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Differentiating Non-Asbestiform Amphibole and Amphibole Asbestos by Size Characteristics

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Mining or processing asbestos minerals can liberate isolated fibers or fiber bundles regulated as airborne asbestos fibers. Coarsely crystalline amphibole minerals are more common than asbestos in many geologic environments, and disturbance can result in the release of prismatic or acicular single crystals or cleavage fragments resembling asbestos fibers or fiber bundles but that are not currently regulated as asbestos. Bulk samples of six coarsely crystalline amphiboles and their five asbestos analogs were processed to maximize the number of particles meeting the criterion for counting under the current U.S. National Institute for Occupational Safety and Health Method 7400 "A" counting rules (>5 μm long with an aspect ratio ≥3:1) and also within the respirable width range, i.e. <3 μm width. The length distributions of the particles produced showed substantial overlap between cleavage fragments and asbestos fibers. Available data sets generally confirmed the relevance of the size distributions of particles generated from reference materials to airborne particles. The length criterion in the current ASTM International standard D7200-06 causes a large proportion (e.g., 40% grunerite and 39% tremolite) of the non-asbestiform particles to be considered potential asbestos. An alternative procedure may be to use a distinction based on width alone as some, but not the majority of, cleavage fragments were thinner than 1 μm (e.g., 9% of actinolite and 20% of grunerite particles), and not many amphibole asbestos particles were wider (e.g., 5% of crocidolite and 18% of amosite particles). This proposal would need further testing. This research should not be considered as addressing any controversy with regard to the toxicity of non-asbestiform amphibole particles of similar dimensions to asbestos particles.

Keywords asbestos, cleavage fragments, mineral fibers, mining

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The findings and conclusions in this report are those of the authors and do not necessarily represent the official position of the Centers for Disease Control and Prevention or the views of RTI International.

INTRODUCTION

Amphibole Asbestos and the Non-Asbestiform Varieties

The term asbestos was defined by industry to refer to the minerals being exploited for their fibrous habit, the fibers being flexible with high tensile strength (the term amosite is actually derived from the acronym of the Asbestos Mines of South Africa). Asbestos is characterized by the parallel growth of crystals called fibrils, which are extremely long relative to their width.(1) Asbestos fibrils are generally very thin and may not all be resolved even by optical magnifications of 400–450×, and so, by eye what is seen as fibers are actually bundles of fibers.

Commercially exploited asbestos normally occurs in veins, and the width of the vein determines the fiber length, generally between 0.5 and 20 mm but with fibers up to 250 mm long being common.(2) Asbestos ore is processed after mining to remove impurities and to produce a consistent product. This processing shortens the length of fibers, but the majority in the commercial product is still of the order of millimeters in length.(2) However, as a result of this or subsequent processing, fibers small enough to remain airborne are formed, and these are generally <100 μm in length and <3 μm in width.

The most common type of asbestos mined and used commercially is the serpentine mineral chrysotile, which will not be further considered in this article. Several amphibole minerals also may crystallize in an asbestiform habit, including crocidolite (which is the asbestiform version of the mineral riebeckite), amosite (the asbestiform version of the mineral series cummingtonite-grunerite), asbestiform tremolite-actinolite (tremolite asbestos, actinolite asbestos), and asbestiform anthophyllite (anthophyllite asbestos). There is very little production of amphibole asbestos today, but there are issues associated with past extraction, production, and use, and with the occurrence of these minerals in rocks and soils (naturally occurring asbestos).

The silicate minerals in the amphibole family may crystallize in an asbestiform habit, but whether they do so depends on the geologic conditions during their formation.[3] More coarsely crystalline analogs of the amphibole minerals are more common than asbestos and can be found in many geologic environments. The individual crystals can also be quite long and thin (and described as prismatic, acicular, or fibrous), and although they do not meet the commercial definition of asbestos, they can be mistaken for individual asbestos fibers.

In addition, amphibole crystals of any size and shape can break along preferred cleavage planes, leading to splinterlike cleavage fragments that can also be mistaken for asbestos. Thus, any exploitation of rock and mineral deposits that include amphiboles may result in an exposure to airborne particles with the morphology associated with asbestos fibers. Airborne fibers have typically been defined as particles elongate in a single direction. The third dimension is not easy to ascertain under the microscope, so it is possible that tablet-shaped or platelike objects on their sides might also be considered to be fibers. To be included as a fiber, the length must be at least three times the width ($\geq$ 3:1), although a ratio of 5:1 has also been used.

Fibers considered as reportable under regulations must also have a minimum length, which for most jurisdictions is greater than 5 μm (although the Environmental Protection Agency's [EPA] Asbestos Hazard Emergency Response Act regulations[4] include fibers with the appropriate aspect ratio down to 0.5 μm in length under the electron microscope). In this paper, the definition of the "A" rules in the National Institute for Occupational Health (NIOSH) Method 7400 is used.[5] These are similar, but not completely identical with the rules in the Occupational Safety and Health Administration (OSHA) Method ID-160.[6] In the production of reference materials, a preference for fibers less than 3 μm will be stated. This is a requirement for reporting under the World Health Organization and International Organization for Standardization methods.[7,8]

Size Distributions of Airborne Amphibole "Fibers"

