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PRACTICAL ASPECTS OF TALC AND ASBESTOS

C. J. Parmentier and G. J. Gill

Cyprus Industrial Minerals Company
Talc Division
Los Angeles, California 90071

Abstract

The present day controversy and misunderstanding regarding talc and asbestos has existed for many years. This paper reviews some of the reasons for the widespread public misconception that all talcs contain asbestos.

The experiences of a major talc producer are discussed in relation to occurrences of talc and asbestos, and the analytical techniques required to substantiate a talc-asbestos relationship are reviewed.

Key Words: Asbestos; scanning electron microscopy; talc; transmission electron microscopy.

Introduction

Numerous environmental, occupational safety, and health agencies have recognized the medical hazards associated with asbestos minerals and have issued regulations for their use. However, during the past five years an increasing amount of controversy has been encountered regarding terms, definitions, and measurement methods.

Due to the ambiguities in these criteria and various misconceptions regarding the mineralogy of talc, disagreements also exist between industry, medical researchers, state and federal regulatory agencies concerning talc and asbestos.

This controversy affects a variety of industries, ranging from the talc producers themselves to the industrial consumers, their insurance carriers, and even the household consumer of the numerous products containing talc.

This presentation will highlight some observations and experiences of a major talc producer, in relation to occurrences of talc and asbestos. Analytical techniques used for identification of asbestos minerals and fibers in a talc matrix are also discussed.

Definitions

Asbestos:

As used in this presentation, the term "asbestos" pertains to fibrous forms of actinolite, tremolite, anthophyllite, and chrysotile. All of the six recognized asbestos minerals are not included in this review as the above mineral types are the only asbestos minerals this laboratory has found to occur in talcs to date.

The terms acicular, lath, bladed, cleavage fragments, rods, needle, and columnar are often used as synonyms for the term fiber or asbestiform. It is the term "fiber" which appears in need of a uniform definition. As used in this presentation, a fiber is described as a particle with a length to width ratio of 10:1 or greater. The present OSHA definition [1]¹ allows a length to width ratio of 5 or more to 1, with a maximum diameter of 3 μ m. Figure 1 illustrates and compares the simulated appearance of particles with the aforementioned ratios. The 5:1 length to width ratio allows blocky particles to be classified as fibers, illustrating the need for more realistic definitions and size criteria of a fiber.

