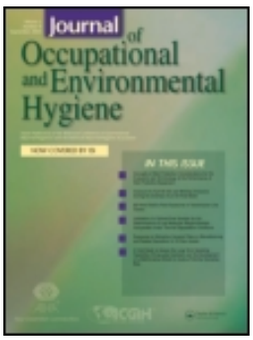

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Kevin Ashley , Martin Harper , Eun Gyung Lee , Bruce Harvey & Michael Beard

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Analytical Performance Criteria
The Effect of a Proposed Change to Fiber-Counting Rules in ASTM International Standard D7200-06

Column Editor
Kevin Ashley

Reported by
Martin Harper,[1] Eun Gyung Lee,[1] Bruce Harvey,[2] and Michael Beard[2]

[1]Exposure Assessment Branch, Health Effects Laboratory Division, National Institute for Occupational Safety and Health, Morgantown, West Virginia
[2]Microanalytical Sciences Department, RTI International, Research Triangle Park, North Carolina

INTRODUCTION

Asbestos is a form of certain serpentine or amphibole minerals that has crystallized in a particular habit known as asbestiform.[1] Amphibole minerals are often encountered in metamorphic geological environments, but the majority will not have crystallized in the asbestiform habit.[1] Thus, ore deposits in these environments may include coarsely crystalline amphiboles that can produce cleavage fragments when the rocks are crushed. These fragments may meet morphological criteria that cause them to be designated as "fibers" but without their being asbestiform *per se*.

In an attempt to distinguish cleavage fragments from asbestiform fibers, the American Society for Testing and Materials (ASTM) International Standard D7200-06[2] includes a procedure for determining whether the particles observable under the phase contrast microscope that meet a morphological definition of a fiber are likely to be asbestiform fibers or cleavage fragments. (The morphological definition of a fiber is that described in the National Institute for Occupational Safety and Health [NIOSH] 7400[3] "A" counting rules, i.e., >5 μm in length and with aspect ratio [length:width] $\geq$3:1). Under ASTM D7200, any particle meeting the definition of a fiber that is curved or has any morphology that suggests 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.

In addition, particles meeting the definition of a fiber and >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 A4.3 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. An alternative definition for Class 2 of length >10 μm **and** width <1 μm is being proposed in an upcoming ASTM member ballot. It is important to assess the impact of both the current and proposed criteria.

Taconite is an iron ore that can contain amphiboles in the tremolite-actinolite and cummingtonite-grunerite amphibole series.[1] We have recently analyzed 77 air samples from a taconite mine ore-processing mill. The mine samples have an average of 83.8 fibers (NIOSH 7400 "A" rules) per 85.6 fields, or approximately one fiber per field area. Twenty-eight percent of these fibers met the ASTM International D7200-06 definition of Class 1, and a further 18.6% met the dimensional characteristics of the current criteria for Class 2 (the remainder being Class 3). However, under the microscope, many of these Class 2 fibers have other morphological features (e.g., aspect ratios at the low end of the range, and nonparallel sides) that indicate they might be mineral cleavage fragments (see Figure 1). Therefore, we hypothesized that the rule used in the current ASTM Standard to classify fibers appears to underestimate proportion of fibers (defined by NIOSH "A" rules) that are actually cleavage fragments,

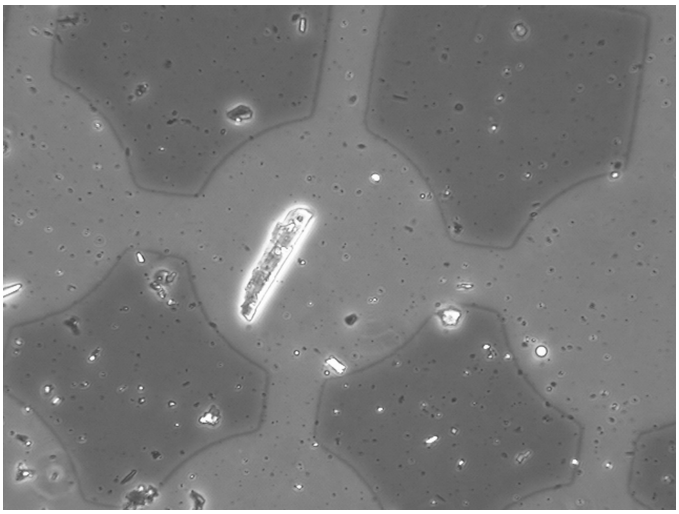

FIGURE 1. Typical taconite mine ore mill air sample under phase contrast microscopy (450×) showing a particle very likely to be a mineral cleavage fragment. The area bounded by the darker arcs is 100 μm across.

with many obvious cleavage fragments being placed in Class 2 (potentially asbestiform).