Disturbing asbestos by extraction or processing leads to airborne fibers, which may be individual fibrils or fibrillar bundles. Due to comminuting, these fibers are not as long as those found in the orebodies. In general, airborne asbestos particle length distributions can be modeled as a log-normal distribution, with the majority below 100 μm and more than 50% below 10 μm length. Some early studies of the size distribution of airborne asbestos include those of Griffis,[9] where only 34% of airborne crocidolite particles found to be <1 μm wide (but >0.2 μm wide) were also >10 μm long; Lynch, Ayers, and Johnson,[10] where only 25–31% of fibers from air samples taken from textile, friction, pipe, and insulation product manufacture or finishing were found to be >10 μm long; Wylie, Virta, and Russek,[11] where the log mean of amosite fibers on air filters collected by OSHA was 10.3 μm (indicating approx 50% were shorter); Siegrist and Wylie,[12] where almost all crocidolite fibers were thinner than 1 μm, while less than 40% were longer than 10 μm; and Beckett and Jarvis,[13] where 26–44% of airborne amosite particles from cutting boards were >10 μm long. The crocidolite in the case of the Siegrist and Wylie study was "air jet milled," with the implication that the size distributions resulting from this type of disaggregation would be appropriate to airborne materials. The results reported for airborne amosite particles from the same work are 75.6% <1 μm wide and 55.7% >10 μm long, but the data shows a bimodal distribution. Finally, Myojo[14] also aerosolized amosite fibers. The median length was 5.4 μm, with a geometric standard deviation of 2.0, so the percentage >10 μm length can be calculated at 18.7%.

Disturbance of coarsely crystalline amphiboles can result in the release of prismatic single crystals or cleavage fragments, which as noted may resemble asbestos fibers. Although there has been less work considering the size distributions of airborne prismatic crystals and cleavage fragments, the general opinion is that they are shorter and thicker than airborne asbestos fibers. The three most studied types of non-asbestiform amphibole fibers have been at the Homestake, South Dakota, gold mine (cummingtonite); at the Gouvernor, New York, talc mines (tremolite); and at the Peter Mitchell taconite mine in the Mesabi Range of northeastern Minnesota (cummingtonite-grunerite).

In general though, it is hard to tease out exact dimensional data from the summary statistics in old publications on these studies. For example, it is common in published papers to provide median lengths and diameters and geometric standard deviations for all fibrous particles (generally defined as those with aspect ratio >3:1) observed under transmission electron microscopy (TEM) or scanning electron microscopy (SEM), even though 90% or more of these may be <5 μm long, and thus would not be counted under NIOSH 7400 "A" rules.[15] In fact, when comparing total particle counts under TEM or SEM and phase contrast microscopy (PCM) in this work, it became apparent that particles <2–3 μm long tend to be overlooked under PCM but are counted under the electron microscope.

In general, the widths of airborne cleavage fragments are greater than those of airborne asbestos fibers, although there is some overlap in the case of very fine cleavage fragments or thick fibrillar bundles of asbestos. However, the difference in lengths may be exaggerated. There appears to be substantial overlap in the distributions of cleavage fragment and asbestos fiber length. To test this hypothesis, we measured fibers from samples taken at a taconite processing operation, as detailed below.

Differentiating Amphibole Asbestos from Non-Asbestiform Amphiboles

Currently, neither non-asbestiform amphibole crystals nor cleavage fragments of non-asbestiform amphibole crystals are regulated as asbestos by Occupational Safety and Health Administration (OSHA)[16] or the Mine Safety and Health Administration (MSHA).[17] Therefore, even though there remains some controversy regarding the relative toxicity of amphibole cleavage fragments and individual crystals of non-asbestiform amphiboles with respect to asbestos,[18,19] a methodology for distinguishing these species in airborne dust samples would be useful for laboratories providing results that are to be compared against the standards promulgated by the regulatory agencies.[20]

The standard method for assessing airborne asbestos is PCM at a magnification of 400 or 450×.[5–8] TEM is less used, as it involves greater cost and cannot be carried out in the field. PCM is restricted by the ability of the optics of the microscope to resolve objects. Resolution is also a function of the difference in refractive index between the mounting medium and the object under view. A resolution of 0.2 or 0.25 μm is generally quoted for the widths of chrysotile fibers, but this may actually be as low as 0.15 μm, and for amphiboles with a large difference in refractive index compared with the mounting medium, the same calculation gives a resolution of 0.05 μm.[21] Under PCM it may thus be possible to observe individual amphibole asbestos fibrils, which may be up to 0.6 μm in width, but some of the visible fibers are actually bundles of fibrils. Fibrillar bundling is observed under polarized light microscopy (PLM) because of the different extinctions of the fibrils but is less easily visible under PCM, except where fibrils partially split off from the bundle (splayed ends).

The greater resolution of TEM causes more individual fibrils to be seen than are visible under PCM.[22] Thus PCM is considered an index of asbestos fiber exposure. Under standard PCM there may be very little difference in appearance between a cleavage fragment or prismatic crystal and an individual fibril or bundle of fibrils, and so, by default, all objects meeting certain dimensional criteria as described above may be assumed to be potentially asbestos. These dimensional criteria do not separate asbestos fibers from non-asbestiform particles well. The NIOSH method[5] does not attempt to differentiate because NIOSH does not recognize there is currently sufficient evidence for a different toxicity for non-asbestiform amphibole particles that meet the morphological criteria for a fiber.[18,19] The EPA has a similar position.[23] However, OSHA and MSHA have had to use different criteria in their consideration of the studies leading to the NIOSH opinion.

