

Slide 1.

Amphibole: Is it asbestos? Is it hazardous? How do we identify it?

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I would like to recognize Alan Segrave from Maxxam Analytics with whom I am collaborating and who provided dimensional analysis for several mineral populations I will discuss today. The work has also benefitted from conversations with Andrey Korchevskiy and Andrew Duane of Chemistry and Industrial Hygiene, Inc. and Mark Utell of the University of Rochester. Together we are working on a project on mineral potency for cancer. (Segrave et al, in preparation)

Slide 2. EMP characteristics that are known to influence the development of asbestos-related diseases include dimension, chemical composition, atomic structure and surface area. The first three factors influence biodurability, control access to lung pleura and other locations in the body while we know from the work of Timbrell et al. (1988) that high surface area exposures strongly influence asbestosis. You have heard Dr. Brooke Mossman describe how Fe oxidation may contribute to inflammation. However, while many factors may play a role, today I will talk primarily about dimensional characteristics of amphibole EMPs. I will return to the subject of fibrous talc at the end of the talk.

In my possession I have a bottle of body powder from a time when my children were babies. In preparation for this talk, I examined some of this powder under the light microscope. By putting the talc product in immersion oil 1.578, the relief of talc goes almost to zero because 1.578 closely matches the indices of refraction gamma and beta of talc. I found particles of tremolite in the body powder, as shown in Slides 3 and 4. This particle is about 83 μm in length. The view in crossed polars (Slide 4) reveals that this particle is not composed of smaller fibrils. It has the morphology of a particle formed by fracture.

Slides 5 and 6 are photomicrographs of tremolite asbestos from Metsovo Greece, where mesothelioma and other asbestos-related diseases are found in excess among residents who use this material for whitewash (Langer et al. 1987). This bundle is about 77 μm in length and it is shown in both plane light (Slide 5) and under crossed polars (Slide 6). This particle displays the anomalous optical properties exhibited by asbestos under polarized light. While properties of bundles may be clear, what about the individual fibers and smaller bundles? Can we use dimensions to tell these from fragmented amphibole?

As described in Slide 7, width has significant relevance. Long asbestos fibers are narrow with modal widths $\leq 0.35 \mu\text{m}$ for $L > 5 \mu\text{m}$ fibers. The significance of length is outlined in Slide 8. Because exposures used to assess dose response are to fibers of $L > 5 \mu\text{m}$, it is the width distributions of these longer fibers that I will discuss in this talk.

Slide 9 shows the distribution of the width of tremolite found in body powder by analysts employed by Materials Analytical Services, LLC, (MAS). These data have been entered into the public record through the legal process as MAS report dated [August 2, 2017](#), entitled Analysis of Johnson & Johnson Baby Powder and Valiant Shower to Shower Talc Products for Amphibole (Tremolite) Asbestos Expert Report.

Several aspects are of note. First, the frequencies are low but uniform over a wide range of widths. Most fall between about 0.2 and 1.0 μm with a small mode at 0.4 μm , and another mode at about 1.5 μm .

Slide 10 shows the distribution of width of tremolite-asbestos from Metsovo Greece. Most EMPs are between 0.05 and 0.3 μm with a well-defined mode at 0.175 μm . These data were collected by Alan Segrave (Segrave et al, in preparation).

Slide 11 shows the frequencies from Slide 9 and Slide 10 plotted on the same scale. The shape and position of the frequency distribution of the body powder tremolite is typical of cleavage fragments of amphibole and the shape and position of the frequency distribution of the Metsovo tremolite asbestos is typical of commercial asbestos. (Wylie 2016).

If the tremolite in body powder is asbestos, should it not look like asbestos? Slide 12 provides the width distribution of crocidolite from Australia and from the Cape SA. These data were taken from Shedd (1985) who carefully measured four different crocidolite samples by TEM with high precision. The two parts of the figure demonstrate that high precision measurements show the distribution to be bimodal, with a set of fiber < 0.05 μm in width but when bin intervals are closer to 0.1 μm , as they normally are, this detail is lost.

