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Amphibole Content of Cosmetic and Pharmaceutical Talcs

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Pharmaceutical and cosmetic-grade talcs were examined for asbestiform amphibole content using a new density-optical method. Talcs under the Food and Drug Administration are not regulated as to asbestos content; however, all talcs were well below the level mandated by the Occupational Safety and Health Administration for industrial talcs. Only one was found to contain an amphibole particle size distribution typical of asbestos.

Introduction

In 1973 the Food and Drug Administration (FDA) proposed a regulation on the permissible asbestos content of talc (*1*). This regulation proposed to limit the amount of amphibole minerals to less than 0.1% and chrysotile to less than 0.01%. However, the optical microscopy method proposed was so complicated, lengthy, and subject to error that the proposed method was never finalized. Since then no final ruling has been issued.

The Occupational Safety and Health Administration, on the other hand, has been more rigorous and has instituted regulations despite the lack of methods to carry out the required measurements. One regulation, instituted in 1986, defines amphibole minerals as asbestos if the length to width ratio is 3:1 or greater. Because many nonfibrous cleavage fragments of amphibole minerals have a 3:1 aspect or greater and because there is no good evidence for adverse effects of these particles, a stay has been in affect on this part of the regulation (*2*). The second applicable regulation is the Hazard Communication Regulation (*3*), which applies to all chemicals used in the workplace. Specifically, it requires labeling of substances containing $>$ 1% of a chemical hazardous to health and $>$ 0.1% of a carcinogenic chemical.

Unfortunately, asbestos and amphiboles cannot be measured using currently developed methods to the level of 0.1% in the presence of talc. Some investigators have suggested that tremolite can be measured to that level by X-ray diffraction. But others have shown that the peak intensities vary between nonfibrous and fibrous tremolite (*4*) so that the 0.1% level of detection and measurement is doubtful except in cases where the sample has been spiked so that the exact nature of the tremolite is known. For anthophyllite there is little argument about the fact that detection cannot be made to 0.1%. However, the main problem with using X-ray diffraction for detection of amphibole minerals is that it gives no information about the shape of the particles, and shape is important in view of the uncertainity in the outcome of the asbestos regulation pertaining to nonfibrous amphiboles.

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The talcs that are pharmaceutical grade fall under the domain of the FDA and are therefore nonregulated in regard to fibrous mineral content. In the course of developing a technique to facilitate quantification of amphiboles in talc (*5*), pharmaceutical and high-grade talcs were examined. They were found to have very low amphibole content and, because of this, were extensively used in examining the lower limit of detection of the new method. The purpose of this paper is to describe the results of analyses for content and shape of amphibole mineral fragments in cosmetic and pharmaceutical talc powders of the United States.

Methods

The method proposed by the FDA in 1973 for analysis of talc was an optical procedure as described below (*1*):

> Weigh out 1 milligram of a representative portion of talc on each of two microscope slides. Mix the talc with a needle on one slide with a drop of 1.574 refractive index liquid, and then the other with 1.590 liquid, and place on each a square or rectangular cover glass sufficiently large so that the liquid will not run out from the edge (ca. 18 mm square) and will provide a uniform particle distribution. Fibers counted by this method should meet the following criteria: (i) Length to width ratio of 3 or greater (ii) length of 5 μm or greater (iii) width of 5 μm or less. Count and record the number of asbestos fibers in each 1 milligram as determined from a scan of both slides with a polarizing microscope at a magnification of approximately 400×. In the 1.574 refractive index liquid, chrysotile fibers with indices less than 1.574 in both extinction positions may be present: in the 1.590 refractive index liquid, the other five amphibole types of asbestos fibers with indices exceeding 1.590 in both extinction positions may be present. Check the extinction and sign of elongation for tentative identification. For specific identification of asbestos fibers, make additional mounts in appropriate refractive index liquids, and refer to the optical crystallographic data in the table. A count of not more than 1000 amphibole types of asbestos and not more than 100 chrysotile asbestos fibers per milligram-slide constitutes the maximum limit for the presence of these asbestos fibers in talc. These limits assure a purity of at least 99.9 percent free of amphibole types of asbestos fibers and at least 99.99 percent free of chrysotile asbestos fibers.

