National Bureau of Standards Special Publication 506. Proceedings of the Workshop on Asbestos: Definitions and Measurement Methods held at NBS, Gaithersburg, MD, July 18-20, 1977. (Issued November 1978)

SELECTION AND CHARACTERIZATION OF FIBROUS AND NONFIBROUS AMPHIBOLES FOR ANALYTICAL METHODS DEVELOPMENT

J. C. Haartz and B. A. Lange

U.S. Department of Health, Education, and Welfare
Public Health Service, Center for Disease Control
National Institute for Occupational Safety and Health
4676 Columbia Parkway, Cincinnati, Ohio 45226

and

R. G. Draftz and R. F. Scholl

IIT Research Institute (IITRI)
10 West 35th Street
Chicago, Illinois 60616

Abstract

More than 50 mineral specimens of fibrous and prismatic (nonfibrous) amphibole species, including tremolite, grunerite, and cummingtonite, were collected and characterized to determine their suitability for use as reference materials in the development of analytical methods. These methods will be used for the detection and measurement of hazardous materials which are found as workplace contaminants. The specimens have been characterized using light microscopy, x-ray diffraction (XRD), and differential thermal analysis (DTA). Some of these specimens have been purified by appropriate physical or chemical techniques and then ground to provide a material with a mass median particle size of less than 10 μm (major) diameter. The results of characterization studies of the minerals, including a comparison of the properties determined for each of the specimens, are presented. Differences in physical properties of the fibrous and prismatic tremolite specimens are indicated by the data obtained from DTA and XRD studies. While the prepared quantity of each mineral is quite limited, the source of each of the specimen materials and the appropriate methods of sample preparation have been carefully documented should additional quantities be desired.

Key Words: Amphibole asbestos; cummingtonite; grunerite; thermal analysis; tremolite; x-ray diffraction.

Introduction[1]

Under the provisions of the Federal Occupational Safety and Health Act of 1970 (PL 91-596), the National Institute for Occupational Safety and Health (NIOSH) is charged with the responsibility for research related to occupational health, including the development and evaluation of analytical methods for the determination of hazardous workplace contaminants. To meet this charge, the Measurements Research Branch of NIOSH has a program concerned with the development of new analytical methods as well as with the

---

[1] Mention of product or trace names does not constitute endorsement by the Public Health Service.

evaluation and improvement of existing methods. Many mineral dusts, such as those of the silica polymorphs, talc, and asbestos minerals, are included in the hazardous materials for which analytical methods are needed. Earlier work in the NIOSH laboratory showed that it was feasible to quantitatively determine by x-ray diffraction techniques (XRD) chrysotile, amosite, and crocidolite using either samples of the bulk material or of airborne dust collected on filters [1][2]. However, further work rather graphically demonstrated the fact that specimens of a mineral originating from different deposits often exhibit significant variations in impurity content and crystallinity [2], and consequently also exhibit vast differences in their response to analytical measurement techniques. It was obvious that reference materials were needed for the development of analytical methods, that these materials should be from natural sources, and that they be selected on the basis of purity, especially as to an absence of other similar minerals. Pure minerals could then be mixed with other materials to simulate the mixtures found in samples collected from occupational environments.

For asbestos, the International Union Against Cancer (UICC) Standard Reference Samples [3] are available as reference materials for chrysotile, amosite, anthophyllite, and crocidolite. These samples have been well characterized with respect to overall chemical composition (elemental weight o/o) and fiber length distribution [4]. There are also some data relating to sample response to heat treatment, and the electron and x-ray diffraction properties [4,5]. However, since these materials were collected and prepared to provide reference samples for inhalation and injection experiments, they were chosen not for phase purity but to be representative of the various types of asbestos used by industry. Further, the UICC samples do not include specimens of the prismatic (nonfibrous) forms of the minerals.

Other reference materials were also needed by NIOSH for the methods development and evaluation program. Consequently, an effort to collect and characterize at least four representative specimens of each of eighteen minerals from different geographical locations was initiated. Table 1 lists the minerals sought and the techniques used for preliminary characterization of the samples. Following the preliminary evaluation and characterization of these samples, the "best" source specimens were chosen for beneficiation, grinding to a respirable size range, and for further characterization and analysis for impurities. A one kilogram quantity of the ground material was established as the final, processed amount to be prepared of each mineral. It was expected that this amount would suffice as reference material for NIOSH analytical research; the source of selected specimens and the appropriate methods for sample preparation were carefully documented should additional quantities be desired.

The following discussion will cover the selection, preliminary separation techniques, beneficiation, grinding, and characterization of some of the amphibole species. Details concerning the other minerals will be published separately.