Various laboratories have devised their own criteria for determining whether a particle under PCM is asbestos or a non-asbestiform analog and this leads to variability in analysis. These criteria may also be subjective and may require skill to apply, which leads to variation even in the application of the same procedure. Thus the OSHA method for counting fibers under PCM declares that "differential counting" should be "discouraged unless absolutely necessary."[6]

The American Society for Testing and Materials (ASTM International) has published a standard, D7200-06 Standard Practice for Sampling and Counting Airborne Fibers, Including Asbestos Fibers, in Mines and Quarries, by Phase Contrast Microscopy and Transmission Electron Microscopy,[24] which includes a procedure for determining whether the particles observable under PCM and that meet the NIOSH definition of a fiber as above are likely to be asbestiform fibers or cleavage fragments (or possibly also fine, prismatic crystals, although this is not stated).

ASTM D7200 Class 1

Under the ASTM D7200-06 method, any particle meeting the definition of a fiber, that is, curved, has split ends, or has any other morphology suggesting that it is a bundle of fibrils, is automatically assigned to a class of particles (Class 1), defined as potentially asbestiform, whatever its actual dimensions. This definition may work reasonably well for chrysotile, which is often seen as wavy, polyfilamentous particles but is less likely to be applicable to amphiboles. Crocidolite tends to show more curvature and split ends than the other amphibole asbestos. Fibrillar bundling, while relatively easy to see under PLM, is less obvious under PCM.

ASTM D7200 Class 2

Particles meeting the definition of a fiber in the NIOSH method and that are also >10 μm in length *or* <1 μm in width are also assumed to be potential asbestiform fibers and are assigned to Class 2 (see Sections 4.2, 13.13.2, and A 4.3 of ASTM D7200). Thus, the potentially asbestiform population is considered the sum of Classes 1 and 2. All other particles that meet the definition of a fiber, including possible cleavage fragments, are assigned to Class 3.

Recently, ASTM International Subcommittee D22.04 (Workplace Air Quality), which has jurisdiction over this standard, balloted a revision to Class 2 to become >10 μm in length *and* <1 μm in width. Interestingly, this latter classification is compatible with an older version of the Berman and Crump[25] risk model for lung cancer and mesothelioma where the dose of particles meeting these criteria may be the greatest predictor of risk for lung cancer and mesothelioma (although not for fibrosis). The Berman and Crump model is not universally accepted, and more recently, attention has focused on even thinner fibers,[25] but the possibility of a size classification based on a proposed risk was worth evaluating. The effect of this change on non-asbestiform cleavage fragments has been examined, but it was also noted that *"the extent to which asbestiform fibers might also be designated as Class 3* [i.e., all other fibers considered non-asbestos] *under the proposed change has not been addressed."*[26] The ASTM ballot to change the definition of Class 2 returned some negative votes that were considered persuasive, and thus, the existing version of Class 2 in D7200-06 remains in effect.

The clearest distinction between cleavage fragments and asbestos fibers is that the width of cleavage fragments is a function of length, whereas the width of asbestos fibers is relatively constant whatever the length.[12] This leads to rather lower aspect ratios (length divided by width) for the cleavage fragments. Virta et al.[27] provides data for aspect ratios for airborne particles from the Homestake mine (cummingtonite), the Peter Mitchell mine (grunerite), and for a crushed stone quarry (actinolite) in Charlottesville, Virginia, and compares them with aspect ratios of airborne particles in two industrial facilities (shipyard and electric company) where asbestos was used commercially (data in Table VI).

Reference Materials to Test D7200-06

All ASTM International standards should contain a statement on uncertainty where applicable. A component of method uncertainty is inter-laboratory reproducibility. ASTM International has an Inter-Laboratory Study (ILS) group to assist committees to obtain data to support statements concerning uncertainty. Standard D7200-06 includes the statement:

> 16.2. The intra-microscopist and inter-microscopist precision of differential fiber counting has not been established, and may be larger than the values encountered in fiber identification alone. It is the intention of the ASTM committee responsible for this practice to encourage an investigation of this issue at the earliest possible date, and to ballot to withdraw this practice from publication if an acceptable precision is not established within a reasonable period of time.[24]

Thus an intra-laboratory study (ILS #0282) has been registered for the purpose of providing precision data. It was first necessary to obtain asbestiform and non-asbestiform amphibole mineral specimens. This has not been a straightforward task. Mineral identifications were sometimes in error (e.g., tremolite that was actually the mineral inesite; anthophyllite that was actually enstatite). Sometimes non-asbestiform amphiboles had poorly developed cleavage and did not produce fiberlike cleavage fragments on crushing. Sometimes asbestos specimens were not particularly asbestiform. Particles generated from the most appropriate specimens are to be used to create mixtures of asbestos fibers and cleavage fragments that can be used in the ILS.

However, one potential criticism of the procedure used to generate the particles is that the asbestos fibers and cleavage fragments may not have the same size distributions as their airborne counterparts. The objective of the study reported here is to examine this possibility. The cleavage fragments generated from the non-asbestiform amphibole specimens and the fibers generated from the asbestos reference materials were transferred to filters and examined under PCM and TEM and measured. This also allowed the binning into different size categories to see the effect of using different size criteria to differentiate them.

Additional Supporting Evidence

Amphibole asbestos has not been produced or used in the United States or Europe in recent years. Thus, it is difficult to obtain air samples of amphibole asbestos for examination. Amosite proficiency test filters from the AIHA Proficiency Analytical Testing (PAT) program were originally developed using liquid slurry deposition,[28] but this was changed to using air-generated fibers for the Asbestos Analysts Testing (AAT) program for the Asbestos Analytics Registry (AAR) because the deposition of the fibers on the filters was regarded as more representative of that found in workplace samples.[29] In fact, the fibers produced by both methods were considered to have similar dimensions, presumably representative of the fibers found in air filter samples. Since 2005, the AAT filters have also been created by wet deposition of milled asbestos. Some recently acquired PAT samples were examined under PCM and the fiber dimensions recorded for comparison with the materials created for the ILS.

