *Accuracy of the Double Variation Technique of Refractive Index Measurement*; J. Res. Natl. Inst. Stand. Technol., Vol. 97, p. 693 (1992).

[7] Stuart, A.; Ord, K. J.; *Kendall's Advanced Therory of Statistics*, Vol. 2, Oxford University Press, NY, (1991).

[8] *Guide to the Expression of Uncertainty in Measurement,* ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC (1994); available at http://physics.nist.gov/Pubs/.

[9] Bowen, O.E., Jr.; *Geology and Mineral Deposits of the Barstow Quadrangle*; San Bernardino Co., CA, State of California, Division of Mines, Bulletin 165, p. 140 (1954).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.