

**The Joint Institute of Food Safety and Applied Nutrition  
(JIFSAN) symposium on  
“Asbestos in Talc”  
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**Invited presentation by  
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**Overview: My Topics assigned by JIFSAN Organizers**

1. Overview of current asbestos testing methodology
2. Limitations of X-ray diffraction (XRD), polarized light microscopy (PLM), and phase contrast microscopy (PCM)
3. Advantages of electron microscopy (EM) methods (SEM, TEM)
4. Methods of mineral identification

I was asked by JIFSAN organizers to describe and explain in thirty minutes the testing methods currently used and proposed for asbestos in commercial talc ores and products. The anticipated audience is "diverse". There are government regulators who oversee the scientific and industrial community; professional organization administrators; biologists; mineralogists; and highly competent specialists- e.g., "talcologists" and "asbestologists". It became clear to me that the audience would have highly varying background knowledge of asbestos in talc, ranging from understanding of mining, product history, nomenclature and analytical methods.

So, and limited by allotted time of thirty-five minutes, I decided to make a presentation that focused on the key issues affecting the techniques used for the materials being analyzed, and with overarching constraint of analyte definition accuracy and consensus.

In the oral presentation at the JIFSAN Talc Symposium, I sought to provide listings of pros, cons and "issues" to summarize what I deem as key aspects of the assigned "limitations" and "advantages" (especially since those terms can be perceived as introducing a bias. Time constraints preclude an extensive discussion of sample submittal and preparation, but remember GIGO- garbage in, garbage out!

There is extensive literature on the topics assigned for me to discuss. I have provided a number of papers that appeared to me to be over-arching and representative of the issues being considered. I apologize for leaving out what are certainly other important papers, but the citations provided should provide links to search the literature.

In my oral and written presentations, I conclude with the proposals that consensus analyte definitions must precede whatever analytical method(s) become selected and that multiple methods are better than a single technique.

## **Title: How do we measure & characterize the elongate “stuff” in talc products?**

What is a "MINERAL"? This is part of a long-standing area of disagreement between members of the mineralogical community and the regulatory/legal community when it comes to how asbestos is defined. Part of it seems to be whose turf we are acting on and part is a serious and marked disagreement over terms. And when terms get entrenched it can be that they are founded on errors in original usage and meaning. This has led to many ongoing debates about what is asbestos? what is a fiber? and what's asbestos?

Mineralogists define a mineral in several ways, but most agreed-upon definitions use aspects of typically chemistry, structure, state of matter and formation. A long-standing definition is "A mineral is a naturally occurring, homogeneous solid, inorganically formed, with a definite chemical composition and an ordered atomic arrangement" (Berry and Mason, 1959, pp 3-5). The International Mineralogical Committee has adopted procedures for mineral nomenclature (Ernest et al, 1998). For a recent discussion of the definition of a mineral, see Ch. 2, Dyer and Gunter, 2008). Also, even within the mineralogical community, there can be strong differences of opinion on how nomenclature is derived, decided and applied (see White, 2004)

Asbestos has been defined in several ways, but predominantly on the basis of commercial applications as an insulator for sound and/or thermal protection. The most relevant definition is "regulatory asbestos" or the so-called regulated six species. The six (chrysotile, and five amphiboles- "amosite", crocidolite, tremolite, actinolite and anthophyllite -subsequently revised to the asbestiform morphologies of the last three), were defined on chemistry, usage and shape/size (the so-called LAF or long asbestos fibers-  $>5 \mu\text{m}$  long, diameter  $<0.3 \mu\text{m}$ , aspect ratio  $>3:1$ ). Over time, additional interpretations based on factors such as medical, bioactivity, legal arguments and emotional interpretations have pushed forward differing definitions.

Mineralogists have traditionally defined "asbestos" in terms of morphology and commercial mining. Traditionally, asbestos and asbestiform have not meant exactly the same thing (see Skinner, 1988) and this has caused sharp disagreements over how to develop and implement regulations to best protect human health and the environment..

Mineralogists generally consider morphology prefixes to add to the mineral name. For example, shape and dimensions can be describe during the general terms of massive, needle-like, acicular, bladed, prismatic, lamellar, etc. These terms can be use to describe broken crystal fragments and also individual crystals and multiple crystal masses.

