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ASBESTIFORM MINERALS IN INDUSTRIAL TALCS: COMMERCIAL DEFINITIONS VERSUS INDUSTRIAL HYGIENE REALITY

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Abstract

As part of its industry-wide study of the talc industry, the National Institute for Occupational Safety and Health (NIOSH) has conducted Detailed industrial hygiene studies of mine and mill operations processing talcs contaminated with asbestiform minerals. The principal analytical method used for studies of asbestiform minerals in talc bulk samples and airborne dust samples is analytical transmission electron microscopy utilizing selected area electron diffraction and microchemical analysis for fiber identification. This presentation includes a discussion of the methods of analysis being used by NIOSH and comparisons of results of analysis with other analytical techniques. Also included are results of NIOSH industrial hygiene studies in asbestiform talc operations and comparisons of airborne fiber characteristics (fiber length, diameter, aspect ratios, etc.) in these operations with other industrial processes using asbestos fibers.

Key Words: Amphiboles; anthophyllite, asbestiform minerals; industrial talc; occupational health; tremolite.

Introduction

The mineral talc is a pure hydrous magnesium silicate $Mg_6(Si_8O_{22})(OH)_4$ which has a theoretical chemical composition of 63.5 percent SiO_2, 21.7 percent MgO, and 4.8 percent H_2O [1,2][1]. However, this ideal chemical structure is rarely found in nature due to ionic substitution in the talc structure and due to common association with other minerals such as tremolite, anthophyllite, calcite, magnesite, quartz, dolomite, diopside, and serpentines (chrysotile, antigorite, and lizardite) [1,2]. Most talcs, as mined, are associated with varying proportions of some of these minerals [1] and sold as industrial talcs. In 1974 over 1.4 million short tons of talc were produced in the United States with major uses being in ceramics, elastomers, foundry facings, insecticides, paints, paper, roofing and toilet preparations [3].

The National Institute for Occupational Safety and Health (NIOSH) in cooperation with the Mining Enforcement and Safety Administration has underway an industry-wide study of the talc mining and milling industry. These studies include both epidemiological studies of exposed worker populations to determine health effects which may be attributed to occupational exposures and detailed industrial hygiene studies to characterize the various agents to which workers have been exposed.

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

Since many talc deposits contain asbestiform amphiboles and in some cases chrysotile (a serpentine), a large portion of the NIOSH environmental studies is directed toward determining mineral fiber exposure patterns and characteristics. For such studies, the primary method used is analytical transmission electron microscopy. This report includes a description of the equipment and procedures used by NIOSH for its environmental studies of industrial talc exposures and results of industrial hygiene studies in a talc mine and mill producing talcs containing asbestiform amphibole minerals. Also discussed are commercially employed definitions of what constitutes asbestos and the relationship of these definitions to observed industrial asbestos exposure characteristics.

Analytical Methods

Equipment

A number of methods are available and have been used to identify and quantitate asbestos concentrations in environmental samples. These methods include x-ray diffraction, differential thermal analysis, phase contrast and bright field optical microscopy, petrographic microscopy, scanning electron microscopy, and transmission electron microscopy. Each of these methods have certain advantages and disadvantages [4,5]. However, many researchers today consider analytical electron microscopy to be the method of choice for studies of occupational and environmental asbestos exposures.

For NIOSH studies of industrial talc exposures, analytical transmission electron microscopy is employed along with other standard mineralogical techniques such as x-ray diffraction and petrographic microscopy. The analytical system consists of a combination transmission-scanning electron microscope with a side entry stage equipped with an energy dispersive x-ray detector which is fitted through a port in the microscope column parallel to the specimen holder. The specimen-to-detector distance is approximately 10 mm with the specimen tilted 39 degrees to the electron beam for optimum x-ray collection. The energy dispersive x-ray detector has an actual energy resolution of less than 170 electron volts, and spatial resolutions of less than 0.5 micrometers are easily realized. This combination of analytical instrumentation permits visual characterization of particulate morphology such as fiber shape, length, and diameter as well as fiber identification using both selected area electron diffraction and x-ray microchemical analysis. In addition, surface topography may be further studied with this instrument by use of the scanning mode of operation using secondary electron images.

Procedures

Either bulk quantities of materials of interest, such as talcs, or environmental samples collected on membrane filters are routinely analyzed. The majority of samples studied consists of airborne particulates collected in industrial operations for the purpose of determining occupational exposure patterns. These samples are routinely collected on Millipore AA, 37 mm diameter membrane filters at sample rates of 1.5-2.0 liters per minute. Sample durations may vary from 15 minutes in very dusty operations to six hours for operations with little visible dust.