Selection of Minerals

More than 80 sources were contacted to obtain the approximately 50 samples of mineral specimens containing amphiboles which were received and inspected. Of these samples, 12 were discarded based on macroscopic examination; 38 were carried through the preliminary characterization steps prior to the final selection of the eleven "best" amphibole samples. Since the final quantity of each mineral needed was large (one kilogram), specimens were chosen based on (1) the least contamination by other minerals and the contrasting habit, and, (2) the amenability of the specimen to beneficiation for removal of contaminant phases.

---

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

Table 1. Reference materials sought.

Mineral	Characterization Techniques
Silica	X-ray Diffraction Infra-red Spectroscopy Thermal Analysis (TG and DTA)
-Quartz	
-Cristobalite	
-Tridymite	
Beryl	
Bunsenite (NiO)	
Fluorite	
Talc	
Fibrous Serpentine	Macroscopic Habit Light Microscopy X-ray Diffraction Thermal Analysis
-Chrysotile	
Platy Serpentine	
-Antigorite	
Fibrous Amphiboles	
-Crocidolite	
-Grunerite ("Amosite")	
-Anthophyllite	
-Tremolite	
Prismatic Amphiboles	
-Riebeckite	
-Grunerite	
-Cummingtonite	
-Anthophyllite	
-Tremolite	

After a macroscopic inspection of the specimens as received, using a hand magnifier, portions were hand ground in an agate or diamonite mortar and pestle. The ground samples were dry sieved to pass a 325 mesh screen and were further characterized using polarized light microscopy, qualitative x-ray diffraction (XRD), and qualitative differential thermal analysis (DTA). The types and quantities of impurities were noted for each of the specimens, and careful scrutiny was given to the mineral morphology, especially for the samples needed for the fibrous and prismatic (or nonfibrous) habits.

For macroscopic specimens, the mineralogical criteria distinguishing the fibrous from the prismatic habit are unequivocal. This is illustrated by the samples of tremolite which are shown in figures 1 through 4. The origin of the fibrous tremolite shown in figure 1 is Alaska, while that of figure 2 is a small sample from Italy which was collected in approximately 1890 and has since been in the collection of the Field Museum of Natural History in Chicago, IL. It was not possible to locate a contemporary source of fibrous tremolite in Italy. The prismatic tremolite in figure 3 is from South Dakota and is a fairly pure sample with an acicular radiated structure which is quite evident in the hand specimens. The sample shown in figure 4 contains interlaced prismatic tremolite, talc and other impurities. Although the individual tremolite "needles" are colorless, the sample has a lavender color which may be due to manganese substitutions [6].

Figure 1. Fibrous tremolite: Alaska, 1X.

Figure 2. Fibrous tremolite: Tuscany, Italy, 1X.

Figure 3. Prismatic tremolite with calcite: South Dakota, 0.57X.

Figure 4. Prismatic tremolite with talc and other impurities, 0.5X.

Distinguishing between the fibrous and prismatic habits is less straightforward with microscopic specimens. The photomicrographs of tremolite (figures 5 and 6) illustrate the appearance of fibrous and prismatic tremolite specimens ground to a mean particle size of 3.1 µm and 1.7 µm respectively. Similarities in particle shape are evident, although the mean aspect ratio of the fibrous tremolite particles is greater than that of the cleavage fragments of the prismatic material.

Figure 5. Fibrous tremolite: Rajasthan State, India, 407X.

Figure 6. Prismatic tremolite: Gouverneur, New York, 407X.

Table 2 lists the amphiboles, and their sources, which were chosen for any necessary beneficiation and final grinding. The impurities listed are those contaminants determined by microscopic analysis of the hand-separated portions of the desired phase. Some of the amphiboles, including the samples of prismatic and fibrous tremolite as well as crocidolite, were obtained as nearly pure, single phase specimens. Others, such as the prismatic grunerite, anthophyllite, and cummingtonite were intermixed with accessory minerals. Hand specimens of the amphiboles selected for preparation as reference materials are illustrated in figures 7-14.

Table 2. Amphibole sources.

Mineral	Geographical Origin	Representative Impurities
Tremolite		
Fibrous	Udaipur District Rajasthan, India	Plant fragments (carbonaceous) & other minerals, ≤3%
Prismatic	Gouverneur, N.Y.	Talc, Limestone, Hematite, ≤2%
Cummingtonite	Homestake Mine, Lead, So. Dakota	Calcite, Quartz, other minerals, ∼30%
Grunerite		
Fibrous ("Amosite")	Lydenburg District Transvaal, South Africa	Magnetite & other minerals, ≤11%
Prismatic	Luce #1 Mine Newfoundland	Quartz, Magnetite, other minerals, ∼50%
Anthophyllite		
Fibrous	Bozeman, Montana	Magnetite, Calcite & other minerals, ≤11%
Prismatic	Bamble, Norway	Quartz, Mica, Rutile, Magnetite, other minerals, ∼25%
Crocidolite	South Africa	Phases which are too fine to identify, ≤2%
Riebeckite	St. Peter's Dome El Paso County, Colorado	Quartz, feldspar, iron oxide, and other minerals, ∼15%

Figure 7. Fibrous tremolite: Rajasthan State, India, 1X.

