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Alice M. Blount, Ph.D.

Mineralogist

GMW

April 23, 1998

M. Raymond Hatcher
MEHAFFY & WEBER
2615 Calder Avenue
P.O. Box 16
Beaumont, Texas 77704

RECEIVED

APR 27 1998

MEHAFFY & WEBER
BEAUMONT, TEXAS

Dear Mr. Hatcher:

According to your letter of March 31, 1998, I have written and enclosed a report on the occurrence, regulation and up-to-date scientific view of asbestos, amphiboles and "intermediate" fibers. I have also enclosed copies of my 1990 and 1991 papers, one of which I am sure that you already have. The 1991 paper was written because I became aware that it was a common opinion among industrial hygienists that industrial talcs were better than pharmaceutical and cosmetic talcs because there was a regulation for the former and not for the latter. I knew that this was not the case and wanted to set the record straight.

Although my papers report an improved method for analysis, the determinations for the sample labeled I (Johnson & Johnson's Vermont talc) have been done by the traditional methods as well (see Table 2, page 567 in the 1990 paper). As I told you, I believe that Johnson & Johnson's Vermont talc contains trace amounts of asbestos which are well below those specified by OSHA. It should be noted that the proposed FDA regulation, which was never finalized, also specified the same 0.1% limit for amphibole asbestos as OSHA.

I may be away for short periods during the coming weeks, but I do check for messages on my work phone at the number you have been using.

Sincerely yours,



Alice M. Blount, Ph.D.

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PROCESS MINERALOGY IX:

*Applications to Mineral Beneficiation,
Metallurgy, Gold, Diamonds,
Ceramics, Environment and Health*

Proceedings of International Symposium on Applied Mineralogy (MAC-ICAM-CAM) held at Montreal, Quebec, Canada on May 14 to 17, 1989, and of the Process Mineralogy Symposium held at Las Vegas, Nevada, February 27 to March 2, 1989. The MAC-ICAM-CAM symposium was held during the annual meeting of the Geological Association of Canada and Mineralogical Association of Canada, and the Process Mineralogy symposium was held during the annual meeting of the Metallurgical Society of the American Institute of Mining and Metallurgical Engineers.

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A Publication of

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DETECTION AND QUANTIFICATION OF ASBESTOS AND OTHER TRACE MINERALS
IN POWDERED INDUSTRIAL-MINERAL SAMPLES

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Abstract

In 1983, a final ruling of the U.S. Occupational Health and Safety Administration (OSHA) specified that chemicals containing >0.1% of a carcinogenic substance or >1% of a substance hazardous to health must be labelled. Amphiboles of the fibrous or asbestos-type must be quantified at levels of 0.1% whereas the status of non-fibrous types is not finally determined at this time. Tremolite and actinolite can possibly be measured to 0.1% by XRD, but anthophyllite cannot be so measured. Recently the International Agency for Cancer Research (IARC) has designated quartz as a suspected carcinogen, and its quantity must be measured to the 0.1% level. Thus, it has become increasingly important to be able to detect and quantify minerals in bulk samples down to low or "trace" levels. It has been found that minerals needing to be quantified can often be concentrated by microcentrifuge and counted by means of polarized-light microscopy. For example, amphiboles in talc samples can be separated by using a heavy liquid (thallium formate-malonate) of density 2.810. Talc floats, and amphiboles sink. After centrifuging, the denser particles are removed from the bottom with a Pasteur pipette and placed in a clean centrifuge tube. Distilled water is added to the tube containing the denser minerals and the tube centrifuged again. After several washings with distilled water, the sample is removed along with a drop of water to a clean glass slide. A drop of 1.584 refractive index liquid and a cover glass are placed over the dried sample. The slide is scanned for particles greater than 5 μm in length and having an aspect ratio greater than 1:3. These are checked for refractive index, sign of elongation and extinction angle to differentiate them from talc particles on edge and other minerals. Amphiboles have all indices greater than 1.584, and talc has all indices equal to or lower than this value. Alternately the heavies at the bottom of the centrifuge tube can be filtered through a nucleopore filter. After the filter has been washed with distilled water and dried, a 1.584 refractive index liquid renders the filter transparent in two of the four extinction positions so that a count of amphibole particles can be made directly on the filter. The refractive index-density method which is so useful for talc powders should be equally useful for other nonsoluble industrial minerals.

Process Mineralogy IX
Edited by William Petruk, Richard D. Hagni,
Susanne Pignolet-Brandom, and Donald M. Hausen
The Minerals, Metals & Materials Society, 1990

Introduction

By the late 60's and early 70's the health community became firmly convinced of the danger of asbestos to workers involved in mining and manufacturing, and the United States government agencies moved to regulate this substance in the workplace. The airborne fiber limit was first set at 5 fibers/cm³ in 1972, then at 2 fibers/cm³ in 1976, and finally to 0.2 fibers/cm³ in 1986 (1). In addition, bulk minerals and chemicals were subject to a "Hazard Communication" regulation, which specified that a substance must be labeled if it contains >1% of a chemical hazardous to health or >0.1% of a carcinogenic substance (2).

