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September 23, 2008

Ms. Diane Miller
NIOSH Docket Office
Robert A. Taft Laboratories
4676 Columbia Parkway, MC-C34
Cincinnati, Ohio 45226

RE: Revised Draft: Asbestos Fibers and Other Elongated Mineral Particles: State of the Science and Roadmap for Research

Dear Ms. Miller:

The National Institute for Occupational Safety and Health (NIOSH) has requested public comment on the captioned revised draft. R. T. Vanderbilt Company, Inc. (RTV) provided extensive comment on the prior draft and is pleased to provide additional commentary.

For close to four decades, RTV has played a prominent role in regard to the proper characterization and understanding of elongated mineral particles (EMPs) - elongated amphibole cleavage fragments in particular. RTV has supported research in this area and was among the first to call attention to a number of issues raised in this NIOSH initiative. As stated in our earlier submission, RTVs talc miners and millers in upstate New York may be the most exposed work population in the world to amphibole cleavage fragments.

Also noted in our prior submission, RTV believes that NIOSH has helped create many of the questions it now seeks to answer. We also believe that the science base concerning nonasbestiform amphibole particulate is more complete then NIOSH previously or currently suggests. After reviewing public comments, written submissions and the comments of Peer Reviewers in response to the first Roadmap draft, it is clear RTV is not alone in holding this view.

Documents RTV finds most pertinent were provided previously in nine file folders in our response to the prior draft. A summary of our experience and position on these issues was also provided in our "White Paper" document. In commenting on the current draft document, reference to documents previously provided will be made. If requested, copies of these documents will be resubmitted.

An effort will be made to link comments to pertinent sections, page(s) and line(s) in the present draft as NIOSH has requested. The expanded comments are appended to this correspondence.

As a general comment, however, RTV believes two orientations or "philosophies" have emerged more distinctly in regard to asbestos fibers and EMPs. Evidence of these positions can be seen in the comments of the peer reviewers selected by NIOSH as well as in public input following issuance of the first NIOSH Roadmap draft. The two views might simply be described as: (a) those who feel elongated mineral particulate should be "defined" on the basis of risk, and (b) those who feel all substances should be accurately and fully characterized, regardless of risk. We believe the view that certain elongated cleavage fragments (nonasbestiform) might be considered asbestos fibers is an example of utilizing risk to define a mineral particulate.

RTV supports the latter view because we feel allowing overly-broad, non-scientific and at times emotion driven exposure characterization serves only to retard the understanding of risk. Further, the fear that accurate exposure characterization may preclude the ability to control identified risks seems misplaced.

NIOSH does make it clear in the current draft that it does not define any nonasbestiform elongated mineral particulate as "asbestos" (page vii). NIOSH has, however, historically emphasized a "risk based" orientation in regard to addressing elongated minerals and we believe this has contributed significantly to broad brush generic fiber thinking and a willingness to disregard proper nomenclature. This thinking remains a prominent theme in the present draft although an improvement in the recognition of the complexities of mineral science is noted.

It is notable that one NIOSH peer reviewer ***pleaded*** with NIOSH "to describe minerals accurately and to employ mineral-related terminology rigorously." The reviewer further points out that: "The correlation between health effects and properties of mineral particles is a classic interdisciplinary problem." Since NIOSH does not have in house mineral science expertise, the reviewer then strongly encouraged NIOSH to obtain help in this critically important area going forward. We could not agree more.

Very truly yours,

R. T. VANDERBILT COMPANY, INC.

John W. Kelse, Corporate Industrial Hygienist
Director, Occupational Health

<u>Page Specific Comments:</u> *Revised Draft* – Asbestos Fibers and Other Elongated Mineral Particles: State of the Science and Roadmap for Research

Prepared by: John Kelse, Corporate Industrial Hygienist
Director, Occupational Health

<u>Page 3</u> **(Figures 1 & 2):** An effort is made in these figures to provide a visual comparison between tremolite cleavage fragments and "Commercial Asbestos – Tremolite" from the NIST Standard Reference Material 1867a. While pictures are worth a thousand words and were requested by several reviewers, this is a very poor pictorial comparison. The "asbestos" figure predominantly shows cleavage fragments and not the asbestiform crystal growth habit the picture text says it shows.

In our prior Roadmap submission, File # 7 contained clear evidence that the NIST Standard Reference 1867a for tremolite used in figure #2 contains considerable nonasbestiform tremolite (cleavage fragments) – along with actual tremolite asbestos. NIOSH is encouraged to look to a purer tremolite asbestos standard for photomicrographs of actual tremolite asbestos so that the reader is provided a true photomicrograph comparison. Lapses of this sort serve only to further confuse and obscure the issues.

RTV often recommends the work of the US Bureau of Mines as a viable reference source in terms of demonstrating the difference in crystal growth habit and clarification of mineral terms. Interestingly, it is believed that even the BOM counted and sized cleavage fragments in an asbestos standard material when attempting to size amphibole asbestos fibers by aspect ratio (IC 8751 BOM Circular/1977 – Select Silicate Minerals and Their Asbestiform Varieties: Mineralogical Definitions and Identification-Characterization – Figure 42 page 44). Even so, the work of the BOM provides a much improved comparison and discussion of the difference in crystal growth habit and various crystal shapes and patterns (single and aggregate) then does the present NIOSH draft. The drawings on page 24 (Figure 21) of this BOM document might be particularly helpful. A copy of this BOM Circular is appended.

<u>Page 16-17</u>: There is discussion on these pages regarding the regulation and control of asbestiform minerals (amphiboles in particular) that fall outside the six asbestos regulated minerals but known to pose the same pathogenic effect as asbestos. RTV would agree that enough flexibility in the regulatory system should exist to apply appropriate standards and controls when any significant risk is reasonably identified. As stressed in our cover correspondence, however, this should not be accomplished by abandoning the need to maintain accurate exposure characterization.

Like NIOSH, RTV believes it is reasonable that the Libby asbestiform amphiboles winchite and richterite be included with the 5 listed asbestiform amphiboles and regulated under the generic term "asbestos". Health evidence and similarity in mineralogy support this. Other asbestiform or highly fibrous minerals outside the amphibole mineral group that reasonably show a "same as" asbestos risk (e.g. fibrous erionite – a zeolite), might also be regulated as stringently as asbestos (e.g. "apply asbestos std.") but should not lose their identity as a discrete mineral species or simply be referred to as asbestos due to a broader difference in the mineral species (not an amphibole or serpentine).

Most importantly, care must be exercised not to "lump" nonasbestiform elongated mineral particulates of any kind under a single generic term that infers a single risk without adequate evidence for doing so (a debatable practice even then as the definition of "adequate" is likely to be different one researcher to the next). We believe to error on the side of safety is commendable, but also that prudence can be stretched too far – especially when it involves viewing major portions of the earth's crust as or "like" asbestos.

Epidemiological Studies (pages 18-27)

NIOSH was responsive to commenter requests that the human cohort studies involving exposure to elongated amphibole cleavage fragments receive expanded discussion. Predictably, much of this discussion is devoted to RTV tremolitic talc mined in northern New York State. Though the discussion is generally balanced, there are a few misleading and speculative statements in addition to data omissions which should be addressed.

On ***Page 18*** this sections begins with the statement "Epidemiologic studies of populations with exposures to EMP's ***that have been reported to be nonasbestiform*** have been conducted in the talc mining region of upstate New York, the Homestake gold mine in South Dakota, and the taconite mining region of northeastern Minnesota." (emphasis added). At least in respect to RTV talc, previously submitted File #1 provides ample evidence that RTV talc workers are exposed to copious nonasbestiform amphibole particulate (mostly tremolite). Further, it was shown that many of these fragments satisfy the commonly applied description of an EMP particle as one with a 3 to 1 aspect ratio (a.r.) or greater that is also 5 micrometers or more in length. This elongated amphibole particulate easily exceeds the existing PEL and REL applied to asbestos fibers (0.5 – 6.0 fibers/cc range recorded in the mine and mill).

The nonasbestiform amphibole nature of this exposure has been repeatedly confirmed over a span of decades in analytical report after analytical report by highly regarded mineral scientists. Some 40-60% of this talc is **clearly** known to be nonasbestiform amphibole. We believe the phrase "***have been reported to be nonasbestiform***" is therefore disingenuous in respect to RTV talc and likely also in respect to the other mining exposures mentioned. The composition of RTV talc is very well defined and NIOSH should not suggest otherwise.

The nonasbestiform nature of amphiboles encountered at Homestake and in the taconite studies (cummingtonite-grunerite) are reasonably well documented and generally recognized as well. Further discussion on these nonasbestiform amphibole exposures, however, should be addressed by others.

On ***Page 19*** and again on ***Page 23,*** NIOSH references early literature reports of elevated mesothelioma rates in certain NY counties in which talc mining is said to have occurred. NIOSH also correctly notes that this elevation no longer exists. NIOSH further notes, in regard to these past rates, that "Mesothelioma rates have been reported to be significantly elevated in Jefferson County, which is the site of much of the talc industry in New York." The suggestion is made in the references cited that these talc mines may have posed (or contributed to) an environmental mesothelioma risk.

Regional records indicate that at least 30 talc mining companies operated in the region with the earliest beginning sometime in the mid 1800's (we believe this to be the first talc mined in the United States). With the possible exception of one or two mines (though we know of none specifically), all of these mines are located in St Lawrence County – **not** Jefferson County.

We also know this mining region presents a complex and varied geological deposit that is by no means one large homogenous rock north of Syracuse, New York. RTV does not know the exact mineral composition of other, now long defunct, talc mines in the region. As noted, however, the composition of RTV talc is well documented and since 1974, it is the only talc mined in the region. To make this point, the following excerpt from a geological description of the Balmat-Edwards talc mining district in St Lawrence county (location also of the RTV talc mine) can be cited:

> "*The metamorphic alternation of the sedimentary Grenville limestones (over a billion years old) resulted in major deposits of tremolite, some of which was further altered to nearly pure talc, including many variations between. Plastic flow, faulting, and shearing added to the complexity of the ore-body. Efficient mining required a great deal of prospect drilling and professional geological mapping in order to predict the highly complex deformation.*" Fred Totten - Geologist

We feel it is important that NIOSH make it clear when referencing observations involving an entire region (especially on a historical basis) to carefully note that the mining exposures addressed no longer exist and may not reflect those of current talc mining in the region (now limited only to RTV). Without this cautionary note, the reader is left with the impression that "a region" poses a single, homogenous mineral particulate exposure when we do not know that to be the case. Given the varied and complex geology of that region, a homogenous particulate exposure is, in fact, highly unlikely.

On ***Page 19*** NIOSH references the Hull paper in regard to lung tissue analysis findings. John Addison performed a critical review of the analytical techniques used to perform the particulate identification in this paper in 2006. While the relevance of this case study to RTV talc remains uncertain (no work histories provided), Mr. Addison finds a number of methodological issues which he feels leaves some conclusions drawn by the authors as "unreliable". A copy of this review is appended and bears review in respect to reliance on this particular case study (published as a supplement paper) in respect to analytical methods applied. Additional critical review on other aspects of the Hull paper has also been completed by researchers with varied disciplines (pathologists, statisticians, etc.). If desired, a copy of this extended critique can be provided upon request. Additional review may be found in previously provided File # 5 (Gibbs and Gamble overview of human studies involving nonasbestiform amphiboles).

On ***Page 20*** in referring to the persistently elevated rate of lung cancer among RTV talc workers, NIOSH notes that the most "persuasive argument against a causal interpretation of these findings is that the lung cancer excess in this study population did not increase with duration and measures of exposure to talc dust." And that "the excess of lung cancer in this cohort has been reported to be limited to workers with short employment (< 1 years)". .

Toward the bottom of that page, however, NIOSH then offers "plausible" explanations for these inverse dose-response findings which it feels might still cast suspicion upon the dust as the causative factor. The two "plausible" concerns are expressed as follows: (a) short term RTV talc worker lung cancer cases may have been exposed to similar talc dust in other "neighboring" talc mines prior to RTV talc employment causing an unreported/unrecognized long term exposure and (b), the respirable dust data used to gauge direct exposure-response relationships in the Honda et al study may not have adequately represented quantitative exposure to elongated mineral particulate. The latter concern was expressed on ***Page 22.***

Fortunately, RTV talc workers are among the most studied talc miners in the world so a reasonable amount of dust exposure data and work histories are available. This information appears sufficiently complete to provide at least a basic "plausibility" check of the alternative explanations provided by NIOSH.

In respect to the issue of tenure and prior talc mining exposure, the following table shows the prior work histories of all lung cancer decedents reported in the most recent RTV talc mortality study (Honda, et al.) with less than 1 year of employment at the RTV talc mine and mill. This short tenure sub-group (17) represents 55% of the total 31 lung cancer decedents reported in this study. Histories were obtained from employment applications.

Tenure At RTV Mine	All Prior Mining	Prior Talc Mining
1 day	Iron Mine 1 yr.	0
3 days	0	0
6 days	Zinc Mine 1 yr.	0
6 days	0	0
8 days	Iron Mine 1 yr.	0
16 days	? (unclear record)	?
18 days	0	0
27 days	? (unclear record)	?
1 ½ months	0	0
2 months	Zinc (1y), Iron (1y)	0
2 ½ months	0	0
2 ½ months	0	0

2 ¾ months	Zinc 16 yrs	0
4 months	zinc, coal, talc (18 yrs total)	Yes (but unspecified time)
6 months	Zinc 1 ½ yrs	0
8 months	Zinc 2 yrs, talc 1 yr	1 Year
10 ½ months	? (unclear record)	?