Fibrous and asbestiform are special discriminatory terms that have been used to describe mineral shapes and aggregations, but which have resulted in much controversy, especially from the non-geological community. An excellent description of mineral morphologies is given by Dyer and Gunter, 2008, Ch. 2 and non-mineralogist are strongly urged to familiarize themselves with how morphological terms are usually broad descriptive terms such those to describe the appearance of a human being (e.g., short, tall, stout, thin lanky etc.,....).

Fibrous is a general term used by geologists to describing a mineral that consists of fibers- however, such a mineral does not have to be asbestiform nor even regulatory asbestos.

Asbestiform described a mineral that has the properties of "asbestos fibers"- long, thin, flexible, unique terminations and surfaces, etc..

Some crystals have structural weaknesses in one or more directions of the structure. Then, breakage can occur along these surfaces. Such breakage is described by mineralogists as "cleavage" and the aggregate shape can be described using one or more of the above morphological terms e.g., prismatic, bladed, lamellar, etc).

A widely cited, but often mis-understood description of mineral and regulatory shapes arose from the then rather simplistic description of hand sample mineral morphologies by Campbell et al, 1977); also, for airborne materials, see Wylie et al, 1985)

Summarizing the shape and morphology issues relevant for particulate analysis, cleavage fragments are broken crystals; acicular/prismatic shapes are (long & narrow crystals or cleavage fragments; and asbestiform minerals are mineral masses of fibers formed by crystal growth.

It is clear to many that we need agreed-upon protocols to discriminate asbestos from non-asbestos particulates (see 2014, 2015 papers by Gordon *et al* vs. Lee & Van Orden, re “disagreements” on what mineral shapes are relevant for bio-hazard and how the masses are measured analytically). Science should always be used to give correct answers, but what happens when it is done wrong and results are misinterpreted? Confusion and disagreement run rampant! So, let's examine the current major and developing methods of particulate analysis in talc ores and products.

Finally, as one approaches a review of talc analytical methodologies, it should be kept in mind that there are TWO categories for talc- USE and ORIGIN

Historically, talc ores have been mined form two major geologic environments- a higher temperature regime (regional metamorphic and metamorphosed ultrabasic) and a lower temperature water-alter<sup>3</sup>ed (hydrothermal) regime (VanGosen et al, 2004). Amphiboles are much more common in some deposits of the former and very limited to absent in the latter. Additionally, usage has been industrial applications (fillers, ceramics, etc...) and cosmetic/pharmaceuticals (e.g. make-up, hygienic powders, excipients, etc..). Source materials for the former have largely come from the higher temperature ores whereas the latter has typically, but not always, been sourced form the lower temperature ores.

### **Initial Visual Sample Observations for Asbestos, talc & “fibers”**

A first step in analyzing a talc sample for suspect asbestos should be to examine available information about source geology. Has the ore deposit that was mined been documented to be "free" of asbestos; was the supplier a single or multiple sources; does the product have a

documentable history of prior analyses; and is there any other available relevant information that will assist the analyst in characterization.

A first analytical step should be via visual examination- the bulk sample or powder using a stereozoom microscope accompanied by a turning of a massive sample or stirring of the powder. While a magnification of 10-30x which is common for standard stereozoom microscopes will not show extremely small elongate particulates. There is the possibility that some "larger" mineral fragments will be visible. The change in orientation might demonstrate a reflectance from a surface and if the "shape" is that of an elongate particulate, the analyst will become alerted that that the sample has suspect fragments.

### **Summary Visual/Eyesight observations for Asbestos, talc & “fibers”**

(Note: for each methodology, a listing of Advantages and Disadvantages/Limitations/ "Issues" are provided. The category of Issues is emphasized in quotation marks so as to not sway the arguments pro and con. For the more important and obvious (e.g., widespread usage) issues, an asterisk is used and often with a subsequent discussion to follow.

#### **Advantages:**

Permits rapid sorting of some samples

#### **Disadvantages/Limitations/"Issues":**

Magnification limits

Only "sees" shapes and not chemistry, asbestiform physical properties

### **LIGHT MICROSCOPY- Polarized Light Microscopy (PLM) and Phase Contrast Microscopy (PCM) for Asbestos, talc & “fibers”.**

PLM is based upon differences in optical properties from structure and composition and is especially useful for building “bulk materials. PCM is based upon the minute differences in phase of light passing through a sample to exaggerate phase boundaries; especially useful for “fibers” in industrial site air samples.