Area air samples taken using the NIOSH 7400 sampling method at various locations within a taconite iron ore mill used for processing ore from the Peter Mitchell pit were also examined under PCM.[30] The ore at the taconite mill contains fibrous amphiboles in the series cummingtonite-grunerite, and ferroactinolite, but the ore is not considered to contain much, if any, asbestos,[31] so that the mill air contains mostly cleavage fragments or prismatic or acicular crystals rather than asbestos fibers. Finally, the results were compared with historical measurements of airborne asbestos particles and cleavage fragments published in the literature and summarized above.

METHODS AND MATERIALS

Actinolite (near Wrightwood, San Bernardino Co., California); cummingtonite (Homestake Mine, South Dakota); grunerite (Tras os Montes, Portugal); and anthophyllite (Kopparberg, Sweden) were obtained from mineral dealers; riebeckite was collected from St. Peters Dome, Colorado. The mineralogical identifications and purity were verified by X-ray diffraction (XRD) analysis. However, the anthophyllite also contained nickeliferous talc. In addition, a previously crushed tremolite had been acquired by Research Triangle Institute (RTI International) from a project for the National Institute of Environmental Health Sciences. The provenance of this tremolite is currently unknown, but its identity was verified by XRD.

At RTI International, small portions of the specimens were ground and processed in a series of steps. These minerals were crushed in a sequential operation using first a hydraulic press to create cm-sized particles. These large chunks were screened through 1-mm, 360-μm, and 250-μm sieves, and in each case, larger material was returned for recrushing. The screened material was ground by hand in a mortar and pestle using little or no hand pressure for 1-min intervals. After each minute interval, a glass slide mount was prepared with the mineral and a 1.550 refractive index liquid to provide maximum contrast. The slide was then inspected by polarized light microscopy to observe the range of particle sizes present.

When a sufficiently large portion of the particles appeared to meet acceptable size criteria ($>5\ \mu$m long, $<3\ \mu$m wide, aspect ratio $\geq$ 3:1), the material was suspended in water and allowed to settle. Aliquots were extracted from the water column at different times until the optimal fractionation point was found to maximize the recovery of preferred particles. The optimal sedimentation was then repeated and the suspension filtered until several milligrams of appropriately sized material was harvested. RTI International also holds the asbestos source materials that were used to prepare the Standard Reference Material (SRM) asbestos samples now obtainable from the National Institute for Standards and Technology (NIST).[32,33] Henceforth, these will be referred to as raw-NIST SRMs.

In addition, alternative actinolite asbestos and tremolite asbestos specimens were sourced from the U.K. Health and Safety Executive (HSE).[34] Samples of these materials were treated in a similar fashion as the non-asbestiform amphiboles, except that the initial crush in the hydraulic press was not necessary. PCM and TEM analyses were carried out at RTI International. Approximately 300 particles from each material were examined under PCM to determine the percentage of particles meeting NIOSH 7400 "A" rules definition of a fiber. Approximately 300 particles from each material (except the HSE actinolite asbestos and tremolite asbestos) were examined under TEM to determine the total size distribution. The size-selective binning procedure in ASTM International D7200-06 was then employed, i.e., the particles that met the NIOSH 7400 "A" rules definition of a fiber were further classified as to whether they met the definition of D7200-06 Class 2 (potential asbestiform fibers) for both the current definition and the proposed revision. TEM was used for this part of the analysis because of the greater accuracy of this technique in sizing fibers around the 1 μm width criterion. This is somewhat similar but not exactly equal to a PCM-equivalent count (PCMe) per NIOSH Method 7402.[35]

Note that particles meeting the definition of Class 1 were not first removed from this analysis. This is because observation of the morphological requirements for Class 1 under TEM would not give the same result as under PCM. However, ignoring Class 1 particles appeared to be justified after observing the particles under PCM because few particles appeared as fibrillar bundles or had splayed ends or curvature, except in the case of crocidolite.

A potential criticism of preparing reference materials in this manner is that the size distribution of the ground materials may not be relevant to that of airborne particles found in and around mines or other industrial locations. For example, individual fibrils may be released that are not resolvable by PCM. When the prepared asbestos materials for this study were examined under TEM, only crocidolite showed any substantial proportion of fibers thinner than those resolvable by PCM (58% if the observable width limit is assumed to be 0.25 μm, 29% if the width limit is assumed to be 0.15 μm). However, the concern here is with the relevance of the size distribution of particles actually meeting the PCM definition for inclusion as fibers.

Three hundred forty-four particles meeting the NIOSH "A" counting rules for a fiber from three standard asbestos sampling cassette filters from the taconite ore mill were sized at the NIOSH Health Effects Laboratory under PCM.

AIHA asbestos PAT program samples are generated by depositing a slurry containing fibers (e.g., amosite) in liquid suspension onto membrane filters. Although these were not generated from sampling airborne fibers, it appears they are considered to be representative of such. Slides produced from the proficiency test filters using the dimethylformamide/Euparal mounting technique and cover slips with relocatable grids are commercially available (REF slides; Asbestos QA Program, School of Occupational Public Health, Ryerson University, Toronto, ON, Canada). Slides from three such filters containing amosite asbestos from different rounds were examined at the NIOSH Health Effects Laboratory under PCM. On each slide, more than 100 particles meeting the NIOSH "A" counting rules for a fiber were measured to distinguish the portion with length >10 μm. Width was also measured on these particles.