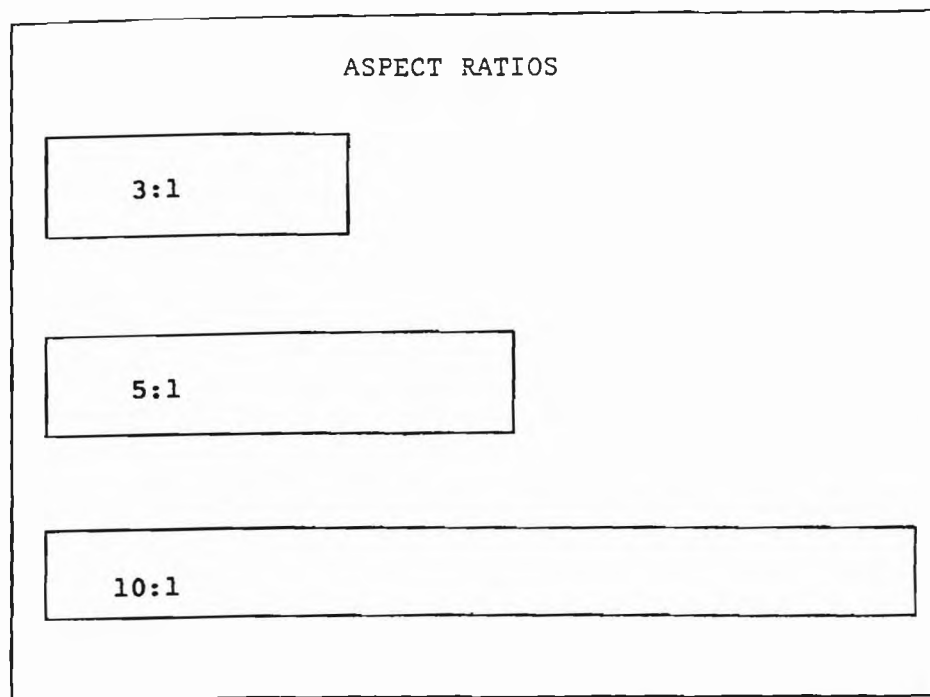


Figure 1. Illustrated by the above drawing are the geometric shapes which have been defined as fibers. The Asbestos Regulation CFR 1910.100L stipulates that a particle with an aspect ratio of 2:1 or greater is a fiber. However, these criteria were further modified by OSHA field information memorandum #74-92, November 21, 1974. The OSHA modification changed the criteria for a fiber from a 3:1 aspect ratio to a 5:1 length-to-width ratio with a maximum diameter of 3 μ m.

The 10:1 aspect ratio is used at the Cyprus Industrial Minerals laboratory.

Talc

The term "talc" has also been misused in published studies and requires definition. Talc refers to a specific mineral with a hydrated magnesium silicate composition of laminar or sheet structure. The natural platelet diameter of a talc varies, depending on deposit location and geological condition. Both the size and orientation of the laminar plates, with respect to each other, determine the characteristic form of the talc, e.g., massive or steatite talc versus foliated or micaceous talc. It is this characteristic talc structure which most often determines end use applications. The large micaceous or foliated talcs are ideal for cosmetic applications; while the finer particle size, massive or steatite type talcs are most often used in filler and extender applications. The purity and locations of the talc deposit, in addition to the degree of beneficiation, also have a significant effect upon the end use application.

¹Figures in brackets indicate the literature references at the end of this paper.

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Talc and Asbestos

Many misconceptions and misunderstandings of talc have existed due to improper use of terms such as tremolite talc, asbestiform talc, and fibrous talc. The majority of these improper terms were used to describe talc from the New York State talc district. In many earlier medical studies, which indicated a high incidence of talcosis, pneumoconiosis and mesothelioma among talc miners and millers from St. Lawrence County, New York, the supposition is made that talc dust was the cause. Kleinfeld and workers in 1967 [2] compared the mortality among talc miners and millers in New York State to findings among asbestos workers and noted similar pathological findings, to quote, "Rather characteristic was the presence of elongated, terminally clubbed bodies indistinguishable from asbestos bodies as seen in asbestosis." The talc dust exposure consisted predominantly of talc admixed with other silicates such as serpentine and tremolite, carbonates and a small amount of free silica, according to Dr. Kleinfeld. Additionally, Dr. Kleinfeld cites numerous other publications in which medical studies were performed on workers engaged in the milling or mining of the "fibrous variety of talc" [3-6]. However, none of these studies have differentiated between the mineral talc and the asbestiform variety of tremolite.

In a later publication [7], studies of New York State talc deposits and their asbestos contents were carried out. Mineralogical analyses of these materials indicated that all samples were predominantly asbestos.

The amount of asbestos impurities encountered in commercial talc ranges from <0.1 percent to over 50 percent. The majority of commercial talc samples analyzed have asbestos contents ranging from <0.2 percent to a maximum of 2 percent. Commercial production talc samples from New York State show a range of 20 percent to over 50 percent asbestos. A few additional commercial talcs have an asbestos content of 5 to 15 percent.

Figures 2 and 3 illustrate the asbestos contents of typical New York commercial talcs.

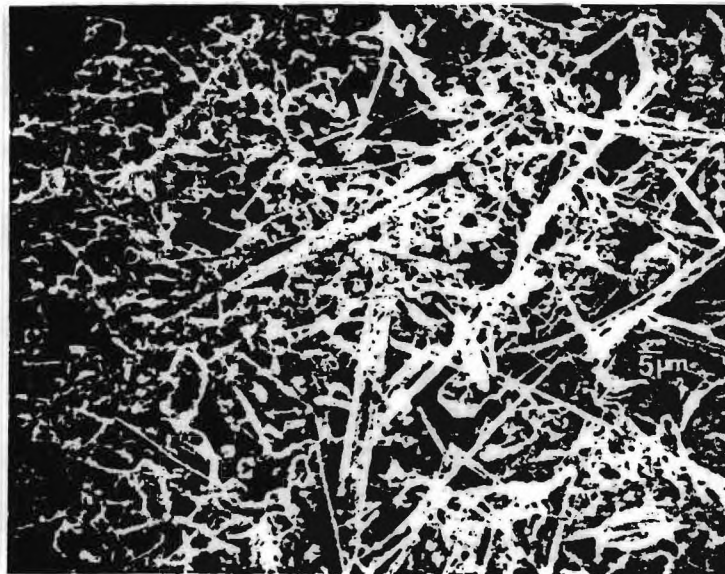


Figure 2. Talc-Fiber No. 1 from International Talc Co. Analyzed as asbestiform tremolite with minor talc content.