While there are some uncertainties due to incomplete records, there is little support for the argument that pre-RTV talc mining work histories (regional talc mining in particular) might explain elevated lung cancer among this short tenure sub-set. In the Gamble case control study (File # 2) this potential was also addressed, supports this observation and further lends no support to this NIOSH concern.

NIOSH's second expressed concern, that the cumulative respirable dust exposure (33% lower for lung cancer decedents than all decedents) may not have been a reliable indicator of EMP exposure, can be tested as well. This can be checked with available respirable dust to "fiber" count data (3:1 a.r. or more – 5 um or greater lengths) through several sources. It is assumed EMP means particulate that satisfy this counting criteria.

<u>NIOSH (1980) Resp. Dust to Fibers/cc</u> (Fibers >5 um 3:1 or greater a.r.)
For select mine and mill jobs with at least 2 data points – <u>personal samples</u>
One sampling effort (over a period of several days in late 1970's)

		Resp. Dust	**Fibers/cc**	**Mppcf (Impingers)**
Mine:	Scrapper/slusher	1.29	?	11.8
	Driller	.98	3.0	11.7
	Trammer	.64	5.6	10.1
Mill:	Wheeler Op.	1.56	8.4	3.1
	Hardinge Op.	1.09	7.9	3.4
	Packers	.60	5.1	3.6
	Forklift Op	.35	4.0	1.6

<u>All Available Data: Resp. Dust to Fibers/cc</u> (Fibers >5um 3:1 or greater a.r.)
For select mine and mill jobs with at last two data point comparisons –
Personnel & fixed samples – all information sources – all avail. years
( ) = number of samples used in average.

		Resp. Dust	**Fibers/cc**	**Total Dust**	**Mppcf (Imp.)**
Mine:	UG Crusher	1.2 (5)	1.5 (13)	6.9 (13)	17.2 (23)
	Scrapper/slusher	1.1 (6)	0.3 (2)	3.2 (4)	15.0 (34)
	Drillers	1.1 (8)	1.7 (12)	3.6 (4)	14.1 (19)
	Trammer	.9 (5)	1.4 (18)	3.1 (4)	7.9 (21)
Mill:	Wheeler Op.	.92(24)	5.5 (23)	3.8 (32)	10.1 (32)
	Packers	.64(49)	1.7 (7)	2.9 (48)	16.0 (69)
	Hardinge Op.	.57(25)	1.4 (2)	1.7 (28)	8.7 (29)

Forklift Op.	.54 (8)	4.0 (2)	2.0 (12)	1.6 (2)
Drier/crusher Op.	.54(23)	0.8 (6)	3.2 (24)	8.1 (31)

It is difficult to use these data with high confidence as respirable dust and fibers/cc data should represent the same sampled activity on the same day, same sample duration, either be personnel samples or fixed samples (not mixed) and be processed/analyzed by the same laboratory. The NIOSH data is closest to satisfying these features but the data is limited to a relatively short sampling period (several weeks).

In preparing the "all available data" table, personnel samples were predominantly used for respirable dust and fibers/cc data points. Total dust tended to be fixed samples as did impinger samples. Indicators of data variability are not included in this table but could be produced (ranges, standard deviations, etc.). Data sources included NIOSH data, company sampling data, insurance company data, regulatory agency data, researcher data and NY State Health Dept. data (early impinger work). The earliest data points appeared in 1952 (RTV talc mining began in 1948) and continued through 2007.

It would seem reasonable that as respirable dust levels increase, respirable elongated mineral fibers (EMPs) present it the airborne dust, would also increase. Available comparison data does not appear to contradict this. The data set indicates that higher respirable dust levels "generally" do tend to reflect higher EMP levels. Admittedly, the difference in respirable dust levels between various common job activities is not very significant (particularly in the mill) and there are several fiber count levels that seem to buck the correlation trend - but these involve only a few data points. In general, available dust data does not, in my opinion, support the unreliability of using respirable dust in the dose-response analysis referenced in the Honda, et al paper. Certainly no clear, significant disparity is seen between comparative levels of respirable dust versus EMP levels

Though the two explanations for the lack of exposure-response in RTV Talc worker lung cancer mortality studies offered by NIOSH are plausible considerations, available data does not support either one.

Importantly, there is additional evidence of the absence of a "same as asbestos" risk among RTV talc previously provided in file #'2 which was ***not addressed*** in the updated NIOSH draft ***pages 18-23.*** Given the importance of the Vanderbilt talc cohort in regard to elongated non-asbestiform amphibole exposure (EMP), each piece of evidence should be addressed in some form and recognized as part of a data base that either supports a dust causal link or does not. It is the totality of evidence in respect to RTV talc that we feel is most compelling – not one single study.

Not mentioned in the draft are references to the fact that nonmalignant respiratory disease among Vanderbilt talc workers does follow the classic dose-response pattern (noted by NIOSH, Honda, et al and other researchers). RTV talc workers most exposed show the highest levels of nonmalignant respiratory disease said to be dust linked - or likely to be over the study period (1960-1989). Elevated exposure to any talc over time can cause “talcosis”. NIOSH should explain, if NIOSH still believes exposure to RTV talc dust caused the excess lung cancer, how these two contradictory exposure findings can exist in the same cohort.

Also provided in File #2 but not mentioned, are more up-to-date non-malignant respiratory disease observations summarized by Brian Boehlecke, MD (a former NIOSH pulmonary specialist and Professor of Medicine at the University of North Carolina). These observations were derived from the results of RTVs semi-annual medical surveillance program. Observations of this nature covering a span of 15 years (1985 – 2000) for existing employees made by an occupational pulmonary specialist of some note would seem worthy of mention. Again, this is a cohort likely exposed to the highest concentration of elongated amphibole cleavage fragments in the world (in addition to talc fiber in several forms).

It should be noted, in this regard, that RTV talc miners are a union work force with a seniority system. Upwards of 60% of Vanderbilt talc workers have worked 20 years or more at the Vanderbilt talc mine and mill (30% 30 years or more). As reflected in the status summary, Dr. Boehlecke does not find the pulmonary experience of these talc workers to be consistent with that of a workforce exposed to asbestos. This observation can not be dismissed due to an over abundance of short term workers who had not yet had time to manifest dust linked pulmonary abnormalities.

Over 30 years have passed since the extremely elevated dust exposures recorded in other area talc operations ended (see data previously submitted). As a result, Vanderbilt talc workers now reflect the effects of much lower over-all dust levels experienced only at the Vanderbilt mine. In 2000 Dr. Boehlecke records only one case of irregular opacities consistent with a dust etiology at a 1/0 profusion level - with no change to 1994. The most recent medical surveillance findings (2008) continue to show only this one pneumoconiosis case which has now advanced to a 1/2 profusion level after 34 years of employment.

Pleural plagues, found among both asbestos and talc workers (and perhaps linked to other mineral exposures), are also observed among approx. 10% of workers with an excess of 20 years of talc work (without decrements in lung function observed). We believe this is among the best pulmonary experience in the mining industry – not among the worst.

Also not mentioned as yet another indicator of the absence of a “same as asbestos” effect is the comparison made by Lamm (1988) between RTV talc workers and Vermont talc workers in regard to lung cancer and non-malignant respiratory disease (see previously provided File # 2). Please note that this is not the same reference to Lamm (1988) which addresses short term workers. While cohort to cohort comparisons are admittedly difficult to make, this VT to NY comparison appears to be among the most defensible (i.e. same years of operation, similar cohort numbers, absence of quartz as a confounder, similar over-all dust levels, etc.). The key distinction between these two talc exposures is the absence of non-asbestiform amphiboles in Vermont talc versus the 40-60% prevalence in RTV NY talc.

For workers with more than one year exposure in both mining populations, the lung cancer rate is no different among Vermont talc workers than among Vanderbilt NY talc workers. Interestingly, the rate of non-malignant respiratory disease linked deaths is actually much higher among Vermont talc workers. When considering whether non-asbestiform amphiboles do or do not pose an asbestos like risk, this comparison would seem worthy of mention (even if only to debate the strength or significance of the comparison). This comparison study should be addressed when discussing Vermont talc worker studies on ***page 22.***

Lastly, also not mentioned but provided in File # 2, is the link between several of the animal and cell studies referenced in the NIOSH draft and the use of Vanderbilt talc in these studies. This will be addressed further in the animal and cell studies section as this is an important part of the science base in respect to RTV talc and should be addressed. These studies lend no support to RTV talc as a "same as" asbestos exposure.

On ***Page 21*** there is a discussion of the Gamble (1993) nested case control study of RTV talc workers. As noted in this study, Dr. Gamble addresses several possible explanations for the persistent lung cancer rates among RTV talc workers such as smoking and prior employment. The author concludes that smoking provides a more plausible explanation for the persistent excess lung cancer observed than does exposure to RTV talc dust (whatever its composition) or prior employment exposures.

NIOSH and others, however, have stated that while smoking prevalence is elevated, it can not account for the entire excess. In contrast, Dr. Gamble has stated that it could because the prevalence recorded does fit within the confidence intervals of his study. Dr. Gamble notes that there is a 73% smoking prevalence among the non-case controls – not just the 100% prevalence found among the cases. This conflict of interpretation might also be linked to the selection of which smoking "model" is used to calculate cancer rates (apparently there are conservative models and less conservative models). Dr. Gamble does defend his position in his case control study (page 453) and RTV does not feel NIOSH has adequately addressed his assessment.

On ***Page 22 – second paragraph*** NIOSH states "NIOSH (1980) reported that dust from these talc mines contains chrysotile, tremolite EMPs, and anthophyllite EMPs. However, the identification of these EMPs as asbestiform has been the subject of debate." This statement does not accurately reflect what NIOSH actually said in 1980.

In this NIOSH 1980 Technical Report, elongated amphibole particulate in RTV talc was not simply described as EMPs. On the Abstract page (iv) of this 28 year old report the industrial hygiene portion of the study flatly states: "Time-weighted average (TWA) exposures to asbestiform amphiboles (anthophyllite and tremolite) were found to be in excess of present U.S. Occupational Safety and Health (OSHA) and Mine Safety and Health Administration (MSHA) occupational exposure standards - etc.- -". There is no "debate" or confusion reflected in that statement. NIOSH clearly described the tremolite and anthophyllite found in RTV talc as "asbestos" – not EMPs. In roughly the same time period, NIOSH did exactly the same thing with nonasbestiform cummingtonite-grunerite exposures at the Homestake mine (which NIOSH referred to as "amosite"). RTV believes this occurred because NIOSH maintained this "policy" of viewing the elongated cleavage fragment analogs of asbestiform minerals as the "same as asbestos" based on perceived risk (the utilization of risk – real or imagined - to justify improper mineral characterization).

In respect to the minor talc and transitional fiber found in RTV talc, confusion is understandable. In the mid 1970's analytical expertise and instrumentation was not as sophisticated as it is today in respect to the ability to properly identify these unusual particulates. However, the identification of nonasbestiform amphibole cleavage fragments in RTV talc (tremolite in particular) as asbestos should not have occurred. NIOSH is to be commended for withdrawing this dated Technical Report from its document library. It is unfortunate, however, that it continues to be referenced.

Also on ***page 22*** in the same paragraph, NIOSH quotes the mineral composition of RTV talc provided in an RTV document known as the "White Paper". In reporting this composition the NIOSH draft states: "Up to 5.6% of the total product was comprised of talc and talc/amphibole fibers, and up to 1.8% (typically about 0.5% by weight) of the total product ***was reported to be asbestiform amphibole fibers."*** (emphasis added).

That ***is not*** what was reported in the White Paper. The statement in this document reads as follows: "The combined weight of those fibers that do exhibit an asbestiform growth habit typically falls around 0.5%. These fibers are not cleavage fragments nor are they asbestos." Above this statement it is pointed out that the "fibers" referenced are talc and transitional fibers – **not amphibole asbestos fibers.** This misstatement in the NIOSH document should be corrected. We know of no "asbestiform" amphibole fibers that have ever been confirmed in RTV talc (only mischaracterizations of cleavage fragments or transitional fibers).

Part of the transitional or mixed fiber is amphibole and part (typically the majority) is talc. The properties of these mixed fibers are not those of pure amphibole or pure talc. Again, it is important that proper mineral characterization always be applied. NIOSH did a reasonable job of describing transitional minerals in this draft document.

On ***page 22*** bottom paragraph, the NIOSH cohort study of Vermont talc miners and millers is discussed. As noted above, we believe the Lamm (1988) comparison study with RTV talc should be addressed in this section. Beyond that, however, NIOSH describes in this section its rational for not linking the moderate excess lung cancer observed among Vermont talc workers to the talc dust. In part, this was due to the contradiction seen between the non-malignant respiratory disease (NMRD) experience versus the lung cancer experience. Higher dust levels in the mills produced higher NMRD but not lung cancer. Instead, excess lung cancer was observed among the miners who experienced lower dust exposure than the millers.

NIOSH findings among RTV talc workers are very similar in respect to millers versus miners. Yet, despite this similar observation, NIOSH makes no mention of this for RTV talc and suggested in the late 1970's that the excess lung cancer noted among RTV talc workers was dust linked. RTV believes this occurred because NIOSH incorrectly believed there was asbestos in RTV talc. Bearing that possible bias in mind, the observations of Lamm (1988) comparing NY talc workers with Vermont talc workers should be of particular interest.