The method presently used by NIOSH for preparation of membrane filter samples for electron microscopic analysis is a modification of a direct clearing method first described by Ortiz and Isom [6]. The NIOSH method has been described in detail elsewhere [4]. Briefly, this method consists of the following steps:

1. A section of the membrane filter is cut with a cork bore (8 mm diameter) or a scalpel. This section is removed and placed sample side up on a clean microscope slide with the edges fastened to the slide with either a gummed binder ring or tape.

2. The slide assembly containing the sample is placed in a glass petri dish on top of four Whatman filters which have been saturated with acetone and covered. The acetone vapors destroy the microporous structure of the filter by slow dissolution, producing a fused, microscopically smooth surface on

the sample side of the membrane filter. A 10-minute fusion time has been found to be generally acceptable for Millipore AA filters.

3. After fusion of the filter surface, the slide assembly is placed in a vacuum evaporator on a rotary stage where the sampled side of the filter receives a fairly heavy (~200 Å) carbon coat. This carbon coat aids in retaining particles during subsequent filter dissolution and also provides for greater thermal stability during microscopic examination.

4. The final step is dissolution of the membrane filter and deposition of the particles onto electron microscope grids. A modified Jaffe Wick method is used whereby four Whatman filter papers are saturated with acetone. Two-hundred mesh carbon filmed grids are used and the coated filters are placed sample side down on them. The petri dish is then covered. Complete filter dissolution takes 8 to 16 hours. Acetone is replaced as necessary.

Using this method, many filters may be prepared as a "batch". Particle losses have been low and estimated at less than 10 percent [6].

Samples prepared by the preceding method are analyzed using analytical transmission electron microscopy whereby three pieces of data are gathered and used to identify each fiber (3 to 1 aspect ratio particles) observed. These include: (1) visual identification of single fiber electron diffraction patterns, (2) visual identification of semiquantitative elemental analysis spectra using x-ray microchemical techniques, and (3) observation of morphological characteristics, such as diffraction fringes, which may aid in identification. In addition, fiber length and diameter are also recorded. For most studies an accelerating voltage of 100 kilovolts is used with a screen magnification of approximately 17,000X. Beam currents are usually fixed at 100 microamps (not to be confused with specimen current).

Fiber concentrations are estimated using the average grid opening area as the calibrated counting area. To optimize statistical accuracy of the analysis while keeping analysis time to acceptable limits, 10 grid openings or 50 fibers are analyzed for each sample with a minimum of 5 grid openings. Analysis times range from 90 minutes to 3 hours per sample. Using this counting criterion for a typical 90 minute sample collected at 2 liters per minute, the lower limit of detection is estimated to be less than 0.1 fibers/cc. Precision and accuracy estimates from studies of the NIOSH phase contrast method [7] are considered generally applicable with a coefficient of variation of approximately ±25 percent for most samples.

Environmental Studies of Talcs Containing Asbestiform Minerals

Methods

As previously mentioned, a large portion of the NIOSH industry-wide study of the talc industry involves industrial hygiene studies of worker exposures, including exposures to asbestiform minerals. One such operation recently studied involved a mine and mill producing industrial talcs certified by the mining concern to be free of asbestos. Apparently, the prime analytical methods relied upon by this company to conclude that its products were asbestos free were gross methods such as observation with a common hand lens or at best low power stereomicroscopy both of which were claimed to be sufficient and proper mineralogical techniques.

In order to evaluate these claims, a detailed industrial hygiene study was conducted at the mine and mill in question to evaluate worker exposures using best available sampling and analytical technology. Although a number of different sampling and analysis methods were employed, only results of the fiber samples are presented in this report.

In order to evaluate fiber exposures and exposure characteristics, personal, breathing zone samples were collected from workers in the mine and mill using 37 mm diameter, Millipore AA membrane filters operated at a flow rate of 1.7 liters per minute. Sample

filters were changed periodically throughout the work shift to prevent filter overloading. During the study, more than 220 such samples were collected and used to determine both peak and time-weighted-average exposures. All samples were analyzed for fiber concentrations (>5 μm) using the standard phase contrast method recommended by NIOSH [7].

In addition, approximately 15 percent of these samples were analyzed by the electron microscopic methods previously described.

Results

Results of the fiber concentrations in the mine and mill as determined by phase contrast optical microscopy are shown in Table 1. Highly elevated fiber concentrations were observed in both mine and mill operations with time-weighted-average exposures ranging from 0.8 to 9.8 fibers >5 μm/cc in the mine and 0.2 to 16.0 fibers >5 μm/cc in the mill. Peak exposures as high as 29.1 fibers >5 μm/cc were observed.

