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Characterization of Lone Pine, California, Tremolite Asbestos and Preparation of Research Material

Martin Harper[1,*], **Bradley Van Gosen**[2], **Owen S. Crankshaw**[3], **Stacy S. Doorn**[3], **Todd J. Ennis**[3], and **Sara E. Harrison**[3]

[1] Exposure Assessment Branch, Health Effects Laboratory Division, National Institute for Occupational Safety and Health, 1095 Willowdale Road, MS-3030, Morgantown, WV 26505, USA

[2] US Geological Survey, MS-973, Box 25046, Denver, CO 80225, USA

[3] RTI International (a trade name of Research Triangle Institute), 3040 Cornwallis Road, Research Triangle Park, NC 27709, USA

Abstract

Well-characterized amphibole asbestos mineral samples are required for use as analytical standards and in future research projects. Currently, the National Institute for Standards and Technology Standard Reference Material samples of asbestos are listed as 'Discontinued'. The National Institute for Occupational Safety and Health (NIOSH) has a goal under the Asbestos Roadmap of locating and characterizing research materials for future use. Where an initial characterization analysis determines that a collected material is appropriate for use as a research material in terms of composition and asbestiform habit, sufficient amounts of the material will be collected to make it publicly available. An abandoned mine near Lone Pine, California, contains a vein of tremolite asbestos, which was the probable source of a reference material that has been available for the past 17 years from the Health and Safety Laboratory (HSL) in the UK. Newly collected fibrous vein material from this mine was analyzed at Research Triangle Institute (RTI International) with some additional analysis by the US Geological Survey's Denver Microbeam Laboratory. The analysis at RTI International included: (i) polarized light microscopy (PLM) with a determination of principal optical properties; (ii) X-ray diffraction; (iii) transmission electron microscopy, including energy dispersive X-ray spectroscopy and selected-area electron diffraction; and (iv) spindle stage analysis using PLM to determine whether individual fibers and bundles of the samples were polycrystalline or single-crystal cleavage fragments. The overall findings of the study indicated that the material is tremolite asbestos with characteristics substantially similar to the earlier distributed HSL reference material. A larger quantity of material was prepared by sorting, acid-washing and mixing for sub-division into vials of ~10 g each. These

[*]Author to whom correspondence should be addressed. Tel: +1-304-285-5823; fax: +1-304-285-6041; mharper@cdc.gov.

SUPPLEMENTARY DATA Supplementary data can be found at http://annhyg.oxfordjournals.org/.

DISCLAIMER

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vials have been transferred from NIOSH to RTI International, from where they can be obtained on request.

Keywords

asbestos; reference material; tremolite

INTRODUCTION

Well-characterized amphibole asbestos mineral samples are required for use as analytical standards and in future research projects. Currently, the National Institute for Standards and Technology (NIST) lists as ‘Discontinued’ their Standard Reference Material (SRM) for two sets of samples of asbestos. NIST recommends that those interested in obtaining reference asbestos should contact Research Triangle Institute (RTI International) for possible assistance. The asbestos material which has been most widely used and which is most commonly encountered is the serpentine mineral chrysotile. Amphibole asbestos is much less common. Amosite and crocidolite were used in building products and thus may be found in building rehabilitation projects. These asbestos types were therefore designated ‘common’, whereas tremolite–actinolite and anthophyllite asbestos were designated ‘uncommon’. Recent mapping of asbestos mines and prospects in the USA by the US Geological Survey (USGS) (Van Gosen, 2005, 2006, 2007, 2008, 2010; Van Gosen and Clinkenbeard, 2011) has demonstrated a considerable past interest in small-scale mining of tremolite and anthophyllite asbestos, although the quantities produced are difficult to estimate. Mining of tremolite asbestos and anthophyllite asbestos was driven in part through most of the 20th century by the needs of the Powhatan Mining Co. (Woodlawn, Maryland) for their ‘Powminco’ product, which was used as the filter in Gooch crucibles and in other applications (EPA, 2009).

Note that terms such as fiber, fibril and asbestiform are not well-defined in the literature and there is a need for consensus agreement (NIOSH, 2011). For the purposes of this article, particles with an aspect ratio of 3:1 or greater are considered fibers, fibrils are considered to be elongate individual crystals with nanometeror micrometer-size widths, and fibers that can be separated into fibrils are considered to be asbestiform. Amphibole crystals can grow as fine fibrils, providing characteristics of flexibility and high tensile strength to the bulk product, which is then in commerce referred to as asbestos. However, there is a great deal of latitude in the fibrous habit of amphiboles, even within a single asbestos vein, so that the product may include larger individual fibrils, prismatic non-flexible crystals and cleavage fragments derived from these crystals. Asbestos is a commercial, and not a geological term, and materials referred to as asbestos may have been mined, sold, and used for a variety of purposes. Amphibole asbestos without a large proportion of fine fibrils may not have the flexibility and tensile strength desirable for weaving, but other properties, such as acid-resistance and fibrous particles are useful, for example in the manufacture of cement, battery cases, and filtration products. Among the ‘uncommon’ varieties of asbestos offered through NIST as SRMs, the tremolite asbestos SRM, even though it was considered to be ‘asbestos’ in the commercial bulk source product, has been criticized as having a less asbestiform

character than other materials more highly prized as asbestos (Brown and Gunter, 2003). In an earlier study of asbestos and cleavage fragment particle characterization (Harper *et al.*, 2008), a tremolite asbestos reference sample (Tylee *et al.*, 1996; Addison *et al*., 1997) provided by the Health and Safety Laboratory (HSL) in the UK was used in place of the NIST SRM. A portion of this HSL material was also included in the proficiency test program for the National Voluntary Laboratory Accreditation Program, and no comments were received regarding a 'poorly asbestiform' nature.