Figure 8. Prismatic cummingtonite with associated minerals: Homestake Mine, Lead, South Dakota, 0.8X.

Figure 9. Fibrous grunerite ("Amosite"): Lydenburg District, Transvaal, South Africa, 0.8X.

Figure 10. Prismatic grunerite with quartz: Luce No. 1 Mine, Newfoundland, 0.8X.

Figure 11. Fibrous anthophyllite: Bozeman, Montana, 1X.

Figure 12. Prismatic anthophyllite with quartz: Bamble, Norway, 0.8X.

Figure 13. Crocidolite (fibrous riebeckite): South Africa, 0.57X.

Figure 14. Prismatic riebeckite (black) with quartz and feldspar: St. Peter's Dome, El Paso County, Colorado, 1X.

Separation and Grinding Techniques

For those samples which required beneficiation to produce the pure minerals, separation techniques were chosen which would adequately liberate the desired phases and least adversely affect their purity. In order to conserve the selected minerals, techniques were chosen which could be applied to material varying widely in size. The preliminary size reduction necessary for beneficiation and grinding of the fibrous amphiboles was accomplished using a rock saw with diamond-impregnated blades. For the nonfibrous amphiboles, a large mortar and pestle were fabricated from strongly magnetic stainless steels so that metals abraded from the equipment during crushing could be removed from the ground material using a magnet. All beneficiation steps were done before the final grinding to allow efficient use of the mineral extraction methods, which are severely limited if the particle size is too small. To avoid chemical alteration of the desired phases, beneficiation was generally limited to physical methods [7]. The final grinding was designed to produce nonfibrous materials which had a mass median aerodynamic diameter between 0.5 and 5.0 µm, and a maximum size of 10 µm. For the fibrous materials, the desired median length was the range 2-10 µm, with a maximum length of 200 µm.

Beneficiation Methods

Simple, primarily physical methods of mineral extraction were employed. Three types of hand separation were used: (1) With a mason's hammer and chisels, the available specimen material was "high-graded" to obtain pieces with the greatest concentration of the desired phase; from these, the larger masses of impurities were cobbed. (2) The rock saw was used to cut cross-fiber vein materials into slabs one centimeter thick measured along the fiber length. The slabs were then chipped into small pencils of fibers for further beneficiation and/or preparation for milling. The saw was also used to cut wall rock from the margins of cross-fiber vein specimens of fibrous grunerite, anthophyllite, and crocidolite. (3) Hand picking, or for ferromagnetic minerals a powerful hand magnet, was used to remove small quantities of obvious contaminants at any stage in the size reduction procedure.

Only two beneficiation techniques were used in which mineral specimens were exposed to the risk of chemical alteration. Slow dissolution of carbonate minerals from specimens of tremolite and actinolite was accomplished by digestion in dilute (∿3 N) acetic acid. Bromoform and tetrabromoethane were used for density separations of quartz, micas, and other silicates from tremolite, cummingtonite and grunerite. After separation, the samples were rinsed repeatedly, with acetone or ethanol and then distilled water, to remove residues of the organic liquids.

Grinding Techniques

Research has shown that some grinding mechanisms degrade the crystalline structure of minerals, particularly asbestiform species, to a considerable degree. Shearing and cutting (in the sense of pinching) actions are reported to be very destructive to crystallinity [8]. Initial attempts in this program to grind asbestos in ball mills equipped with lifter bars confirmed this observation. Impact between air-suspended particles and/or impact of elongate fragments on cutting edges accomplished size reduction with much less reduction in crystallinity, as shown by x-ray diffraction studies. Therefore, grinding tests were made to identify milling devices which exploit the free impact principle and which could efficiently produce large quantities of respirable size particles.