Table 1. Summary of fiber exposures in talc mine and mill operations as determined by optical microscopy.

Operation	Fiber Concentrations (fibers >5 μm/cc)			
	Time-Weighted Averages			Highest Peak
	Mean ± SE	Median	Range	Conc. Observed
Mine (N=54)	4.5 ± 0.8	4.4	0.8- 9.8	18.2
Mill (N=168)	5.0 ± 0.5	4.3	0.2-16.0	29.1

N = Number of individual samples collected SE = Standard Error

Time-Weighted averages represent full shift determinations

While the above fiber concentrations, determined by phase contrast microscopy, may include some fiber types other than asbestos (e.g., talc "fibers"), they nevertheless represent minimum estimates of true exposures to asbestiform minerals as most asbestiform fibers are less than 5 μm in length and, in addition, some fibers, although longer than 5 μm, may escape detection due to resolution limits of optical microscopy. These facts are demonstrated in Table 2, which show concentrations of positively identified asbestiform mineral fibers as determined by electron microscopy. Time-weighted-average exposures were found to range from 9.5 to 25.0 fibers/cc in the mine and 7.3 to 102.7 fibers/cc in the mill. The highest concentration observed on a single sample was 102.7 fibers/cc.

Table 2. Summary of asbestiform mineral fiber exposures in talc mine and mill operations as determined by electron microscopy.

Operation	Fiber Concentrations[a] (fibers (all lengths)/cc)			
	Time-Weighted Averages			Highest Peak
	Mean ± SE	Median	Range	Conc. Observed
Mine (N=8)	16.4 ± 0.9	15.3	9.5- 25.0	25.0
Mill (N=19)	30.0 ± 1.4	24.1	7.3-102.7	102.7

N = Number of air samples randomly chosen and analyzed by electron microscopy

SE = Standard Error

[a] Concentrations reported include only those fibers positively identified as one of the asbestos minerals by analytical electron microscopy.

A typical electron photomicrograph of fibers in these operations is shown in figure 1 demonstrating the fibrous morphology of these particulates. The asbestiform habit of many of these fibers is evidenced by the "fiber bundle" effect. Results of the electron diffraction and microchemical studies on these fibers clearly demonstrated the presence of two amphibole fiber types; these being tremolite and anthophyllite. Analytical data for typical tremolite and anthophyllite fibers are shown in figures 2 and 3, respectively. The anthophyllite is seen to be low in iron content.