Slide 13 illustrates the point that asbestos ore varies from place to place, even if it has the same name. These data from Shedd (1985) are from four sources of crocidolite. In addition, the disease patterns among miners vary from place to place. The first recognition of the importance of width may have been the publication by Timbrell et al. (1971) pointing out the near absence of mesothelioma among the miners of amosite in contrast to the experience of Cape asbestos miners and attributing this difference to the differences in width. These differences are illustrated in Slides 14 and 15.

Slide 14 contrasts the width distributions of two samples of riebeckite in different habits: one is Australian crocidolite (Shedd 1985), and the other a glassy nonfibrous riebeckite from California which we crushed to form cleavage EMPs (Wylie et al. 2015b). The curves are similar in shape to those depicted in Slide 11. Cleavage fragment population is characterized by a broad poorly defined mode between 0.3 and 1.2 μm , while asbestos has a narrow range of width and a well-defined mode, in this case at 0.05 μm . The curves in Slide 14 are a bit smoother than those shown in Slide 11 because of the larger number of particles in the riebeckite studies.

Slide 15 illustrates the width distribution of airborne amosite from Wylie et al. (2015c). The modal width for amosite is broader than that for crocidolite but the vast majority of particles are narrow. Amosite fibers tend to adhere to one another and bundles are more difficult to break apart. There may be sheet silicates that bridge fibrils, or there may be some structural continuity between fibrils. The individual fibrils are very narrow.

Slide 16 displays a pattern in the frequency of width that is characteristic of noncommercial asbestos. The Libby vermiculite deposit was once prospected as an asbestos deposit but the quality of asbestos was poor and it was wasted in the mining of vermiculite. The bimodal nature of the width distribution suggests two distinct populations. The one with modal width at 0.6 μm may be due to brittle fragments, as this mode is common among such populations of amphibole. Data are from EPA (2006).

Slide 17 illustrates the frequency of width of the fluoro-edenite fibers from Biancavilla Italy constructed from the data provided by Paoletti and Bruni (2009). The frequency distribution is similar to that of winchite-richterite asbestos from Libby.

Slide 18 illustrates another bimodal population: anthophyllite asbestos from Paakkila Finland. The data were provided by Alan Segrave (Segrave et al. in preparation). Like Italian fluoro-edenite and Libby winchite-richterite asbestos, the first mode is at 0.33 and the second is at about 0.6 μm .

Fibers of asbestos longer than 5 μm fall into three groups: To the first group belong tremolite asbestos from Metsovo and crocidolite from Australia and the Cape; there is a single prominent mode over a very narrow range of very small width. In these two examples, 50-60% of the fibers having width $\leq 0.15\mu\text{m}$. Amosite represents the second group. The fibrils are somewhat larger, but still quite small but they resist disaggregation. The third group includes asbestos from Paakkila, Finland, and fluoro-edenite from Italy. Two distinct modal widths are found in these populations, one near 0.3 and one larger at about 0.6 μm . While these differences among asbestos types are noted, there are also significant and defining similarities. For example, populations of asbestos fibers from mines, mining and industrial settings studied by TEM contain long fibers $\leq 0.15\mu\text{m}$ in width, fibers longer than 8 with widths less than or equal to 0.25 and abundant long fibers $\leq 0.3\mu\text{m}$ in width.

Does the width of long EMPs define their potency for asbestos-related disease? To answer that question, we can use single location exposures to examine the relationship between toxicity and dimension. Slide 19 summarizes current data that are published as recently as 2018 (Garabrant and Pastula). We know that for the same occupational exposure on a fiber/cc-year basis, mesothelioma mortality varies by asbestos type and occurrence, as shown in Slide 20. These differences may reflect dimension, durability, chemical composition, atomic structure or an unknown surface related property of particles in the exposure. By using only amphibole, the durability and atomic structure are largely constrained. Fe remains an unconstrained variable and so do surface properties.