The problem with the proposed method is that talc flakes are often oriented vertically or at a sufficient angle that they appear to be needles and thus must be tested for refractive index (Fig. 1). A typical number of such particles is five per field of view. This

FIGURE 1. Talc flakes in 1.584 refractive index liquid. Note that there are particles in this field that have aspect ratios greater than 3:1. Width of view 0.13 mm.

means that some 20,000 particles would need to be examined in a typical case. In additon, chlorite is often present and when on edge must be examined in two extinction positions. This is clearly beyond what could be expected of any sane microscopist for a routine analysis. Since no other procedure has been developed as an alternative, a compromise has been to count 100 fields of view (FOV). In this way one need only examine about 500 particles in detail.

Because 500 particles is still a lengthy process, a more rapid and equally accurate method has been developed based on concentrating the amphibole particles by density difference. Figure 2 illustrates that there is a distinct break in density ranges between talcs and amphiboles. A heavy liquid of intermediate density is used, either Klein's (cadmium borotungstate) or Clerici's (thallium formate-malonate) solution. Experimentation showed that a heavy liquid of density 2.810 gives good separation even though values given in the literature and shown in Figure 2 would suggest that the density should be slightly higher. Because the density difference between particles and liquid is small, to get separation in a reasonable length of time a microcentrifuge is used with tubes containing 1.5 mL liquid. The height of the liquid column is, in this case, about 10 mm.

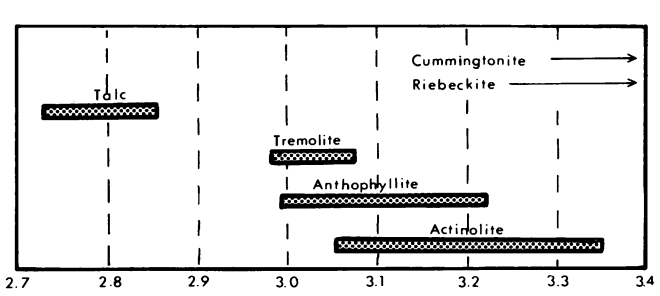

FIGURE 2. Specific gravities of talc and amphibole (6).

The general procedure involves weighing about 60 mg sample into a microcentrifuge tube and adding heavy liquid of density 2.810. After these are mixed, the tube with sample is placed in a vacuum for 3 min to remove the small bubbles adhering to the particles. After centrifuging the sample for 10 min at 7000 rpm, the heavy particles are removed from the bottom of the tube with a micropipette.

The counting of particles can be done either on a membrane filter (Nuclepore, 1.0 μm pore size) which has been placed on a microscope slide or as particles directly on the glass slide. In the first case, the heavy liquid with sample is forced through a membrane filter followed by distilled water to clean out the heavy liquid. The filter is then placed on a glass slide while wet. When dry, 1.584 refractive index liquid is placed on the filter followed by a cover glass. The photographs shown in this paper are of particles on filters.

The second case, particles directly on the microscope slide, requires transferring the heavy particles and some of the heavy liquid to a second centrifuge tube. Distilled water is added and the sample centrifuged. The liquid is pipetted off and more distilled water added. This is repeated several times to clean out the heavy liquid. Finally, the particles with several drops of water are transferred to a glass microscope slide. The advantage of this procedure is that any refractive index liquid can be used, whereas, in the former case, the refractive index is constrained by having to match the index of the membrane filter (either 1.584 or 1.625). The 1.584 value is good for analyzing amphiboles in talc, but the centrifuge method described has application to other mineral combinations, such as talc-quartz. With other combinations, refractive indices other than the two exhibited by the membrane filter may be more appropriate.

The particles are counted in 20 FOV. Being concentrated from 60 mg or more of sample, one will see more amphiboles than in 100 FOV using the old method. The number of amphibole particles per milligram (ppmg) is calculated:

$$\text{ppmg} = \text{amphibole particles/mg} = \frac{(\text{number of amphibole counted/number FOV counted}) \times \text{total number FOV}}{(\text{efficiency}) \times (\text{number of mg of sample})}$$

Efficiency of the spin-down is determined experimentally. For more details of the method see Blount (5).

Figure 3 illustrates the results obtained when testing the method using known mixtures. Because it is difficult to measure and mix in very small weights of amphibole, a sample containing 2% tremolite in talc was mixed with pure talc to make mixtures containing very low percentage values of tremolite. For example, sample A (Fig. 3) consisting of 0.06% tremolite was made by weighing 58.9 mg of pure talc with 1.7 mg of talc containing 2% tremolite (1.7 mg/60.6 mg $\times$ 2% = 0.06%). It is not necessary to make a homogeneous mixture since the entire sample was used in the experiment. Also, the talc containing amphibole was put in the tube second in order not to give the amphibole any "head-start" in sinking to the bottom.

