Moderator Session C – Interpretation of Testing Data

Goal: To establish consensus on the interpretation of microscopy measurements for mineral fibers in cosmetics containing talc.

Moderators: Mickey Gunter (*in absentia*), Matthew Sanchez, and Brooke Mossman

At the outset we believe the title of this goal was miswritten by whatever group organized this session. We reach this conclusion in that there should be no "interpretation" of testing of microscopy data, but rather verification of it, especially for correct mineral identification and the differentiation of talc from anthophyllite. To that end any lab or analyst must provide written documentation and photographs for every particle. Also, no competent mineralogist would rely on only one method to identify a mineral where the observed data was ambiguous. So it was recommended that PLM and TEM and/or SEM be used, along with the non-microscopic method of powder XRD.

Providing more detail to the above, it was pointed out that no one had defined a mineral, but for brevity herein we can stick with only part of that definition - a fixed range of chemical composition and a specific atomic arrangement. Thus, given these two criteria, it should be obvious that to identify a mineral both its chemical composition and crystal structure must be determined. Providing the details of how to completely accomplish this is beyond the scope of this document, but suffice it to say this is a routine task for a competent mineralogist. However, the issue we find in many of the "methods" used by commercial testing labs is that they were developed for asbestos-containing materials, primarily used by individuals that do not understand the scientific principles they are based upon, and thus fall short to correctly identify minerals in environments where one cannot assume asbestos is present.

While the PLM provides indirect methods to determine a mineral's composition and structure by the optical properties of a mineral, the TEM can provide compositional data with the EDS -- albeit it only semi-quantitative -- and diffraction data (i.e., SAED) that can be used for near-unequivocal particle identification. However as mentioned above these microscopic methods require thorough documentation, both written and photographic. For the PLM this would mean photomicrographs in both cross-polarized and plane-polarized light, as well as observation of Becke lines in refractive index matching fluids in multiple directions of the observed particulates. For the TEM three sets of data are required: 1) a photograph of the particle, 2) the corresponding EDS, and 3) two indexed zone axis SAEDs with the corresponding tilt angle between them. Given this type of information, there would be no interpretation of the "microscopy measurements" instead there could be verification of them. These microscopic results should be verified with each other and powder XRD.

To our recollection no one at the meeting defined asbestos or the asbestiform habit. As such we thought it best to provide one. And we believe the most succulent one is give in EPA/600/R93/116 page A-1[1]) as follows:

[1] See also ISO 22262-1 “Part 1: Sampling and qualitative determination of asbestos sin commercial bulk materials”, and current USP talc Monograph.

Asbestiform (morphology) Said of a mineral that is like asbestos, i.e., crystallized with the habit of asbestos. Some asbestiform minerals may lack the properties which make asbestos commercially valuable, such as long fiber length and high tensile strength. With the light microscope, the asbestiform habit is generally recognized by the following characteristics:

* Mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than 5 µm. Aspect ratios should be determined for fibers, not bundles.

* Very thin fibrils, usually less than 0.5 micrometers in width, and

* Two or more of the following:
  Parallel fibers occurring in bundles,
  Fiber bundles displaying splayed ends,
  Matted masses of individual fibers, and/or
  Fibers showing curvature

These characteristics refer to the population of fibers as observed in a bulk sample. It is not unusual to observe occasional particles having aspect ratios of 10:1 or less, but it is unlikely that the asbestos component(s) would be dominated by particles (individual fibers) having aspect ratios of <20:1 for fibers longer than 5 µm. If a sample contains a fibrous component of which most of the fibers have aspect ratios of <20:1 and that do not display the additional asbestiform characteristics, by definition the component should not be considered asbestos.

Asbestos – A commercial term applied to the asbestiform varieties of six different minerals. The asbestos types are chrysotile (asbestiform serpentine), amosite (asbestiform grunerite), crocidolite (asbestiform riebeckite), and asbestiform anthophyllite, asbestiform tremolite and asbestiform actinolite. The properties of asbestos that caused it to be widely used commercially are: 1) its ability to be separated into long, thin, flexible fibers; 2) high tensile strength; 3) low thermal and electrical conductivity; 4) high mechanical and chemical durability, and 5) high heat resistance.

Other than the above synopses on the verification of mineral identification by microscopic methods, the following points were also made.

* The USP Talc Expert Panel has spent considerable time dealing with similar issues as discussed at this meeting; to gain an overview of them their two stimuli articles need to be considered.

* The USP Talc Expert Panel is also working on PLM, XRD, SEM, and TEM methods that should be used by FDA.

* Concentration methods could be used to lower the detection limits for amphibole minerals in bulk talc samples, but the detection for amphiboles is already approximately 0.1% by powder XRD, and concentrations in the parts per million rage are reached by routine PLM and TEM microcopy testing.

* Also note: 1) Concentration methods cannot be used to test for chrysotile. 2) Concentration methods do nothing for the correct identification of the minerals present. Unless mineral identification is performed correctly it does not matter how you prepare the samples for analysis.

* Using compositional tests (e.g., Ca as a proxy for tremolite) could be used to define upper limits of potential tremolite content, however it does not account for other minerals such as chrysotile and anthophyllite (to name a few). Also, due to the unknown mineral composition of talc samples expected to be encountered during routine screening one must use a microscopy technique where discrete particles are observed and identified.

* Finally it was pointed out that there are methods used by certain commercial labs to count a kinked particle as anthophyllite asbestos when they have an EDS and SAED that might also match talc. This is clearly incorrect and must not be tolerated.