On **page 23** under summary comments, we believe this summary would not read as it does if the above omissions had been addressed. We believe studies of RTV talc (which include the results of animal and cell experimentation - see below) more convincingly point away from this talc as a "same as" asbestos exposure. NIOSH discussion of RTV talc studies is, however, better balanced than it has been in the past. RTV believes NIOSH continues to maintain a bias when it comes to studies of RTV talc.

Animal& Cell Studies – Pages 28 – 29 and 54

On ***Page 28*** NIOSH again outlines the position it has historically held in regard to elongated non-asbestiform amphiboles and serpentines articulated at the OSHA hearings in 1990 (i.e. treat as asbestos). NIOSH first points to animal studies as the basis of this position or "policy". The animal studies cited by NIOSH, however, do not actually show a "same as asbestos" risk for elongated non-asbestiform particles but rather merely support the importance of mineral fiber length, width and bio-persistence.

In effect, the "dimensional" argument presented by NIOSH was applied to non-asbestiform particles in a broad-brush way. The animal studies cited did not, in actual fact, support the position NIOSH took because the "dimensions" most implicated belong to those most commonly found among asbestos fibers – not elongated nonasbestiform amphibole cleavage fragments. Differences in biologic effect and particle dimension between asbestos fibers and elongated cleavage fragments in animal and cell studies have been reviewed by several authors (see previously provided examples in File # 5) and in papers cited by NIOSH such as Wylie et al (1993).

As discussed at some length in our prior submission, discussion of "similarly sized" or "appropriately sized" asbestos fibers and elongated cleavage fragments are believed to be greatly exaggerated in terms of what is commonly found. The expanded discussion with references in previously provided File # 6 in our prior submission should be reviewed in this regard. RTV has come to believe that much of this "similarly sized" argument originates from sizing elongated cleavage fragments found in asbestos standard samples and/or excessive grinding in sample preparations. The prevalence of "similarly sized" is grossly exaggerated – especially when the fibrillar bundling structure of asbestiform fibers is ignored.

NIOSH cites Stanton ***Page 28 and again on page 40*** as one of the supporting animal studies for its earlier position. In doing so, however, NIOSH fails to mention that talc samples #6 and #7 are RTV tremolitic talc samples which contain upwards of 50% nonasbestiform amphibole (tremolite in abundance). These samples produced 0% tumors in the experimental animals. Interestingly, sample #6 also recorded a log number of Stanton critical dimension fibers (longer then 8 micrometers and thinner than 0.25 micrometers). This sample should have produced upwards of a 60% tumor response based upon the Stanton hypothesis – yet no tumors were observed.

Contradictions of this type to the "Stanton hypothesis" suggest to other researchers that mineral type is also plays a role - as NIOSH does point out. These "critical dimension" fibers were not cleavage fragments but rather the talc and transitional fiber found in RTV talc. File #2 previously provided contains documentation confirming the identity of those two talc samples. When discussing RTV talc, NIOSH failed to point this out as additional evidence for or against RTV talc as posing a "same as" asbestos risk.

Although on ***Page 40*** NIOSH references an *In Vitro* study by Wylie, Mossman, et al (1997). This study is discussed in regard to the importance of experimental design and exposure metrics such as fiber mass, fiber number, or fiber surface area. NIOSH does not mention, however, that this cell study is yet another study that specifically involved a comparison between RTV talc and asbestos. In this instance the purpose of the study was to contrast a concentrate of RTV talc fiber (with transitional fibers) against an equivalent (or as close to equivalent) concentrate of asbestos fiber (crocidolite and chrysotile in this case). FD-14 (RTV talc used in the Smith animal study – see below) was used in the experiment as well as a whole product comparison.

In this study, all elongated particulate in each sample were carefully sized. The researchers noted a difference in cell proliferative effects (in Hamster Tracheal Epithelial cells) that "could not be explained by differences in fiber dimensions or surface areas, indicating an important role of mineralogical composition rather than size of fibers." Cytotoxicity for all fibers > 5 um and similar surface area was similar but cytotoxicity is not generally viewed as a measure of carcinogenicity and can be produced with many non-asbestos materials with no demonstrated carcinogenic effect (as we understand it).

NIOSH also references Smith (1979) (***<u>Page 28 and again on page 47)</u>*** and points out some possible limitations in this study. This IP injection study contrasted tremolite asbestos to nonasbestiform tremolite. Whatever the limitations, this study did show tumor production above background for the asbestos samples and 0% tumors for the nonasbestiform tremolite tested in the same way. One of the nonasbestiform samples (FD-14) was an off-the-shelf sample of RTV talc and the other a concentrate of nonasbestiform tremolite of respirable size that originated from the RTV talc ore body. The presence of these two RTV samples in this study was not mentioned. File #2 (previously provided) contains documentation confirming the identity and origin of these samples. NIOSH does point out that the FD-14 (RTV talc) sample did contain tremolite with a 13% fiber content as defined by a 3:1 aspect ratio.

These are two early animal studies (pre 1990) that directly involved RTV talc and independently produced the same result – no tumors. In contrast, tremolite asbestos and other forms of asbestos tested under the same test conditions produced tumors. While there may or may not be protocol lapses in these studies (to few animals, not enough dose variations, experiment too short, etc.) neither provide a shred of "same as" asbestos evidence for elongated nonasbestiform amphiboles or the talc and transitional fiber found in RTV talc. Failure to point out that these animal studies produced negative results for RTV talc in contrast to tremolite asbestos does not inform the reader in respect to additional evidence that RTV talc may or may not pose a "same as" asbestos exposure (a "weight of evidence" consideration possibly more important than the strengths or weaknesses of a single study).

On ***Page 29*** ***(and again on Page 48)*** NIOSH discusses the study by Davis et al. 1991. This study contrasted three tremolite asbestos samples to three tremolite samples described as nonasbestiform but of differing morphologies (granular to highly elongated or acicular). Early into this IP injection study numerous animal deaths and tumors were observed for the asbestos exposed animals while none was observed for the non-asbestos tremolite exposed group.

As NIOSH notes, however, at the very end of the study one non-asbestos tremolite sample (Italian Sample) produced a significant 67% pleural tumor rate. The authors found and reported an asbestos subpopulation in this sample and attributed the late hour tumors to this low dose asbestos exposure. A second non-asbestos sample (also containing unusually high aspect ratio material) produced a 12% late hour tumor prevalence when the authors consider fewer than 10% to be background at a 10 mg dose. It was noted ***(page 48)*** that this sample also contained some long, thin asbestiform tremolite fibers though fewer than observed in the Italian sample. The authors concluded this sample (uncharacteristic of typical cleavage fragment populations) "is unlikely to show evidence of carcinogenicity following administration by the more natural route of inhalation" and that this material "is probably to be considered harmless to human beings." The third non-asbestos tremolite sample (more granular/prismatic) produced below background tumors.

Though these observations are mentioned by NIOSH, there is a note of reservation made in this presentation suggesting that the asbestos subpopulation might not simply explain the tumors observed in the predominantly nonasbestiform tremolite samples. There is the suggestion that the high acicular nonasbestiform tremolite in these samples may be fully responsible or have acted in a contributory manor. It is not possible to "prove" this one way or the other without sample purification and retesting of these high acicular tremolite samples. As the authors point out, however, relatively minor amphibole asbestos subpopulations in otherwise nonasbestiform exposures is likely the more plausible explanation in an animal study in which materials are deposited directly on or into the highly sensitive pleura.

Concluding Statement

NIOSH's discussion of health studies continue to promote fiber size over crystal growth distinctions and other particulate characteristics as most key to asbestos and other EMP risk. This orientation, however, appears significantly less pronounced than that seen in the first NIOSH draft.

As earlier noted, RTV does not dispute the key role particle dimension plays in particulate risk but does believe crystal growth habit, mineral type and linked physio-chemical properties also play a key role. RTV agrees with NIOSH that these variables (***section 1.6.3 pp 34-38)*** deserve additional investigation.

In regard to nonasbestiform elongated minerals NIOSH does note the conclusions of researchers on ***Page 48, 49, 51 and 52*** who have both reviewed the literature and directly studied the distinction between elongated particulate and asbestos fiber. These researchers conclude that a distinct difference in biologic effect is consistently demonstrated in the human, animal and cell work completed to date (Addison, McConnell, Mossman, Berman, Gibbs, Gamble and others). The health link to fiber dimensions most characteristic of asbestos fibers is further cited by NIOSH through the work of other researchers (Lippmann, Stanton, Dement, etc.).

RTV believes that while improved, NIOSH continues to subtly "imply" an asbestos fiber risk for elongated cleavage fragments throughout the draft document. This occurs each time a study showing a difference between asbestos fiber and non-asbestos particulate is critiqued by NIOSH as potentially faulty - or worse, when pertinent studies are not addressed at all. The strengths and weaknesses of studies should be recognized but care should be taken to avoid implying results may not be meaningful - especially if consistent with other studies and in the absence of evidence to the contrary.

Another example, is the advancement of general fiber risk theories that can be interpreted as suggesting non-asbestos EMPs might pose an asbestos risk "if" of appropriate size or "if" in sufficient concentration, etc. Pointing out the fact that the vast majority of asbestos fibers are typically shorter than 5 micrometers and that this is consistently observed in lung tissue (Dodson et al.) is an example of an implied risk. Such an observation (as NIOSH did recognize) does not necessarily mean these short fibers "cause" all or some asbestos disease end points. The health literature generally does not support this short fiber association however prevalent short asbestos fiber may be. NIOSH recognizes this as well.

Extrapolations from asbestos fiber mechanism studies to elongated particulate can be viewed as speculative as well. To the extent nonasbestiform elongated particulate may share some compositional similarities to tested asbestos fibers, these similarities should be recognized and not presumed to be risk linked characteristics until proven to be.

RTV would not argue against any insufficiently explored hypothesis being tested, or against the value of identifying a universal EMP risk linked characteristic (if one exists).

RTV believes, however, that theoretical constructs should not overshadow direct study results or the "trend" direction of these results. Since no study is without fault, the significance of the totality of evidence (human, animal and cell) takes on extra importance in our view. RTV does not believe NIOSH would argue with this general concept, but the tenor of the draft document indicates that the Institute is not yet ready to fully embrace it when it comes to EMP or when addressing RTV talc specifically - even though the objectivity of NIOSH in both areas shows improvement in our view. RTV believes the objectivity of NIOSH in this area does, however, show improvement.

IC 8751

Bureau of Mines Information Circular/1977

Selected Silicate Minerals and Their Asbestiform Varieties

Mineralogical Definitions and Identification-Characterization

UNITED STATES DEPARTMENT OF THE INTERIOR

Information Circular 8751

Selected Silicate Minerals and Their Asbestiform Varieties

Mineralogical Definitions and Identification-Characterization

By W. J. Campbell, R. L. Blake, L. L. Brown, E. E. Cather,
and J. J. Sjoberg

This current report on asbestos has been prepared by the Bureau of Mines, U.S. Department of the Interior to—

1. Provide precise nomenclature and information on selected silicate minerals and their asbestiform varieties.
2. Invite comment, revisions, or additional information on the subject.

Please direct communications to the author—
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UNITED STATES DEPARTMENT OF THE INTERIOR
Cecil D. Andrus, Secretary

BUREAU OF MINES

As the Nation's principal conservation agency, the Department of the Interior has responsibility for most of our nationally owned public lands and natural resources. *This includes fostering the wisest use of our land and water resources*, protecting our fish and wildlife, preserving the environmental and cultural values of our national parks and historical places, and providing for the enjoyment of life through outdoor recreation. The Department assesses our energy and mineral resources and works to assure that their development is in the best interests of all our people. The Department also has a major responsibility for American Indian reservation communities and for people who live in Island Territories under U.S. administration.

This publication has been cataloged as follows:

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Bibliography: p. 54-56.

1. Silicate materials. 2. Asbestos. 3. Asbestos fibers. I. Campbell, William Joseph, 1926- . II. United States. Bureau of Mines. III. Series: United States. Bureau of Mines. Information circular - Bureau of Mines ; 8751.

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SELECTED SILICATE MINERALS AND THEIR ASBESTIFORM VARIETIES

Mineralogical Definitions and Identification-Characterization

by

W. J. Campbell,[1] R. L. Blake,[2] L. L. Brown,[3] E. E. Cather,[4] and J. J. Sjoberg[5]

ABSTRACT

This report by the Federal Bureau of Mines Particulate Mineralogy Unit recommends mineralogical definitions and identification-characterization concepts for selected silicate minerals and their asbestiform varieties. Precise definitions acceptable to mineral analysts, regulatory personnel, and medical scientists are essential because of the present lack of conformity in terminology concerned with measuring and controlling asbestiform particulates and their related health effects. Because of the complexity and variability of crystal morphology in different mineral groups, the descriptive terms are generally explained by illustration rather than by numerical values. Applications and limitations of several analytical techniques for particulate identification and characterization are discussed.