The National Institute for Occupational Safety and Health (NIOSH) has a goal under the NIOSH Current Intelligence Bulletin 'Asbestos fibers and other elongate mineral particles: state of the science and roadmap for research' (NIOSH, 2011) of locating and characterizing research materials for future use: 'To support the needed research, a national repository of samples of asbestos and related minerals will be required. … Currently, no national repository exists …. These reference samples should be well-characterized researchgrade materials that are made available to the research community so they can be used for testing and standardization.' NIOSH has been collaborating with the USGS, which as noted earlier, conducted a program of mapping asbestos mines, prospects, and occurrences in the continental USA. Locations of promising materials were investigated in concert with USGS personnel, and samples were collected for further analysis. If an initial characterization analysis determines that a collected material is appropriate for use as a research material in terms of composition and asbestiform habit, then sufficient amounts of this material will be collected to make it publicly available. The probable source of the reference material from the HSL was tracked to an abandoned mine near Lone Pine, California, where a vein of tremolite asbestos had been worked. Asbestiform material was collected from this mine for study to determine if it would meet the NIOSH goal.

RTI International was contracted to conduct a thorough characterization of an initial sample from the collected material using polarized light microscopy (PLM), X-ray diffraction (XRD), transmission electron microscopy (TEM) with energy-dispersive X-ray spectroscopy (EDS) and selected-area electron diffraction (SAED), and spindle stage analysis. The PLM analysis was conducted on five subsamples, with a determination of principal optical properties and photomicrography. The XRD analysis was also conducted on five subsamples and includes qualitative scans and determination of unit cell dimensions. The TEM/EDS/SAED analysis includes determination of chemical composition and verification of crystal structure and unit cell data. Spindle stage analysis under PLM was used to determine whether individual fibers of the sample were polycrystalline bundles or single fibrils, or cleavage fragments from larger crystals. The bulk of the collected tremolite asbestos has now been prepared and is available from RTI International for use as an analytical standard or for other research purposes, such as in studies relating physical and chemical properties to mechanisms of cellular toxicity.

The material described in this article is not a certified reference material, which is defined by the International Organization for Standardization (ISO, 2008) as 'a reference material characterized by a metrologically valid procedure for one or more specified properties, accompanied by a certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability'. NIOSH and RTI International do

not intend to imply that the material was developed and characterized by organizations accredited to ISO Guide 34 (2009): General requirements for the competence of reference material producers. However, this material may serve as a reference material, which is defined by ISO (2008) as ‘material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process (Note 2: Properties can be quantitative or qualitative, e.g. identity of substances or species)’. It is possible that this material may become generally accepted by users as a reference material once sufficient evidence has accumulated with respect to the homogeneity of properties between subsamples.

The mine

The mine from which material was removed was located by GPS at 36° 32′ 11.3″ N, 117° 54′ 58.0 W (36.53647 N, –117.91611 W, using map datum of NAD27, CONUS). The mine is shown as an adit symbol on the Dolomite, California 7½-minute topographic quadrangle map in the SE¼ of Section 14, T. 16 S., R. 37 E. The nearest major town is Lone Pine, ~13 miles to the northwest in Owens Valley, California. The horizontal shaft or adit (Fig. 1) is at an elevation of 3800 ft above sea-level. The adit is driven into dolomite, part of the Hidden Valley Dolomite, which is described as thick-bedded, fine- and evengrained, mostly light in color, with a thickness 300–1400 ft of Silurian–early Devonian age. At the location of the adit, the dolomite shows no evidence of metamorphism, in contrast to the marbles along strike in each direction, but it has been fractured, and the geologic map for the area (Ross, 1967) draws a fault through the area of the adit. Above the adits are the remains of a tower supporting the Saline Valley Aerial Tramway. The tramway was constructed between 1910 and 1913 to move evaporite minerals mined in the Saline Valley, adjacent to Death Valley, across the Inyo Mountains to Owens Valley, where it was milled and shipped by narrow gauge railway to Nevada (Mt. Whitney Packers and Owens Valley History web-site, accessed December 2013). The foundations of the terminus of the Tramway and the milling area can also be seen near the road. The Tramway was also referred to as the Salt Works Aerial Tramway; this clue suggested the mine was the location referred to as the ‘Saltworks Mine’, listed as the source of the tremolite asbestos that has been used as a reference by the UK HSL for the past 17 years. (Sample 5 in Tylee *et al.*, 1996). This clue was important because no mine by the name of Saltworks is listed in the US Gazetteer of mining and because this property is in Inyo County, CA, and not San Bernadino County, CA, as reported in the reference.

When the mine was working, it was listed as the McIlroy property, named after the first owner S.J. McIlroy, who died in the mine a few years prior to 1951 (Norman and Stewart, 1951). It was then worked by his son, who according to local residents later died, leaving no relatives in the area. At the time the mine was visited by USGS and NIOSH personnel, the claim was listed as inactive and no recent payments had been made on the claim. The asbestos deposit is recorded in 1951 as follows (Norman and Stewart, 1951): ‘The white-colored tremolite, occurring along a fissure in limestone, is exposed in a 4–20 ft width zone in the lower outcrop for a distance of 100 ft and in a 3-ft wide zone for a distance of 40 ft in the upper outcrop. Both long slip-fiber and short crossfiber materials are present.’ and ‘the deposit has been developed by numerous prospect pits, cross cuts and an adit of

indeterminate length'. It is mostly likely that hydrothermal fluids containing silica moving along the fractures formed the tremolite by reaction with the host dolomite. EDS analysis of host rock particles around the vein indicated the presence of calcite and talc, consistent with this hypothesis.