For size reductions of fibrous amphiboles, a fiber mill (Retsch Ultracentrifugal-mill, Type ZM-1) was chosen. In this device a rotor with vertical pins at the periphery spins at 10,000 or 20,000 rpm impelling fibers outward against the perforated wall of the grinding chamber (sieve ring) on which cutting edges are angled toward the oncoming particles. The non-fibrous amphiboles were ground using a jet mill (Micron-master Jet Pulverizer) in which tangentially inward-directed jets of dry, filtered air (50 scfm at 90 psig) circulate the feed material in an annular grinding chamber. Size reduction is accomplished by impact between particles; the air stream minimizes particle contact with the walls of the grinding chamber. Additional advantages of the fiber mill and jet mill for this work are: (1) the carrying air stream controls heat build-up in the equipment thereby reducing the risk of thermal degradation of the material being milled. (2) Virtually all particles are subjected to size reduction with each pass of material through the mill. (3) Each mill is provided with a cyclone collector, thus providing coarse and fine fractions. (4) The continuous processes permit efficient size reduction of kilogram quantities of fibrous and nonfibrous amphiboles to the specified size by iterative milling without additional size classification steps. Table 3 presents particle size distributions for fibrous and prismatic tremolite reduced to final size by the respective milling devices.

Table 3. Particle sizes[a] of "reference" tremolite samples after grinding.

Fibrous (India)		Prismatic (New York)	
Size Range (μm)	Number Percentage	Size Range (μm)	Number Percentage
<2	37.4	<1	28.0
2-6	35.8	1-3	47.0
6-10	15.4	3-5	18.3
10-20	6.9	5-7	5.9
20-80	3.6	7-10	0.8
80-160	0.9	>10	0.0
>160	0		
3.1 μm Geometric Mean		1.7 μm Geometric Mean	

---

[a] Particle sizes determined using optical microscopy. For fibrous tremolite, fiber length is reported; for prismatic tremolite, Feret's diameter.

Analytical Studies

Analytical studies have been initiated using two of the "reference" materials from this program, the fibrous and prismatic tremolite samples. In addition to these "reference" samples, which were processed by IITRI, and which were carefully characterized as to identity, source, and particle size, a number of samples from the NIOSH mineral collection were used. These samples were included in the analyses to allow comparisons of tremolite specimens from various sources and geographical locations to determine if general characteristics of tremolite specimens could be delineated by obtaining additional experimental data. The NIOSH specimens were ground in a SPEX freezer mill at liquid nitrogen temperatures, sieved through a 10 μm sieve, and sized using electron microscopy techniques. The ground material had a mean particle length or diameter of <3.0 μm. The following sections summarize the preliminary results obtained in the studies of tremolite.

Chemical Analyses

The relative iron, magnesium, and calcium content of several of the specimens used in these studies was determined in order to confirm the designation of these amphiboles as tremolite. To minimize contamination which could occur from contact with metallic surfaces during grinding, pieces of the hand specimens instead of ground material were used for the analyses. These pieces were dissolved by heating in a mixture of HF and concentrated HCl. Blind replicate analyses were done for each of the specimens using atomic absorption spectrophotometry. The results (table 4) for the ratio (Fe + Mg : Ca) and the calculated weight percent FeO indicate that all of the samples fall within the empirical composition limits for tremolite [9], including a specimen previously identified as prismatic actinolite. In general, the specimens of fibrous tremolite contain more iron than the prismatic form although the South Korean sample of fibrous tremolite was an exception.

Table 4. Chemical analyses.

Amphibole	Atom Ratio Fe : Mg : Ca	Atom Ratio Fe + Mg : Ca[a]	Wt. % FeO
Prismatic Tremolite			
Gouverneur, N.Y.[b]	1 : 205 : 78	5.3 : 2.0	0.21
South Dakota	1 : 44 : 21	4.3 : 2.0	1.03
Fibrous Tremolite			
Rajasthan, India[b]	1 : 13 : 6	4.7 : 2.0	2.87
Alaska	1 : 13 : 6	4.7 : 2.0	3.03
Korea	1 : 33 : 16	4.3 : 2.0	0.69
Italy	1 : 31 : 14	4.6 : 2.0	1.43
Prismatic Actinolite[c]			
South Dakota	1 : 15 : 7	4.6 : 2.0	2.63

---

[a] Theoretical limit of ratio = 5:2.

[b] "Reference" material, supplied by IITRI.

[c] Classification based on color and location of source.

X-Ray Diffraction Studies

For the x-ray powder diffraction studies of the "reference" tremolites, both bulk powder samples (packed in cups) and thin layers on silver membrane filters were used. For the filter studies, homogeneous suspensions of known tremolite concentration in isopropanol were prepared using ultrasonic agitation to ensure dispersion. Aliquots of this suspension were filtered through 25 mm, 0.45 µm pore size silver membrane filters. The calculated weight of tremolite deposited was confirmed by weighing, using a microbalance. For both fibrous and prismatic tremolite the 310 and 110 peaks (3.14 Å and 8.38 Å, CuKα radiation) were step scanned to determine the integrated peak intensities. The calibration curves (figure 15) were prepared by plotting the net normalized integrated intensities of these peaks versus the amount of tremolite on the filters.