INTRODUCTION

Concurrent with concerns within the Federal Government over future availability of minerals to meet our expanding needs is the requirement that minerals and mineral commodities be mined and processed with minimum environmental impact. Traditionally, matters related to "... inquiries and scientific and technologic investigations concerning mining, and the preparation, treatment, and utilization of mineral substances with a view to improving health conditions and increasing safety...." have been within the province of the Bureau of Mines as authorized in the amended Organic Act of 1913 (Public Law 62-386). Since its establishment by Congress, the Bureau of Mines has long been deeply involved in investigating the explosive characteristics of dusts in the mineral industries in its mining and metallurgy research centers, and has established analytical and mineralogical laboratories in seven metallurgy research centers. These laboratories are essential to solving the increasingly complex

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safety, health, and environmental problems posed by dusts within the mineral system.

Various legislative actions and public concerns within the past decade have had, and will continue to have, an impact upon the mineral industry. As a result, control of mineral particulates is becoming increasingly important, with much recent attention focused on asbestiform particulates in both air and water. Figure 1, from an Environmental Protection Agency report, shows the widespread occurrence of common amphibole and serpentine minerals that, according to existing regulatory definitions, may be classified as asbestiform minerals (16).[6] With such possibly overwhelming implications to both mineral producer and mineral consumer, it is essential that existing ambiguities regarding silicate minerals and their asbestiform varieties be resolved. Until recently, adverse health effects associated with asbestos were focused on occupational exposure in asbestos-related industries. Now there is international concern regarding the effect on health from long-term low-level, or short-term high-level, exposure to mineral particulates by the general public (5, 28, 36). These particulates may include both the common and the asbestiform varieties of certain silicate minerals. In many instances, cleavage fragments of common amphibole minerals have been mistakenly identified as microscopic fibers of the related asbestiform variety. Such lack of precision in identifying these particulates is a handicap to scientific decisionmaking by regulatory agencies and medical researchers. The Particulate Mineralogy Unit was created to work on problems such as this.

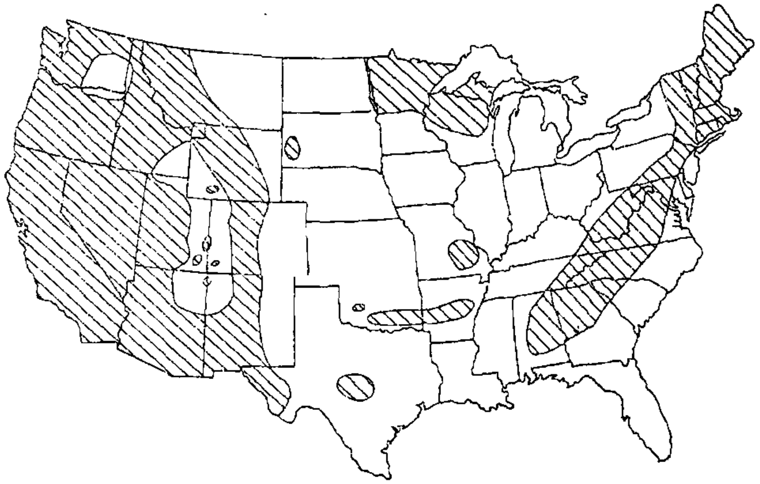

FIGURE 1. - Regions of the United States (shaded area) reported by the Environmental Protection Agency to contain asbestiform minerals in the bedrocks (16).

The Particulate Mineralogy Unit

The Bureau of Mines established the Particulate Mineralogy Unit in September 1976. The College Park Metallurgy Research Center in College Park, Md.,[7] is the focal point for this unit, but substantial support will be supplied by the other Bureau of Mines metallurgy and mining research centers, which are located throughout the United States. The unit is to assist local, State, and Federal agencies in establishing precise and workable mineral definitions and to improve or develop methods of particulate identification and quantitative measurement. The unit is also providing characterized serpentine and amphibole minerals for use by Federal health agencies in their asbestos-related research programs.

Scope of Report

This Bureau of Mines report is intended to clarify some of the terminology used in identification and characterization of asbestiform minerals, and to sharpen the distinction between common rock minerals and their asbestiform varieties. It defines certain mineral terms related to asbestiform minerals and discusses mineral-characterization techniques on a strictly mineralogical basis. The report then discusses the identification of silicate particulates and suggests how to apply this information to asbestos-related problems. Suggested areas for further research are summarized at the end of this report.

ACKNOWLEDGMENTS

The following College Park Metallurgy Research Center personnel are acknowledged for their invaluable contributions. Photographic assistance was provided by Garrett Hyde, research physicist, and Lawrence Johnson, geologist (mineralogy). Application data were provided by Raymond Brown, physical science technician; Charles W. Huggins, research chemist; and Eric Steel and Robert Virta, geologists (mineralogy).

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[6] Underlined numbers in parentheses refer to items in the list of references at the end of this report.

[7] The research center is scheduled for relocation to Avondale, Md., in June 1978.

NOMENCLATURE OF SELECTED SILICATE MINERALS AND THEIR ASBESTIFORM VARIETIES[a]

Background

In August 1976, a preliminary paper (1) was presented from which the Bureau of Mines hoped to elicit suggestions from the mineralogical community for critically defining silicate minerals and their asbestiform varieties. Significant terms such as "asbestos," "asbestiform," and "fibers" have different meanings to the various concerned groups--mineralogists, regulatory agencies, medical scientists, and industry. This has resulted in situations where erroneous conclusions have been drawn. Thus, precise definitions are required that will be uniformly accepted and applied by all personnel involved with silicate minerals that have asbestiform varieties (4, 11, 24-25, 31, 39).

Mineralogical nomenclature recognizes the historical origin of terms, and changes are made only when they are proven incorrect by new information (10, 32). The science of mineralogy is constantly being advanced with newly examined mineral occurrences and with new and improved instruments and techniques that provide more details on chemical composition, crystal structure, and morphology. To prevent constant revision, nomenclature has to have a certain flexibility, yet must be definite enough to be scientifically useful. The objective of this part of the report is to summarize mineralogically acceptable terms that relate to asbestos.

There is no "group" of asbestos minerals. "Asbestos" is a general term applied to certain minerals (that are themselves classified under crystal-structure-based groups) when these minerals crystallize as the asbestiform variety. Table 1 lists some common silicate minerals and their asbestiform varieties, together with their relationships and formulas. Although discussion in this report is limited to these minerals, appropriate terms and statements also apply to other silicate minerals that have rare fibrous varieties such as talc, some clay minerals such as attapulgite, and other amphiboles such as arfvedsonite, eckermannite, and richterite.

TABLE 1. - Selected silicate minerals and their asbestiform varieties

Mineral	Asbestiform variety
AMPHIBOLE GROUP	
Anthophyllite: $(Mg,Fe^{+2})_7$ $Si_8O_{22}(OH,F)_2$...	Anthophyllite asbestos.
Cummingtonite-grunerite: $(Mg,Fe^{+2})_7$ $Si_8O_{22}(OH)_2$.	Cummingtonite-grunerite asbestos.
Tremolite-actinolite: $Ca_2(Mg,Fe^{+2})_5$ $Si_8O_{22}(OH,F)_2$.	Tremolite-actinolite asbestos.
Riebeckite: $Na_2Fe_3^{+2}$ Fe_2^{+3} $Si_8O_{22}(OH,F)_2$....	Crocidolite.
SERPENTINE GROUP	
Serpentine: $Mg_6Si_4O_{10}(OH)_8$................	Chrysotile.

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[a] Tibor Zoltai, Professor, Department of Geology and Geophysics, University of Minnesota, made significant contributions to this section of the report.

Amphibole minerals and, to a lesser degree, serpentine minerals occur widely distributed in the earth's crust in many igneous or metamorphic rocks. In some rare instances, the mineralogical occurrences contain sufficient quantities of usable asbestiform minerals to be economically minable for commercial asbestos.[9] The soft, silky fibers of asbestos (sometimes called mineral silk) are so flexible that they can be spun into threads from which cloth can be woven. The resulting material is fireproof, is a good thermal and electrical insulator, and has moderate to good resistance to acids. It has been used from Roman times, and is most familiar in daily use in brake linings for automobiles and as the "asbestos" siding used in residential construction.

Only a very small quantity of the amphibole and serpentine minerals under particular geologic circumstances occur as the asbestiform variety of the mineral. The asbestiform varieties occur in veins or small veinlets within rock containing or composed of the common (nonasbestiform) variety of the same mineral. Macrophotographs of the minerals of table 1, both asbestiform and nonasbestiform varieties, are shown in figures 2 through 7.

The serpentine group of minerals is limited to serpentine as the common variety and to chrysotile as the asbestiform variety. Antigorite and lizardite are not listed as separate varieties, but are understood to be included in the term "serpentine" because they represent 2 frequently named polytypes of about 10 recognized polytypes of serpentine (20). The polytypes differ only in minor structural stacking of components and are not sufficiently different to have a separate mineral status. Chrysotile generally occurs segregated as parallel fibers in veins or veinlets, although a recent study (6) has shown fine chrysotile intimately intergrown with the lizardite polytype.

The minerals and mineral series of the amphibole group in table 1 have variable compositions with extensive elemental substitutions and are found in forms ranging from massive to blocky to very fibrous. Crocidolite is the varietal name given to the fibrous habit of the mineral riebeckite (fig. 7), as shown by at least one study (35). It is retained here as a useful and correctly identified term.

Cummingtonite-grunerite asbestos is the mineralogically proper terminology for the commercial material commonly known as "amosite." Amosite, implied to be a mineral variety, is really an acronym--Asbestos Mines of South Africa--for a fibrous mixture of minerals; namely, cummingtonite-grunerite with variable tremolite-actinolite. Amosite has been discredited as a mineral species (22), and its use as a mineralogical term should be discontinued; however, it is still useful as a commercial term.

Mineral compositional series such as cummingtonite-grunerite involve replacing one cation for another in a crystal structure without significantly altering the structure. There may be a gradation in the structure in some series, and minor changes in physical characteristics may occur with elemental substitution. Usually a series involves two named compositional end members with intermediate substitutional compounds being separately named (if the members were recognized by early mineralogists), given a varietal name (for similar reasons), or just qualified by being referred to as members of the series. Members of the tremolite-actinolite-ferroactinolite series are hydroxylated calcium-magnesium, magnesium-iron, and iron silicates, respectively. Their series is named in table 1 for two of its members, and when its composition is known, it should be called by the specific name, such as

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[9] Clifton, R. A. Asbestos. BuMines MCP-6, in preparation, 1977.

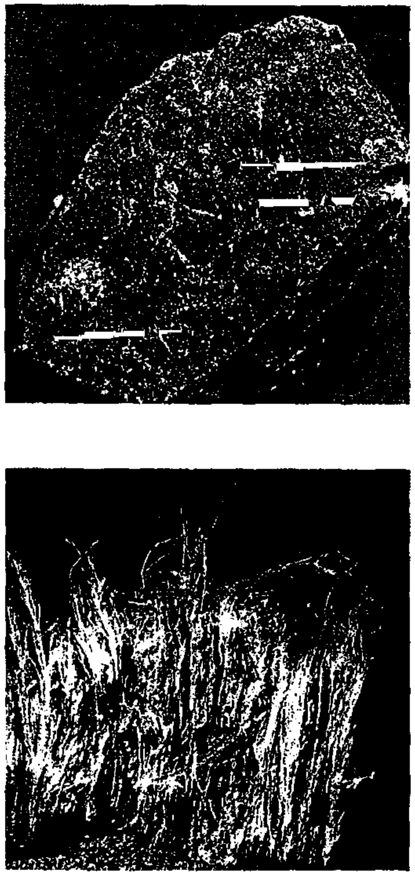

FIGURE 2. - *Macrophotographs of serpentine (top, X 1) and chrysotile (bottom, X 3).*

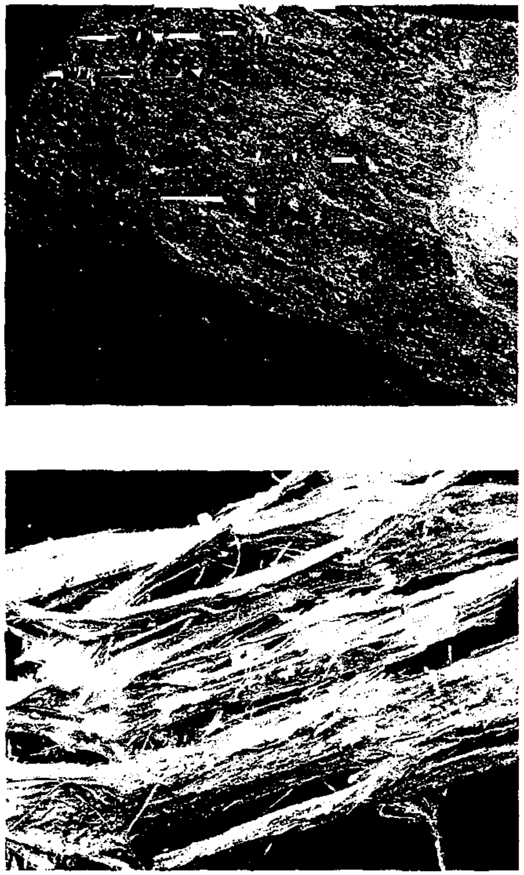

FIGURE 3. - Macrophotographs (X 3) of tremolite (top) and tremolite asbestos (bottom).