The centrifuge method was also tested with a commercial talc. 100 FOV were counted in ten 1-mg samples according to the FDA procedure for amphibole. This was compared with 20 FOV counts on 60-mg centrifuge samples (Fig. 4). The agreement is quite good. The standard deviations were determined in two

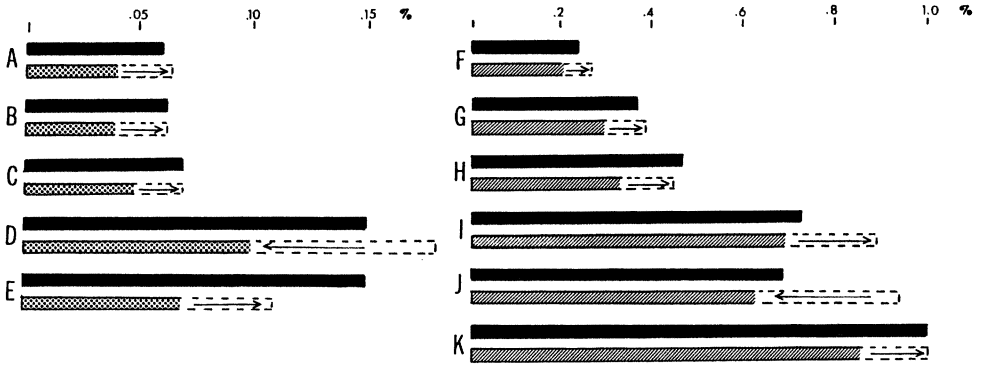

FIGURE 3. Percent tremolite in talc as determined by the centrifuge/optical method (shaded bars) compared with that actually present in experimental mixtures (black bars). The dashed part of the shaded bars indicates +2 SD (right arrow) or −2 SD (left arrow).

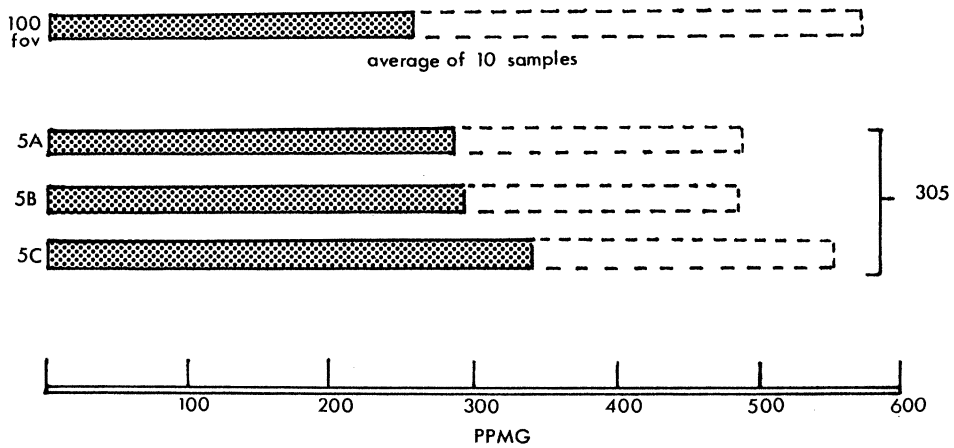

FIGURE 4. Comparison of traditional (100 FOV) count with centrifuge/optical count of same talc. The three lower bars indicate the values in particles/mg obtained by the centrifuge/optical method for three 60-mg samples. The top bar is the average of ten 100 FOV (traditional method). The dashed part of all the bars is +2 SD.

ways: for the traditional method by calculating in the usual way from multiple analyses and for the centrifuge method by means of the Poisson distribution from single counts. Standard deviations are high for the centrifuge method because of the very few particles counted. These could be decreased by making a larger count, but isnce the purpose of the study was to find a reasonably rapid method of monitoring amphibole content of talcs, larger counts were not generally made.

Results

High-grade talc products from five deposits in Montana, three in Vermont, and one each in North Carolina and Alabama were examined using the centrifuge/optical method. In addition, four talcs from outside the U.S. but available in the U.S. market were included in this study. Talcs from other districts in the U.S. were examined, but these talcs had grades with less stringent requirements and are not included in this report.