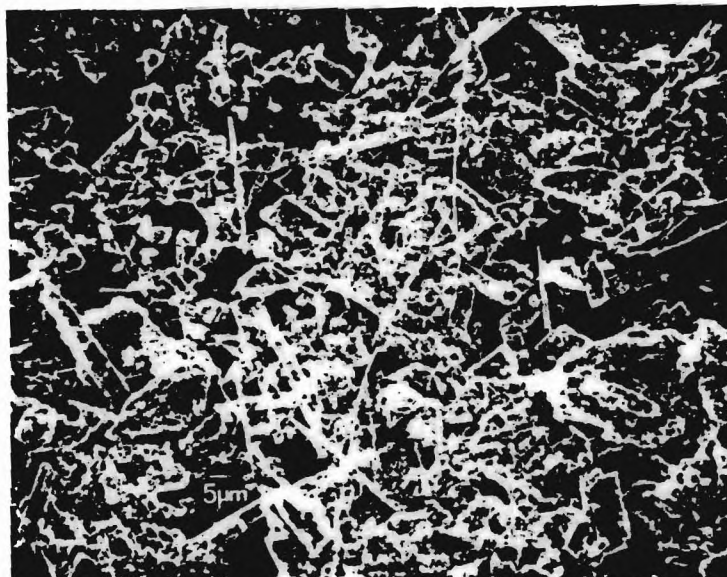


Figure 3. New York talc product used in the paint industry. Actual talc content is less than 40 percent. Majority of fibers are asbestos-tremolite, identified by TEM/SAED.

Cyprus Industrial Minerals Co. is one of the largest talc producers in the world. We have a continuing exploration program and over the past ten years have characterized talc deposits worldwide, in addition to analyzing numerous commercial talc samples. With this background and experience we feel justified in stating that not all talcs contain, or are associated with, asbestos. We also feel confident in stating that our ultra-fine grind Montana talc is the standard of talc purity throughout the world. Although high-purity talc deposits such as the Montana talcs are rare, asbestos-free talcs are not uncommon.

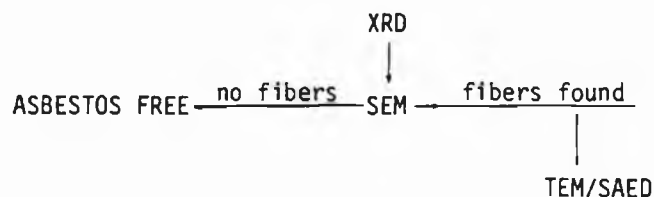
Based on our experience and bank of analytical data, we are aware that some talcs on the market do contain varying amounts of asbestos minerals. The most commonly encountered asbestos impurities in talc are tremolite and anthophyllite. There have been occurrences in which chrysotile asbestos has been found in selected talc samples. However, these occurrences are considered unusual but not unlikely.

Analytical Techniques

The methodology used for the analysis of asbestos fibers in talcs is a subject of considerable controversy. Following is a summarized description of the techniques used by the Cyprus laboratory and the reasons for their use.

Illustrated in Table 1 is the typical test procedure used to analyze talc for possible asbestos contamination. Although x-ray diffraction is shown as the initial method of analysis, it must be remembered that the primary function of the Cyprus Industrial Minerals laboratory is the characterization of talcs. If we were concerned with analyzing talcs for the presence of asbestos only, we would consider scanning electron microscopy as the most applicable technique to initiate analyses by screening samples for the presence of fibers.