Based on the current exposure that reveals a short length of a thin, mixed long-fiber and cross-fiber vein, the deposit may have been almost fully worked out before being abandoned. The earlier description of the deposit includes a comment on the calcite content of this asbestos, which the authors felt might make the deposit uneconomic (Norman and Stewart, 1951). No definite reports of production were available, but it was noted that the asbestos that was exploited had mainly been sold to the Powhatan Mining Company. Bowles (1955) states that small quantities had been shipped to the eastern USA for filter use in 1940 and 1941.

A photograph of the tremolite asbestos vein as first encountered in the course of this study is shown in Fig. 2. Although a considerable quantity of asbestos occurs in the spoil from the mine, only material from the vein within the mine was collected to ensure the mineral's chemistry had not been modified by external weathering.

CHARACTERIZATION OF MATERIAL

An initial sample of 17.6 g from the bulk material collected was sent to RTI International under contract for the sample to be processed and characterized by PLM, XRD, TEM, and spindle stage analysis. Five subsamples of the material were initially selected for further preparation by the four methods. Under microscopy, tremolite minerals can be identified and analyzed separately from accessory minerals, but certain analyses still show the presence of these minerals.

PLM analysis

The initial sample was prepared for PLM analysis by dividing it into five representative subsamples, including the largest most coherent bundles and fluffy masses of smaller fibrils. The five subsamples were separately placed in sealed plastic containers. Random pinch mounts were made from these separate subsamples in 1.605 and 1.635 refractive index (RI) liquids. Using an Olympus BX50 polarized light microscopes (Olympus America, Center Valley, Pittsburgh, PA), three analysts measured fiber length, fiber width, aspect ratio, extinction angle, and refractive indices of 10 separate fibers from each of the five subsamples. RI measurements were made by observing the fibers in plane polarized light using the dispersion staining objective of the PLM. For RI measurements, RTI Standard Operating Procedure (SOP) EMSD 0993-001 was utilized. The 1.605 and 1.635 RI liquids were used to measure the refractive indices of the fibers in the perpendicular (α) orientation and in the parallel (γ) orientation with respect to the PLM analyzer. For each measurement, the analysts recorded the stage temperature of the microscope with a NIST-traceable thermometer and the wavelength corresponding to the dispersion staining colors observed for the fibers in the two orientations. The observed wavelengths were then converted to refractive indices using published reference tables [Su, 2003].

Five fibers were measured in each subsample by one analyst in both α and γ orientations, and these measurements were repeated by a second analyst for duplicate quality control of the aliquots. The results were compared, which showed only minor differences between the analysts (<0.03% difference in RI measurements). The RI liquids used for this study were verified at the beginning of the project using both a Fisher refractometer (Fisher Scientific, Pittsburgh, PA) and NIST RI glass beads. The Olympus microscopes are aligned daily (including Köhler illumination) upon use as well as serviced and cleaned annually.

Figure 3 shows a characteristic PLM image. A summary of the results is presented in Table 1 in what follows. Tremolite refractive indices have been graphed against iron (ferro-actinolite) content for amphiboles generally (Deer *et al.*, 1997) and for amphiboles divided according to whether they were considered to be asbestiform, 'byssolitic', or massive (Verkouteren and Wylie, 2000). In the latter reference, typical asbestiform tremolites with low ferroactinolite contents from Easton, PA and Korea were reported as having n_α of 1.603 and 1.605, respectively and n_γ of 1.631 and 1.633, respectively, which are within the range of values in Table 1. Given that the accuracy of the correlation is limited to some 3 mole percent of the end-member components and that lower values can result from the replacement of OH with F (Petersen *et al.*, 1982) that is relatively common in the more Mg-rich members, it is gratifying that the range of values for n_α in Table 1 includes 1.602, which is the 'mean value for synthetic and almost pure natural tremolite' (Deer *et al.*, 1997). Results from analyses of the HSL tremolite asbestos using the same microscope are included in the table, along with results published in the reference (Tylee *et al.*, 1996). The median aspect ratio for the particles examined in the Lone Pine material was 48:1. These measurements include polyfilamentous bundles and any individual asbestiform fibrils large enough to be observed under the optical microscope, but also any non-asbestiform crystals and cleavage fragments that may be present.

XRD analysis

Aliquots of each of the five subsamples described earlier were individually ground with a SPEX (Metuchen, NJ) 8000D mechanical ball mill using a clean tungsten carbide sample holder and ball. Each of the Lone Pine subsamples was ball milled in a clean sample holder for 5 minutes; however, after stereoscopic examination, the subsample was deemed insufficiently powdered because large bundles remained intact. An additional 8 minutes of ball mill grinding was required to prepare this material for analysis by XRD. The Lone Pine material required a total of 13 minutes of grinding for each of the five aliquots because of the tenacious nature of the long, splayed, white fibers constituting that material. Each sample was packed into a standard XRD sample holder for qualitative analysis.

The aliquots from each subsample were analyzed using XRD following RTI SOP 0801-001, 'X-Ray Diffraction (XRD) Analysis of Bulk Materials'. Crystalline phases of each aliquot were determined by full qualitative scans. These broad scans were qualitative in nature to identify all mineral phases present. All scans were performed on a Shimadzu XRD-6000 X-ray diffractometer (Shimadzu America, Columbia, MD) with a five-position sample changer. XRD scans for all samples were run from 5 to 62° 2θ at a scan rate of 0.02° with a dwell time of 2 seconds per step. The diffractometer is calibrated each day upon use by

scanning a National Bureau of Standards traceable powdered silicon standard (640a) and comparing the resulting peak data to the known reference information for that standard. If the result is within 0.050° 2θ, the unit is cleared for use on that day and considered sufficiently aligned. A duplicate analysis was conducted and showed ideal repeat results.