Asbestos, tremolite, actinolite and anthophyllite are regulated at 0.1% according to the 1986 OSHA regulation. This has proven to be a problem in industrial minerals, such as talc, because this limit is below present detection levels by X-ray diffraction. It may be possible with very time-consuming step-scans to detect tremolite to this level, but this is not possible with anthophyllite. In addition, tremolite, actinolite and anthophyllite occur in fibrous and non-fibrous forms, and X-ray diffraction does not distinguish between these forms. At the time of this writing there is a court stay on the part of the Final Rule of 1986 covering non-fibrous tremolite, actinolite and anthophyllite (3).

In addition in 1987, the International Agency for Research on Cancer (IARC) determined silica (quartz, tridymite and cristobalite) to be a suspected human carcinogen (4). This automatically triggered the provisions of the 1983 Hazard Communication, and all industrial minerals containing >0.1% of free silica must be labeled as containing a carcinogenic substance.

From this brief outline, the increasing importance of detection and measurement of minerals at trace levels is apparent. The writer considers these to be "trace minerals" as they are analogous to "trace elements." Under present government regulations, several hundred or less particles are counted in a million particles. A method to do this has been developed and is presented here. It consists of standard techniques of optical and X-ray analysis in combination with heavy liquid separations.

Whereas an individual mineral species has variable density because of chemical substitutions, it is often possible to find a density which will effectively separate minerals of interest. Fig. 1 shows that talc has a distinctly different density from the amphibole minerals of concern. The major part of our effort has been devoted to experimentation with talc containing amphibole. This paper will discuss experimentation with talc and amphibole mixtures although the method is applicable to other powdered industrial minerals.

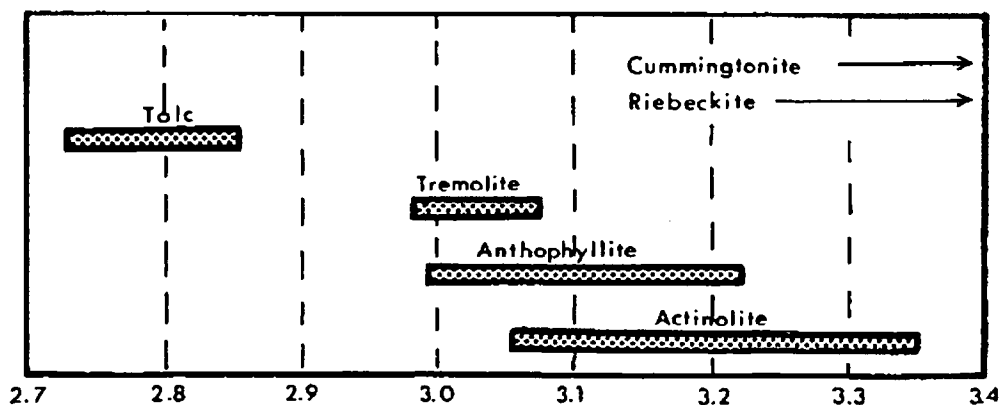


Figure 1 - Chart showing range of density for talc and amphibole minerals (10).

Description of Analytical Methods for Talc-Amphibole

Detection of amphiboles in powdered talc is very difficult. Attempts were made to measure tremolite to 0.1% by using X-ray step diffraction scans with long counting times on each step. Since very low counts are obtained and since the intensity of the X-ray diffraction peak is dependent upon slight changes of chemistry due to substitutions of elements in the mineral structure and to the fibrous or nonfibrous condition of the mineral (5), this method was found to be unsatisfactory. For anthophyllite, the major peaks have such low X-ray intensities that the method was not attempted. The only viable method of analysis has been an optical particle count such as that proposed in 1973 by the Food and Drug Administration (6). Although the original method proposed analyzing an entire 1 mg sample, most users of the method scan 100 fields of view (f.o.v.). The procedure is stated below:

"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 μ m or greater (iii) width of 5 μ m or less. Count and record the number of asbestos fibers found in each 1 milligram as determined from a scan of both slides with a polarizing microscope at a magnification of approximately 400X. 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 talc at least 99.9 percent free of amphibole types of asbestos fibers and at least 99.99 percent free of chrysotile asbestos fibers."

A major difficulty with this method is that very many talc flakes are on edge or at such an great angle that the edge of the flake is seen. These particles then show an aspect ratio greater than 3:1 and must be tested for refractive index by checking the Becke line often in both extinction positions. The procedure is very time-consuming for 100 f.o.v. Using a standard petrographic cover glass there are generally about 10000 f.o.v. per immersion mount (1 mg of sample). Thus to be sure that less than 0.1% amphibole was present the count needed to be 5 particles or less (5 plus two standard deviations = 9.47) in 100 f.o.v.

Several investigators have pointed out the utility of using density along with other optical properties for identification of unknown minerals using the polarizing microscope (7, 8, 9, 10) Their approach is to observe the sink or float characteristics of particles in immersion liquids under the microscope. The approach used in this study was to separate the particles of interest using heavy liquids and a microcentrifuge.

Selection of Heavy Liquid and Density

Many heavy liquids are available to the mineralogist. Whereas there is some risk associated with such substances (11, 12, 13, 14), with proper care they can be handled safely. An advantage of the method used here is that only small quantities of the liquid are used. An important consideration in selecting the heavy liquid used in this investigation was the desire to be able to remove the liquid from the particles by washing with distilled water. Clerici solution (thallium formate-malonate) and Klein's solution (cadmium borotungstate) diluted with water were selected. Both proved satisfactory

but Clerici solution is less expensive.