Table 1. Analytical procedure flow sheet



Scanning Electron Microscopy (SEM) has two very unique advantages compared to optical microscopy techniques. First is the capability to accurately identify a fiber by tilting the specimen, thus viewing a particle from various angles. Additionally, the added depth of focus characteristic of SEM allows the complete particle to be studied at the same time. Figure 4 is included to illustrate this advantage, and shows the typical curled edge of a talc platelet. If a similar talc platelet was observed by optical microscopy, the limiting depth of focus would dictate that only the curled edge was in focus and would appear as a fiber, or the plate would be in focus and appear as a separate particle.



Figure 4. This micrograph illustrates the morphology of a typical curved talc platelet. The problems associated with attempting to define a fibrous structure by optical microscopy and its limited depth of focus can be seen. The optical microscope would make the above particle appear to be two separate particles, one of which would appear as a fiber and the other as a platelet.

The second advantage of SEM is the easily varied magnification capabilities with sufficient resolution to distinguish the presence of very fine fibers. In contrast, optical microscopy techniques can allow a large number of very fine fibers such as chrysotile to go undetected. Langer, Selikoff, et al., state "Many particles found in lung tissue are submicroscopic, measuring as little as 200 Å (0.02 microns) in diameter, therefore requiring electron microscopy" [1]). Suzuki and Churg also stated the preponderance of submicroscopic asbestos fibers observed in the successive steps in the development of the asbestos body were less than 1 µm in length, necessitating the use of the electron microscope [14].

Figures 5 and 6 are included to illustrate the SEM capabilities by revealing numerous chrysotile fibers present in a talc sample spiked with 1.5 percent chrysotile. Many of these fibers can be observed in figure 6 with sizes approaching single fiber diameters of ~20 nm (200 Å), well below the theoretical resolution limit of optical microscopy.

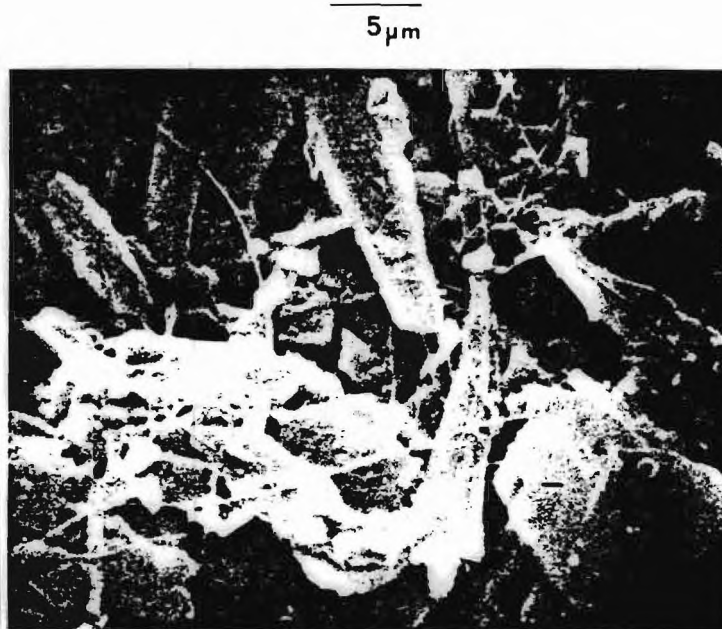


Figure 5. CTFA spiked talc by SEM. 1.5% chrysotile, 0.5% tremolite x 2800 at 40°.