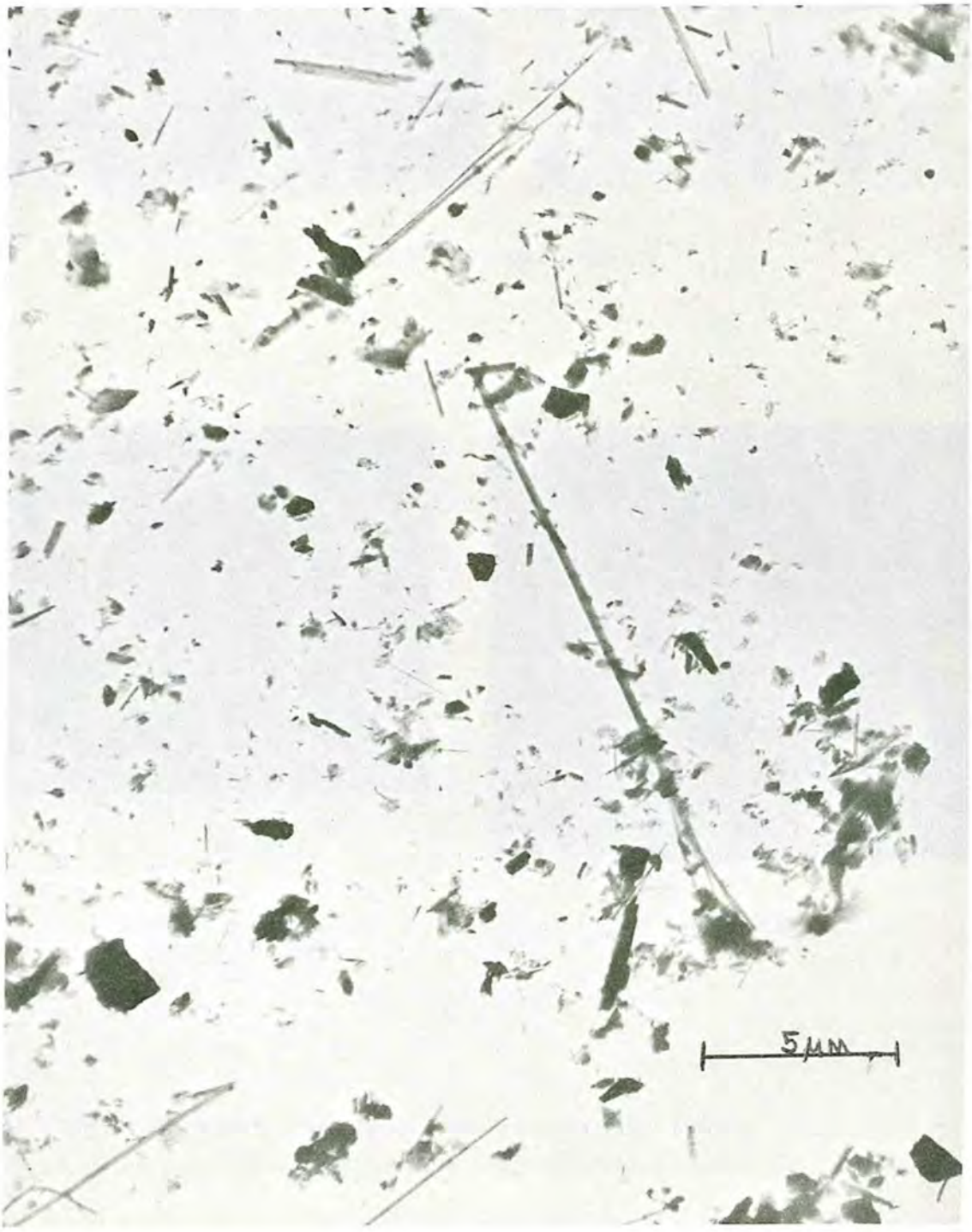
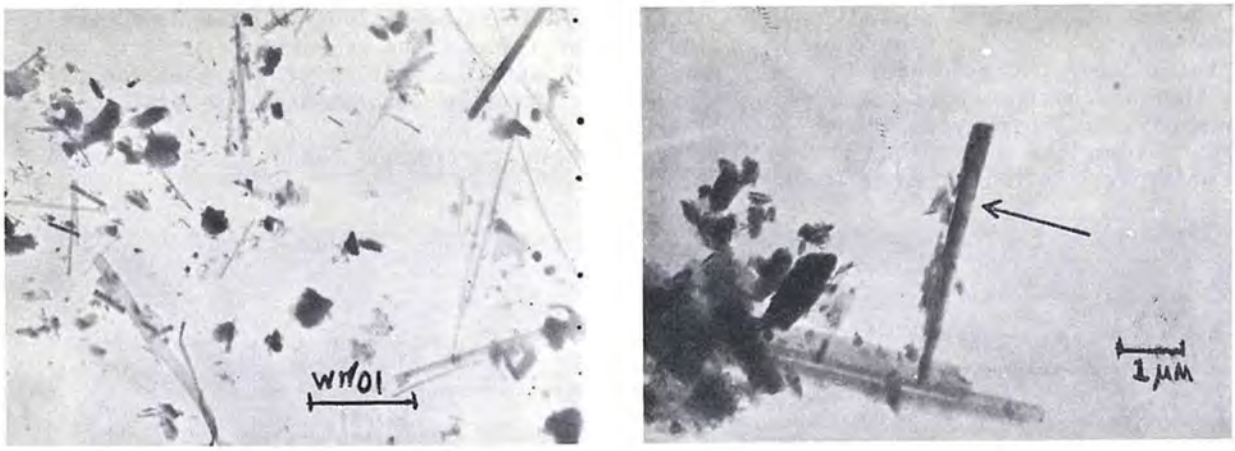
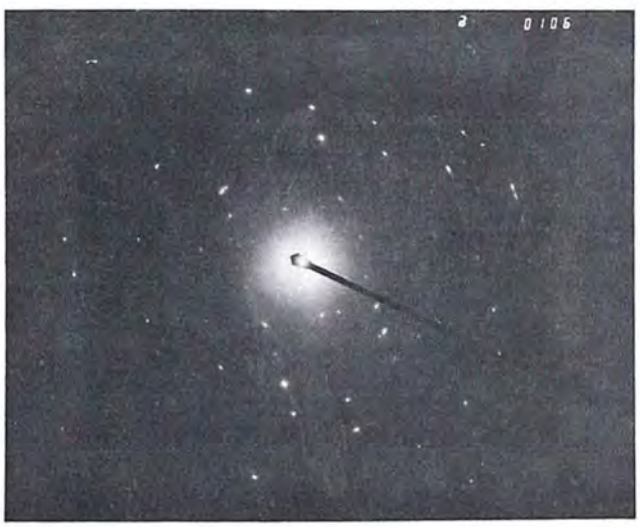