Results of particle counts are shown in Table 1. The FDA has equated 0.1% with 1000 particles per milligram. In order for amphibole particle content to be less than 0.1%, 20 or less particles must be observed in 20 FOV (5). Since all were well below this value, more extensive counts were not generally made.

It should be borne in mind that the 0.1% indicated is percent by count and not percent by weight or volume. The question of the validity of this relation has been considered (5). Briefly, the relation implies (1000 amphibole particles)/(1,000,000 total particles). Counts of total particles per milligram of talc have shown that 1 million particles per milligram of talc is a low value. Most show at least 2 to 3 times this number. The only exception was a baby powder with very large flakes which showed 0.4 to 0.8 million particles per milligram. It was not clear, however, whether this was a true value or due to the problem of counting where large, flakey particles could potentially hide other particles even in the most carefully prepared samples. Using 1000 particles/mg = 0.1% would, in most samples, give a percentage value on the high side and in this sense be a conservative answer.

The counts shown in Table 1 were made of regulatory fibers i.e., aspect ratio > 3:1. In some samples there were as many or more nonregulatory particles of amphibole as regulatory fibers. The shape of the amphibole varies greatly and seems to be highly characteristic of each deposit. In Table 1, the particles having aspect ratios less than 6:1 are designated cleavages and prismatic pieces. Those greater than 6:1 and less than 15:1 are labeled

Table 1. Counts of regulatory fibers in processed talcs.

Sample	Counts, particles/mg	SD	Particle shapes	Particles/FOV[a]
A	38	25	Cleavages	3/100
B	ND[b]			0/20
C	ND			0/20
D	< 25[c]		Cleavages	0/20
E	ND			0/20
F	ND			0/20
G	ND			0/20
H	17	17	Cleavages and needles	2/20
I	226	59	Needles and fibers	17/20[d]
	283	100	Needles and fibers	8/20
	291	98	Needles and fibers	9/20
	341	108	Needles and fibers	10/20
	102	51	Needles and fibers	3/20
J	25	14	Cleavages	1/20
	27	27	Cleavages	3/20
K	25	25	Cleavages	1/20
L	< 10[c]		Needles	0/20
M	39	21	Cleavages and fibers	4/20
N	25	17	Prismatic pieces	3/20
O	ND			0/20

[a]FOV, fields of view.
[b]ND, none detected.
[c]No particles seen during a 20 FOV count, but some particles could be seen during a random scan of the filter. Value shown is the lower limit of detection.
[d]Large sample used for this analysis (305 mg).

"needles." The remainder, which are greater than 15:1, are labeled "fibers." Whereas in many samples only a few particles were counted as shown in the right-hand column of Table 1, it should be remembered that even if only one particle was present in 20 FOV that about 300 were present on the slide. Because of the low interference by talc particles, these were seen so that it was easy to get a sense of the general particle shape.

The shape distribution of particles for several samples was determined. Figure 5 shows a photograph of a particle of tremolite in sample *I*. The particle is composed of fibrils. The length and width of 100 amphibole particles in this talc were measured. The resulting distribution of aspect ratios is shown in Figure 6. The results when compared with the aspect ratios determinded for tremolite asbestos with SEM by Campbell et al. (*7*) show sample *I* has a distribution similar to asbestos. Sample *M* was analyzed in the same way (Figs. 6 and 7). The graph of aspect ratio verses percent is compared with Campbell's results for nonfibrous tremolite. The similarity of the curves indicates that the tremolite in this talc is of the nonfibrous type.

Because the fractions produced by centrifuge are not generally pure after a single spin-down, a sample containing a variety of particle shapes was tested to see if the aspect ratio distribution results become biased in favor of larger, chunky grains (low aspect ratio) over small, long grains (high aspect ratio). The sample used contained 6.5% tremolite, a sufficient quantity that the traditional optical method could be used to compare with the centifuged sample. The resulting aspect ratio distribution curves (Fig. 8) do not show significant differences. With the traditional method, 69% of the amphibole particles have an aspect ratio of 3:1 or greater, whereas for the centrifuged samples the value is 64%, a variation which is not significant. The differences shown for 5:1 and 10:1 are probably due to the limited number of particles measured, in this test 100 particles in each sample.