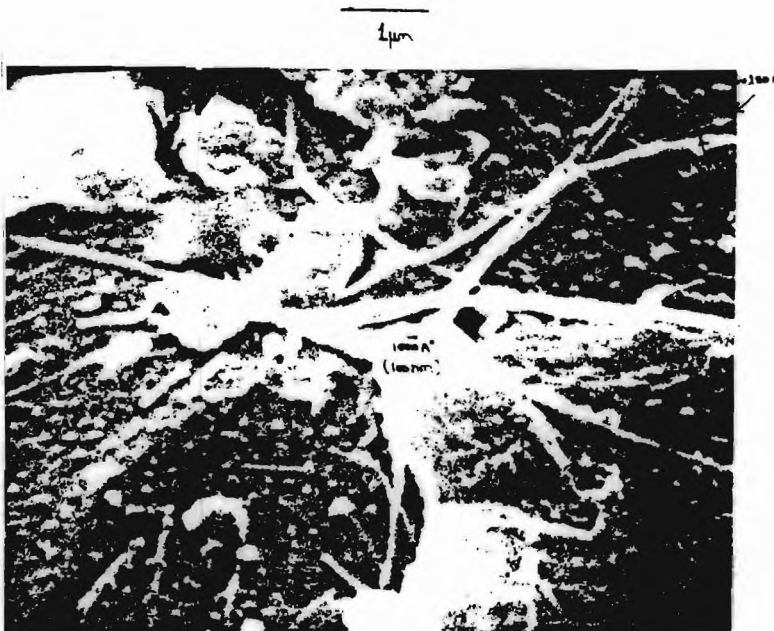


Figure 6. Spiked talc by SEM. 1.5% chrysotile x 13,500 at 35°.

Scanning electron microscopy for initial sample screening also has advantages compared to transmission electron microscopy methods. The sample preparation is considered easier and less time-consuming than TEM, which requires preparation of filmed specimen grids for sample mounting. Additionally, and most significant is the fact that by SEM screening of talc samples for fiber presence, the amount of sample examined is approximately an order of magnitude greater than TEM methods (0.1 mg vs. <0.01 mg). This difference becomes a major factor when investigating talc samples for trace fiber content (<1000 ppm.).

Transmission Electron Microscopy and Selected-Area Electron Diffraction

This powerful analytical technique is capable of furnishing a combination of morphological and crystal structure data of small single fibers which can result in very conclusive identification. However, this method requires a good deal of operator expertise and accuracy of data. We feel that fiber analysis via electron diffraction, supplemented with morphological data, provides a positive identification. We have observed, however, many cases where workers have identified minerals through using only partial diffraction patterns; that is to say, only the high angle reflections (low numerical spacings). These spacings are very similar between silicate minerals making absolute identification impossible without the more conclusive low angle reflections (high numerical spacings). Additionally, due to the unique possibilities of unusual mineral occurrences, which are more the expected than the unexpected in mineralogy, the morphology of a fiber does not, and should not indicate asbestos.

Along these lines, some examples of unexpected trace mineral fibers which have been encountered and subsequently identified in specific talc samples or deposits are: zeolite fibers (mordenite), clay mineral fibers (attapulgite-polygorskite), and a rare fibrous variety of antigorite-serpentine known as picrolite [12,15]. These materials are not asbestos by definition.

All of the indicated fibers have similar d-spacings in the high angle reflections (numerically lower than $\sim 5.00 \text{ \AA}$), indicating the necessity for using only the numerically high, low angle spacings for positive identification.

Although SAED allows positive and undisputed identification of mineral fibers, only under specific conditions are SAED patterns considered positive identification. The following criteria must be met before an SAED pattern is considered accurate:

1. The camera constant must be determined on the same grid and same sample area as the SAED pattern, using a known standard. Preferred accuracy is obtained by shadowing the specimen preparation with a gold or aluminum metal which allows the camera constant to be calculated directly on the SAED pattern and acts as an internal camera constant.
2. The indexed d-spacings should include the most intense low angle spacings. Any pattern without d-spacings numerically higher than $\sim 5.00 \text{ \AA}$ (low angle) should be questionable, as many silicate minerals have similar d-spacings in the higher reflection angles.
3. Where applicable, unit cell parameters should also be used to supplement fiber identification.

X-Ray Diffraction

X-ray diffraction (XRD) is a standard mineralogical technique. Within levels of detectability and lack of interfering reflections, it allows the identification and quantitation of asbestos minerals in talc. The major limitations of XRD are the lack of morphological data and the lower levels of sensitivity can allow a large number of asbestos fibers to go undetected.

The information derived from XRD is invaluable for the characterization of a talc, and is of considerable help in the understanding of the mineralogical processes of forma-

tion and the possible impurities present. The background information of mineral phases present aids and expedites the TEM-SAED interpretation of unknown fibers.

Optical Microscopy

Optical microscopic techniques are considered of minimal value in the analyses of fine-grained complex silicate mineral mixtures, and often result in ambiguous data of questionable value. As previously mentioned, optical microscopy techniques can allow a large number of very fine fibers such as chrysotile to go undetected [7,13]. Due to these findings and the health and economic considerations involved, optical microscopy is not used at the Cyprus laboratory for asbestos in talc analyses or airborne asbestos fiber analyses.