However, the usual Walton-Beckett graticule for sizing fibers was replaced in this exercise by the RIB graticule (Klarmann Rulings, Litchfield, N.H.) because the Walton-Beckett graticule has no single micrometer gradations, whereas

TABLE I. Results of Different Size-Binning of Crushed Non-Asbestiform Amphibole Particles

		Percentage of PCMe Fibers Under TEM[B] Meeting Different Criteria		
Non-Asbestiform Amphibole Type	**Percentage of Fibers[A] (%)**	**Current ASTM D7200 Class 2 – Percentage >10 μm long *or* <1 μm wide (%)**	**Proposed ASTM D7200 Class 2 – Percentage >10 μm long *and* <1 μm wide (%)**	**Width Only Classification – Percentage <1 μm wide (%)**
Riebeckite	62.0	37.7	0.0	16.4
Cummingtonite	71.3	49.1	0.0	33.3
Grunerite	64.7	40.0	0.0	20.0
Actinolite	58.3	29.4	2.9	8.8
Tremolite	62.3	39.0	4.9	24.4
Anthophyllite[C]	91.3	74.4	6.8	50.4

[A] Percentage meeting the NIOSH 7400 method "A" counting rules definition of a fiber out of 300 particles observed under PCM.
[B] For greater accuracy, measurements were made under TEM. 300 total particles were measured and the number of PCM-equivalent (PCMe) fibers counted. Percentage is the number meeting the stated classification out of the 300 PCMe fibers measured under TEM.
[C] Anthophyllite contains talc, probably fibrous.

TABLE II. Results of Different Size-Binning of Crushed Amphibole Asbestos Particles

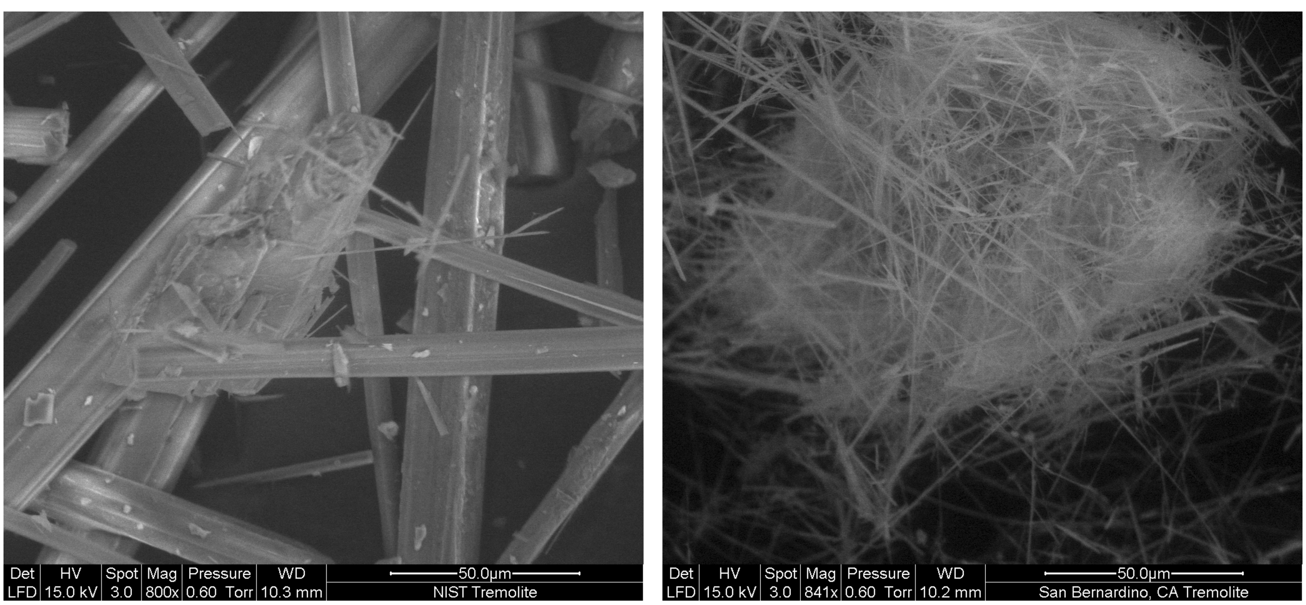
		Percentage PCMe Fibers Under TEM[B] Meeting Different Criteria		
Asbestos Type	Percentage Fibers[A] (%)	Current ASTM D7200 Class 2 – Percentage >10 μm long *or* <1 μm wide (%)	Proposed ASTM D7200 Class 2 – Percentage >10 μm long *and* <1 μm wide (%)	Width Only Classification – Percentage <1 μm wide (%)
Crocidolite	77.3	98.7	31.0	94.7
Amosite	59.0	89.3	15.1	82.2
Actinolite asbestos	64.0	73.3	10.7	59.0
HSE actinolite asbestos	80.3			
Tremolite asbestos	65.3	50.7	2.7	27.2
HSE tremolite asbestos	86.0			
Anthophyllite asbestos	74.3	58.9	3.7	37.5

[A] Percentage meeting the NIOSH 7400 method "A" counting rules definition of a fiber out of 300 particles observed under PCM.
[B] For greater accuracy, measurements were made under TEM. Three hundred total particles were measured and the number of PCM-equivalent (PCMe) fibers counted. Percentage is the number meeting the stated classification out of the 300 PCMe fibers measured under TEM. (Procedure not carried out for HSE material.)

the RIB graticule does. Even with the RIB graticule, it was difficult to determine whether a fiber of 1 μm nominal thickness was actually greater or less than 1 μm, so that a criterion of ≤ 1 μm was used for inclusion.