Many experienced microscopists believe they can distinguish cleavage fragments from asbestiform fibers by visual clues, although there is no established reference procedure; the ASTM criteria are the first attempt to establish criteria acceptable by consensus. Given that there is no prior procedure against which the ASTM criteria can be tested, we decided to test the criteria by examining the results of their application to crushed fragments from samples of massive or coarsely crystalline amphibole minerals that do not have the finely fibrous nature associated with the asbestiform habit. One sample of actinolite was obtained from a mineral dealer who stated it was from near Wrightwood, San Bernardino County, Calif. (Figure 2); the mineralogical identification was verified by X-ray diffraction (XRD) analysis.

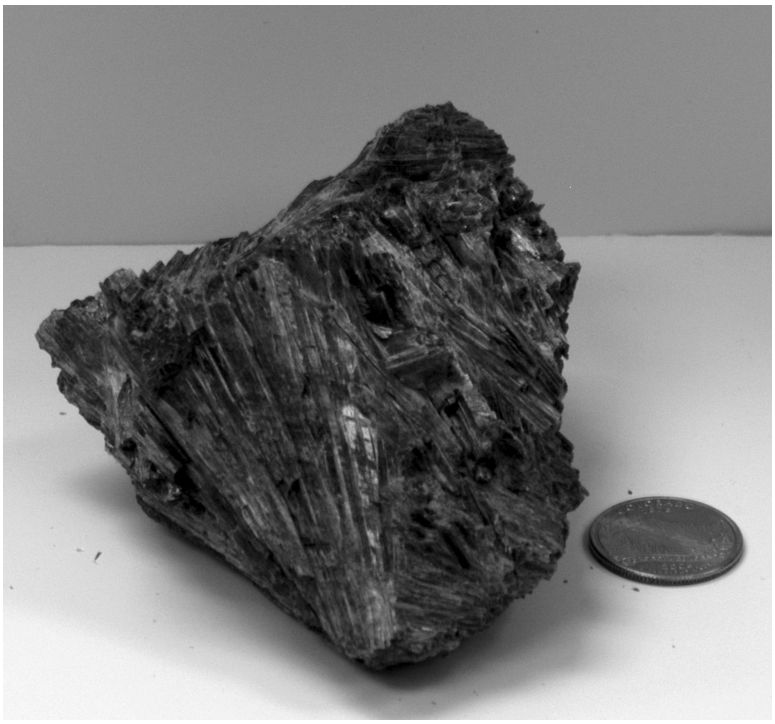

FIGURE 2. Coarsely crystalline (non-asbestiform) actinolite (scale indicated by a 25¢ piece).

The actinolite was crushed at the RTI International laboratory in a sequential operation using a hydraulic press and a mortar and pestle, in an attempt to maximize the percentage of particles that would meet the definition of a fiber under phase contrast microscopic analysis. In addition, RTI has a stockpile of a previously crushed tremolite acquired from the National Institute of Environmental Health Sciences (NIEHS), whose provenance is currently unknown.

The identity as tremolite was also verified by XRD. A weighed portion of each material was suspended in water, stirred, and aliquots were taken at various time intervals to determine the optimum loading and particle sizing. Approximately 300 particles from each material were examined according to the procedures in ASTM International D7200-06. That is to say, 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). The results are shown in Table I. Whereas the presence of a small amount of asbestiform fiber contamination in the mineral specimens used cannot be ruled out, it is unlikely to be more than a few percent at most. As expected, almost none of the particles had the characteristics of Class 1; therefore, it is likely that the large proportions of fibers considered as asbestiform according to the current definition of Class 2 are actually mineral cleavage fragments (along with the remaining fibers that fall into Class 3).