Conclusions

This presentation has reviewed the misunderstandings and widespread misconceptions that all talcs contain, or are associated with, asbestos. Some of the reasons for these misconceptions were shown to be the lack of mineral background in the use of terms and definitions. However, the use of inadequate analytical methods has been shown to be a major cause. The use of ambiguous analytical techniques such as optical microscopy for reasons of expense, lack of experience, or ease of analysis has far-reaching economic and health implications. Only TEM-SAED techniques supplemented by SEM appear to give the necessary degree of accuracy needed for positive results.

The existence of large deposits of high-purity, asbestos-free talc are well documented, and it is hoped that future references to talc will be more clearly defined as to proper mineral content.

In view of the evidence presented from both the medical and mineralogical science fields, it is evident that state and federal regulatory agencies need to redefine terms, definitions, and analytical methods for the assessment of asbestos and talc. This applies to both airborne exposure and bulk samples containing asbestos.

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Asbestos in Talc

by Arthur N. Rohl*

Talc deposits include asbestos minerals such as chrysotile and amphiboles that may be carried over into consumer products. Optical microscopy and x-ray diffraction analyses may not reveal their presence. Examples are given of electron microscopy procedures that permit detection and measurement.

The mineral talc is a hydrous magnesium sheet silicate that occurs in both platy and fibrous crystal forms. Talc tends to occur in rock masses coexisting with a number of other hydrous magnesium silicate minerals. Typically, talc deposits consists of fine-grained, intergrown mixtures of minerals which may contain considerable amounts of asbestos. In addition, talc deposits often show complex mineral zonation, which adds to the difficulty of selective mining. For example, in the talc deposits of the Gouverneur District of New York State, talc occurs with the asbestos minerals chrysotile, tremolite, and anthophyllite in addition to other silicate minerals.

Since the mining of talc rock almost invariably includes the mining of asbestos as well, the asbestos contaminant may be carried over into the consumer product and thus introduce the risk of asbestos disease. This possibility leads to an important public health question: is asbestos present in consumer talcs, and if present, which mineral fibers and in what concentrations?

Among the standard mineralogical techniques which may be used for identification and quantitation of asbestos in talc are optical microscopy, x-ray diffraction, and electron microscopy (EM).

Optical microscopy, employing polarized light optics, is useful for determining the optical

properties of particles. However, in the instance of talc, the extremely fine grained intergrowths of different minerals and the extensive overlapping and similarities of their optical properties limit this technique to a preliminary or screening function. Since large numbers of fibers may go undetected, optical microscopy would not be capable of quantitative analysis.

X-ray powder diffraction is a routine technique for analyzing crystalline materials. It is relatively simple in principle, but the results may be difficult to interpret. The limitations of precision and accuracy must be given careful consideration.

The identification and quantitation of asbestos fibers in talc by x-ray diffraction may be achieved by comparison of known dilutions (fiber type and quantity) of asbestos in a talc matrix with unknowns. The preparation of standard dilutions of asbestos minerals in talc for quantitative analysis requires: (1) a talc matrix completely free of contaminating asbestos minerals, (2) pure asbestos fiber as the sought adventitious phase, (3) a preparation method for insuring homogeneity and reproducibility of the standard dilution material, and (4) selection of x-ray reflections with no superimposed interferences. Condition (1) requires the selection of pure talc matrix material. In the first stage of screening, material was first scanned by x-ray diffraction to identify the major mineral phases rapidly, especially asbestos minerals. If no asbestos phases were detected, the material was

*Environmental Sciences Laboratory, Mount Sinai School of Medicine, New York, N.Y. 10029

re-examined in a more sensitive mode of x-ray diffraction called step scanning, and finally by electron microscopy. In this way a pure talc was selected (pure with respect to asbestos; small amounts of chlorite and phlogopite mica were tolerated).

In a similar way, pure samples of anthophyllite, tremolite, and chrysotile were screened and selected for use in preparing the talc-asbestos standard dilutions.