Slide 21 provides the data from Garabrant and Pastula (2018) of Rmeso, the % of expected mortality due to mesothelioma per fiber/cc-year of exposure. All exposures are to EMPs $> 5\mu\text{m}$ but as we have seen, there are large and important differences in the EMPs in terms of dimension. The exposure to EMPs at Homestake Gold mine resulted in no deaths from mesothelioma despite long term exposures to EMPs $> 5\mu\text{m}$ in length. Chrysotile has such a low Rmeso because it has a different atomic structure, different surfaces and different biodurability. For these reasons it cannot be mixed with amphibole exposures when the only consideration is dimension.

Slide 22 lists three dimensional parameters chosen to compare to mesothelioma mortality. These were based loosely on the following: Lippmann (2016) proposed that mesothelioma was caused by fibers with $w \leq 0.15\mu\text{m}$, and Stanton et al. (1980) found that the number of fibers $L > 8 W \leq 0.25\mu\text{m}$ correlated most closely with carcinogenicity in rats following implantation; Pooley (2018) found abundant amosite fibers of 7 μm in length at the lung edge; and Lenz et al. (2003) concluded that fibers must be $< 0.4\mu\text{m}$ to enter the pleura. These parameters should be seen as preliminary; others may be more sensitive.

These three categories are first approximations of possible metrological indices for toxicity. My colleagues and I are working to refine this model. (Segrave et al. in preparation). We have additional data on both Rmeso and metrology to report. It is the goal of the work to find parameters that correlate

with toxicity not only for mesothelioma, but for other asbestos related diseases as well in order to predict toxicity of new sources of EMP dust.

Slide 23 plots the three possible metrological toxicity indices. In this graph, Cape and Australia are grouped together, and a single point along the x-axis is established for each type of asbestos by the weighted averages of populations from different sources, different labs, etc. All data were gathered by SEM or TEM (Segrave et al, in preparation). The lines represent a best fit. R^2 for all are greater than 95%. Homestake data plot at the origin, since there are no particles in the populations with the metrological toxicity indices chosen and studies have shown no excess mesothelioma.

Slide 24 shows the range of error. Dr. Phil Piccoli, Department of Geology, prepared this slide for me to visually display error. For Rmeso the 95% confidence intervals are shown. For the % fiber, the standard deviations derived by combining populations are shown.

Slide 25 shows the curves generated from the correlations shown in Slides 23 and 24. If these curves are predictive, the metrological parameters from three locations where asbestos exposure occurred and has resulted in disease: Biancavilla Italy, Metsovo Greece, and Paakkila, Finland, should plot on them. (PLEASE NOTE: The reference given in the Legend for Slide 25 is incorrect. It should be Paoletti and Bruni, 2009, and Paoletti et al. 2000). The three metrological parameters predict for Metsovo Greece an Rmeso of about 0.28, for Biancavilla Italy, an Rmeso of about 16%, and an Rmeso of about 0.08% for Paakkila, Finland. (The low percentage of fiber with $w < 0.15 \mu\text{m}$ from Italy may reflect the fact that no fibers with $W < 0.1 \mu\text{m}$ were counted.)

Two of the metrological toxicity indices, $w \leq 0.15 \mu\text{m}$ and $L > 8 W < 0.25 \mu\text{m}$ are very sensitive indicators of Rmeso. Very small proportions of these fibers in the exposure raise Rmeso. The third category, $L > 7 W > 0.4 \mu\text{m}$ is not as sensitive and less useful. The model suggests that a population must have at least 10% of these fibers to measurably increase mortality from mesothelioma. It may be useful for pleural plaques. More complex models are expected to enhance the predictive capacity of toxicity indices (Segrave et al, in preparation).