PLM is generally considered to be a more advanced and technically more difficult method than Dispersion Staining. While PLM is the better analytical method, its use is often overshadowed by the cookbook techniques of DS. One major advantage of DS is that the sought after analyte is a member of the regulated asbestos species. Therefore, "just" the color effects and grain outline can serve as a diagnostic character. In contrast, for analytes other than building materials (e.g., talc ores and products), there is the possibility that many EMP's, both relevant and non-relevant, could be present.

Both methods are acceptable to confirm "Regulatory Asbestos". However, over the past decade or two, university training in PLM has undergone a marked downturn in numbers of Geology Departments offering a full semester course and also for those who do offer training, a much

reduced time allocation (Rutstein, 2012). Hence, what many experienced and older mineralogists would consider to be the preferable method, fewer trained microscopists will be available for using this standard method of analysis (Gunter, 2004).

Under AHERA, the US EPA codified PLM methods and techniques for bulk materials. It was revised in 1993, but those guidelines are deemed advisory (U.S. EPA, 1993). Given the widespread need for the analysis of bulk building materials, PLM (both Becke Line and Dispersion Staining) has gained wide popularity. The initial set-up is relatively inexpensive and with trained, competent analysts, rapid turn-around times for identification and quantitation of less than one hour are readily achievable.

While an upper level of magnification of circa 400-450X can be achieved, most routine magnification works are done at 100-300x. While this level of magnification may preclude the identification of the very small asbestos fibers, it is not especially critical for friable building materials. Such materials have relatively larger sized asbestos masses and with most exceeding the regulatory threshold of 1%, magnification is not a major issue. Furthermore, if the material is concentrated by methods such as mechanical sieving or elutriation/fluidized bed separation, particles at the lower end of size may begin to be discernible and thus, give the analyst adequate reason to proceed to electron microscopy techniques.

### **Summary Polarized Light Microscopy (PLM)**

#### **PLM Advantages:**

- Codified by EPA \*
- Widespread usage \*
- Relatively inexpensive
- Rapid turn-around
- Standardized rules
- Dispersion staining
- Becke Line
- Good for building materials
- Good for “bulk materials”
- “Sees” range of fiber sizes
- Skill levels

#### **PLM Disadvantages/Limitations/"Issues":**

- Magnification limit ~400x \*
- Smallest fibers can be masked by matrix
- Non-friable materials opaque
- Variations in mineral chemistry changes RI
- Becke Line techniques “harder”  
(pleochroism, extinction, RI, ...)
- Quantification

## **X-ray Diffraction (XRD) for Asbestos, talc & “fibers”**

X-ray diffraction (aka powder XRD as opposed to single crystal and selected area electron diffraction via TEM) fell into disfavor circa 1980. Regulators decided that XRD yielded no morphological information and thus, could not distinguish asbestiform morphology from cleavage fragments.

### **XRD Advantages:**

- Rapid turn-around
- Standardized rules
- Reference standards
- Good for gross phase ID
- Identifies sample mineral assemblage
- Semi-quantitative for amounts
  - improvable by concentration (sieving, elutriation)*
- More quantitative with standards (slow scan)
- “Sees” almost all size fractions

### **XRD Disadvantages/limitations/”issues”:**

- Measure atomic spacings
- Phase ID errors
- Expensive
- Need reference standards
- Sample “mounts”
  - powder “packing”
  - grain orientation
- Analysts expertise & skills
- Radiation protocols
- Instrument calibration
- “Poor” shape information
- Overlap 2 theta scan peaks \*
- Detection limits \*

### **Detection Limits for XRD**

Currently, XRD is being increasingly used as a tool to discriminate talc products that might contain amphiboles and serpentine (especially chrysotile).

A major advantage for using XRD for the analysis of talc-based products is that it allows for the quick and relatively easy determination of the presence of serpentine (chrysotile) and with some special effort, the presence of amphiboles.

Relevant amphiboles have a diffraction peak located just above the major talc diffraction peak present at 10 degrees 2 theta (via Copper K alpha radiation). However, if the scan is done at slower speed or by step scanning, it is possible to observe the amphibole peak on the shoulder of the talc peak and the analyst can conclude that additional work is necessary to characterize amphibole morphologies.

### **Scanning Electron Microscopy (SEM) for Asbestos, talc & “fibers”**

Scanning Electron Microscopy is currently experiencing resurgence in use after having fallen out of favor when electron microscopy methods were being adopted under AHERA (Asbestos Hazard Emergency Response Act) in the late 1980's. SEM yielded excellent images of morphological properties, albeit at lesser magnifications than Transmission Electron Microscopy (TEM). Also sample elemental chemistry could be determined via energy dispersive spectroscopy (EDS).