Despite the similarity of the curves, the mean length and mean width of the amphibole particles measured using the centrifuge method are greater than those obtained using the traditional method (Table 2). Analysis of size distribution indicates that the proportion larger than 15 μm is greater in the centrifuged sample. This difference in dimension distribution does not appear, however, to affect the aspect ratio distribution. Other investigators have found that as particles increase in length, the aspect ratio shifts to higher values (*8,9*). This applies to both asbestos and nonasbestiform amphiboles, so presumably the effect of centrifuging down longer particles would be to force the aspect ratio distribution peak to higher values.

Discussion

The high-grade talc powders are uniformly low in amphibole content. Counts obtained were 0 to 341 particles/mg. Indeed, talc from some districts appears to be completely free of such minerals. In those containing amphibole minerals there are two distinct types: cleavage type and asbestos type. These two types show distinctly different aspect ratio distributions as demonstrated in Figure 6 (samples *I* and *M*). The aspect ratio difference probably accounts in a large part for the higher particle count per milligram of sample *I* compared with the others which show cleavage fragments. It is easy to see that the number of particles showing greater than 3:1 aspect ratio would be greater in the former case even if the total number of particles of amphibole were equal. This observation reinforces the original decision to count particles visually rather than attempting to use X-ray diffraction. It is not wise to try to convert information on dimensions to percent by weight or volume because a few very large particles can drastically affect the resulting value. Campbell et al. (*8*) discuss this in some detail.

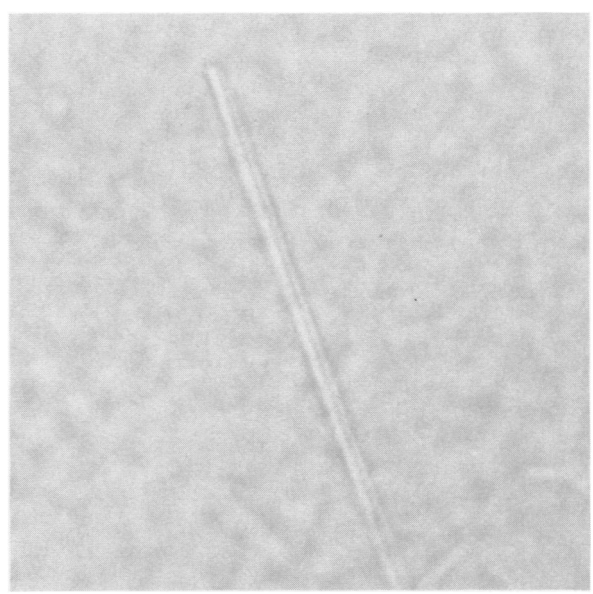

FIGURE 5. Particle of amphibole in centrifuged sample *I*. Width of view 0.07 mm and 1.584 refractive index liquid. Particle is on a membrane filter.