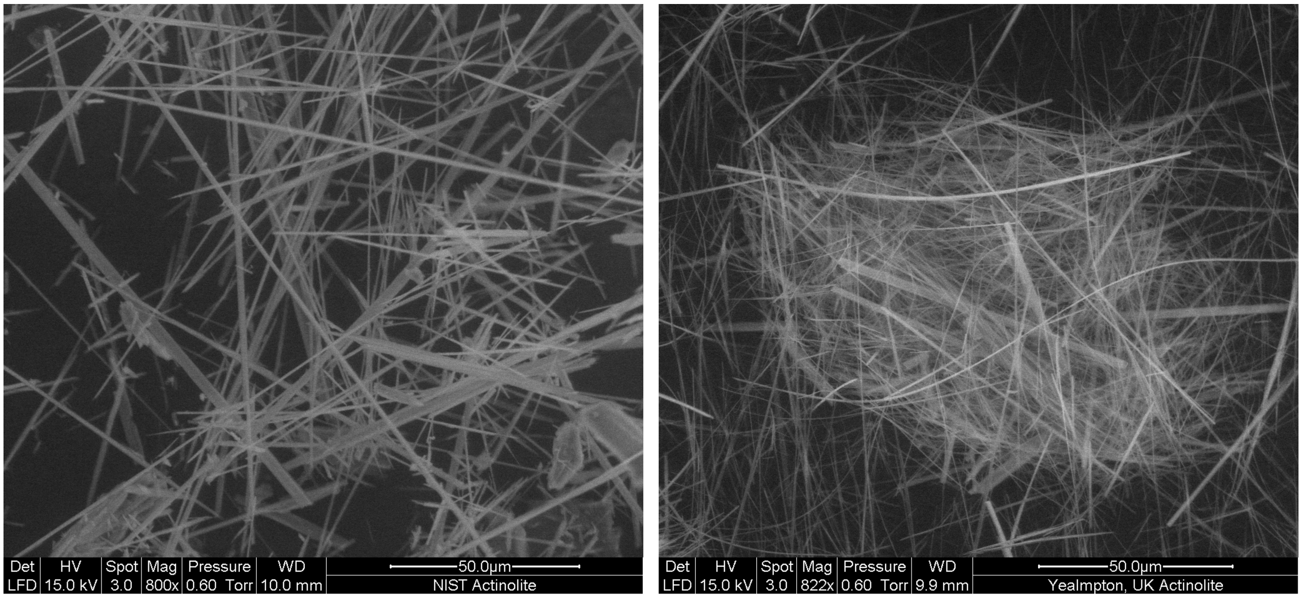
RESULTS

Table I shows results from size determination of particles produced from crushing non-asbestiform amphiboles. Exempting anthophyllite, which also may contain fibrous talc, Table I shows that as many as almost half the "fiber" particles would meet the Class 2 definition of "potential asbestos" under the current Standard (Column I), but that this would reduce to almost nothing under the revised Class 2 definition (Column II). The results of examining particles from milling of the five raw-NIST SRM amphibole asbestos materials are given in Table II. For crocidolite and amosite, the most common commercial amphibole asbestos materials, where most of the material should be expected to be asbestiform, almost all of the particles would be classified as potential asbestos under



(A) (B)

FIGURE 1. Comparison of (A) raw-NIST SRM tremolite, and (B) HSE tremolite (both 800×). (Photo courtesy RTI International)



(A) (B)

FIGURE 2. Comparison of (A) raw-NIST SRM actinolite, and (B) HSE actinolite (both 800×). (Photo courtesy RTI International)

the current standard, but many fewer would be classified so under the proposed revision.

The majority of particles from the NIST actinolite asbestos and tremolite asbestos SRMs would be considered as unlikely to be asbestos under the revised ASTM D7200 standard. This was the reason behind considering the HSE reference materials. The difference between the HSE reference materials and the raw-NIST SRMs can be seen in the photomicrographs (Figures 1 and 2) for materials that had received the same degree of preparation.

It is clear that the fibers of the HSE materials are thinner and show more curvature (i.e., they are more asbestiform) than their NIST SRM equivalents. Photographic examination alone does not invalidate the labeling of the NIST SRMs as asbestos

TABLE III. Comparison of NIST and HSE Actinolite Asbestos and Tremolite Asbestos Aspect Ratios with Other Published Aspect Ratios

	Percentage of Fibers with Aspect Ratio >10:1(%)	Percentage of Fibers with Aspect Ratio >20:1(%)
Homestake Mine, S.D.[A]	4	0
Peter Mitchell mine, Minn.[B]	7	1
Charlottesville quarry, Va.[C]	6	1
NIST tremolite asbestos	13	2
NIST actinolite asbestos	28	8
Shipyard asbestos (amosite)	72	41
Electric co. asbestos (amosite)	83	59
HSE tremolite asbestos	83	51
HSE actinolite asbestos	71	38

Note: Aspect ratios from Virta et al.[27] calculated for all particles with aspect ratios ≥ 2:1 (mining) or ≥ 3:1 (industrial) using SEM, while those from the current work are calculated only for particles >5 μm long using PCM. For cleavage fragments where there is a correlation between length and aspect ratio, this will not bias the aspect ratio, but for asbestos fibers it will have the effect of slightly increasing the average aspect ratio in this work.
[A] Cummingtonite cleavage fragments.
[B] Grunerite cleavage fragments.
[C] Actinolite cleavage fragments.