Diffraction patterns were interpreted using Jade version 9 software to identify peaks and generate reports. Unit cell dimensions were determined for each of the five subsamples and are presented in Table 2 in what follows. The search for matching mineral phases was performed using the PDF4+ 2010 relational database from the International Centre for Diffraction Data (ICDD). The PDF4+ database is a licensed, copyright-protected, non-transferable product available through a lease agreement with ICDD (JCPDS, 2010). The resulting peaks from each diffraction pattern were identified through search and match functions of the database.

Scans of the Lone Pine material produced typically complex diffraction patterns, which included an array of crystalline peaks. The vast majority of identified peaks matched those of tremolite (ICDD database reference card 00-013-0437). Peak intensities were consistent across the replicates and this material was therefore considered to be tremolite. A comparison of peaks with the reference is available in the Supplementary information, available at *Annals of Occupational Hygiene* online, accompanying this article. Minor peaks, identified as calcite were observed across all replicate scans of this material. The reference card number for calcite is 00-005-0586.

TEM analysis

RTI SOP 0512-001 SOP for Particulate Analysis by TEM was used. Further aliquots from the five subsamples from the initial sample of Lone Pine tremolite were prepared for TEM by hand-grinding each aliquot with a clean mortar and pestle, suspending the ground material in isopropyl alcohol, sonicating the suspension, and preparing drop mounts. The drop mounts were analyzed by TEM at magnifications of ×10 000 to ×20 000 using a Hitachi H-7000 scanning transmission electron microscope (Hitachi America, Tarrytown, NY) with a Kevex X-ray detector (Kevex, Burlingame, CA). The Hitachi H-7000 and the X-ray system are calibrated on a quarterly basis, including X-ray peak resolution, energy, and relative height, and microscope camera constant and magnification calibration. Instrument calibration records are maintained in a notebook in the RTI Electron Microscopy Laboratory. Individual fibers were micrographed, and EDS and SAED were utilized to further characterize individual fibers.

From each aliquot from the five subsamples, 10 photomicrographs were collected to show fiber size and morphology, five EDS patterns from fibers were used to show elemental composition (more were collected; all have similar relative peak heights), and five SAED patterns from fibers were collected to obtain crystal structure information. A portion of the SAED patterns were indexed to determine *d*-spacings, interplanar angles, and zone axis information. The *d*-spacing data obtained were then used to determine unit cell dimensions (Table 3).

The average unit cell dimensions determined by SAED are in general agreement with those determined by XRD analysis; however, the SAED patterns were measured by hand and are not as precise as the XRD measurements. Thirteen of the 25 zone axis diffraction patterns collected were [100] zone axis patterns indicating a possible preferred orientation of the (100) plane of the mineral, which is perpendicular to the electron beam (Fig. 4). The orientation tendency is also reflected in the higher number of data points collected in the determination of the *b* and *c* unit cell dimensions.

The β angle used for the calculations was the angle determined by XRD unit cell refinement. In some cases, unit cell dimensions were not determined for every fiber because SAED patterns had not been obtained for all orientations.

TEM observations indicate that the Lone Pine material consists primarily of individual fibers and bundles of fibers that have aspect ratios ranging from 5:1 to >50:1. EDS of the Lone Pine material indicates that it has chemistry consistent with tremolite and it is also consistent with the HSL tremolite asbestos reference. A comparison of atomic percentages derived using the same microscope conditions is presented in Table 4. The EDS patterns for the Lone Pine samples exhibit the silicon, oxygen, magnesium, calcium, and minor iron peaks (Fig. 5) characteristic of tremolite. The iron content is very low (~0.5%). Figure 6 is a typical image showing the very fine fibrils of tremolite, which make up the fiber bundles that are the ‘fibers’ observed at lower magnifications.

Spindle stage analysis

Aliquots of the five subsamples of the initial sample of Lone Pine tremolite were prepared by minimal hand grinding to obtain bundles and fibers small enough to mount on the needle of the spindle stage and individual particles were selected at random for analysis. Using guidance provided in ‘Morphological and Optical Characterization of Amphiboles from Libby, Montana, USA, by Spindle Stage Assisted Polarized Light Microscopy’ (Brown and Gunter, 2003) and an in-house procedure developed for this project, a minimum of 50 spindle stage analyses were performed on the Lone Pine material with a second analyst performing the same measurements on 10% of the particles for each of the samples. The measurements obtained by the two analysts were consistently in agreement. Twenty spindle stage measurements were initially performed on the NIST SRM tremolite; these data were compared to those obtained by Brown and Gunter (2003) on the same material for calibration purposes. The results were in agreement, that is comparable portions of particles showed inclined extinction and fragment morphology.

Length, width, thickness, and extinction angle on width and thickness were obtained for each particle measured. The particle type was also recorded based on morphology as either ‘fragment’ or ‘fiber bundle’; in this case ‘fragment’ referred to a particle that is a single crystal and ‘fiber bundle’ referred to a polyfilamentous particle. Although all observed fiber bundles were asbestiform, the single crystals may have been larger individual asbestiform fibrils, non-asbestiform prismatic single crystals, or cleavage fragments derived from larger crystals. Tables 5 and 6 summarize the morphological and optical characteristics of the Lone Pine sample from the spindle stage analysis.