To determine mineral densities, talc and amphiboles were obtained from all the major talc producing regions of the United States and tested. The lowest density at which talc floated was determined by slowly increasing the density of Clerici solution by adding, drop-by-drop, more dense liquid to a less dense liquid which contained talc. At the point at which the talc floated, the index of refraction of the liquid was determined using an Abbe Refractometer. The density was determined using a density-refractive index chart (15) (Fig.2). A similar procedure was followed for amphiboles except that the proper density for the amphibole to sink was determined. These tests indicated that the best density for a heavy liquid to separate talc from amphibole was 2.810.

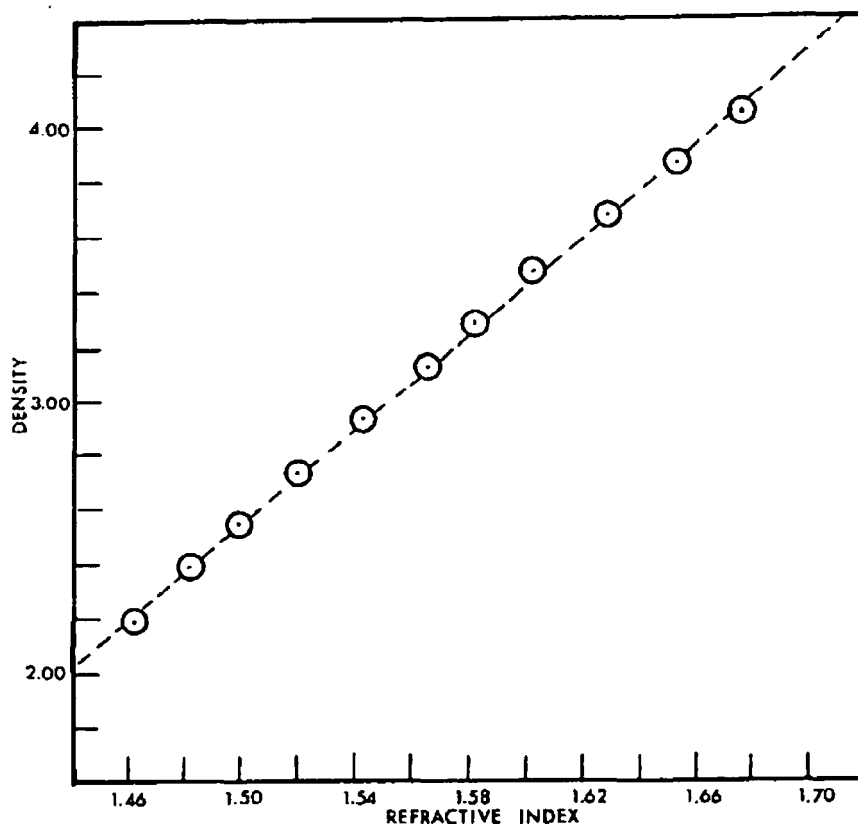


Figure 2 - Density-refractive index graph for Clerici solution at 23° C (15).

To avoid having to continuously use the refractometer to adjust the density, commercially produced density standards adjusted to 2.800, 2.810 and 2.820 were purchased. The liquid could then be conveniently adjusted to 2.810. The adjustment is easier if "stock solutions" are made up, one with slightly higher density and one with slightly lower density. Using such solutions eliminates the rapid change of density that occurs with the addition of a single drop of distilled water or very heavy liquid. It is important that the solution is well mixed after each addition of more or less dense liquid. If not well mixed, density stratification occurs.

Selection of Centrifuge

Once the proper density heavy liquid has been selected, the mineral particles are separated by means of a centrifuge. Several points need to be considered when selecting a centrifuge. These are: (1) In order to separate very small particles in liquids which differ from them only slightly in density, it is important for the particles to move through a short column of liquid or the centrifuge time will be impractically long. A microcentrifuge with 1.5 ml tubes was, therefore, selected. (2) The centrifuge must have a variable speed setting on the spin. The reason for this requirement is that microcentrifuges are designed to handle low-density liquids (< 1.2 g/ml). Heavy liquids at high spin speeds exert forces which are great enough to break the centrifuge tubes and tube holders which causes major damage. When using a variable speed centrifuge it is important not to run the machine at top speed while using heavy liquids. The maximum safe speed can be determined using the following approximation (16):

$$\text{RPM}_{\text{max}} = \text{rated rpm} \cdot \sqrt{1.2/D}$$

*information supplied with centrifuge and dependent upon tube type and capacity

D is density of heavy liquid

For the present work:

$$\begin{aligned}\text{RPM}_{\text{max}} &= 13,250^{**} \sqrt{1.2/2.810} \\ &= 8,659\end{aligned}$$

** 1.5 ml polypropylene tube

Sample Preparation

60 to 100 mg of talc are weighed into a disposable centrifuge tube. About 1.2 ml of heavy liquid adjusted to 2.810 is added, and the sample with liquid is mixed by shaking. The tube is then placed in a bell jar which is evacuated with a vacuum pump for three minutes. This removes air bubbles adhering to the particles. The charged tubes are placed in a centrifuge and spun for 8 minutes at a speed of 7000 rpm.