TABLE IV. Aspect Ratios of Crushed Non-Asbestiform Amphibole Particles

Non-Asbestiform Amphibole Type	Percentage Fibers[A]	Percentage Fibers Under TEM[B] with: Aspect Ratio >5:1	Aspect Ratio >10:1
Riebeckite	62.0	31.1	3.3
Cummingtonite	71.3	59.6	8.6
Grunerite	64.7	48.6	5.3
Actinolite	58.3	41.2	2.9
Tremolite	62.3	53.7	9.8
Anthophyllite[C]	91.3	83.8	35.0

[A] Percentage meeting the NIOSH 7400 method "A" counting rules definition of a fiber out of 300 particles observed under PCM. From Table I.
[B] For greater accuracy, measurements were made under TEM. 300 total particles were measured and the number of PCM-equivalent (PCMe) fibers counted. Percentage is the number meeting the stated classification out of the 300 PCMe fibers measured under TEM.
[C] Anthophyllite contains talc, probably fibrous.

TABLE V. Aspect Ratios of Crushed Amphibole Asbestos Particles

Asbestos Type	Percentage Fibers[A] (%)	Percentage Fibers Under TEM[B] with Aspect ratio >5:1 (%)	Percentage Fibers Under TEM[B] with Aspect ratio >10:1 (%)
Crocidolite	77.3	99.7	94.3
Amosite	59.0	98.0	66.8
Actinolite asbestos	64.0	84.0	36.7
Tremolite asbestos	65.3	58.1	14.8
Anthophyllite asbestos	74.3	67.2	17.4

[A]Percentage meeting the NIOSH 7400 method "A" counting rules definition of a fiber out of 300 particles observed under PCM. From Table II.
[B]For greater accuracy, measurements were made under TEM. Percentage is the number meeting the stated classification out of the 300 PCMe fibers measured under TEM.

but rather highlights the fact that the term asbestos can include a range of morphologies. On the other hand, the actual asbestos content of the NIST SRM tremolite asbestos has been criticized as being low, with as little as 19% of particles being considered to be asbestos.[36] It should be noted that NIST SRM 1867a is not currently available from NIST.

In the final column of Tables I and II are the percentages of fibers less than 1 μm in width. Nearly all fibers of crocidolite and amosite, the major commercial amphibole asbestos types, meet this criterion. On the other hand, the number of cleavage fragments meeting a $<1 \mu$m criterion is between 9 and 33% (with the exception of anthophyllite), as shown in Table I. Thus, a 1 μm width criterion was considered a potentially reasonable compromise for separation purposes.

However, 41% of the actinolite asbestos fibers and 73% of the tremolite asbestos fibers are >1 μm wide. The tremolite asbestos, at least, is thought to be heavily loaded with cleavage fragments or large prismatic crystals, and this may also be true of the actinolite asbestos. At TEM magnification, no additional fibers are seen in either material. TEM size analyses have not yet been performed on the HSE materials; however, PCM analyses confirm a larger number of particles meeting the NIOSH 7400 "A" rules than is the case with the raw-NIST SRM materials. By PCM examination, the HSE actinolite asbestos had 66% of the NIOSH "A" rules fibers <1 μm wide and the HSE tremolite asbestos had 77.5% of the NIOSH "A" rules fibers <1 μm wide. The corresponding PCM measurements for the NIST actinolite asbestos and tremolite asbestos are 2.6% and 1.0%. (Note these figures are substantially different from those in Table II, which were obtained from TEM measurements. Under PCM, many particles close to 1 μm width are attributed a width of 1.0 μm, while under TEM they might be rendered as 0.9 or 0.8 μm.)

Aspect ratio (length:width) is another measure allowing discrimination of asbestos fibers and amphibole cleavage fragments. The results of attempting discrimination based on two example divisions, 5:1 and 10:1, are given in Tables III and IV. Whereas a >10:1 ratio excludes almost all non-asbestiform amphibole fragments, except in the case of anthophyllite, and includes nearly all crocidolite and much amosite, it also excludes a lot of the raw-NIST SRM actinolite asbestos, tremolite asbestos, and anthophyllite asbestos fibers. However, this also may be a function of limited asbestiform particles in the tremolite asbestos and actinolite asbestos and the presence of fibrous talc in the anthophyllite asbestos.

In Table V, the PCM measurements of aspect ratio for the crushed raw-NIST SRM asbestos are compared with similar measurements of the HSE materials and TEM data from actual air samples (but see note in table concerning direct comparison of PCM and TEM data). A >5:1 ratio includes most asbestos but also includes a lot of the non-asbestiform amphibole fibers. Combining the 5:1 aspect ratio with the 1.0 μm width criterion (not shown) gives no better differentiation than using the width criterion alone.

For the taconite ore-processing air samples, out of 344 fibers meeting the NIOSH fiber definition under PCM, 48.8% met the criterion of >10 μm long *or* <1 μm wide, while only 3.8% met the criterion of >10 μm long AND <1 μm wide. It is interesting to note that the count median length (L) of 7.5 μm and width (W) of 1.3 μm compared extremely well with L = 7.5 μm and W = 1.5 μm of the crushed bulk cummingtonite (n = 214) and L = 7.0 μm and W = 1.5 μm for the crushed bulk grunerite (n = 194), also under PCM. The percentage of fibers meeting a criterion of <1 μm, 24.4%, is also similar to the percentage found in the crushed bulk cummingtonite (33.3%) and grunerite (20.0%). However, the proportion of fibers with aspect ratios >5:1 is rather large, so that aspect ratio may not help in the differentiation. For the three amosite PAT sample