The tendency for the Lone Pine sample to commonly display complete, parallel extinction on its widest dimension indicates that the amphibole crystal is flattened and is lying on its (100) plane rather than the typical (110) plane. Amphiboles with a polycrystalline, fibrous morphology have been found to display parallel extinction. The SAED results for the Lone Pine tremolite sample agree with the spindle stage results. Figure 7 shows lattice fringes consistent with the *d*-spacing of (010) on the wide dimension suggesting orientation on (100). The tendency for the Lone Pine tremolite to be flattened parallel to the (100) plane resulted in a high rate of measurements of parallel extinction and a high frequency of obtaining [100] zone axis diffraction patterns. Possible polysynthetic twinning and/or stacking faults on the (100) plane of Lone Pine tremolite may result in the mineral parting preferentially on the (100) plane due to weakening on the twin or slip planes. Parting on the (100) plane would lead to the 'flattening' of the mineral on the (100) plane (Veblen, 1980).

SUMMARY AND CONCLUSIONS OF RTI INTERNATIONAL CHARACTERIZATION

PLM: The fibers in the Lone Pine samples have optical properties, including RI and angle of extinction, that are consistent with tremolite asbestos. All subsamples had consistent results.

Spindle stage: The fibers in the Lone Pine samples have optical properties, including RI and angle of extinction, that are consistent with tremolite asbestos. All subsamples had consistent results.

XRD: Analysis of the diffraction patterns using Jade software indicates that the fibers in the Lone Pine samples have tremolite crystal structures. Average unit cell dimensions calculated by the software indicate that unit cell dimensions and β angles for each subsample of Lone Pine samples are consistent with tremolite (see Table 7 for a comparison between XRD and SAED results with the ICDD reference card). TEM: Analysis of EDS spectra and SAED patterns of fibers in each subsample concludes that the Lone Pine samples have chemistry and crystallography that is consistent with tremolite.

In summary, optical microscopy, electron diffraction, X-ray spectroscopy, and XRD are all in agreement with the conclusion that the Lone Pine material is tremolite and the aspect ratios and spindle stage results indicate that ~90% of fibers are made up of polyfilamentous fibrillar crystals (asbestiform).

PREPARATION OF BULK RESEARCH MATERIAL

The larger quantity of raw tremolite asbestos extracted from the mine was sent to RTI International in June 2012. Once the material was unbagged from transport it was weighed; a whole quantity of ~7.7 kg was determined for the tremolite asbestos and accessory minerals. The material was processed by cutting the tremolite asbestos fibers and bundles into 1–1.5 cm maximum widths and lengths via scissors and shears of various types, all by hand. Bulk elemental analysis suggested the presence of substantial calcite, in line with observations (Fig. 8). To remove this calcite, the cut material was then acid washed in a 10%

HCl/90% deionized water solution (Fig. 9). The HSL reference material is also described as having been acid-washed to remove calcite.

The material was submerged and constantly manipulated by hand for 15 minutes; each batch weighed ~100 g. While the tremolite was in the HCl solution, any granular pieces of calcite or marble were located by the reaction trail of fizzing and removed from the sieve pan sitting in the acid bath. Once each batch had been submerged for 15 minutes it was removed from the acid bath and rinsed with fresh water for at least three rinses. Some batches were rinsed more than three times, but no batch had less than three rinses to remove the HCl and cease the reaction.

Once the material dried completely inside the HEPA filtered fume hoods, it was mixed by hand and bagged in re-sealable bags for packaging into polypropylene scintillation vials with screw-top lids. The materials were weighed in each bag. The final quantity of dried, processed tremolite asbestos was determined to be 5.3kg. Each empty vial was weighed, tared, and filled with between 9.5 and 10.5g of fibrous mineral; lids were then secured tightly. Once all of the material was packaged, the total count was 487 vials ready for labeling and shipping.

The vials are labeled as follows:

CDC/NIOSH Tremolite Asbestos Reference

Lone Pine, CA

Approx. 10 grams

December 2012

Characterized and prepared by RTI

International

Each vial is then placed in a metal paint can with the same label affixed to the outside. The sealed paint cans are then placed inside individual cardboard boxes rated at 200 pounds burst strength for secure shipping. The vials and their contents are now in the custody of RTI International through a Materials Transfer Agreement with NIOSH. Researchers, analysts and others can request single vials from RTI International at no cost, although RTI International can request a nominal charge for shipping and handling. This material is intended primarily for use as an aid to analysts in the identification of asbestos, but it is recognized that it may be used for other purposes. However, users should be aware that although the bulk material was extracted from the same vein as the initial material tested for compatibility with the HSL tremolite asbestos reference material, there has been no attempt to determine consistency of properties between the bulk material and the initial material or to determine the consistency of properties between the vials of subsample from the bulk material, as would be necessary for a Certified Reference Material.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