The amphiboles should sink leaving the talc floating, so that the particles that have sunk are of interest. The liquid and particles lying at the bottom are removed with a micropipette and transferred to a syringe. The syringe is attached to a filter holder, and the liquid with heavies is filtered using a Nucleopore filter (pore size 1.0 μm) which is held in a plastic filter holder. About 40-50 ml of distilled water is washed through the filter to remove the heavy liquid. The filter (plus residue) is then carefully transferred to a clean glass slide with a tweezers and permitted to dry.

The amphiboles can be examined without removing them from the filter because the Nucleopore filter has an index of refraction of 1.584 in two of the four extinction positions. This is the proper index of refraction to distinguish between talc and amphiboles. Talc has indices less than or equal to 1.584, whereas amphiboles have indices greater than this value. A drop of 1.584 refractive index liquid renders the Nucleopore filter transparent when scanned in the matching extinction position. The fields are scanned for particles having an aspect ratio > 3:1. These can then be checked for sign of elongation and relative index of refraction in the usual way. The determination of extinction position is slightly different when looking at particles on an anisotropic filter. Since the filter is anisotropic it will change colors on rotation. The extinction position of a particle is the angle at which it matches the color of the filter. Occasionally a particle will appear dark or the same color at all rotations. This simply indicates that the extinction position of the particle and the filter are the same.

Two comments should be made. First, it is easier to do an optical count if the carbonate minerals are removed from the talc by acid treatment before analysis. Calcite will float in 2.810 liquid but magnesite and dolomite will sink. Second, the heavy liquids that are left in the centrifuge tubes can be reused by filtering them through a filter with 0.4 μ m pores using a syringe and plastic filter holder as used before. Also heavy liquids if left standing for a period of time crystallize and get cloudy. The crystals can be removed in the same way as the heavy minerals.

Analytical Results

To determine the level of amphibole contamination of a sample the efficiency of the spin-down for the amphibole particles and the number of talc particles in our sample must be known. Before examining results from actual processed talc samples, these two factors are considered.

Efficiency of Spin-down

In order to determine the percentage of total amphibole that would be brought down during a single centrifuge run, talc-dolomite samples were analyzed. Dolomite has a density of 2.86 which is comparable to that of the amphiboles (see Fig. 1). Mixtures of talc plus dolomite at various percentage concentrations could be spun down, transferred to a filter as previously described and the filter plus sample weighed. After putting the filter (plus sample) back in the holder and washing with acid followed by distilled water, the sample plus filter is reweighed. The weight difference indicates the amount of dolomite brought down. Fig. 3 shows the results of these analyses. The bottom line on the graph shows the values obtained when a micropipette removing 0.15 ml was used and the top when 0.6 ml was removed. The top line shows more scatter in the data, so most of the results reported in this paper are obtained with a 0.15 ml sample and using an efficiency factor of 14%.

The efficiency results of 14% and 33% show that the proposed method could be improved by finding a better method of removing the sample from the centrifuge tube. Several investigators have used liquid nitrogen to freeze the tube contents partly (17) or completely (18). These methods were not tested; however, it was found to be advantageous in some cases to put the heavies from two tubes onto a single filter to get a higher fiber count.

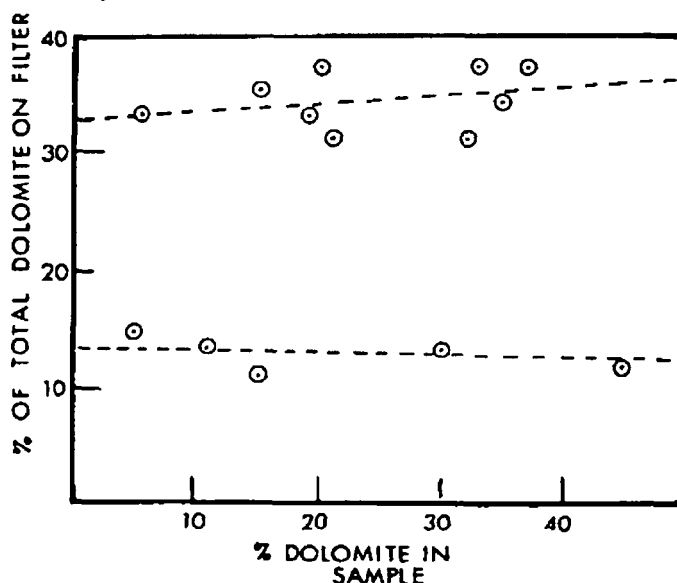


Figure 3- Graph showing the percentage of dolomite caught on the filter under two different experimental conditions. See text for further explanation.

Number of Amphibole Particles in One Milligram

The 1973 Food and Drug Administration's proposed method is based on the assumption that a one milligram sample of talc contains one million particles.