The data clearly indicate that quantitation of pure samples as small as 20 µg is feasible. However, the ratios of the reflections, $I_{(110)}:I_{(310)}$, are different for filter deposits of fibrous and prismatic habits. The peak ratio (8.38 Å:3.4 Å) for prismatic tremolite is approximately 1.0 while that for the fibrous tremolite is approximately 0.40. Packed bulk samples of both tremolite habits give the same peak ratio, the value of which is 0.20. Information in the Powder Diffraction File [10] indicates a peak ratio of 1.0 for tremolite from St. Gotthard, Switzerland. The morphology is described as "white

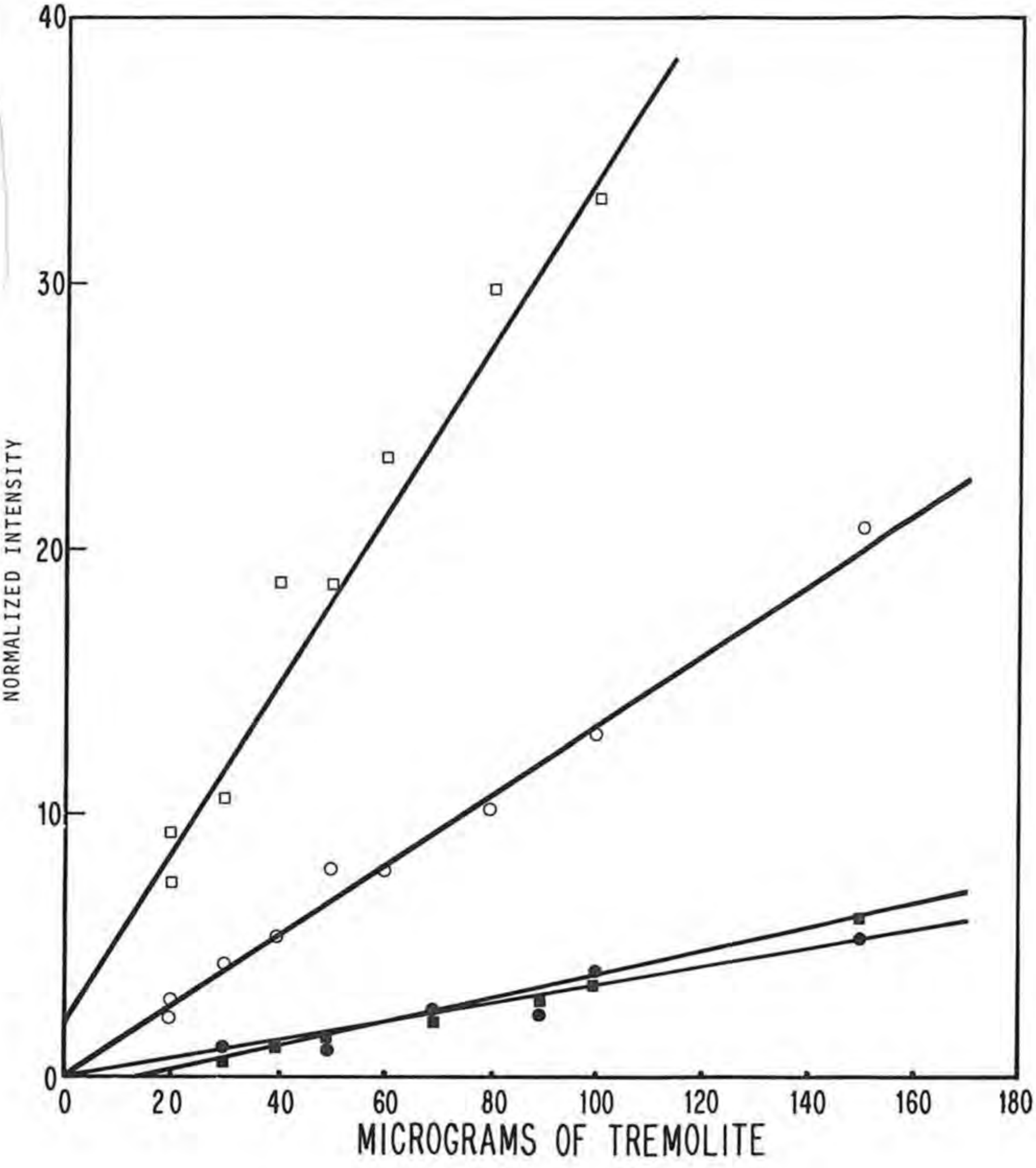


Figure 15. Calibration curves for fibrous and prismatic tremolite:
Fibrous tremolite: □ 3.14 Å; ○ 8.38 Å
Prismatic tremolite: ● 3.14 Å; ■ 8.38 Å

radiating fine fibrous masses," but the term "radiating" suggests it may be a prismatic form. Data obtained for specimens of tremolite from other geographical locations indicate that, for material deposited on filters, the samples of prismatic tremolite in general show a larger ratio for these peaks than do samples of fibrous tremolite (table 5).