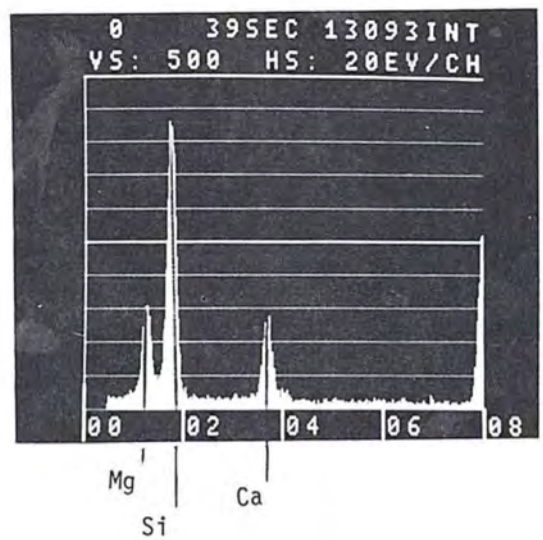
Figure 1. Electron photomicrograph of particles in talc certified as asbestos-free.



Electron photomicrographs

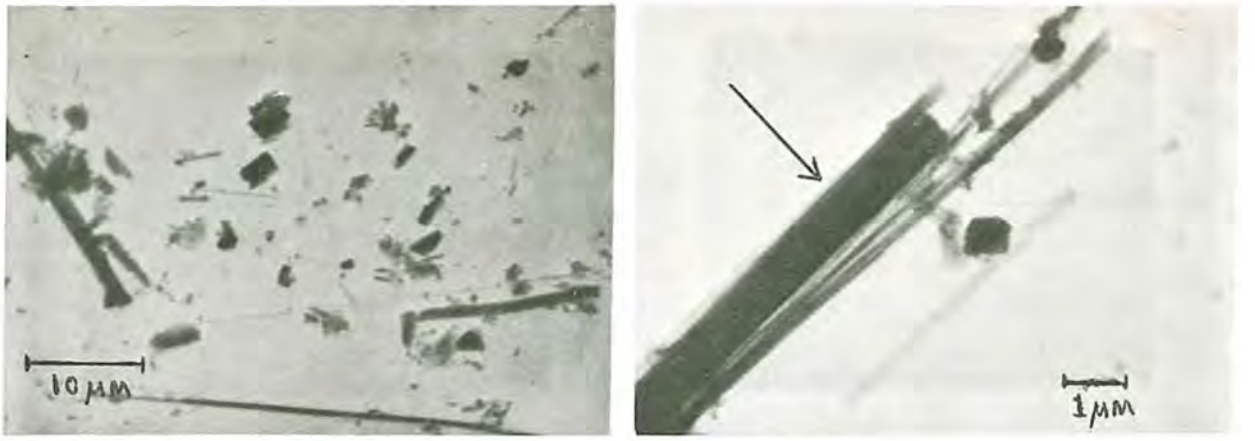


Diffraction pattern

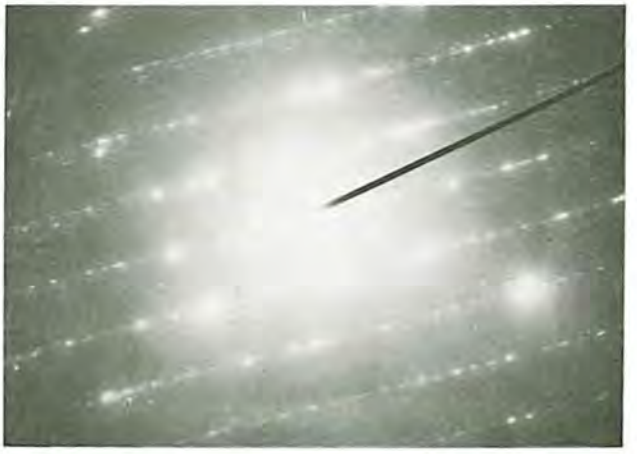


X-ray spectrum

Figure 2. Analytical data for tremolite fibers in talc certified as asbestos-free.