1000 equals 0.1%

$$1000/.001 = 1 \times 10^6 \text{ particles}$$

A quick calculation using weight and volume shows that one million particles per milligram is probably a conservative estimate. In OSHA's regulations for airborne dusts where limits are in Mppcf (million particles per cubic foot) and in Mg/M^3 (milligrams per cubic meter), the two values when equated show that a one milligram sample is considered to contain from 5 million to 200 million particles depending upon the substance. While one may think of dusts as having very small particles, respirable dust is, for the most part, greater than $5 \mu\text{m}$ (19). It is different from processed talc, however, in lacking the large particles $> 10 \mu\text{m}$ which represent a disproportionally large percentage by weight of processed talcs. Since one million particles per 1 mg sample is considered conservative, percentages of fibrous particles will calculate on the high side rather than the low side, so this value is used.

ppmg = fibrous particles per mg =

$$\frac{(\text{number of fibers counted})/(\text{number of f.o.v.}) \times 5740 \text{ f.o.v.}^*}{0.14 \times (\text{number of mg of sample})}$$

*number of fields of view needed to cover entire filter

Percent fiber then can be determined:

$$\% \text{ fiber} = (\text{ppmg}/1 \times 10^6) \times 100\%$$

The results in this paper are generally given in ppmg (particles per milligram).

Counts of total talc particles per milligram can be made on the actual sample. This is done by preparing a standard one milligram sample on a glass slide. One drop of distilled water or methanol is added to the powder (not on top but along side), and the liquid and powder mixed with a small spatula. The mixture is smeared evenly over the glass slide and permitted to dry. The slide is left uncovered so that the talc particles stand out in high relief relative to air. Counts are usually done of particles in a part of the field of view using a grid eyepiece to define the area.

Results of counts as just described have shown that processed talcs generally contain $0.9\text{-}20 \times 10^6$ particles/mg in the optical size range. The only exceptions are some baby/body powders with very large particles. These are often very difficult to count because the flakes are very thin and tend to lie on top of or overlap other flakes making it necessary to exercise some judgement as to whether one is observing separate particles or a step on the surface of a single particle. Obviously some particles would be covered completely by others and not seen at all. Values on these talcs are $0.4\text{-}0.8 \times 10^6$ particles/mg.

Analytical Results Obtained with Mixtures

A number of problems were encountered in testing the proposed method. Foremost is the question of whether pure starting material would adequately represent the minerals in actual processed talc samples. In addition is the problem of how to mix samples so that the very small percentage of particles representing the contaminant are randomly distributed in the sample. To avoid these problems, an actual processed talc known to contain amphibole was chosen and a talc without such minerals. The percent amphibole was determined in the former by performing standard 1 mg -100 f.o.v. counts using traditional methods on eight separate preparations. This yielded a value $2.19 \pm 0.97\%$. Mixtures

were then prepared by first weighing pure talc powder in the centrifuge tube and then adding a small amount of 2.2% talc. The procedures described in the section "Sample Preparation" then were followed. 20 fields of view were counted. The results are indicated in Table 1. Except for samples e and h, all are within 2 standard deviations of the actual value. It should be noted that actual unknowns are generally spun-down and analyzed in triplicate, so that any discrepancies can be detected. Discrepancies can be caused by preparation problems such as the gaskets not being tight on the filter holders so that part of the sample escapes. Fortunately this is generally obvious before reaching the counting stage. In this test, the samples were not run in triplicate because of the difficulty of mixing a sample to put into three centrifuge tubes. If differences had been found during such an experiment it would have been more likely due to mixing problems than to counting procedures.

Table 1. Mixtures of talc contain 2.2% tremolite with pure talc.

No.	actual	measured (σ)
a	0.06 %	0.04 % (0.01)
b	0.06	0.04 (0.01)
c	0.07	0.05 (0.01)
d	0.15	0.18 (0.04)
e	0.15	0.07 (0.02)
f	0.24	0.20 (0.04)
g	0.37	0.30 (0.05)
h	0.47	0.34 (0.06)
i	0.73	0.70 (0.10)
j	0.74	0.94 (0.16)
k	1.03	0.86 (0.09)

Actual Commercial Talc Samples

A sample of commercial talc known to contain very small quantities of amphibole was tested with the traditional 1 mg-100 f.o.v. technique and the centrifuge technique. Ten 100 f.o.v. counts on separately prepared mounts were counted. The results indicated 259 ppmg (range for 95% confidence is 0-573 ppmg). The results obtained using the centrifuge are shown on Table 2.

The standard deviation for particle counts can be calculated using Poisson's distribution $\sigma = (N)^{0.5}$ (20). The standard deviations listed under the centrifuge method in the Table are calculated in this way. Poisson's distribution is the best that can be achieved if the variation is due to random particle distribution on the slide and there is no other systematic sources of error.

Fig. 4 shows the appearance of a typical amphibole in the talc from which the results shown in Table 2 were obtained. It is made up of fibrils. Note that the photographs in this paper generally show very large particles. This is due simply to the fact that they are easier to photograph and are selectively chosen for this reason.

Another talc known to contain amphibole is shown in Fig. 5. It displays the reason for evaluating talc optically rather than using X-ray diffraction. The volume of the amphibole particles in this talc is much greater than the volume of amphibole in the talc of Fig. 4, and XRD would yield a much larger value even if it contained the same number of amphibole particles as the previous example.

The particles shown in Figures 6-9 are from talcs which, although periodically analyzed by SEM, were not believed to contain amphiboles. Amphibole particle counts are extremely low and generally there is no indication of particles being made up of fibrils. The figures show typical particle shapes for each talc type shown. No amphibole particles were found in the talc shown in Figures 8 and 9 during a count of 20 random fields of view; however, particles were noted during a quick scan



Figure 4 - Amphibole particle ($65\ \mu\text{m}$ in length) in talc which measured $259\ \text{ppmg}$ (± 317).