Table 5. Ratio of XRD peaks observed for fibrous and prismatic tremolite.[a]

Amphibole	No. of Replicates	Ratio (8.38Å:3.14Å)
Prismatic Tremolite		
Gouverneur, N.Y.[b]	7	1.04
South Dakota	5	1.08
Newburyport, Mass.	10	1.45
Fibrous Tremolite		
Rajasthan, India[b]	7	0.39
Alaska	10	0.55
Korea	5	0.35
Italy	5	1.25

---

[a] 150 µg on 0.45 µm pore size silver filters.

[b] "Reference" material, supplied by IITRI.

At this point no explanation can be advanced to account for the differences in peak ratios, although the effects observed may be due at least in part to preferred orientation of the particles in some or all of the samples. Regardless of the reason, the effect is seen for a variety of samples and for a wide range of filter loadings as demonstrated by the calibration curves. The distinctions observed using this technique may prove useful in analytical attempts to ascertain the type of material to which a worker is being exposed.

Thermal Analysis Studies

Preliminary differential thermal analysis (DTA) studies on tremolite samples have been completed. These studies included an evaluation of the feasibility of this technique for the quantitative analysis of tremolite and, while good calibration curves were obtained, DTA was not sensitive enough to detect microgram quantities of tremolite. The samples were heated in platinum cups to a temperature of 1150 °C at a heating rate of 10°/min in dry air flowing at 5.7 L/hr; the instrument was calibrated using $SrCO_3$, an NBS-ICTA Standard Reference Material.

In parallel with the XRD studies of the "reference" tremolite samples, differences between these samples (table 6) were observed during the thermal studies of fibrous and prismatic tremolite samples. These differences in peak position and the color of the decomposition product were observed for samples from other geographical locations as well as for the "reference samples." Similar differences were observed by IITRI for those specimens considered for selection as "reference" materials. All samples displayed the strong endotherm which is associated with the loss of structural water and the breakdown of the amphibole structure, which subsequently recrystallizes to a monoclinic pyroxene [11]. However, the data indicate that in general the fibrous tremolite samples dehydrate and recrystallize at a lower temperature than do the prismatic tremolite samples. This behavior is analogous to that noted for serpentine, i.e., chrysotile loses structural water at a lower temperature than does antigorite [12]. Although it is recognized that differences in particle size, grinding techniques and experimental conditions can affect the position of a DTA peak [13], data obtained in both the NIOSH and IITRI laboratories are consistent in showing that the endotherm of fibrous tremolite is lower by approximately 50 °C than that of the prismatic tremolite. It was also observed that the pyroxenes formed from fibrous tremolite were always brown to tan in color while the pyroxenes formed from the prismatic tremolite were always white in color. However, XRD scans of the pyroxenes were virtually the same regardless of color or origin of the specimen and indicated that the final decomposition material was primarily diopside.

Table 6. Thermal analysis of tremolite.[a]

Amphibole	No. Samples	DTA[a] Endotherm, °C	Color of Pyroxene
Fibrous Tremolite			
NIOSH[b]	4	1026 ± 27	tan
IITRI	1	1002	not determined
Prismatic Tremolite			
NIOSH[c]	5	1078 ± 20	white
IITRI	4	1053 ± 11	white

---

[a] NIOSH samples included those listed in Table 5 as well as two additional samples from the Gouverneur, N.Y. area; IITRI samples include those screened as potential "reference" materials.

[b] Geometric mean particle length <3.0 μm.

[c] Geometric mean particle maximum dimension <3.0 μm.

Summary and Conclusion

The analytical studies planned for the reference materials have been initiated using the tremolite specimens. These studies have indicated that x-ray diffraction may turn out to be an even more useful tool than expected. The detection limits obtained and the differences in peak ratios observed for samples of fibrous and prismatic tremolite on silver filters have potential for applications to analyses of hazardous, workplace contaminants.

The authors gratefully acknowledge the guidance and assistance received during this program from J. V. Crable of NIOSH and B. G. Woodland of the Field Museum.

References

[1] Crable, J. V., Am. Ind. Hyg. Assoc. J., 27, 293 (1966).

[2] Nenadic, C. M. and Crable, J. C., Am. Ind. Hyg. Assoc. J., 32, 539 (1971).

[3] Timbrell, V., Gibson, J. C. and Webster, I., Int. J. Cancer, 3, 406 (1968).

[4] Timbrell, V., "Pneumonoconiosis. Proceedings Int. Conf.," Johannesburg, 1969, . 28-36.