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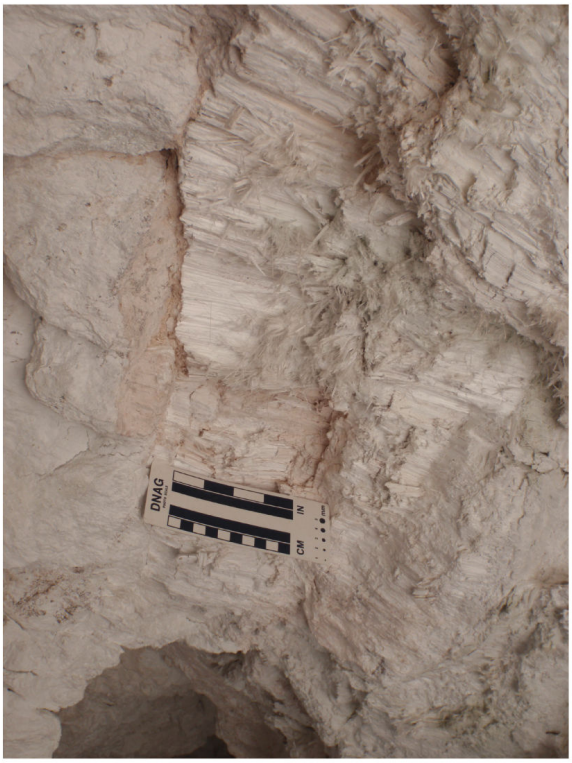
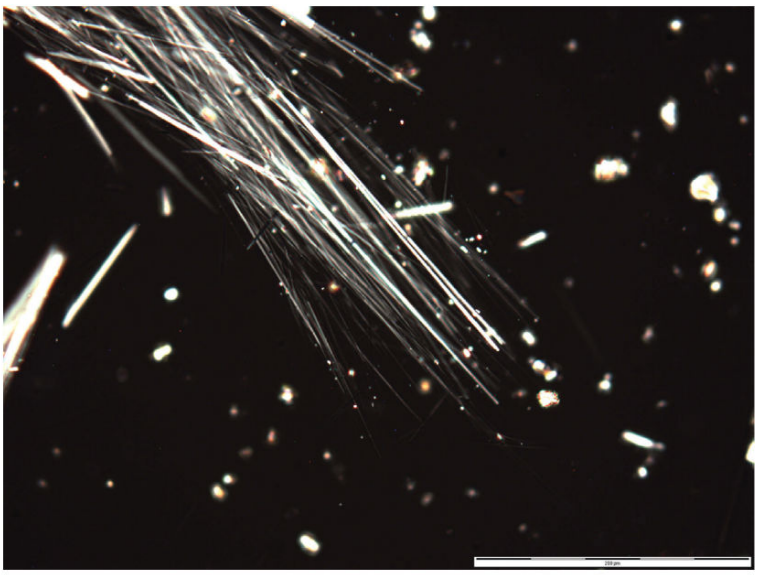
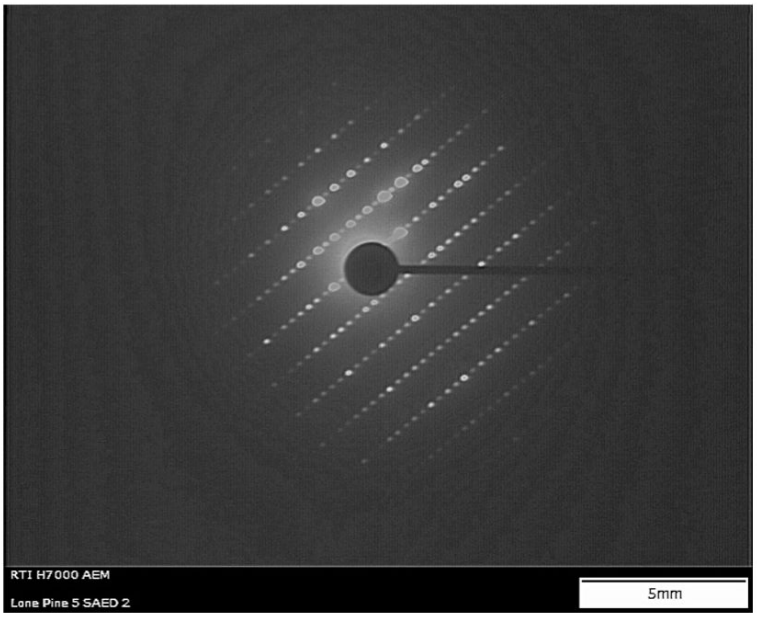
1.
McIlroy property, Owens Valley, California, mine entrance.



2.
Tremolite asbestos vein in mine roof.

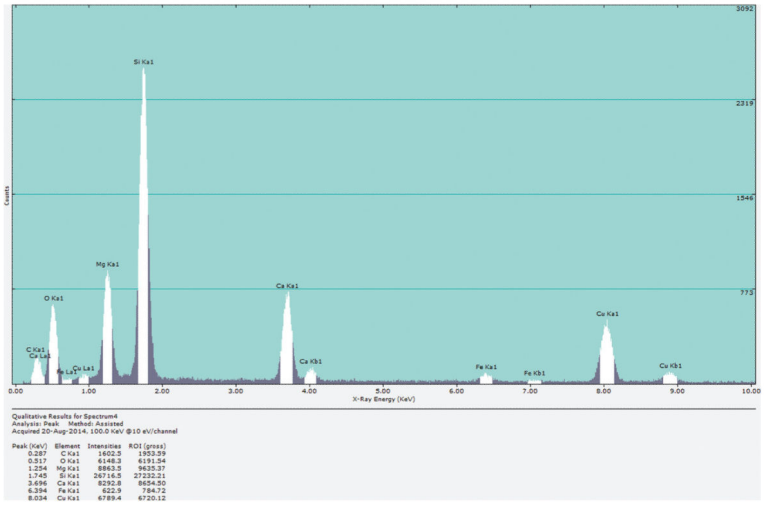


3.
Lone Pine tremolite asbestos, ×400, crossed-polars.



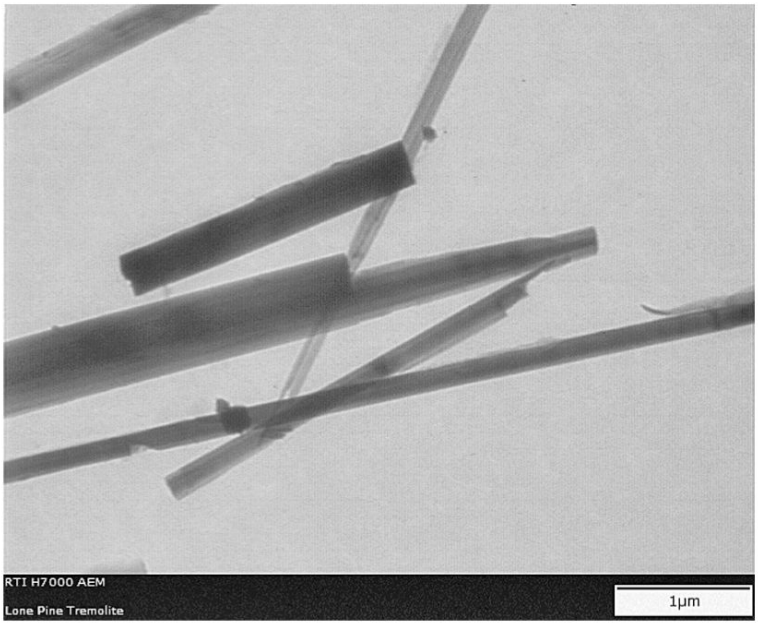
4.