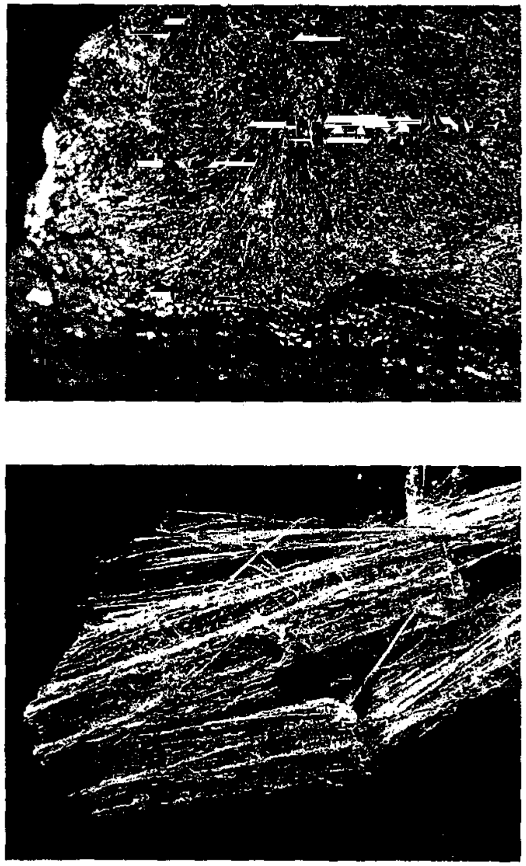

FIGURE 4. - Macrophotographs (X 3) of anthophyllite (top) and anthophyllite asbestos (bottom).

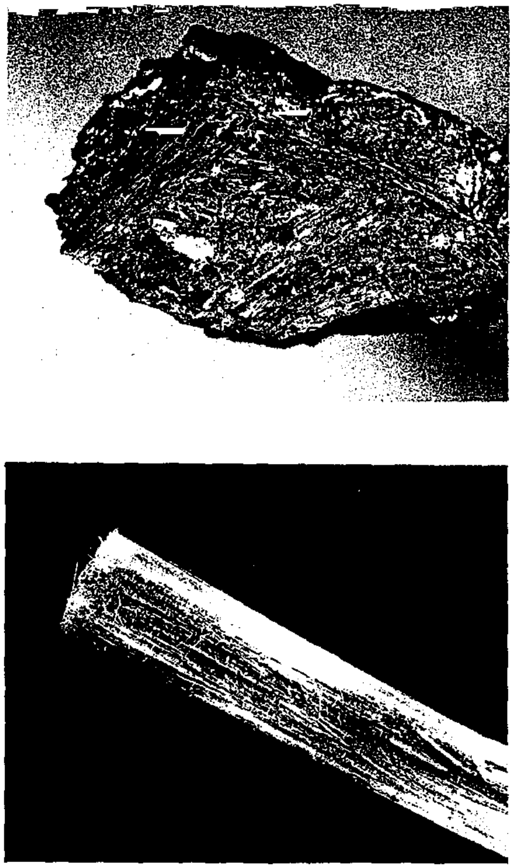

FIGURE 5. - Macrophotographs of actinolite (top, X 1) and actinolite asbestos (bottom, X 3).

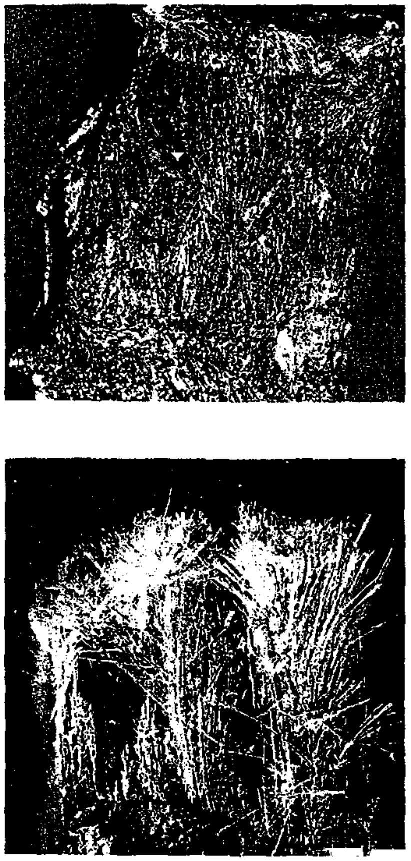

FIGURE 6. - Macrophotographs (X 3) of cummingtonite (top) and cummingtonite-grunerite asbestos (bottom).

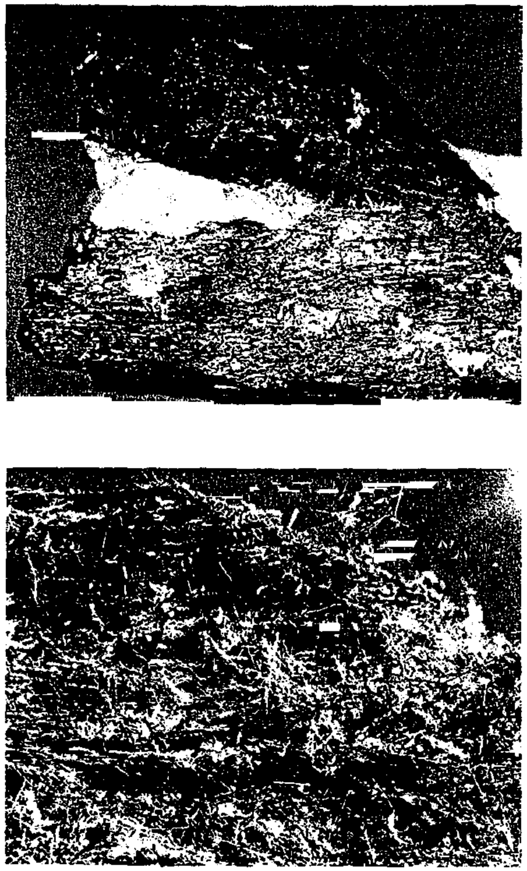

FIGURE 7. - Macrophotographs (X 3) of riebeckite (top) and crocidolite (bottom).

tremolite. The following nomenclature for this series (7) is based on the mole ratio (in parentheses) of $\frac{Fe}{Fe + Mg}$ in the formula $Ca_2(Mg,Fe)_5Si_8O_{22}(OH)_2$: tremolite (0 to 0.2), actinolite (0.2 to 0.8), and ferroactinolite (0.8 to 1.0).

Definitions

Many mineralogical definitions apply to the *entire mineral field*, but the definitions in this report are restricted to those necessary to characterize the fibers and cleavage fragments related to asbestos *identification-characterization*. The terms to be defined are outlined in the following list:

- Mineral terms:
  - Mineral
  - Mineral groups
  - *Mineral series*
  - Mineral varieties
- Asbestos-related terms:
  - Asbestos
  - Fibrous
  - Mineral fiber
  - Fibril
  - Fibril structure
  - Asbestiform
- Crystal terms:
  - Crystal structure
  - Crystalline
  - Crystal
  - Single crystal
  - Twinned crystal
  - Polycrystalline
  - Polymorphs
  - Polytypes
  - Crystal form
  - Crystal morphology:
    - Single crystals:
      - Equant
      - Prismatic
      - Acicular
      - Fiber
      - Fibril
      - Filiform
      - *Bladed*
      - Platy
      - *Lamellar*
    - Crystal aggregates:
      - Asbestiform
      - Columnar
      - Fibrous
      - Lamellar
      - Massive
      - Radiating
      - Reticulated
- Breaking of minerals:
  - Cleavage:
    - Types:
      - Pinacoidal
      - Prismatic
      - Rhombohedral
      - Cubic
      - Octahedral
    - Quality:
      - Perfect
      - Good
      - Fair
      - Imperfect
    - Cleavage fragment
  - Fracture: Types:
    - Even
    - Uneven
    - Splintery
    - *Conchoidal*
    - Hackly
  - *Parting*

Mineral Terms

Mineral.--A homogeneous, naturally occurring, usually inorganic and crystalline substance. *Minerals* with distinct crystal structure types (including lack of crystal structure) and limited variation in chemical composition are given individual *names*.

The early concept of *a mineral* as a natural-history species was gradually abandoned and changed into a chemical and structural definition around the middle of the 19th century, long before crystal structures were understood. The term "species" persisted for a long time with this newer definition, but is now seldom used in mineralogy.

Mineral Group.--Minerals that have essentially *the same* (or comparable) structures, but have different cations in secondary structural position (for example, pyrites, feldspars, and amphiboles).

In silicates, most of the mineral groups have the same *linkage patterns* of the silica tetrahedra, like the characteristic double chains of amphiboles. However, the secondary atomic sites may be occupied by a *relatively wide* variety of cations or some may even be vacant in the actual structures of the members. In some silicate-mineral groups, the identity of the *silica-tetrahedral frame* is less restricted and may be limited to the similarities of some basic characteristics. For example, the silica-tetrahedral frames of the *zeolite minerals* are variable, but they are all characterized by large open channels.

All *mineral* groups have names. In some instances, this name is the universal name of a common or important member of the group (for example, serpentine group).

Mineral *Series*.--*Two* or more members of a mineral group in which the cations in secondary structural position are similar in properties and can be present in *variable*, although frequently limited, ratios (for example, cummingtonite-grunerite). Also known as an isomorphic series.

Some mineral series such as the plagioclases have unique names, but most are identified by the *combined names* of the end-member minerals, such as tremolite-actinolite. The current trend is to simplify long series names by using the mineral name of only *one* (*end or intermediate*) member.

Individual minerals in the *series are either* identified by the names given to compositional ranges (for example, bytownite or oligoclase in the plagioclases), or by the name of the series *followed by a symbol* expressing the mineral's position in the series or the ratios of the variable cations ($Ab_{x\%}An_{100-x\%}$ for plagioclases, where Ab and An *designate the two end* members, albite and anorthite).

Mineral Variety.--Minerals that are conspicuously different from those considered normal or common in crystallization habits, polytypes, *and other* structural variants, or other physical properties such as color. Varieties are named by mineralogists, miners, gemologists, manufacturers of *industrial products*, and mineral collectors.

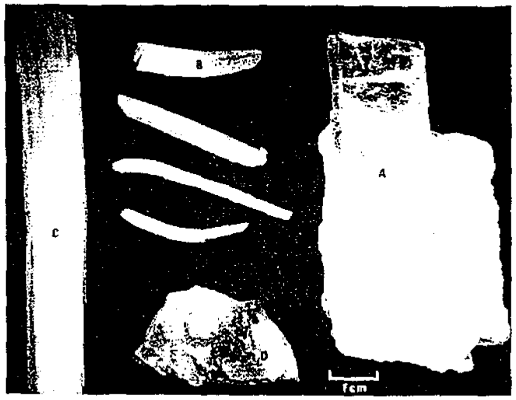

FIGURE 8. - Four varieties of gypsum: (A) Selenite; (B) satin *spar*, fine fibrous; (C) satin spar, coarse fibrous; and (D) alabaster.

Although mineral names are controlled by national and international mineralogical organizations, variety *names are not*. In practice, any variety name that *becomes* sufficiently popular is eventually recognized by these organizations as distinct enough to be used as a mineral-variety name. Typical of these are the various varieties of gypsum shown in figure 8. Figures 2 through 7 illustrate the massive and asbestiform varieties of serpentine and *amphiboles*.

Asbestos-Related Terms

In the following discussion, asbestiform refers only to asbestos. The other terms, "fibrous," "mineral fiber," "fibril," and "fibril structure," apply to both asbestiform and *nonasbestiform* varieties.

Asbestos.--(1) A *collective* mineralogical term encompassing the asbestiform varieties of various minerals; (2) an industrial product obtained by mining and processing primarily *asbestiform* minerals.

The quality of asbestos depends *on the* mineralogy of the asbestiform variety, the degree of asbestiform development of the fibers, the ratio of asbestiform fibers to acicular *crystals or other* impurities, and the length and flexibility of the fibers. The major asbestiform varieties of minerals used for asbestos are chrysotile, *tremolite-actinolite asbestos*, cummingtonite-grunerite asbestos, anthophyllite asbestos, and crocidolite. Asbestos may be marketed by its mineral name such as anthophyllite asbestos, *its variety name* such as chrysotile or crocidolite, or a trade name such as Amosite or Montasite. Two types of commercial asbestos are shown in figures 9 and 10.

The term "asbestos" was first introduced by Plinius Secundus in 77 A.D. The term "*amiant*" *was* previously used for the same mineral by Dioscorides in 50 A.D., and this term was the more common one until the middle of the 17th century. After *that until the* 20th century, "asbestos" was more common, and "amiant" was reserved for the more silky and flexible asbestos. In the 18th century, asbestos was *classified into five* different species. Currently, all asbestos is recognized as varieties of several individual minerals. Thus, in mineralogy, "asbestos" became a collective term, somewhat like "*clays*" *or* "*gems*." During the 20th century, *asbestos* developed into an important industrial *material*. Some asbestos products contain nonasbestiform minerals (for example, asbestos-cement and asbestos-magnesia); consequently, the mineralogical and the industrial definitions of asbestos do not always coincide.

FIGURE 9. - Macrophotograph of tremolite asbestos (X 3).