Also shown in Table I is the effect of using the alternative definition for Class 2 of length >10 μm and width <1 μm. This alternative definition removes the majority of fibers from Class 2 and moves them into Class 3 (i.e., nonasbestiform). When applied to the mine samples, a similar result is obtained. Whereas the proportion of Class 1 fibers is unchanged, the proportion of Class 2 fibers using the proposed alternative definition for Class 2 falls to about 0.8%, i.e., nearly all of the Class 2 fibers are reclassified as Class 3. We have shown that the current definition of Class 2 applied to the nonasbestiform actinolite and tremolite in Table I causes many cleavage fragments to be considered asbestiform fibers. We have further shown that this is likely also to be true for the mixed actinolite-tremolite and cummingtonite-grunerite (nonasbestiform versions of amosite) of the taconite mine samples. We have also shown that this is not the case if the proposed new ASTM International definition of Class 2 is used. However, the question remains as to whether this proposed alternative definition will also cause some actual asbestiform fibers to be classified as nonasbestiform. For example, this may occur when thick bundles of asbestiform fibrils do not show the asbestiform characteristics (curvature or splayed ends) necessary to be included Class 1.

TABLE I. Percentage of "Fibers" from Crushed Nonasbestiform Amphibole Minerals and the Percentage of Those Particles Meeting the Class 2 Definition of ASTM International Standard D7200-06 Before and After a Proposed Change

Mineral	Particles Meeting 7400 Fiber Definition (%)	7400 Fibers in D7200-06 Class 2 (%)	7400 Fibers in Proposed Class 2 (%)
Actinolite	58.3	25.1	0.6
Tremolite	62.7	46.8	1.6

Note: "Fibers" were particles meeting the NIOSH Method 7400 "A" counting rules for a fiber out of the 300 particles examined for each mineral species.

Many of the cleavage fragments with length >10 μm have quite low aspect ratios and widths >3 μm. For a fiber of the density of a silicate mineral up to at least 100 μm in length, applying a maximum width of 3 μm should have the same effect as applying the International Organization for Standardization (ISO) curve for thoracic respirability.[3] It is interesting to examine the effect of using a 3 μm width cut-off as is done in the World Health Organization (WHO) rules.[4] (Note that this is also the case in the NIOSH 7400 "B" rules, but there is a difference in that these rules also include a >5:1 aspect ratio.)

For the actinolite sample, the number of particles classified as fibers is reduced from 58% to 48% of the total, whereas the percentage of those in D7200-06 Class 2 drops even further, from 25% to 14%, or just 7% of the total particles. For the tremolite sample, the number of particles classified as fibers is reduced from 63% to 43% of the total, whereas the percentage of those in Class 2 again drops even further, from 47% to 29%, or just over 12% of the total particles. Thus, using the 3 μm upper width limit (or a sampler with a size-selective inlet that performs in an equivalent fashion)[3] may be an alternative to the proposed change in the ASTM International D7200-06 Standard. However, applying this criterion to the taconite mine samples did not produce as much of a reduction in Class 2 particles as with the crushed amphiboles, reducing the average from 18.6% of 83.8 fibers to 15.2% of 80.4 fibers.

In conclusion, the current classification system for fibers in the ASTM International Standard D7200-06 was tested using materials that were expected to contain a large proportion of particles meeting the current NIOSH definition of a fiber but where those particles would be predominantly cleavage fragments of amphibole minerals rather than asbestiform fibers: crushed samples of coarsely crystalline amphibole minerals and air samples from a taconite ore mill. The current classification rules designate many cleavage fragments as Class 2 (i.e., potentially asbestiform), whereas a proposed change to the definition of Class 2 would place these particles almost exclusively in Class 3 (i.e., nonasbestiform). However, the extent to which asbestiform fibers might also be designated as Class 3 under the proposed change has not been addressed.

Note: Counting rules are often given in terms of less than or greater than a measured value. The precision of measurements under the microscope requires assumptions about rounding errors. The crushed amphibole measurements were made by eye and rounded to the nearest micrometer, except that widths obviously less than 1 μm were assigned a value of 0.5 μm. The analysis provided here assumes all measurements to be greater than the nominal values. For the mine sample data, computer-aided measurements on photomicrographs

were made to one-tenth of a micrometer, so that x.0 micrometers was assumed to be not greater than x.

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