Lone Pine tremolite asbestos [100] zone axis SAED pattern. Extra reflections on the (010) layer lines of the [100] zone axis pattern indicate twinning on (010). The frequency of observing [100] zone axis patterns is the result of the mineral preferentially parting on the (100) plane and may be due to polysynthetic twinning or stacking faults on the (100) plane of the mineral.

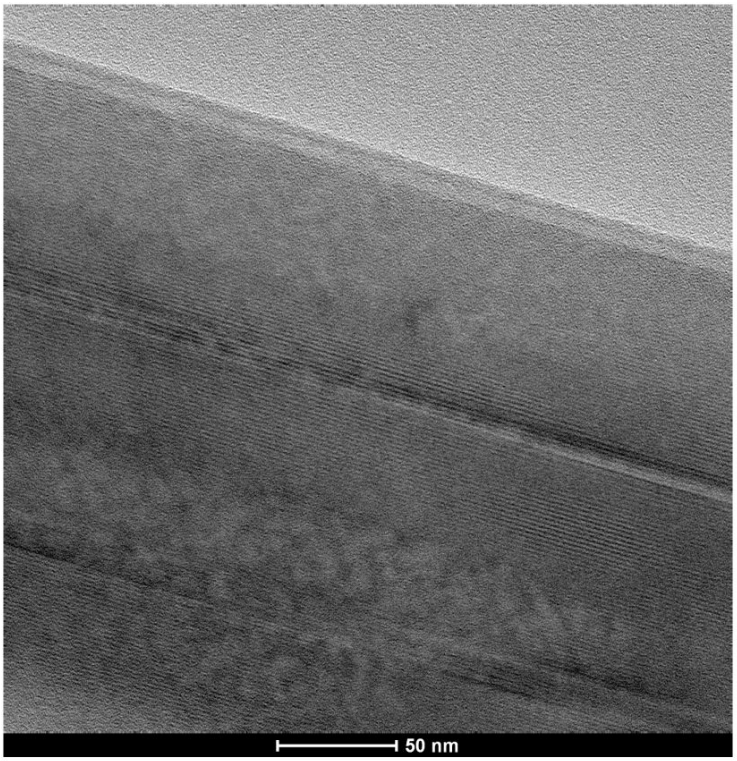


5.

Lone Pine tremolite asbestos EDS spectrum (sample is on a copper grid).

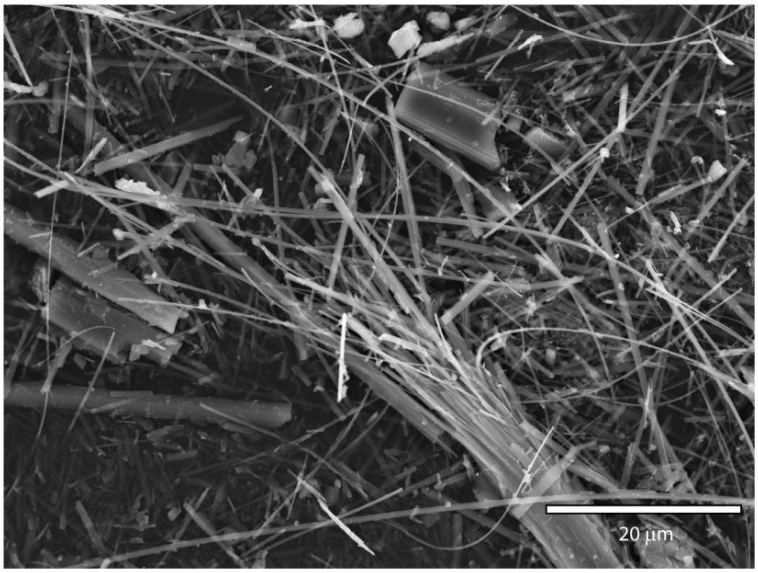


6.
Lone Pine tremolite asbestos TEM micrograph at ~×24 000.



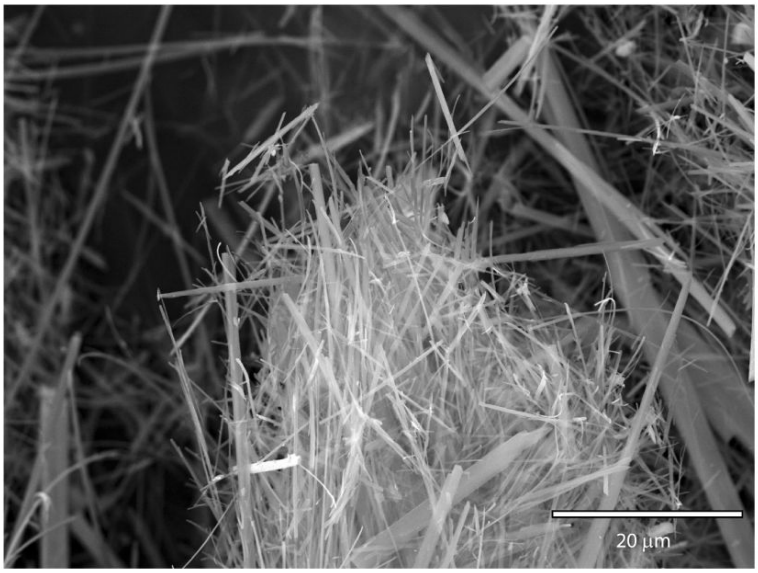
7.