Figure 5 - Tremolite particles ($69\ \mu\text{m}$ in length) in talc. This sample measured $833\ \text{ppmg}$ (± 336).

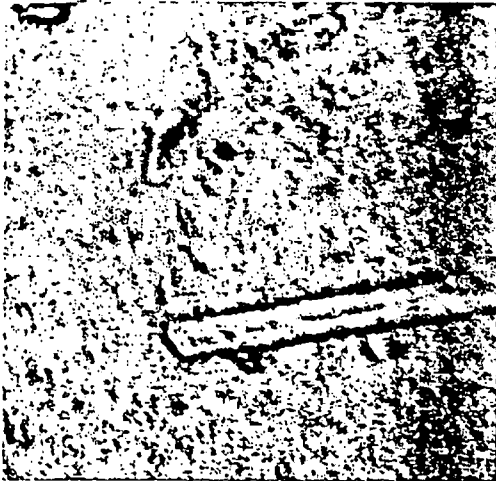


Figure 6 - Amphibole particle (length $63\ \mu\text{m}$) in talc which measured $88\ \text{ppmg}$ (± 24).

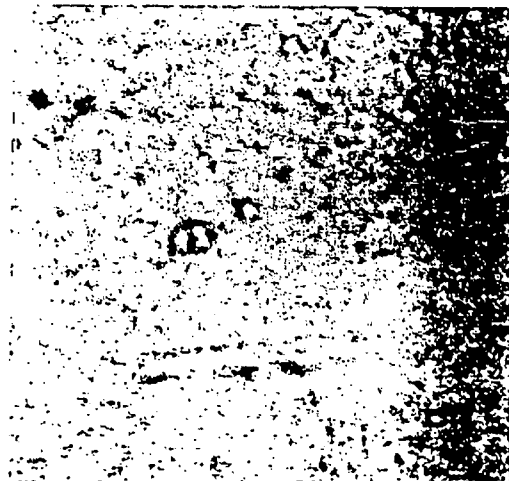


Figure 7 - Amphibole particle ($40\ \mu\text{m}$ in length) in talc which measures $25\ \text{ppmg}$ (± 28).



Figure 8 - This talc showed no amphibole particles during a standard 20 f.o.v. scan. This indicates <25 ppmg. Particles could be found by a quick scan or filter. This one is 22 μ m in length.



Figure 9 - Similar to the talc shown in Figure 8, no amphibole particles were detected during a routine scan. This indicates < 10 ppmg. The particle shown has a length of 66 μ m.



Figure 10 - Amphibole counted on a Nucleopore filter. The count is 102 ppmg (± 102). Length of particle is 133 μ m.

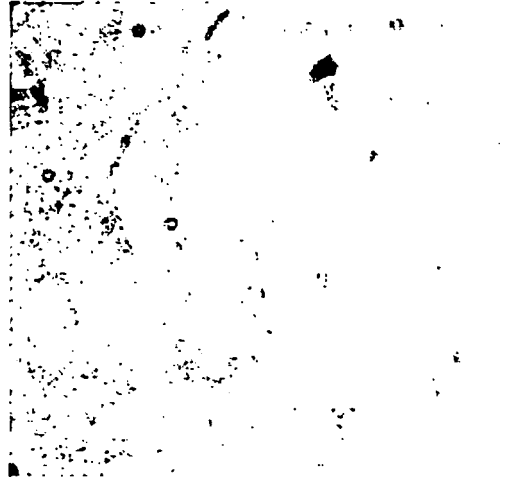


Figure 11 - Same talc as Figure 10. The amphibole is counted on a glass slide without a filter as described in the text. The count is 62 ppmg (± 87). The particle length is 34 μ m.

continues. The density-refractive index chart (Fig. 1) is presented for the convenience of those wishing to apply the method to other mineral associations.

Discussion

The method proposed in this paper permits one to find amphibole particles and to view their shape much more easily and rapidly than standard optical methods. It was found that talc contains <0.1% amphibole if less than 20 particles are observed in 20 fields of view. On the other hand, using the traditional 1 mg procedure, less than 5 particles in 100 fields of view would indicate <0.1%. It is much easier to find the amphibole particles by optical methods after concentrating them with heavy liquids because there is less interference by talc particles. In the 1 mg count many talc particles are seen on edge thereby needing to be tested for refractive index. Although the standard deviations are quite high, the proposed method permits one to routinely analyze industrial mineral samples where it was impractical to do so.

It is not known whether talc that passes the 0.1% Hazard Communication regulation will it pass the airborne asbestos regulation (2 Mg/M^3) (21). Unfortunately, one cannot conclude anything about whether or not a talc powder will pass the fiber regulation if it is below the talc dust limit because the mandated analytical procedure is phase contrast which bears little to no relationship to true fiber content where mixed mineral dusts are involved (22). An additional problem as indicated previously is that the coarser particles of a bulk powder will not become airborne so that a different portion of the sample will be present in the air as compared with the bulk powder. On the other hand, if we consider true fibers greater than $5 \mu\text{m}$ in length it would appear that the 2 Mg/M^3 would offer a good certainty that the airborne level would be less than 0.2 fibers/cm^3 . The only way it could be higher would be if only 1/100 of each milligram of talc could become airborne and if all the fibers were in that 1/100 of the sample. Since this is very unlikely, it appears that the dust limit for talc is sufficiently stringent for fiber as well.