Electron photomicrographs



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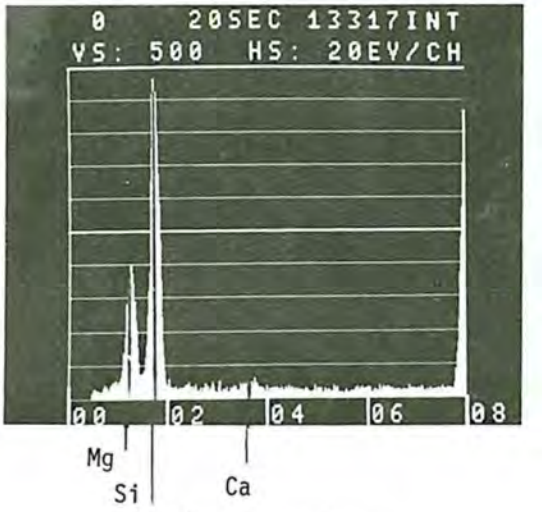

X-ray spectrum

Figure 3. Analytical data for anthophyllite fibers in talc certified as asbestos-free.

Tabulations of results of the fiber identification studies by electron microscopy are shown in Table 3. Of all airborne fibers (3:1 aspect ratio particles), 12-19 percent and 38-45 percent were found to be tremolite and anthophyllite, respectively, while 38-39 percent remained unidentified due to unrecognizable diffraction patterns. Tremolite fibers were observed to be generally shorter in length than anthophyllite fibers as demonstrated in Table 3 when only fibers longer than 5 µm were considered. Only 7 percent of the fibers longer than 5 µm were identified as tremolite whereas 65 percent were anthophyllite. This may also be observed in Table 4 where summary statistics of fiber length are given. While all median fiber lengths were found to be similar and not statistically different, the proportion of anthophyllite fibers longer than 5 µm in length was significantly ($P<0.05$) greater than tremolite (8-10% for anthophyllite versus only 3% for tremolite).

Table 3. Summary of airborne fiber types in talc mine and mill operations as determined by analytical electron microscopy.

Fiber Length	Percent of all Airborne Fibers[a]			
	Tremolite	Anthophyllite	Nonasbestos	Not Positively Identified
All Fibers	12-19	38-45	1-2	38-39
Fibers ≥ 5 μm	7	65	3	25

[a] Total number of fibers analyzed was approximately 1850.

Table 4. Summary of airborne fiber lengths for positive amphiboles in talc mine and mill operations as determined by electron microscopy.

Operation and Fiber Type	Median Length μm	Geometric Standard Deviation	% ≤ 5 μm in Length
Mine			
Tremolite (N=83)	1.6	1.8	97
Anthophyllite (N=164)	1.5	2.6	90-92
Mill			
Tremolite (N=160)	1.5	1.9	97
Anthophyllite (N=687)	1.4	2.9	90

N = Number of individual fibers analyzed

Inasmuch as the NIOSH recommended phase contrast counting method defines countable fibers only on the basis of fiber length and aspect ratio, much controversy has arisen with various industrial and mining groups claiming that this liberal criterion would define many mineral fragments as being asbestos. In this regard, fiber aspect ratios for positively identified amphibole fibers in the talc mine and mill under study are shown in Table 5 for all fiber lengths, and similar data for fibers longer than 5 μm are given in Table 6. These tables demonstrate that anthophyllite fibers in these talcs have larger aspect ratios than tremolite fibers and by comparison of Tables 5 and 6, aspect ratios increase with fiber length. Of interest is the fact that less than two percent of the positively identified amphiboles longer than 5 μm in length had aspect ratios 5 to 1 or smaller.

Table 5. Aspect ratios (length to width) for airborne amphibole fibers (all lengths) in mine and mill operations as determined by electron microscopy.

Fiber Type	Median Ratio	% ≤ 5 to 1	% ≤ 10 to 1
Tremolite (N=164)	7.5	23-24	70
Anthophyllite (N=687)	9.5	15-17	70

N = Number of individual fibers identified and sized

Table 6. Aspect ratios (length to width) for airborne fibers ≥ 5 μm in length in mine and mill operations as determined by electron microscopy.