Lone Pine tremolite asbestos TEM micrograph at ~×400 000. Lattice fringe spacing of ~1.8 nm is consistent with d-spacing for tremolite (010) plane.



8.

Lone Pine tremolite asbestos (×1600). Note this photograph was taken prior to acid-washing; calcite is visible in the picture.



9.
Acid-washed HSL tremolite asbestos (×1600).

Table 1

Summary of PLM data for Lone Pine tremolite

No. of analyses = 50	Mean	Maximum	Minimum	Comments
RI α	1.604	1.607	1.602	HSL reference 1.599-1.620, this study; 1.616 (Tylee *et al.*, 1996)
RI γ	1.6325	1.635	1.630	HSL reference 1.622-1.641, this study; 1.632 (Tylee *et al.*, 1996)
Extinction angle α	7	18	0	20 parallel extinction (out of 50)
Extinction angle γ	5	20	0	33 parallel extinction (out of 50)
Aspect ratio α	57:1	310:1	11:1	
Aspect ratio γ	76:1	420:1	4:1	

Table 2

Unit cell dimension determination by XRD using XRD MDI Jade 9 cell refinement software

Subsample	a (Å)	b (Å)	c (Å)	β angle
1	9.84	18.05	5.27	104.7
2	9.84	18.05	5.28	104.8
3	9.84	18.04	5.28	104.8
4	9.84	18.05	5.28	104.8
5	9.84	18.04	5.28	104.8
Average	9.84	18.05	5.28	104.8
Tremolite ICDD reference # 00-013-0437	9.84	18.02	5.27	104.9

Table 3

Unit cell dimension determination by SAED pattern interpretation[a]

Subsample	a (Å)	b (Å)	c (Å)
1	9.86 (*n* = 1)	18.09 (*n* = 3)	5.27 (*n* = 4)
2	—	18.09 (*n* = 6)	5.27 (*n* = 6)
3	9.85 (*n* = 2)	18.09 (*n* = 1)	5.27 (*n* = 1)
4	—	18.09 (*n* = 4)	5.27 (*n* = 4)
5	9.86 (*n* = 1)	18.09 (*n* = 4)	5.27 (*n* = 1)
Average	9.86 (*n* = 4)	18.09 (*n* = 18)	5.27 (*n* = 17)
Tremolite ICDD reference # 00-013-0437	9.84	18.02	5.27

[a]The unit cell dimensions were calculated using the following formula for monoclinic crystals: $1/d2 = h2/a2\sin2\beta + k2/b2 + l2/c2\sin2\beta - 2hl\cos\beta/ac\sin2\beta$; where: $d = d$ spacing calculated from distance from transmitted beam and reflections on diffraction pattern and camera constant of TEM; h, k, l = Miller indices for crystallographic planes with d-spacing, d; a, b, c = unit cell dimensions; and β = beta angle of monoclinic mineral. *Source*: Hirsch *et al.* (1977).

Table 4

Comparison of atomic percentages by EDS for Lone Pine and HSL tremolite asbestos

	Lone Pine		HSL tremolite asbestos	
Element	**Average (*n* = 5)**	**Std. dev.**	**Average (*n* = 5)**	**Std. dev.**
O	42.1	1.56	40.5	3.51
Si	34.5	1.12	34.9	1.81
Mg	16.4	1.22	17.1	1.54
Ca	6.41	0.493	6.92	0.847
Fe	0.530	0.119	0.570	0.077

Table 5

Classification of particles measured by spindle stage analysis (n = 51)

Aspect ratio	Fibers (%)	Fragments (%)	Not classified (%)	% of Total
<3	0	0	0	0
3–5	2.0	0	0	2.0
6–10	14	2.0	0	16
11–20	35	5.9	0	41
21–50	35	2.0	0	37
51–100	0	2.0	0	2.0
>100	2.0	0	0	2.0

Table 6

Morphology and extinction angle characterization by spindle stage analysis

	Lone Pine (*n* = 51)
Average length (microns)	1377
Maximum length (microns)	6500
Minimum length (microns)	100
Average width (microns)	70
Maximum width (microns)	150
Minimum width (microns)	7
Average thickness (microns)	33
Maximum thickness (microns)	100
Minimum thickness (microns)	2
Average aspect ratio	22
Maximum aspect ratio	130
Minimum aspect ratio	5
Parallel extinction on widest dimension (no. of particles)	19
No extinction on widest dimension (no. of particles)	3
Incomplete extinction on widest dimension (no. of particles)	23
Inclined extinction on widest dimension (no. of particles and angle range)	6 (10° to 20°)
Parallel extinction on thinnest dimension (no. of particles)	16
No extinction on thinnest dimension (no. of particles)	3
Incomplete extinction on thinnest dimension (no. of particles)	24
Inclined extinction on thinnest dimension (no. of particles and angle range)	8 (10° to 23°)

Table 7

Comparison of XRD and TEM SAED data with JCPDS[a]

	a (Å)		*b* (Å)		*c* (Å)		β angle (degrees)
	XRD	SAED	XRD	SAED	XRD	SAED	
Lone Pine tremolite	9.84	9.86	18.04	18.09	5.28	5.27	104.76
Tremolite ICDD reference # 00-013-0437	9.84		18.02		5.27		104.95

[a]JCPDS—International Center for Difraction Data® (http://www.icdd.com/).