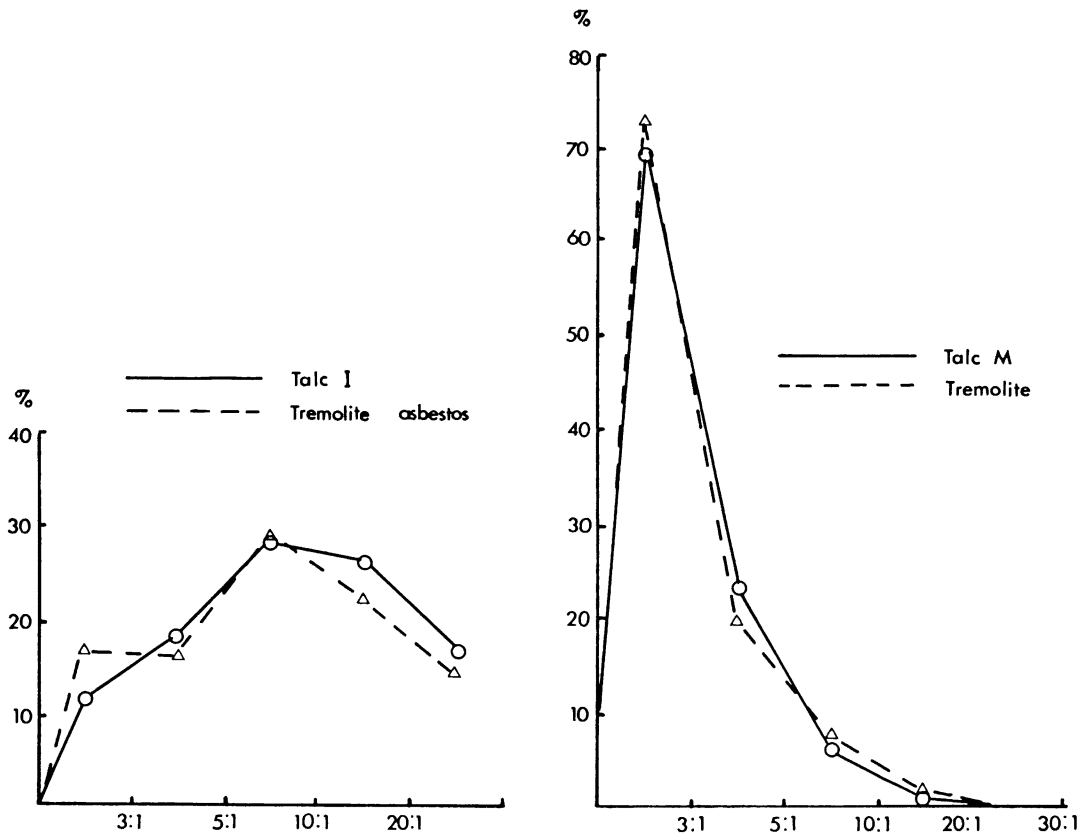

FIGURE 6. Percent amphiboles in each aspect ratio group for talc sample *I* (left) and *M* (right) compared with tremolite asbestos (*7*) and tremolite (nonasbestiform) (*7*).

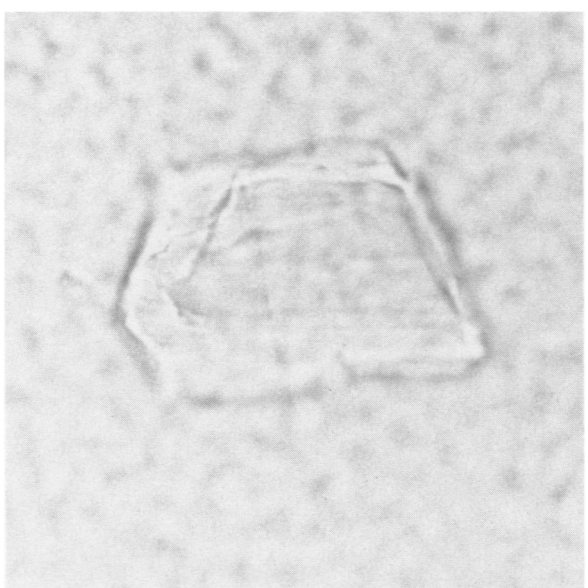

FIGURE 7. Particle of amphibole in centrifuged sample *M*. Width of view 0.07 mm and 1.584 refractive index liquid. Particle is on a membrane filter.

FIGURE 8. Percent amphiboles in each aspect group for a sample handled in two ways: solid line shows results using traditional method and dashed line shows results using centrifuge method. Dimensions of 100 particles measured for each curve.

Table 2. Summary of size and aspect ratio data used to construct Figure 8.

	Size, %		
Method	5–10 μm	10–15 μm	≥ 15 μm
Traditional	57	26	16
Centrifuge	33	27	38
	Mean length, μm	Mean width, μm	Mean aspect ratio, μm
Traditional	12.5	3.0	4.4
Centrifuge	17.5	4.7	4.6

Further, the results from this study demonstrate the utility of the centrifuge method not only for obtaining a count of particles, but also for obtaining information on the shape of particles in a population. It should be emphasized that the aspect ratio curves determined for samples *I* and *M* would have been virtually impossible to obtain using the FDA procedure. The determination would have required examining over 3000 FOV. As indicated previously, many talc flakes on edge appear to be fibers and must be examined during such a scan, making the whole job impossibly tedious.

Finally, even in those cases where one may wish to use the standard 100 FOV count, the centrifuge method offers a way to screen samples between those times when a more lengthy count is made, and it permits a double check of values so determined. In addition, the tendency to bring down a disproportional number of larger particles has the advantage that with true asbestiform amphiboles one generally sees some particles showing bundles of fibrils which removes any doubt about the nature of the amphibole.

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