Slide 26 shows a photomicrograph taken in polarized light of a fiber bundle of tremolite asbestos from Metsovo Greece. Dimensional analysis predicts that this material is mesotheliomagenic. This particle is 165 by 18 μm . It is composed of very fine fibrils that could easily disaggregate, which are evident in this photograph.

Slide 27 is a tremolite particle 77 x 9 μm , taken from my bottle of body powder. It is not a fiber bundle. It cannot break up into fibrils. It cannot be inhaled. This particle is not asbestos. It represents no threat to human health of which I am aware. By PLM, the distinction between asbestos and cleavage fragment in a particle this size is unambiguous.

Slide 28 lists the analysis issues in the mining and use of cosmetic talc. The first question and perhaps the most difficult: "Is the amphibole asbestiform?"

Slide 29. Particles in body powder are large. If amphibole-asbestos is present, it will be seen by light microscopy and, because its properties are distinctive, it will be recognized as asbestos. If heavy liquids are used, the detection limit can be extraordinarily low ($<<0.01\%$), since it only depends on the amount of material examined. If measurements by electron microscopy are the only data, then the population should be compared to that of asbestos according to metrological indices for toxicity.

Slide 30 outlines some of the conditions under which one might expect to find chrysotile. Chrysotile is rarely reported in cosmetic talc, but it has been reported following TEM analysis. Chrysotile should be readily identifiable by TEM.

The identification of anthophyllite asbestos in cosmetic talc has been contentious. Slide 31 depicts a particle 143 x 33 μm in size from the same bottle of body powder in which I found tremolite cleavage fragments. This is the fibrous form of the mineral talc. It is not asbestos.

Primarily because the water content is higher in talc (5%) than it is in anthophyllite (2%), the average index of refraction of talc is lower than anthophyllite. This fact, and the high birefringence of a sheet structure, makes the distinction between fibrous talc, anthophyllite, and even their likely proportions within intergrowths of the two, a trivial problem if a mineral powder is mounted in immersion oils within the appropriate range of indices of refraction and viewed by PLM (Wylie, 2017)

Despite the ease of identification by PLM, distinguishing anthophyllite asbestos from fibrous talc by electron microscopy is not simple. The Mg/Si is indistinguishable and chemical analysis by EDXA is normally insensitive to water content. Morphology may be overlooked. Ultimately, the distinction rests on electron diffraction patterns. ISO 10312 states that a fiber should be classified on the basis of morphology and EDXA composition, and that a list of minerals consistent with that composition be made. A zone-axis SAED pattern should then be tested for consistency with the crystal structure of the suspected type of asbestos, but also tested for inconsistency with crystal structures of the other minerals of similar composition. This necessary step may not be often followed.

One of the questions we were asked to comment on was analysis by weight percent vs particle number/mass. The most important response to this question is that before undertaking any quantitative analysis the presence of asbestos must be established. In other words, the identification that a hazard is present, and the measurement of its concentration should be separated. Before asbestos can be measured it must already have been detected.

In cosmetic talc powders, the mass is overwhelmingly concentrated in particles large enough to be easily seen by PLM. If a mass basis is the goal, then a detection system based on PLM could be designed with a detection limit approaching 0.01%. If heavy liquids are used, the detection limit could be much lower. If asbestos is present, it would not be homogeneously distributed, given the unique circumstances under which it forms, so methods that examine a large amount of material would be preferred. Given its high tensile strength and durability, if amphibole asbestos is present, it will be so in the form of long fibers in bundles. Particle number/unit mass data are very difficult to verify. The amount of material examined is very small, yet extrapolations assume material homogeneity. This may not be a valid assumption for "contaminants" such as asbestos, or bits of hanging wall in advertently scraped into the ore.

If an amphibole is found in the ore, or in reproducible amounts in mineral products, a dimensional analysis may be useful. To be so, there must be a sufficient number of particle measurements that cover the full range of dimension in the product so that a reasonable evaluation for toxicity can be made through comparison to known asbestos. However, it must be accompanied by examination of the material by PLM.

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