

To: J. J. Travers
From: W. C. Forbes — *Dept. Geo. Services U. of Illinois*
Subject: Evaluation of proposed FDA test for "Determination of Asbestos in Talc by Optical Microscopy".

In compliance with your letter of September 14th, I have evaluated the test for "Determination of Asbestos in Talc by Optical Microscopy" which has been proposed by the FDA. In my opinion the test is completely unsuitable for the evaluation of asbestos abundance in talc.

The FDA procedure states that mineral abundances can be determined by counting the number of grains of that mineral which occur within a grain mount. This approach is both naive and totally incorrect. The basic flaw of the FDA procedure is that it completely ignores the size of the grains being counted. A chrysotile grain 1000 x 100 microns in size is treated exactly the way as a grain measuring 10 x 1 microns. In this case the actual volume abundance varies by 4 orders of magnitude.

The only valid method for estimating mineral abundances using the polarizing microscope is the point count method. To briefly describe the method, a thin section or grain mount is examined at regular intervals, the interval spacing being selected to achieve a uniform coverage of the entire sample. At each sampling site, the mineral variety under the cross-hair is identified and individually tabulated. After covering the entire slide, the number of "hits" for each individual mineral species are summed and recalculated to percentage values. The number of times a given mineral is encountered is, of course, proportional to the area occupied by that mineral in the entire sample. The areal percentage obtained is meaningful only when compared to the areas of all other mineral components. Thus, the FDA procedure has absolutely no value if isolated grains are counted with no regard to the size of these grains or the abundance of other mineral species.

Objections to the FDA test on technical grounds are summarized below:

1. The FDA test is predicated on the assumptions of: (a) the "optical uniqueness" of asbestos; (b) uniformity of the sample material and, (c) uniform distribution of the sample over the slide. These assumptions were found to be invalid.

a. Chrysotile is quoted as having indices of refraction substantially less than 1.574 and therefore should be easily detectable in an oil of that index. It should be noted that chrysotile invariably consists as an aggregate of fibers variously disoriented so that a great deal of optical

compensation exists. The true maximum and minimum indices are probably never measured. Also talc flakes and fibers oriented with their c crystallographic axes parallel to the stage will exhibit an index of refraction between 1.536 and 1.590 and on cursory examination may be identified as chrysotile. The probability for operator error is very large and will likely produce over-estimates of chrysotile abundances.

An additional complication is that iron and aluminum substitution in chrysotile will raise the indices of chrysotile to levels close to those of talc. However, the chance of this happening is slight.

b. The mineral contents of the sample must be completely uniform if bias is to be avoided. Asbestiform minerals usually occur as fibrous aggregates even after grinding. If the fibers of one such clot were counted, and this "count" extrapolated over the entire slide area, a highly exaggerated asbestiform content would result. Optical examination of several talc samples supplied by Avon showed substantial inhomogeneity in mineral abundances.

c. A very important assumption is that the sample can be distributed evenly over the slide area (18mm square). This could not be achieved in several attempts. Fine grained materials such as ground talc tend to aggregate into large multi-grain particles which resist mechanical attempts to achieve a uniform particle distribution. Thorough mixing of the talc sample with the immersion oil also failed to achieve a uniform grain distribution. A talc --- water slurry was evaporated onto the slide, however, this did not produce a marked improvement in sample distribution.

The homogeneity of the sample distribution was tested in the following way. One milligram samples of 815-157 and "Western Treated" talc were prepared following the FDA procedure. For 815-157, the number of drop-side grains observed in a 0.64mm^2 field of view were tabulated; the slide was then shifted 0.5mm and the procedure repeated. Fifteen such counts were made per scan. Four other such scans were made at 4mm intervals. The average number of drop-side grains observed at each "sample site" and the standard deviations are listed in Table 1. The large spread in the averages (0.13-5.8grains/site) is immediately evident, indicating an almost complete lack of homogeneity. This is reflected in the standard deviations which are almost as large as the averages themselves. The results for the "Western Treated" talc are listed in Table 2 and show a similar lack of homogeneity.

For this slide, carbonate, quartz and diopside grains were counted. There is a six-fold difference between the maximum and minimum averages with correspondingly high standard deviations. The lack of homogeneity precludes any accurate estimate of mineral abundances based on a "grain count". It should be noted that the scanning and counting procedure is much more rigorous than that called for by the FDA. Only clearly recognizable grains were counted and the scan was made at regular intervals. This avoids the bias introduced by judgement decisions (chrysotile vs. fibrous talc?) and irregular scanning methods.

2. A grain mount is not a two-dimensional surface. The immersion oil + grains may have a thickness of up to 50-75 microns. This can result in mineral segregations based on density differences. As microscopes have a 5-7 micron depth of focus at 400 magnification, apparent mineral abundances will be a function of the depth of the focal plane within the grain mount.

3. The FDA test calls for a 1 milligram sample. As fine grained materials may be hygroscopic, a large error in sample weight can be introduced at an early stage of the test.

4. The test calls for counting of asbestos fibers using an unspecified scanning procedure. When 150 fibers are counted, total asbestos is "determined" by the ratio of area scanned to total slide area. As the area scanned is an estimate and the scan steps may be irregular, large errors will be introduced.

5. Amphibole grains in the 5 x 2 micron size bracket will not be clearly apparent in 1.590 immersion oil as the test procedure implies. the small grain size reduces relief and many small amphibole grains will be overlooked.

6. The test does not specify the grain size of the sample to be examined. The obvious extreme is to have a 1 mg. sample which consists of a single talc flake and thus has 0% impurities.

7. This procedure is extremely tedious and there is a strong possibility of operator error after 2-3 hours work.

8. At one sample site (768-206), 26 grains were found in a .65mm² area which initially met the FDA criteria as chrysotile, i.e. index of refraction less than 1.574, and a length/width ratio greater than 3. Subsequent investigation showed all 26 grains to be talc, either as fibrous talc or the curled edges of talc flakes. There is a very strong possibility

of misidentification, resulting in overestimates of chrysotile abundances.

Respectfully submitted,

September 21 1973

Warren C. Forbes

Table 1

Average number of dropside grains per sample site (0.64 mm^2).

Sample #815-157.

Percentage of slide covered equals 15%.

Scan #	1*	2	3	4	5
Mean number dropside grains	3.2	5.8	1.4	0.13	5.5
Standard Deviation	3.7	4.9	1.2	—	3.3

* Interval between scans is 4mm.

Area of individual scan site equals 0.64 mm^2

Distance between scan sites equals 0.5mm.

Table 2

Average number of carbonate, quartz, and dropside grains per sample site.
Sample # "Western Treated Talc".

Percentage of slide covered equals 30%.

Scan #	1*	2	3	4	5	6	7	8	9	10
Mean number of grains counted	7.3	11.8	10.0	8.0	5.4	7.0	5.8	4.0	3.8	2.0
Standard Deviation	2.7	4.7	3.5	3.1	3.0	5.0	2.6	2.3	2.0	1.5

* Interval between scans is 1 mm.