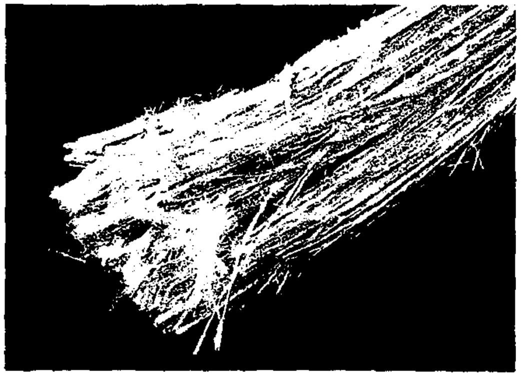

FIGURE 10. - *Macrophotograph* of anthophyllite asbestos (X 3).

Fibrous.--The *occurrence* of a mineral in bundles of *fibers*, *resembling* organic fibers in texture, from which the fibers can usually be separated (for example, satin-spar and chrysotile).

The term "fibrous" has been used during the last 200 years to describe all kinds of minerals that crystallized in habits resembling organic fibers, including asbestos minerals. *However*, the related term "asbestiform" was never used for fibrous mineral habits other than asbestos. Accordingly, "fibrous" is the more general term, *and asbestiform* is a specific type of fibrosity. Figures 11, 12, and 13 show various types of fibrous mineral habits. Examples of fibrous minerals, both *silicates and non-silicates*, that are not classified as asbestiform are shown in figure 13.

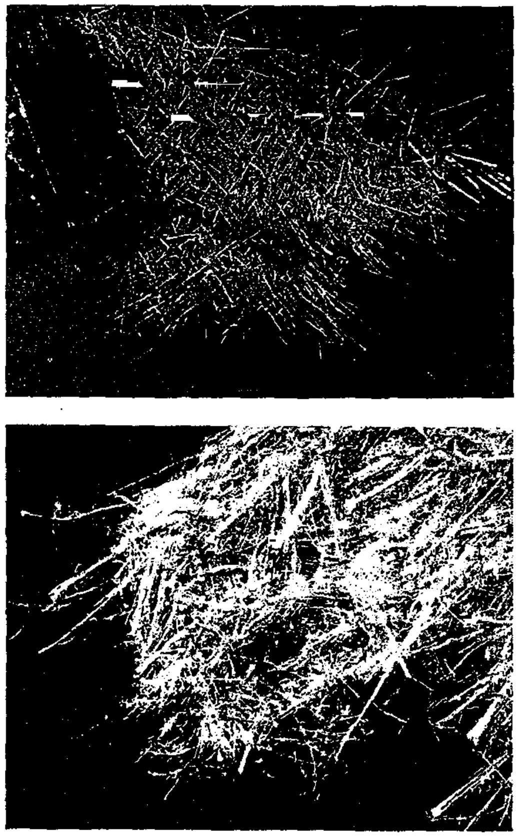

FIGURE 11. - Macrophotographs (X 3) of two fibrous amphiboles showing asbestiform habit: Byssolite (top) and richterite (bottom).

Mineral Fiber.--The smallest elongated crystalline unit that can be separated from a bundle or appears to have grown *individually in that* shape, and that exhibits a resemblance to organic fibers. (*Examples:* fiber bundles, chrysotile and crocidolite; individual fibers, epsomite and millerite.)

FIGURE 12. - Photomicrograph illustrating the separation of a crocidolite fiber bundle into fibers (X 600).

The term "fiber" is not limited to asbestos. However, it is distinct from "acicular" because it requires the resemblance to organic fibers. Figures 14 and 15 illustrate mineral fiber habits. Excellent photomicrographs of organic fibers are illustrated in The Particle Atlas (18).

Fibril.--A *single fiber*, which cannot be separated into smaller components without losing its fibrous properties or appearances.

Most *fibers* are *single structural* entities, such as millerite and nickel sulfide, and some may be called fibrils. However, some fibers are composed of two or more fibrils that are less readily separable from each other than fibers are from bundles (for *example*, chrysotile and crocidolite). Figure 16 *shows* the high magnification necessary to resolve a fibril.

Fibril Structure.--A *systematically deformed* and/or defective crystal structure *of a* fibril. A defect structure would involve various types of dislocation. The fibril structure may be exhibited by a single crystal, a group of single crystals, or a twinned single crystal.

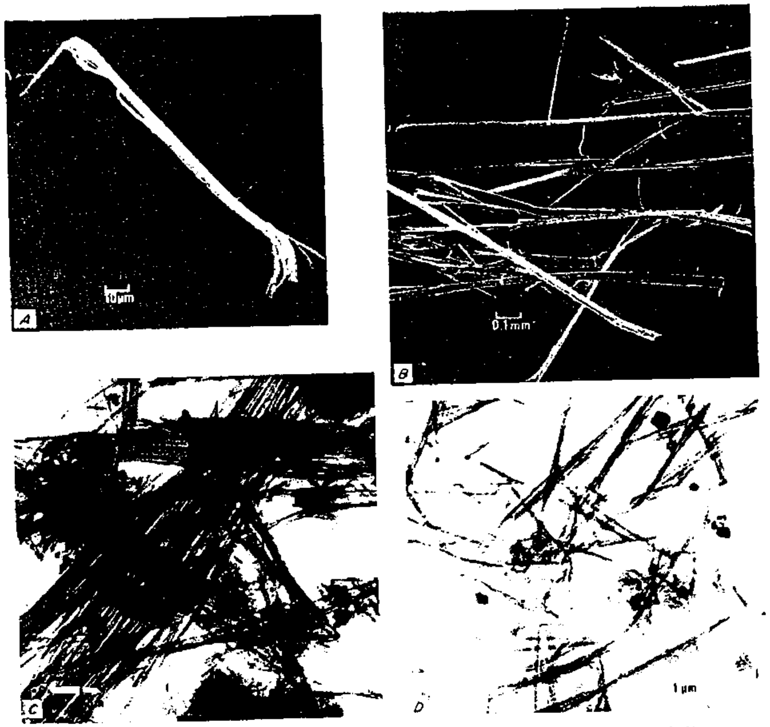

FIGURE 13. - Four fibrous nonasbestiform mineral varieties: *A*, Fibrous talc (X 500); *B*, fibrous brucite (X 50); *C*, polygorskite (X 30,000); and *D*, attapulgite (X 30,000).

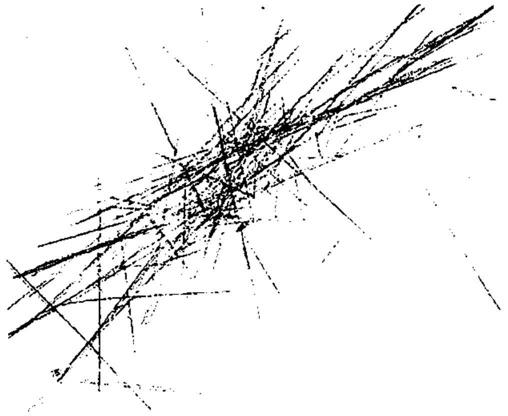

FIGURE 14. - Light optical photomicrograph of fibers from tremolite asbestos (X 115). A few particles are seen to be bundles of fibers.

FIGURE 15. - Fibers of epsomite (magnesium sulfate hydrate) (X 13).

The scroll-like fibril structure of chrysotile (38), the twinned single-crystal fibrils of chrysotile (6), and the incompletely resolved fibril structure of an amphibole (9) are all examples illustrated in the literature.

Some acicular single crystals may have the appearance of fibers and fibrils, yet there is nothing unusual about their crystal structures. Other acicular single crystals may have significant structural deviations in addition to appearance that result in the display of certain properties usually found in fibers such as high tensile strength along the fiber axis. Thus, fibril structure is not limited to asbestiform structures, but may occur in a minor form in nonasbestiform structures.

Asbestiform.--A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.

"Asbestiform" and "asbestos" are essentially synonymous in current usage. Some special properties of asbestiform varieties, including optical extinction and surface charge, are either not fully understood or are not uniformly applicable to all asbestiform fibers; consequently, they cannot be considered fundamental characteristics at this time. The prototype of the expression "asbestiform" was introduced by Werner in 1774. He recognized three subspecies of actinolite and of tremolite. One of these subspecies had the prefix "asbestartiger." Thus, the restriction of "asbestiform" to certain mineral varieties appears to

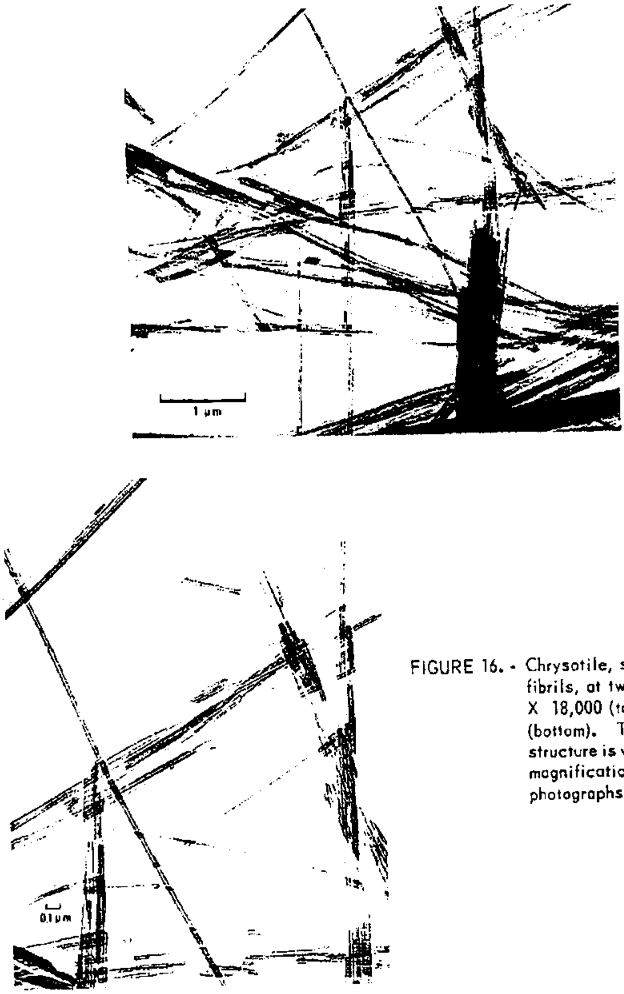

FIGURE 16. - Chrysotile, showing individual fibrils, at two magnifications: X 18,000 (top) and X 35,000 (bottom). The hollow-tube structure is visible at the higher magnification. (TEM microphotographs.)

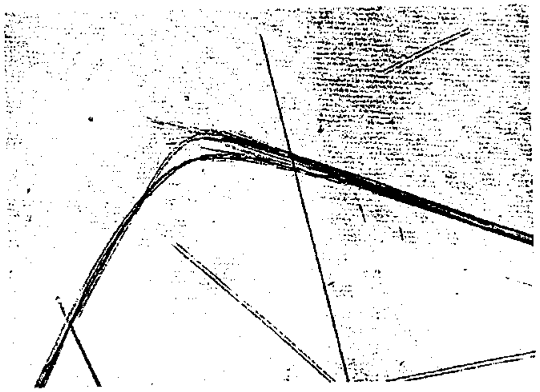

FIGURE 17. - Crocidolite, showing a fiber bundle and fibers (X 300).

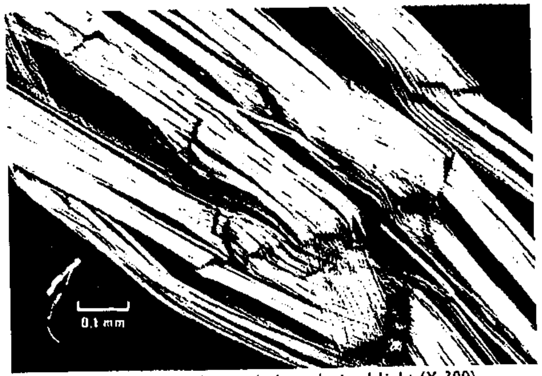

FIGURE 18. - Chrysotile by polarized light (X 100).

be justified on a historical as well as physical basis. Figures 17 to 20 show several asbestiform varieties of minerals.

Crystal Terms

Crystal Structure.--The pattern of the regular arrangement of atoms and ions in space. Usually refers to the basic (or average) structure of solids (and the ordered portions of liquids) without reference to minor, localized deviations.

Crystalline.--A substance possessing a reasonably well developed and long-range ordered crystal structure.

Crystal.--(1) Any single crystal; (2) a single crystal terminated by planar or nearly planar surfaces called crystal faces.

Single Crystal.--A crystal containing an uninterrupted crystal structure in a single orientation.

Twinned Crystal.--A crystal composed of two or more single crystals where adjacent crystals share a plane that is an integral part of both orientations of the crystal structure.

Polycrystalline.--A substance composed of two or more single crystals.

Polymorphs.--Two crystals with identical chemical composition but different crystal structures.

FIGURE 19. - Chrysotile (X 300).

Polytypes.--Polymorphs that contain essentially identical structural components, like layers, which are arranged in various patterns, like stacking of layers.

Crystal Form.--A set of symmetrically equivalent faces in a single crystal. A crystal may display several crystal forms.

Crystal Habit.--The actual shape assumed by a crystal or group of crystals as a result of the growth of dominant crystal forms (faces). Also known as crystal morphology.

Crystal Aggregate.--A cohesive mass of individual crystals or grains.