Fiber Type	% ≤ 5 to 1	% ≤ 10 to 1
Positively Identified Amphiboles	<2	37-38
Non-Asbestos or Unidentified Fibers	18	80

Approximately 1850 fibers analyzed

Discussion

Results of an industrial hygiene study of talc operations producing industrial talcs certified by the company under study to be asbestos free have been presented. Contrary to claims of this company that its products do not contain asbestos, this study demonstrated excessive exposures to airborne fibers of which more than 70 percent of the fibers >5 μm in length could be identified as positive asbestiform amphiboles by best available analytical techniques. Repeated requests have been made of this company to clarify analytical methods and definitions of asbestos used to arrive at the conclusion that its products were free of asbestiform minerals. Apparently, the analytical method used was observation of hand ore specimens with a hand lens or, at best, use of low power stereomicroscopy. The definition of "asbestos" employed is less clear. Apparently the definition used is one which might best be termed a "commercial definition"; that is, in order for an amphibole to be considered to be asbestos it must have commercial value due to its fibrous shape.

This same company also operates another nearby talc mine and mill producing talc products which the company acknowledges as containing anthophyllite asbestos and labels these products with the warning required by the Occupational Safety and Health Administration. The determination made by the company that these talcs should be labeled was again based on macroscopic observation of hand specimens.

Having observed such elevated exposures as were presented in this report in operations considered by this company to be "asbestos free", it would seem logical to evaluate airborne fiber characteristics in this other operation acknowledged as containing asbestos. Such a study has been conducted using 10 airborne dust samples collected by the Mining Enforcement and Safety Administration during a 1975 survey. These samples were analyzed by identical electron microscopic methods which have been previously described and results are given in Table 7 along with comparisons with the other mine and mill operations producing products certified to be "asbestos free".

Table 7 clearly demonstrates that all airborne fiber characteristics between these two operations are remarkably the same. In fact, the mine and mill producing "certified" talcs were found to have a statistically ($P<0.05$) significantly higher proportion of positive amphiboles based largely on a higher tremolite fiber content.

Considerations for what constitutes an "asbestos fiber" from an industrial health point of view warrants further discussion. Many researchers continue to promote unusable definitions based on the microscopic world whereas microscopic mineral fibers are of real concern for the health scientist. The data shown in Tables 4 and 7 demonstrate that more than 90 percent of all airborne amphibole fibers in the talc operations studied were shorter than 5 μm in length. Some individuals might argue that these fibers were mineral fragments and not "asbestos", however, it must be pointed out that all industrial operations using or processing asbestos generate airborne fibers similar to those seen in this study. This fact is demonstrated in Table 8 which compares airborne fiber lengths in various operations.

Table 7. Comparison of airborne fiber characteristics between two operations of the same company, one producing asbestos talcs and the other producing talcs certified by the company as asbestos free.

Airborne Fiber Characteristics	Mine and Mill Producing Labeled Talcs	Mine and Mill Producing Unlabeled Talcs	Statistical Significance
Proportion Positive Amphiboles	0.50	0.58	P<0.05
Proportion Anthophyllite	0.47	0.45	NS
Proportion Tremolite	0.03	0.13	P<0.001
Median Fiber Length			
Anthophyllite	1.61 μm	1.45 μm	NS
Tremolite	---[a]	1.55 μm	--
Median Fiber Diameter			
Anthophyllite	0.16 μm	0.13 μm	NS
Tremolite	---[a]	0.19 μm	--
Median Fiber Aspect Ratio			
Anthophyllite	9.9	9.5	NS
Tremolite	---[a]	7.5	--
% of Fibers ≤ 5 μm in Length			
Anthophyllite	92	90-92	NS
Tremolite	---[a]	97	--

[a] Insufficient number of fibers observed for calculation of size distribution.

NS = Not significantly different at 0.05 level

Table 8. Comparison of airborne fiber length distribution in various asbestos operations.

Operation	Fiber Type	Median Length	% ≤ 5 μm
Textile[a]	chrysotile		
fiber preparation and carding		1.4	4
spinning, twisting, weaving		1.0	2
Friction[a]	chrysotile		
mixing		0.9	2
finishing		0.8	2
Asbestos-cement pipe[a]	chrysotile		
mixing		0.9	2
finishing		0.7	1
Study Talc Mine and Mill	tremolite and anthophyllite	1.4 to 1.6	3-10

[a] Taken from reference 8.

Conclusions

Based on the preceding discussion, the following conclusions are drawn.

1. Commercial definitions of asbestos, whereby asbestos fibers are defined on a microscopic scale, have little or no relevance to actual airborne fiber exposures where fibers of microscopic scale are of concern. Furthermore, those mineralogical or geological methods such as examination of ore specimens with a hand lens or low power microscopy are of limited value for routine identification of asbestiform mineral contamination in minerals or mineral products.