[5] Skikne, M. I., Talbot, J. H., and Rendall, R. E. G., Env. Res., 4, 141 (1971).

[6] Deer, W. A., Howie, R. H., and Zussman, J., "Rock-Forming Minerals, Vol. 2, Chain Silicates," J. Wiley & Sons, N.Y., N.Y. 1963 p. 253.

[7] Muller, L. D., "Laboratory Methods of Mineral Separation," CH. 1, in J. Zussman, ed., Physical Methods in Determinative Mineralogy, Academic Press, London & N.Y., 1967.

[8] Ocella, E. and Maddalon, G., Med. Lavoro, 54 (#10), 628 (1963).

[9] See, for example, Deer, W. A., Howie, R. A., and Zussman, J., "Rock-Forming Minerals, Vol. 2, Chain Silicates," J. Wiley & Sons, N.Y., N.Y. 1963, p. 251.

[10] Powder Diffraction File. Joint Committee on Powder Diffraction Standards, 1601 Park Lane, Swarthmore, PA 19081.

[11] Deer, W. A., Howie, R. H., and Zussman, J., "Rock-Forming Minerals, Vol. 2, Chain Silicates," J. Wiley & Sons, N.Y., N.Y. 1963, pg. 254.

[12] Deer, W. A., Howie, R. H., and Zussman, J., "Rock-Forming Minerals, Vol. 3, Sheet Silicates," J. Wiley & Sons, N.Y., N.Y. 1962, pg. 180.

[13] Martinez, E., Am. Miner., 46, 901 (1961).

Discussion

I. STEWART: Both DTA and x-ray diffraction are very sensitive to packing, and, of course, this can be related to shape. Did you do any tests to determine whether packing or repacking would change the relative ratios of peak heights or peak positions?

J. HAARTZ: No, we haven't.

STEWART: Or spinning the sample in x-ray diffraction perhaps?

HAARTZ: The relative ratios of the peaks in x-ray diffraction were the same for the bulk samples. For the samples that were deposited on a silver filter, that is a very thin layer; we did see the differences in the peak ratios. This was the case not only with samples of different origins, but with a great many replicas of the same material.

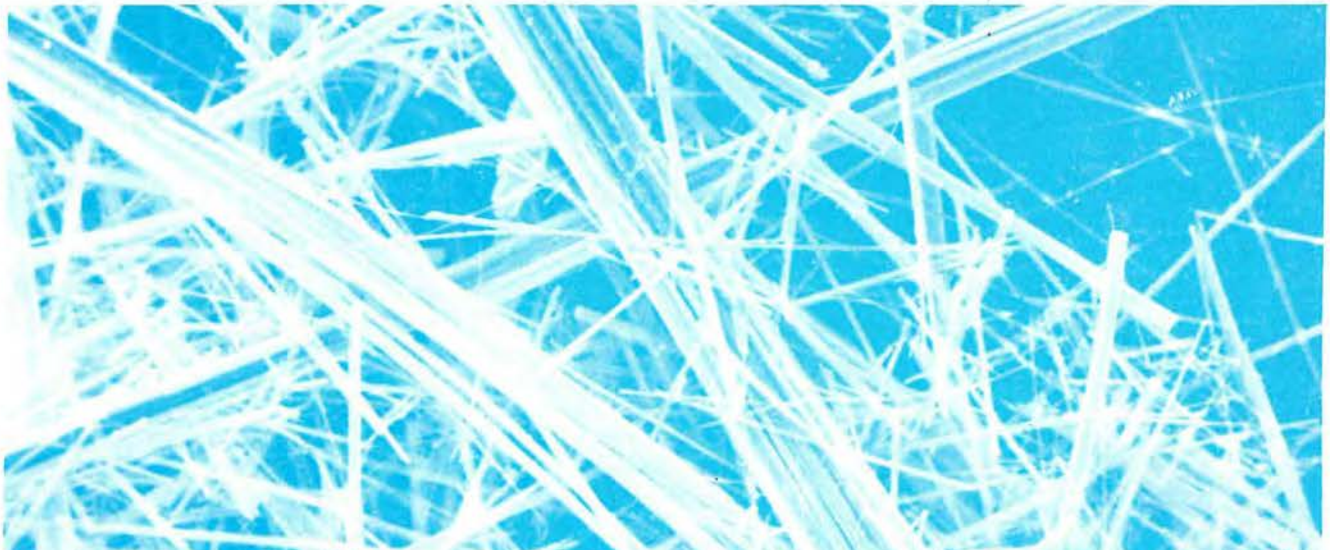
STEWART: I see. So, it was purely the fact that it was fibrous, you think? I didn't quite catch what you meant by your bulk sample. By bulk, I was equating that with "massive." You mean a bulk fiber sample.