TABLE VI. Size Data of Fibers from Amosite Proficiency Test Samples

Slide No.	N	Percentage ≤ 1 μm Wide (%)	Percentage >10 μm Long (%)	Percentage ≤ 1 μm Wide *or* >10 μm Long (%)	Percentage ≤ 1 μm Wide *and* >10 μm long (%)	Aspect Ratio >10:1 (%)
1662-4	133	73.7	27.1	82.7	18.8	54.1
1683-1	111	95.5	27.9	99.1	24.3	53.2
1522-1	126	96.8	34.1	99.2	31.7	86.5

filters, fibers exceeding 10 μm in length constituted only 27–34% of fibers meeting the NIOSH definition, as shown in Table VI, and this was confirmed by independent examination (24–35% by Thomas Pang, Ryerson University, Canada). This is slightly greater than but reasonably similar to the percentage found in the crush of our NIST SRM precursor amosite sample (21.5%). Widths were generally ≤ 1 μm; for two of the slides >95% of fibers met this criterion.

Once again, it can be shown that while the current ASTM D7200 Class 2 criterion includes most asbestos fibers, almost the same result can be achieved by using a simple width criterion, and that the proposed revision of D7200 Class 2 excludes most of the amosite fibers as being potentially asbestos (with the number of fibers showing features of Class 1, i.e., bundling, curvature or split ends, in the PAT samples being negligible). Whereas one of the slides had a high proportion of fibers with a high aspect ratio (>10:1), the other two did not, again showing a likely poor predictive value.

CONCLUSIONS

Particles from processed amphibole asbestos and non-asbestiform amphiboles were prepared to produce samples that are to be used to evaluate the precision of determining size-selection criteria for differentiation in ASTM International Standard D7200-06 in an Inter-Laboratory Study (ILS#0282). Processing of the bulk materials used techniques similar to those used in ore mining and production (crushing, grinding, and milling), and fibers of selected dimensions were separated and concentrated by flotation. On microscopic examination, the degree of overlap in length distributions for the asbestos fibers and for the non-asbestiform cleavage fragments was unexpected. However, the size distributions appear to be relevant to airborne particles as well as to particles used as surrogates for airborne materials. Based on the size distributions observed in this study, an alternative size-selection criterion can be suggested.

For the raw-NIST SRM crocidolite and amosite asbestos and for the HSE actinolite asbestos and tremolite asbestos, it has been shown that most fibers are likely to be <1 μm wide, but also <10 μm long, and this is also true of actual airborne fibers. For most of the non-asbestiform amphiboles, the processing gave rise to particles where a proportion were >10 μm long but where, typically, only around 25% of particles meeting the NIOSH definition of a fiber were <1μm wide. The size-distributions of these laboratory-processed materials can be compared with airborne size distributions of mined materials. For example, under PCM, fibers in taconite ore-processing air samples were very comparable in length and width to the materials prepared for the ILS.

The current Class 1 of ASTM D7200, which is based on the appearance of the fiber, is rather subjective and may not be very useful for distinguishing amphibole asbestos. This hypothesis will be tested as part of the ILS. In a previous publication we had reported the percentage of fibers from the taconite ore-processing air samples meeting the Class 1 definition as 28%.[21] On further examination of these same samples after additional training with a more experienced microscopist, the percentage was lowered to 12.8%. This is an indication of the potential problems involved in using recognition of features rather than objectively verifiable measurements.

The current Class 2 criterion of ASTM D7200-06 (>10 μm long *or* <1 μm wide) tends to designate as asbestos much of the asbestos in air samples by virtue of width, rather than length. It does not completely differentiate non-asbestiform amphibole particles meeting the NIOSH definition of a fiber from asbestos fibers. The proposed revision to Class 2 is likely to exclude many asbestos fibers from being counted as asbestos on the basis of length. A width criterion of <1 μm may adequately include asbestos (although, for example, missing 5% of crocidolite and 18% of amosite) while minimizing the amount of cleavage fragments also included. It would also exclude any prismatic or acicular crystals >1 μm in width. Thus, it is relatively conservative, and since it depends on measurement alone, it is verifiable.

Although bias in microscopic measurements has been observed (e.g., inclusion of particles slightly less than the 5 μm length criteria for a fiber is rather common), it is relatively easy to discover and remedy.[37,38] The Walton-Beckett graticule used as an aid in fiber measurement is of little help, since it does not include 1 μm divisions. An alternative, the RIB graticule, does include 1 μm divisions and was used in this study. The use of the RIB graticule will also be examined in the ASTM ILS. In practice, a number of PAT amosite asbestos fibers were measured using this graticule as 1 μm. Thus, a width criterion of ≤ 1 μm, rather than <1 μm would be an improvement to include these particles.

This width criterion is based on retrospective analysis and should be confirmed through application to additional datasets. Hopefully, this will be accomplished in part through the ASTM ILS, where cleavage fragments and asbestos fibers are being mixed on slides to approximate different percentages and distributed to participants, who will analyze the slides by PCM and make measurements of the fibers to determine measurement precision with both the Walton-Beckett and RIB graticules, since accurate measurement is the basis of any Class 2 definition. (The ILS will also determine the variance in the ability to recognize Class 1 fibers.) Interestingly, a width criterion of 1 μm for fibrous silicate minerals is somewhat equivalent to size-selection according to a respirable convention. This leads to the possibility that a good separation of cleavage fragments and prismatic crystals from asbestiform fibers might be accomplished at the sampling stage by using a respirable size-selective sampler such as a cyclone. However, it should be clearly recognized that a 1 μm width criteria is not being proposed here as a dividing line between safe and unsafe.

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