2. Users of products containing asbestos have a right to know that they have potential for exposures to asbestos or asbestiform minerals such that proper precautions may be taken to eliminate or reduce exposures. Producers of these products have an obligation to provide these data based on appropriate analytical techniques. Regulatory agencies must insist that appropriate techniques be employed and monitor results.

3. Inasmuch as considerable quantities of data are available suggesting that many fibrous materials may be biologically active [8], consideration should be given for establishing exposure standards for "mineral fibers" as a class of materials with similar health effects. The lives and health of American workers, America's most valuable resource, should not be compromised while the health scientist and the mineralogist disagree over definitions. As Dr. Paul Kotin of the Johns-Manville Corporation stated so well at this conference, the body has not read the asbestos regulations to decide which fibers should cause a biological response. Similarly, neither has the body read a mineralogy text to determine which particles of fibrous minerals should be considered "asbestos" or only mineral fragments.

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Discussion

NOTE: Discussion of this paper was included in the General Discussion at the end of this session.

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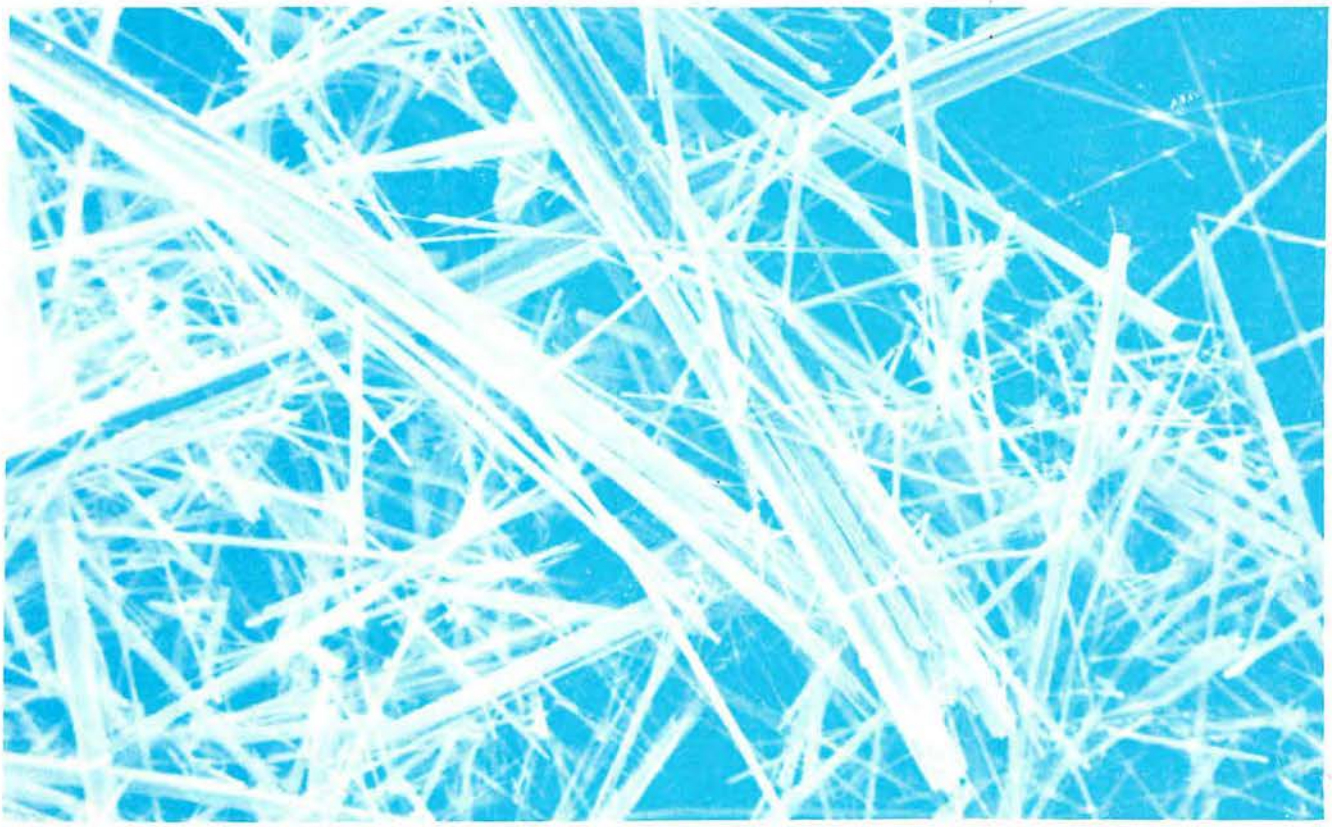
Workshop on Asbestos: Definitions and Measurement Methods



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