HAARTZ: By a bulk sample, I mean a milligram or more, of either the massive or fibrous, showed the same diffraction pattern: identical. When these samples are deposited as a thin layer on a silver membrane filter and the pattern taken, we do see differences in the peak ratios.

NBS SPECIAL PUBLICATION 506

U.S. DEPARTMENT OF COMMERCE / National Bureau of Standards

Workshop on Asbestos: Definitions and Measurement Methods



NATIONAL BUREAU OF STANDARDS

The National Bureau of Standards[1] was established by an act of Congress March 3, 1901. The Bureau's overall goal is to strengthen and advance the Nation's science and technology and facilitate their effective application for public benefit. To this end, the Bureau conducts research and provides: (1) a basis for the Nation's physical measurement system, (2) scientific and technological services for industry and government, (3) a technical basis for equity in trade, and (4) technical services to promote public safety. The Bureau's technical work is performed by the National Measurement Laboratory, the National Engineering Laboratory, and the Institute for Computer Sciences and Technology.

THE NATIONAL MEASUREMENT LABORATORY provides the national system of physical and chemical and materials measurement; coordinates the system with measurement systems of other nations and furnishes essential services leading to accurate and uniform physical and chemical measurement throughout the Nation's scientific community, industry, and commerce; conducts materials research leading to improved methods of measurement, standards, and data on the properties of materials needed by industry, commerce, educational institutions, and Government; provides advisory and research services to other Government Agencies; develops, produces, and distributes Standard Reference Materials; and provides calibration services. The Laboratory consists of the following centers:

Absolute Physical Quantities[2] — Radiation Research — Thermodynamics and Molecular Science — Analytical Chemistry — Materials Science.

THE NATIONAL ENGINEERING LABORATORY provides technology and technical services to users in the public and private sectors to address national needs and to solve national problems in the public interest; conducts research in engineering and applied science in support of objectives in these efforts; builds and maintains competence in the necessary disciplines required to carry out this research and technical service; develops engineering data and measurement capabilities; provides engineering measurement traceability services; develops test methods and proposes engineering standards and code changes; develops and proposes new engineering practices; and develops and improves mechanisms to transfer results of its research to the utlimate user. The Laboratory consists of the following centers:

Applied Mathematics — Electronics and Electrical Engineering[2] — Mechanical Engineering and Process Technology[2] — Building Technology — Fire Research — Consumer Product Technology — Field Methods.

THE INSTITUTE FOR COMPUTER SCIENCES AND TECHNOLOGY conducts research and provides scientific and technical services to aid Federal Agencies in the selection, acquisition, application, and use of computer technology to improve effectiveness and economy in Government operations in accordance with Public Law 89-306 (40 U.S.C. 759), relevant Executive Orders, and other directives; carries out this mission by managing the Federal Information Processing Standards Program, developing Federal ADP standards guidelines, and managing Federal participation in ADP voluntary standardization activities; provides scientific and technological advisory services and assistance to Federal Agencies; and provides the technical foundation for computer-related policies of the Federal Government. The Institute consists of the following divisions:

Systems and Software — Computer Systems Engineering — Information Technology.

[1] Headquarters and Laboratories at Gaithersburg, Maryland, unless otherwise noted; mailing address Washington,D.C. 20234.

[2] Some divisions within the center are located at Boulder, Colorado, 80303.

The National Bureau of Standards was reorganized, effective April 9, 1978.

Proceedings of Workshop on Asbestos: Definitions and Measurement Methods

Proceedings of a Workshop on Asbestos held at
the National Bureau of Standards,
Gaithersburg, Maryland, July 18-20, 1977

Edited by

C. C. Gravatt, Philip D. LaFleur,
and Kurt F. J. Heinrich

National Measurement Laboratory
National Bureau of Standards
Washington, D.C. 20234

Sponsored by
National Bureau of Standards
of the Department of Commerce
and Occupational Safety and Health Administration
of the U.S. Department of Labor

U.S. DEPARTMENT OF COMMERCE, Juanita M. Kreps, Secretary
Dr. Sidney Harman, Under Secretary
Jordan J. Baruch, Assistant Secretary for Science and Technology
NATIONAL BUREAU OF STANDARDS, Ernest Ambler, Director

Issued November 1978

Library of Congress Catalog Card Number: 78-600109
National Bureau of Standards Special Publication 506
Nat. Bur. Stand. (U.S.), Spec. Publ. 506, 490 pages (Nov. 1978)
CODEN: XNBSAV

U.S. GOVERNMENT PRINTING OFFICE
WASHINGTON: 1978

For sale by the Superintendent of Documents, U.S. Government Printing Office
Washington, D.C. 20402 - Price $6.75

Stock No. 003-003-01993-3