Single crystals exhibit growth shapes, while crystal aggregates exhibit growth patterns or arrangements (fig. 21). Some habits imply minor deviation of the crystal structure (deformation of the structure, presence of defects, inclusions, or impurities). Most habits are limited to the appearance of single crystals (crystal forms, irregular terminations, dimensional development, texture) or the pattern of aggregation of single crystals (dimension, orientation of the crystals, and cohesion between them). Under the following heading of "Single Crystals," the growth shapes are arranged first as equant, followed by other shapes that can be thought of as being derived from equant by suppressing or extending one or two of the three space dimensions, thus conveying the gradational nature of various defined crystal shapes.

Single Crystals

Equant.--The shape of a single crystal or grain with three approximately equal space dimensions.

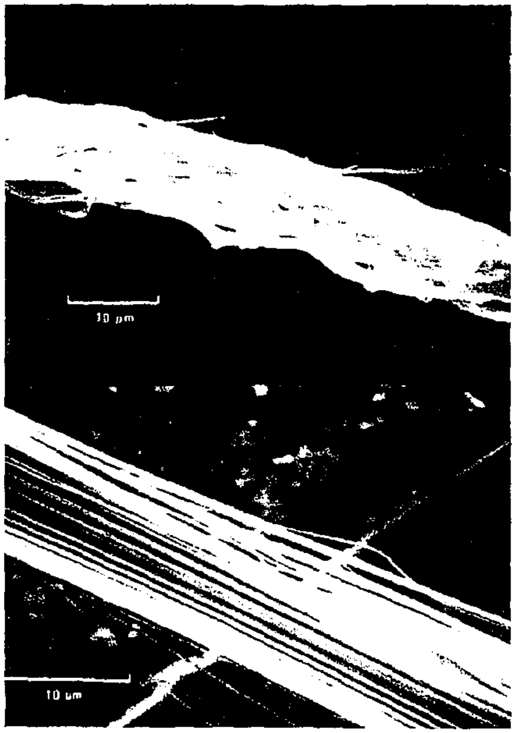

FIGURE 20. - Chrysotile at two magnifications: X 1,960 (top) and X 2,800 (bottom).

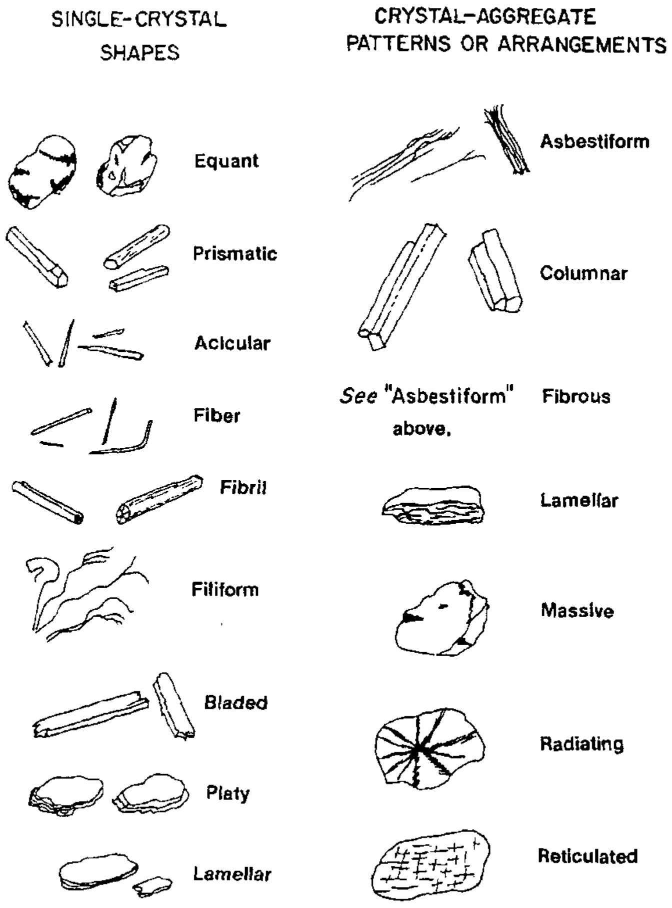

FIGURE 21. - Various shapes of single crystals, and patterns or arrangements of *crystal aggregates.*

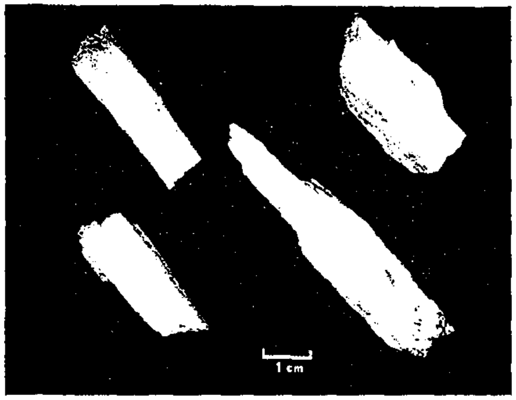

FIGURE 22. - Macrophotograph of spodumene ($LiAlSi_2O_6$) showing *prismatic shape* (X 1).

FIGURE 23. - *Macrophotograph* of tremolite showing prismatic and acicular crystal shapes (X 10).

Prismatic.--The shape of a single crystal with one elongated dimension and two shorter, approximately equal, dimensions.

Prismatic shapes of various single crystals are shown in figures 22 to 25.

Acicular.--The shape shown by an extremely slender crystal with small cross-sectional dimensions (a special case of prismatic form). Acicular crystals may be blunt-ended or pointed. The term "needlelike" refers to an acicular crystal with pointed termination at one or both ends.

Figures 23 and 26 show acicular crystals or crystal shapes.

Fiber.--See definition under "Asbestos-Related Terms."

Fibril.--See definition under "Asbestos-Related Terms."

Filiform.--The shape of threadlike mineral fibers.

Bladed.--The bladelike shape of a crystal with one longer dimension and two unequal, much shorter, dimensions.

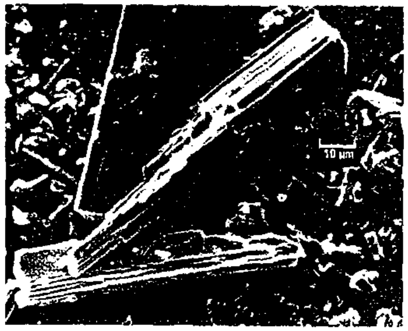

FIGURE 24. - Riebeckite, showing prismatic shape (SEM *photomicrograph* at X 800).

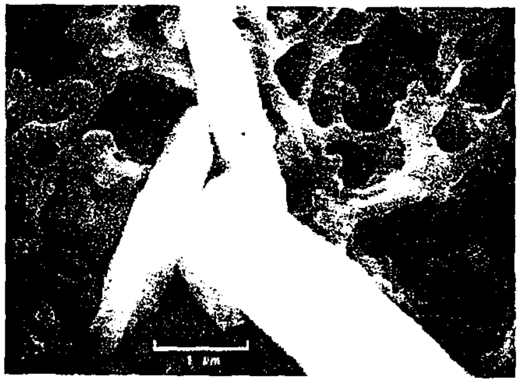

FIGURE 25. - Actinolite, showing *prismatic shape* (SEM *photo*micrograph at X 20,000).

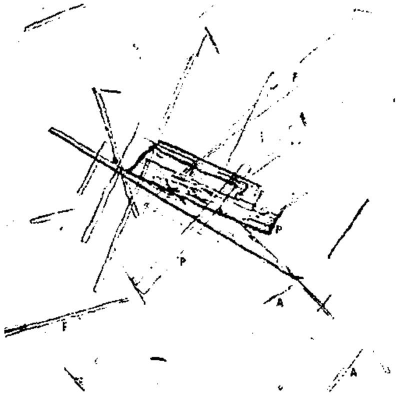

FIGURE 26. - Tremolite cleavage fragments (X 130), showing acicular (A), fibrous (F), and prismatic (P) shapes.

Figure 25 shows the bladed prismatic habit of actinolite.

Platy.--The shape of a crystal with one short *dimension and* two longer, approximately equal, dimensions. *Chlorite*, *micas*, and talc usually crystallize into platy shapes.

The serpentines, *although* possessing layered structure similar to *micas* and talc, rarely grow in platy shapes. Talc is of interest here because some talc deposits have associated asbestiform *and nonasbestiform* minerals. Platy talc, when seen in thin *sections or as fragments* in oil, may occur in various orientations. Plates lying *flat look* like plates but, if standing on edge, could appear *fibrous*.

Lamellar.--The shape of a very thin platy crystal.

Crystal Aggregates

Asbestiform.--See definition under "Asbestos-Related Terms."

Columnar.--The arrangement of a group of approximately parallel, prismatic, acicular, or bladed crystals.

Figure 27 shows columnar aggregates *of coarse* anthophyllite.

Fibrous.--See definition under "*Asbestos*-Related Terms."

FIGURE 27. - Macrophotograph of columnar aggregates of *coarse anthophyllite* (X 1).

Lamellar.--The pattern exhibited by aggregates of very thin platy minerals.

Massive.--Homogeneous structure without stratification, flow-banding, foliation, or schistosity. Also, crystals or crystalline grains that are tightly packed and scarcely distinguishable.

Figure 2 (top) is a good example of massive serpentine.

Radiating.--An arrangement of prismatic, acicular, or bladed crystals that appear to be diverging from a common center.

Figure 28 shows radiating aggregates of pyrophyllite.

Reticulated.--The pattern of a crisscross network of acicular, prismatic, or bladed crystals.

Breaking of Minerals

When a mineral crystal or grain is strained beyond its elastic and plastic limit, it will break in one of several characteristic modes described as *cleavage*, *fracture*, or *parting*.

Cleavage.--The tendency of a crystal to break in definite directions that are related to the crystal structure and are always parallel to possible crystal faces.

Cleavage Types

Pinacoidal Cleavage.--A crystal with only one cleavage plane that yields platy or lamellar fragments (for example, talc and the mica minerals). Also called platy, basal, or lamellar cleavage.

Prismatic Cleavage.--A crystal with two distinct cleavage planes that yield prismatic fragments (for example, the amphiboles and pyroxenes).

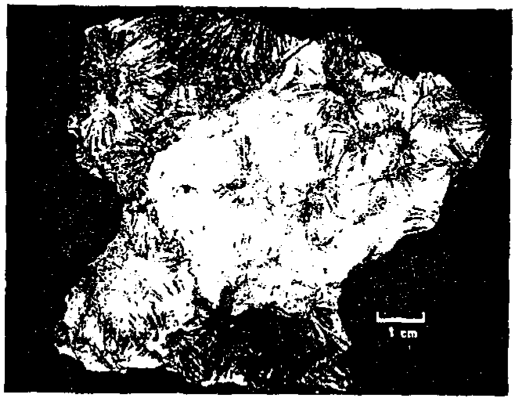

FIGURE 28. - Macrophotograph of radiating aggregates of acicular pyrophyllite (X 1).

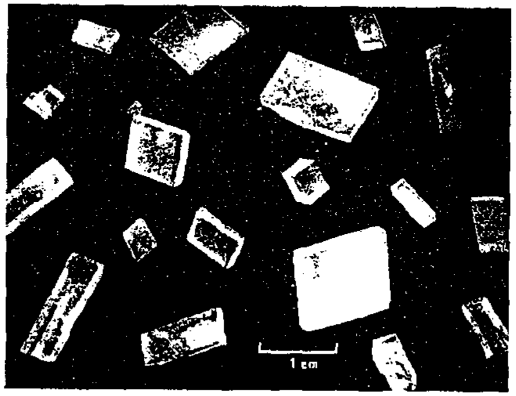

FIGURE 29. - Macrophotograph of calcite rhombohedral cleavage fragments (X 1.7).

Rhombohedral Cleavage.--A hexagonal crystal with three distinct cleavage planes that yield rhombohedral fragments (such as calcite).

Figure 29 shows the excellent rhombohedral cleavage of calcite.

Cubic Cleavage.--An isometric crystal with three distinct cleavage planes that yield cubic fragments (for example, halite).

Octrahedral Cleavage.--An isometric crystal with four distinct cleavage planes that yield octahedral cleavage fragments (for example, magnetite and fluorite).

Cleavage Quality

The quality or persistence of a cleavage is described as follows:

Perfect.--Difficult to break in any other direction; cleavage surfaces are extensive and smooth.

The cleavage of calcite in figure 29 is perfect.

Good.--Breaks readily along one direction, but can be broken in other directions; cleavage

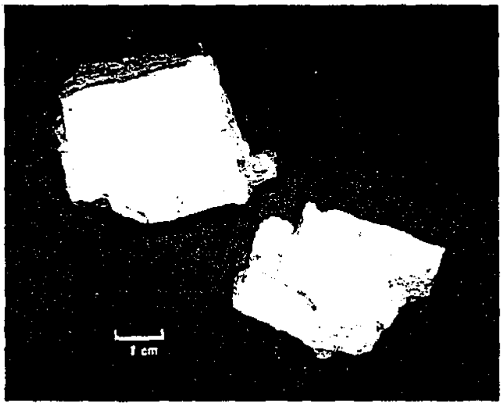

FIGURE 30. - *Macrophotograph* of pyroxene showing good cleavage interrupted by uneven fracture (X 1).