The Hazard Communication regulation generally assumes that percent is either by weight or volume. If percent by weight is used, samples containing stubby particles such as that shown in Fig. 5 would seem to be more dangerous than those samples containing an equal number of long narrow particles similar in shape to those in Fig. 4 because the weight percent would be higher. For this reason percent by count was chosen. One can, however, determine weight or volume percent by measuring amphibole particle dimensions during a count. This would no doubt be necessary in samples where grinding had to be done before analysis.

In the course of this investigation, samples of talc have been found to contain amphibole which were not previously known to contain this mineral. In many cases periodic SEM analyses had been done. The problem with SEM is that little is accomplished in going to sub-optical sizes unless there is also an increase in the ratio of amphibole to talc particles, and there is no indication that this occurs in bulk samples except possibly in the case of "true asbestos" where the particles have broken into fibrils (23, 24, 25). It is possible that the centrifuge technique will be useful for concentrating and separating amphibole from talc for SEM work. This will require the use of less viscous heavy liquids so that reasonable centrifuge times can be used for these very small particles.

It should not be construed from comments in this paper that all talcs contain amphibole particles. There certainly are amphibole-free talcs. There are others in which the amphiboles have not been previously recorded because of their low level. All U.S. cosmetic, pharmaceutical or high grade talcs that were analyzed during this study are in compliance with federal regulations, generally by a large margin.

of the slide. These shown in the photographs were found in this way. The lower limit indicated assumes that the smallest number which could be found in 20 f.o.v. is 1. On samples with such low counts one might scan more fields of view, but no purpose was served by doing this in the present case.

Table 2. Comparison of values obtained by traditional 1 mg-100 f.o.v. count with centrifuge method using various sample weights.

(a) Standard 1 mg-100 f.o.v. counts	
Sample	fibers/mg
a	216
b	392
c	234
d	351
e	113
f	376
g	94
h	309
i	0
j	505

average	259 $\pm$ 157

(b) Centrifuge method (20 f.o.v.)			
Sample	Sample size	fibers/mg	σ
P1	47 mg	253	146
P2	115	277	98
P3	59	296	64
PP1	308	275	108
PP2	60	341	108
	60	291	98
	60	283	100

Application to Other Problems

The results and photographs presented to this point were obtained with particles collected on a Nuclepore filter (1.0 μ m pore size). There may be times, however, when one may wish to separate minerals by density but examine them with refractive index liquid other than 1.584. This can easily be done by modifying the procedure slightly in this way. After the sample has been spun, the material on the bottom of the tube is transferred to a second centrifuge tube by micropipette. If the heavies are of interest, distilled water is added to this second tube. If the float is of interest the distilled water is added to the original tube. Once the heavy liquid is mixed with distilled water the density is lower than the minerals, either float or heavies, so that the sample can be spun down in the centrifuge. The liquid is removed with a micropipette and fresh water added. After spinning down in distilled water five or six times, the sample is transferred with a drop of water to a glass slide. After it is dry, any refractive index liquid may be used. Figures 10 and 11 show an example of the same sample prepared on a filter and on glass. The counts are essentially the same.

This procedure has been used for concentrating quartz from talc samples and for obtaining tremolite counts on acid insoluble residues of carbonate rocks. Work on these and other applications

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Amphibole Content of Cosmetic and Pharmaceutical Talcs

by A. M. Blount*

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 effect 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 uncertainty in the outcome of the asbestos regulation pertaining to nonfibrous amphiboles.

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 μ m or greater (iii) width of 5 μ m 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 $\times$. 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

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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 addition, 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.

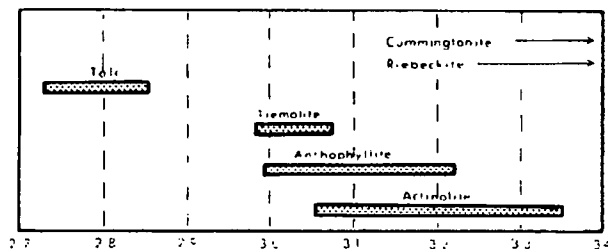


FIGURE 2. Specific gravities of talc and amphibole (6).

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 μ m 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:

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

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 ($1.7 \text{ mg}/60.6 \text{ mg} \times 2\% = 0.06\%$). 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

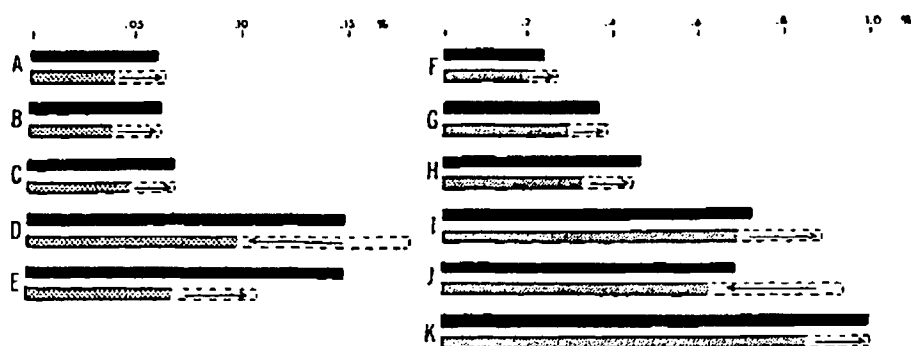


FIGURE 3. Percent tremolite in talc as determined by the centrifuge/optical method (shaded bars) compared with that actually present in experimental mixtures (black bars). The dashed part of the shaded bars indicates +2 SD (right arrow) or -2 SD (left arrow).