#### **SEM Advantages:**

Visual magnification of shape

Chemistry by EDS

#### **SEM Disadvantages/Limitations/"Issues":**

Versus TEM, the “Gold” standard \*

Expensive

Analyst expertise

Instrument calibration

No structural capability \*

Can't discriminate some amphiboles \*

Interpretation of-

*“shapes”/morphologies*

*asbestos present and amount(s)*

### **Transmission Electron Microscopy (TEM) for Asbestos, talc & “fibers”**

TEM methods have the major advantages of magnification to see the very small-sized particulates; energy dispersive spectroscopy for qualitative and quantitative chemical analysis; and selected area electron diffraction capabilities for the determination of crystal structure. Upon the passage of the Asbestos Hazard and Emergency Removal Act (AHERA), TEM became the so-called Gold standard for asbestos analysis (AHERA, 1987). However, it is often not recognized that the method was designed primarily for the analysis of residual asbestos fibers in a defined and enclosed space after asbestos removal/remediation activities were completed. Subsequently, some have used the method for raw bulk materials; treated bulk materials considered as non-friable; and for commercial products such as talc-based compositions (see Kremer and Millette, 1990). Essentially, we have moved from rigorous methods designed for the evaluation of airborne fibers which would be primarily residual regulatory asbestos to

measurement and characterization of elongate mineral particulates (EMP's). (for background, see Rohl and Langer, 1975, 1977; CTFA Cosmetic Talc, 1984; U.S. EPA, 1993; U.S. Pharmacopeial Convention, 2009); Block et al, 2014; Van Orden et al, 2008)

**Advantages:**

Perceived as AHERA “Gold” standard \*  
Relatively widespread usage  
Morphology, Chemistry, Structure \*  
*distinguish amphibole species*

**TEM Disadvantages/limitations/”Issues”:**

“Sees” mostly smaller size particulates on limited number of grid openings  
Expensive  
Need reference standards  
Analyst expertise  
Instrument calibration  
Interpretation shapes/morphologies \*  
*sample preparation (grinding, milling)*  
Population and amounts \*  
*detected/not-detected; confirmed/not-confirmed*  
Talc vs. anthophyllite - Twisted talc ribbons/fibers (“kinky” talc)

**TEM Limited Population Issues**

Unequivocal ID from a single “fiber” and extrapolation to uncounted TEM grids can be VERY misleading! However, it is “likely” to be (regulatory) asbestos on basis of: aspect ratio, population, parallel sides, bent/flexible particulates, terminations, surfaces, nomenclature, unit cell chemistry and litigation

**TEM CHEMISTRY “ISSUES”**

**Talc-Tremolite-Anthophyllite**

anthophyllite  $Mg_7Si_8O_{22}(OH)_2$

Ca:Mg:Si = zero:7:8

talc  $Mg_3Si_4O_{10}(OH)_2$

Ca:Mg:Si = zero:3:4

tremolite  $Ca_2Mg_5Si_8O_{22}(OH)_2$

Ca:Tc & An = infinity

Thus, tremolite can be readily distinguished from talc and anthophyllite by Calcium (Ca) content. However, because of the chemical similarities of Magnesium (Mg) and Silicon (Si) for talc and anthophyllite, chemical analysis alone is insufficient to distinguish which mineral is present. So, characterization via SAED (scanning area electron diffraction) of the atomic structure becomes necessary.



## SAED ID "ISSUE"

The characteristic unit cell dimensions (or structural building blocks/units) of the three minerals are:

tremolite	$\underline{a} = 9.84\text{\AA}$	$\underline{b} = 18.02\text{\AA}$	$\underline{c} = 5.27\text{\AA}$
talc	$\underline{a} = 5.27\text{\AA}$	$\underline{b} = 9.2\text{\AA}$	$\underline{c} = 18.85\text{\AA}$
anthophyllite	$\underline{a} = 18.55\text{\AA}$	$\underline{b} = 18.03\text{\AA}$	$\underline{c} = 5.28\text{\AA}$

**Summary of Analytical Methodologies** Note that the length of 5.27/8 Å (Angstroms) is common to all three minerals.

In common practice, especially for identification of residual asbestos fibers in air samples taken after asbestos remediation, and if no Calcium from tremolite is identified, the 5.3Å (~0.53 nm) dimension is used to conclude that anthophyllite is present. However, this dimension is essentially the same as a talc dimension, so measurement of a single dimensional direction from bulk talc samples can lead to questionable conclusions.