In x-ray diffraction the reproducibility of reflection intensities is strongly influenced by the degree of cleavage of crystalline powders. The minerals under investigation exhibit a high degree of platy and fibrous cleavage. A number of preparation techniques have been developed for reducing preferred orientation effects. These were tested, but none were found to give satisfactory reproducibility. Accordingly a technique was developed and employed which gives a high degree of sensitivity for substances present in minute quantities and with a greater level of reproducibility of reflection intensities.

Binary systems of three asbestos minerals in talc were prepared at varying levels of dilution concentrations. Standard weights of these mixtures were dispersed in water with ultrasonic energy to disperse the phases homogeneously. This slurry was filtered through a membrane filter by use of a hypodermic syringe (Fig. 1). The residue forms a flat cake which is mounted for x-ray analysis. This technique has the advantage of uniformly preparing, mounting and measuring the talc-asbestos dilutions under identical conditions.

Because of the structural similarities between some of the minerals, there was considerable overlapping or interference in many reflections, and this made it necessary to select reflections which could be unambiguously used as indices of the amount of each mineral present. These

diagnostic reflections were step-scanned at $0.01^\circ 2\theta$, in a fixed-count mode. This permits the weak reflections produced at low dilution levels to be determined with precision. From the fixed-count data a profile of the diagnostic reflection is obtained and the area above background is taken to be proportional to the reflection intensity. The results of these analyses are given in Table 1, which shows that: chrysotile at dilution levels less than 0.25% was not detected, tremolite was detected down to 0.1% dilution level, and anthophyllite was not detected at concentrations below 2.0% (diagnostic reflection is at 8.26 Å; $I/I_1 = 55$).

In order to determine the number of chrysotile fibers present at various dilution levels, aliquots of the various dilutions were prepared for EM scanning. A fairly standard technique called the rubout method was used. For each dilution level, 20 fields from three EM grids are photographed at constant magnification and the number of long unit fibrils per field are counted from printed enlargements (Figs. 2 and 3). These fiber counts show fairly good correlation with levels of chrysotile dilution. By using the fiber count data, it is possible to calculate the number of fibers in a unit weight of sample. Thus, at a 1% dilution level there would be about 40×10^8 fibers/mg. Even at the lowest level of detection by x-ray diffraction, i.e., 0.25%, there would be about 10^9 fibers/mg. Cosmetic talcum powder, for example, which had been step-scanned and chrysotile not found might contain billions of fibers released during dusting with a half-gram dose.

Thus, very large numbers of asbestos fibers may be present in talc end products, yet they remain undetected if only optical microscopy and x-ray diffraction are used. On the other hand, EM can be a very sensitive method for detecting extremely minute amounts of asbestos in talc.

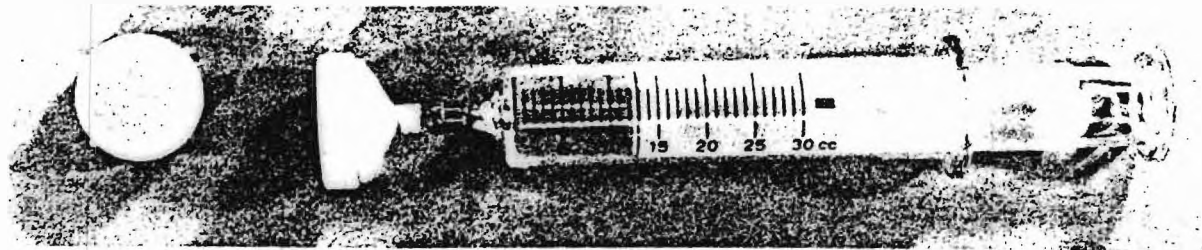


FIGURE 1. Membrane filter holder-hypodermic syringe system of sample preparation.

Table 1. Comparison of lower limits of detection of asbestos minerals in talc by step scanning and continuous scanning.

Asbestos mineral	Diagnostic reflection, Å	Detection limit concentration, %	
		Step scanning (0.01° 2θ) ^a	Continuous scanning (1° 2θ/min) ^b
Chrysotile	3.66	0.25	1.0
Tremolite	8.38	0.10	2.0
Anthophyllite	8.26	2.0	4.0

^a Operating conditions: fixed count rate = 2000; 45 kV, 20 mA.