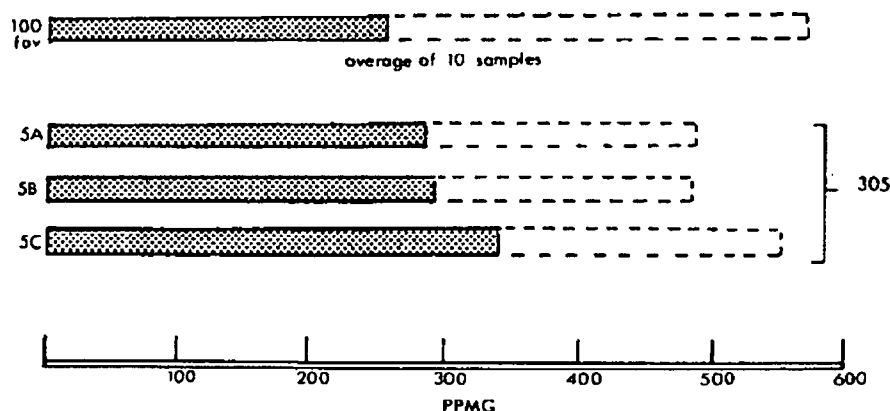


FIGURE 4. Comparison of traditional (100 FOV) count with centrifuge/optical count of same talc. The three lower bars indicate the values in particles/mg obtained by the centrifuge/optical method for three 60-mg samples. The top bar is the average of ten 100 FOV (traditional method). The dashed part of 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 since 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 ^e		Needles	0/20
M	39	21	Cleavages and fibers	4/20
N	25	17	Prismatic pieces	3/20
O	ND			0/20

^aFOV, fields of view.^bND, none detected.^cNo 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.^dLarge 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 determined 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 versus 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 centrifuged 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.

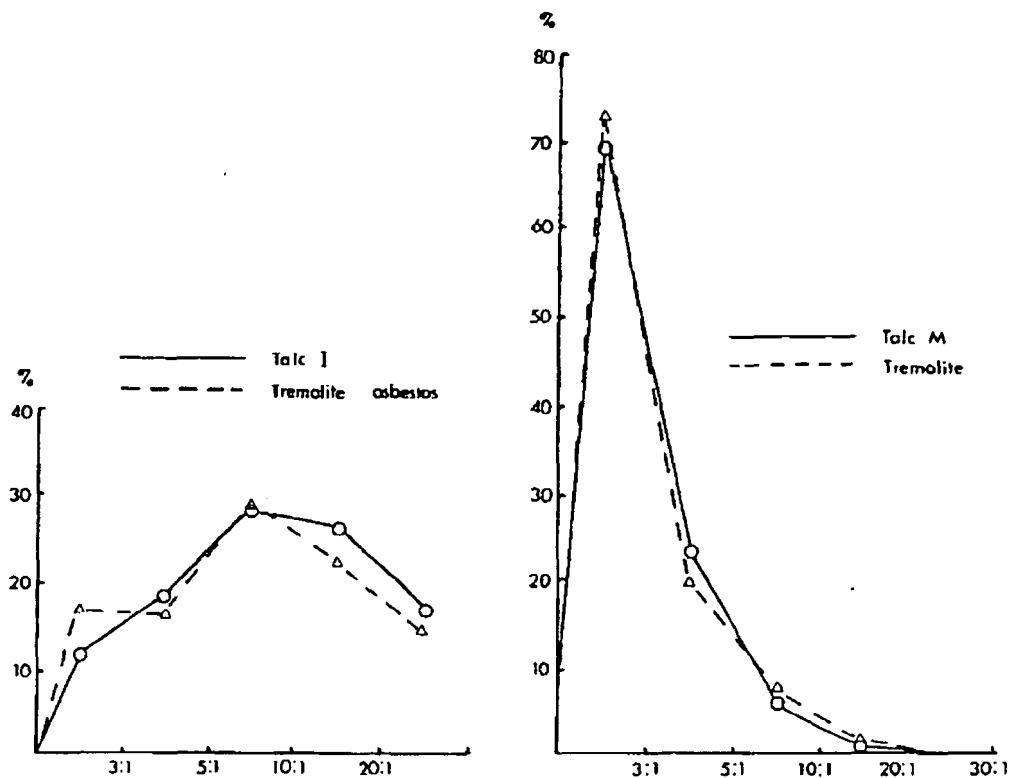


FIGURE 6. Percent amphiboles in each aspect ratio group for talc sample I (left) and M (right) compared with tremolite asbestos (7) and tremolite (nonasbestiform) (7).



FIGURE 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.

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.

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

Method	Size, %		
	5-10 μm	10-15 μm	$\geq 15 \mu\text{m}$
Traditional	57	26	16
Centrifuge	33	27	38
	Mean length, μm	Mean width, μm	Mean aspect ratio, μm
	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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