Millette (2015) concluded that for fibers with kinks (e.g, twisted fibrous talc or talc ribbons), "When using the zone-axis indexing option, a few rare fibers with kinks in them that would normally be dismissed as talc ribbons by morphology may show a zone axis that match anthophyllite. Because the crystal structure matches anthophyllite and the fiber has substantially parallel sides for the majority of the fiber length, the fiber is counted as anthophyllite in this method."

This conclusion can be criticized for making an unknown characteristic default to "asbestos"

Since the  $\underline{a}$  crystal lattice dimension of talc is essentially the same as the  $\underline{c}$  lattice dimension for both tremolite and anthophyllite, a single zone axis measurement is not necessarily conclusive. So, at least two zone axis dimensions and one interfacial angle measurements are necessary for **correct** identification (see Lee et al, 2016)

## CONCLUSIONS

### **Advantages versus Disadvantages/Limitations/"Issues"**

If the goal for talc products is to demonstrate ("prove") the absence of relevant amphiboles and chrysotile, then there is no single method that will always work alone. Rather, a stepped sequence of methods is more likely than not to be capable of generating relevant data for managers, regulators and consumers to decide if the talc product is acceptable for human usage. To accomplish this end point, we will need a full spectrum of analytical tools, applied in context of an analyte definition in the particular product (industrial, pharmaceutical and cosmetic) of concern, to assert problematical levels of concern.

If the targeted goal is to "prove", the absence of relevant amphiboles and chrysotile, the, absolute proof is going to be an elusive goal. Instrumentation improves over time. Analytical techniques change. And what was deemed adequate and sufficient proof decades before will likely not meet the standards and methodologies that have evolved over time. Recall the abundant literature especially cited in legal proceedings by Plaintiffs taken from the 1960's and 1970's describing elongate particles as "asbestos" when in fact they were talc ribbons analyzed using airborne fiber methodologies!

And what is a particulate of concern? Should it be: only regulatory asbestos (The Six); or amphiboles with discernible biohazard manifestations; or EMP's; or asbestiform shapes; or mineral cleavage fragments; and should the regulations for talc products for industrial usage, cosmetic talcs, and pharmaceutical products be the same or different?

And what is a "problematic level of concern" will have to be decided by regulators using the best available techniques which generate scientifically valid data and accepted by consumers and the legal community. Should there be different levels for industrial talc and pharmaceutical/cosmetic talcs? And for our future, if the targeted suspect particulates come to include elongate mineral particulates (EMP's), then proof becomes an almost unattainable goal.

My assessments for analytical techniques that will get us toward scientifically valid truths given present-day analytical methodologies and which are available in for profit and producer laboratories are as follows:

PLM will remain primary technique given its simplicity and widespread availability. SEM can be a very useful supplement. XRD will be especially useful to confirm presence of amphiboles. And TEM is likely to be "ultimate" analytical. It is important to emphasize that for any particular technique to be used to generate valid and useful results, the users of such data must agree on definitions of names and relevant shapes!

And in the absence of sound analytical results, remember that AHERA TEM method allows for "Ambiguous" and/or "Indeterminate" when the analyst just can't be sure. Thus, for some samples, especially those with transitional amphiboles and/or minute concentrations, the analytical instrumentation may not be good enough to generate scientifically sound results!

So, as we seek the "perfect method" and chase after "analytical zero's", let us keep in mind that we are seeking to characterize minerals, and also commercial products; and both vary in physical and chemical properties!

Looking back, it is hard for many to comprehend how we got to deeply into regulating down to the very last trace of asbestos in any product or usage (Rutstein, 1982). If asbestos is really as dangerous as many perceive, is it logic based on developed evidence or a developed bias due to having done it that way before that leads us to be concerned about "everything in any concentration and at any exposure level. Will we strive to regulate: elongate mineral particles, transitional fibers, "the number" of fibers, respirable fibers or EMP's that are merely and barely

"PRESENT"! In particular, when a few (1, 2, 3, 4 or ?) elongate particulates are all that are observed on a small number of TEM grids, is it rational to extrapolate those findings to thousands of unviewed grids to generate a large concentration supposed to be bioavailable?

I suggest that we regulate mineral products on the basis of demonstrated and agreed upon human health hazard and amphiboles be more heavily regulated than chrysotile and that iron-bearing and iron-alkali-containing amphibole asbestiform species be the most heavily regulated.

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