^b Operating conditions: 500 counts/sec; time constant = 2.0; 45 kV, 20 mA.

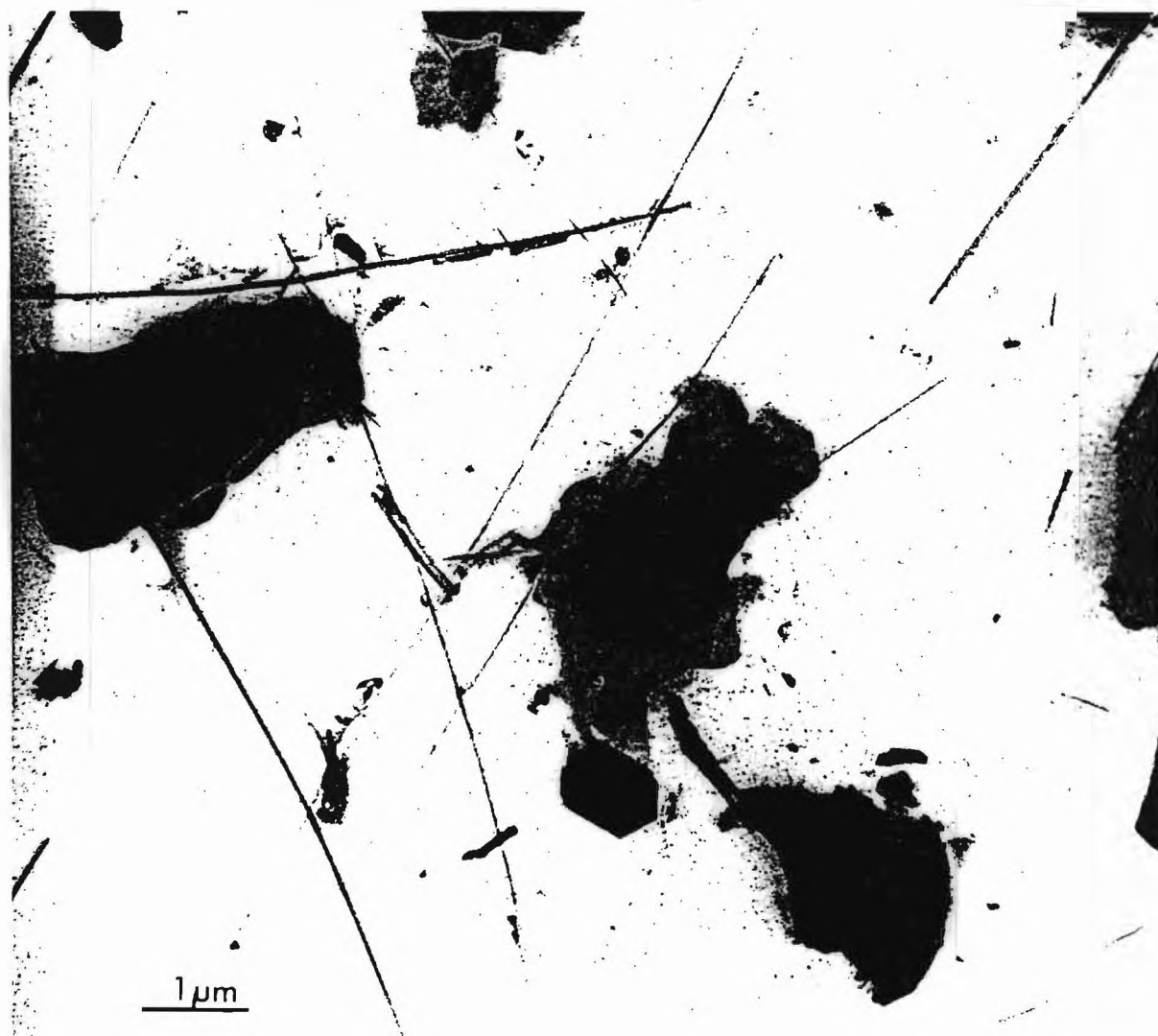


FIGURE 2. Electron photomicrograph of chrysotile-talc (99% talc, 1% chrysotile).



FIGURE 3. Electron photomicrograph of chrysotile-talc (95% talc, 5% chrysotile).