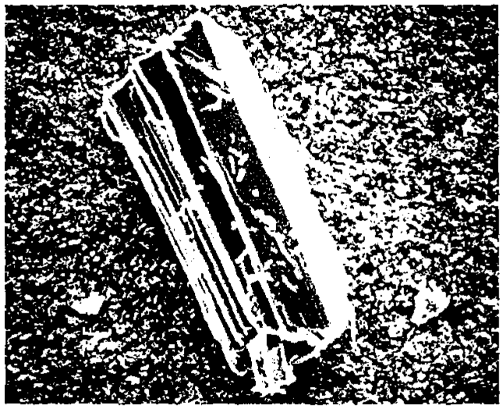

FIGURE 31. - Tremolite, showing good prismatic cleavage (SEM photomicrograph at X 560).

surfaces are smooth but interrupted by other fractures.

Figures 30 and 31 show good cleavage of pyroxene and tremolite.

Fair.--Breaks most readily along cleavage but also fractures easily in other directions; cleavage surfaces are seldom large.

Imperfect.--Breaks about as easily by fracture as by cleavage; cleavage surfaces tend to be small and frequently interrupted.

Cleavage Fragment

Cleavage Fragment.--A fragment produced by the breaking of crystals in directions that are related to the crystal structure and are always parallel to possible crystal faces.

Minerals with perfect cleavage can produce perfect regular fragments. Amphiboles with prismatic cleavage will produce prismatic fragments (fig. 31). These fragments can be elongated and on superficial observation may resemble fibers (figs. 26 and 32). However, because they did not grow as fibers, they cannot have the characteristics of fibers. *Consequently,* cleavage fragments cannot be called fibers. Minerals do not *always* break into the same shapes *as their growth* habits. For example, calcite has many growth habits, but usually breaks into rhombohedral cleavage fragments (fig. 29).

FIGURE 32. - Cleavage fragments of riebeckite (X 500). Particle F has a fibrous appearance.

Fracture.--The tendency of a crystal or grain to break in an irregular manner apparently unrelated to crystallographic directions.

Fracture types are as follows:

Even.--Breaking along relatively smooth planes.

Uneven.--Breaking along irregular planes.

Splintery.--Breaking into irregular, elongated fragments.

Conchoidal.--Breaking along spherical or conical surfaces.

Hackly.--Breaking with a jagged irregular surface.

Parting.--The tendency of a crystal or grain to break along crystallographic planes weakened by inclusions or structural defects. Different specimens of the same mineral may or may not exhibit parting. Twinned crystals often part along composition planes, which are lattice planes and, therefore, potential crystal faces. Parting is similar to cleavage.

MINERAL IDENTIFICATION AND CHARACTERIZATION

Until recently, emphasis in the United States was placed on occupational exposure of employees manufacturing or using asbestos products for insulation and other applications (29, 33). Regulatory procedures were adopted from those used in Great Britain. The industrial-hygiene identification procedures were acceptable to industry, health, and regulatory organizations because the concern was restricted to several mineral products known collectively as asbestos. Although light optical microscopic procedures counted only the larger particles collected on the *air filters*, the procedure was adequate for correlating health effects to the number of fibers observed. Exact definitions for asbestos-related mineralogical terms were not essential since all three groups (industry, health, and regulatory) clearly understood what was being counted and regulated.

The light optical microscopic procedures used by industrial hygienists were designed for control of asbestos-processing operations in which the chrysotile and asbestiform amphiboles are present as bundles of fibers as well as individual fibers (15). These bundles may have an average diameter of 0.75 to 1.5 um for chrysotile and 1.5 to 4.0 um for the amphibole asbestos (3). Particulates of these sizes can be readily observed at a magnification of X 450 to X 500. In contrast, samples from ambient air and personnel air monitors may consist of individual fibrils or small bundles of chrysotile 0.02 to 0.1 um in diameter, and/or amphiboles 0.1 to 0.2 um in diameter (3). Fibrils and small fibers in this size range are not visible using the conventional light optical microscopic procedures (13, 34). Therefore, the identification procedures currently used for regulating the U.S. mineral producing and consuming industries must be reexamined to insure that they are both mineralogically correct and applicable to the size range of the particles being regulated.

This discussion will be limited to the selected silicate minerals and their asbestiform varieties listed in table 1. The objective is to point out the particle size at which the minerals can be identified and characterized by various analytical techniques (17). Detailed descriptions of the various analytical and characterization techniques are available in numerous publications and textbooks and are therefore not included in this report.

A crystalline mineral is defined primarily by its crystal structure and by its definite composition or range of compositions. Therefore, any system of mineral identification should be based principally on crystal structure and chemical criteria. Additional characteristics have to be determined to distinguish varieties. These varieties have similar basic crystal structures and composition, but are usually differentiated macroscopically by the characteristic habits and/or other specific features of the varieties. The objective for this section of the report is to summarize the methodology for identifying the mineral first by mineral group (such as serpentine and amphibole), then by mineral (actinolite, anthophyllite, or chrysotile), and finally by mineral variety.

Macroscopic Samples

At the macroscopic level (easily visible by the unaided eye), the obvious feature of the asbestiform varieties is the presence of fibers that can be easily separated, while the nonasbestiform varieties have a massive, blocky, bladed, or columnar appearance (figs. 2-7). Although chrysotile does occur very rarely in a nonasbestiform habit, in general the distinction between chrysotile and serpentine can be based on the presence or absence of separable fibers. In some serpentine samples where an obvious asbestos texture is not displayed, the distinction between serpentine varieties may require more specialized techniques (6, 19). The distinction between serpentine and

amphibole minerals at the macroscopic level can be made by optical microscopy, elemental analysis, differential thermal analysis, and X-ray diffraction techniques. For essentially pure samples, these techniques should also be sufficient to identify the individual amphibole minerals based on the elemental composition corresponding to the various members of the solid solution series.

Many macroscopic samples of interest to the occupational and environmental health personnel may contain low percentages of asbestiform minerals (for example, chrysotile in serpentine and tremolite asbestos in talc). As a supplement to optical microscopy, the presence or absence of serpentine or amphibole minerals can be determined in 10- to 100-mg samples by instrumental techniques such as X-ray diffraction, differential thermal analysis, or infrared spectrophotometry. In general, the sensitivity of these instrumental methods is approximately 1.0 weight-percent. Sensitivity is significantly affected by the presence of other minerals that give a response at or near the response peak of the serpentine and amphibole minerals. It is important to note that these methods usually only distinguish between mineral groups; light optical or electron optical microscopy is required to obtain morphological characteristics necessary to identify varieties of the same material.

Chemical characterization is generally necessary to assign a specific mineral name to an amphibole whose structure is known. The amphiboles have been described (8) using the structural formula $W_{0-1}X_2Y_5Z_8O_{22}(OH,O,F)_2$. Generally, W = Na, K; X = Na, Ca, Mg, Fe^{+2}, Mn; Y = Al, Fe^{+3}, Ti; and Z = Si, Al. In addition to the variation implied by the structural formula, a chemical analysis must take into account inclusions of other minerals that may be present. In contrast to the more formidable task of amphibole chemical characterization, the serpentine minerals generally show little deviation from the formula $Mg_3Si_2O_5(OH)_4$. For either structural or chemical characterization of a macroscopic sample, sufficient time must be spent in sample preparation to insure that relatively pure minerals are being examined.

Microscopic Samples

The petrographic microscope provides a general method by which particles larger than 5 um can be characterized. By observing the optical properties characteristic of the structure and chemistry of a mineral, an experienced microscopist can distinguish amphiboles from serpentines and, in some cases, distinguish individual minerals within these groups (7). The refractive indices are sufficiently different for the serpentine and amphibole groups to make a distinction between groups by using the appropriate index oil (table 2). There is significant overlap in the range of the three refractive indices among the amphiboles, but a specific index (for example, α, β, or γ) can be determined to aid in identifying the amphibole species. Optical relationships can be confused, however, if the particle consists of fiber bundles or is some other form of crystalline aggregate. The well-known parallel extinction of the commercial asbestos known as Amosite can be used to distinguish that variety from the nonasbestiform varieties of cummingtonite and actinolite. A method of using extinction angles and cleavage directions to distinguish specific asbestiform and nonasbestiform amphiboles has been described (37); however, this technique is limited to particles with diameters greater than about

5 um and cannot be universally applied to all amphiboles. There are many other optical parameters such as pleochroism, sign of the elongation, and color that are easy to obtain. Other parameters such as optic axial angle, optical orientation, and optic sign are relatively more difficult to obtain.

TABLE 2. - Refractive indices for the serpentine group and selected amphibole minerals

	Refractive index	Range of values
Chrysotile	α	1.493-1.560
	β	1.504-1.550
	γ	1.517-1.562
Antigorite-lizardite	α	1.538-1.564
	γ	1.546-1.573
Anthophyllite	α	1.596-1.652
	β	1.605-1.662
	γ	1.615-1.676
Actinolite-tremolite	α	1.599-1.668
	β	1.612-1.680
	γ	1.622-1.688
Cummingtonite-grunerite	α	1.635-1.696
	β	1.644-1.709
	γ	1.655-1.729
Riebeckite	α	1.654-1.701
	β	1.662-1.711
	γ	1.668-1.717

Except for the asbestiform variety, serpentines are usually massive, while amphiboles range from fine-grained massive to columnar or radiating aggregates of prismatic or acicular crystals. Amphiboles in acicular habit may appear to grade into the asbestiform varieties. The characteristic features of this habit may still be seen by electron microscopy. Terms such as "acicular" or "prismatic" may still be applied when seen, but the term "asbestiform" begins to lose its usefulness. For example, how may flexibility be demonstrated in a 2-um bundle of fibers? As particle size decreases, the inability to manipulate the mineral grains restricts the use of the term "asbestiform" without altering the original sense of the word. High magnification necessitates the use of strictly *dimensional terms such as size and* aspect ratios to accurately describe the morphology of the amphiboles and serpentines. The degree of morphologic characterization possibly will depend on the *magnification* being used. An asbestos particle being described as a single fiber at low magnification may be seen to be a bundle of fibers at some high magnification. Therefore, the magnification must be stated in the description. Morphologic characterization using light microscopy can be accomplished on particles as small as a few micrometers. Electron optics can

be used to characterize a wide range of sizes extending down to a few angstroms. Morphologic characterization alone will not identify a mineral without supplemental structural or chemical data.

Structural information on individual particulates can be obtained by use of a transmission electron microscope (TEM) in the *selective area electron* diffraction mode (SAED). The inclination of the single crystal fragments to the electron beam is very critical since a slight tilt of the crystal may change a relatively simple reciprocal lattice pattern into a very complex one. Consequently, a special goniometer or tilting stage is necessary to obtain easily interpretable diffraction patterns. For the identification of the mineral, a goniometer or tilting stage is even more essential since dependable conclusions cannot be made from measurements on one reciprocal lattice plane. The quality of the SAED pattern is a function of fiber diameter (fig. 33). The larger diameter fibers (>0.5 um) strongly absorb the 60- to 100-kev electrons used in a conventional TEM, while the very small-diameter fibers (<0.2 um) do not give sufficient electron-diffraction intensity. A second problem with small-diameter fibers is the degradation of the single-crystal pattern by diffraction lines *from nearby particles. A higher energy TEM, with the* resultant greater penetration of the electron beam, can be utilized for large-diameter particles. However, these costly instruments are not widely available.

Although the magnitude of the characteristic C, the distance between the conspicuous layer lines for chrysotile and the amphiboles, is similar in direct space (d_{001} ~ 5.3A), the chrysotile pattern has very prominent streaks on these layer lines compared with the spot pattern for the amphiboles (27). Researchers indicate the ability to distinguish between the fibrous and non-fibrous variety of amphiboles by SAED is still to be resolved.

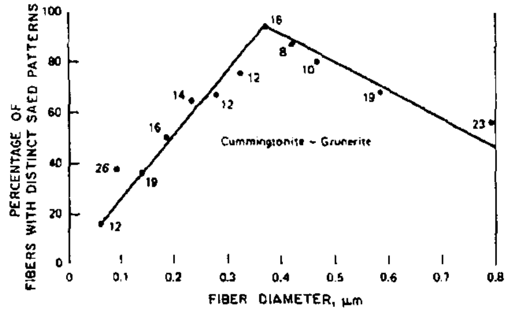

FIGURE 33. - Quality of SAED pattern as a function of amphibole fiber diameter. The values indicated on the graph are the number of fibers examined at each diameter. (Reprinted with permission of D. R. Beaman and D. M. File from Analytical Chemistry (2). Copyright by the American Chemical Society.)

At the very high magnification available with a TEM, chrysotile's hollow-tube (scroll-like) structure, approximately 50 A in diameter, is visible (fig. 16). This hollow-tube structure, together with chemical and structural data regarding the sample, is sufficient to identify the mineral variety. However, the hollow-tube structure is only visible for individual fibrils; fibers (composed of

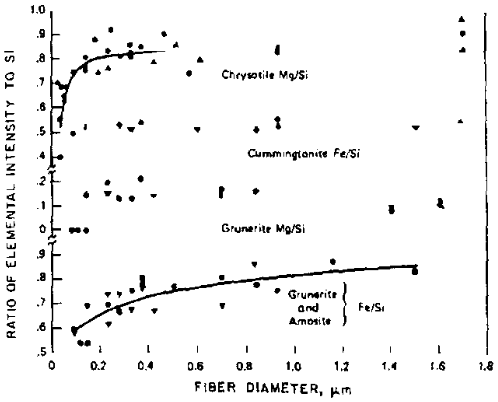

FIGURE 34. - Intensity ratio of FeKα, MgKα, or CaKα relative to SiKα as a function of fiber diameter. (Reprinted with permission of D. R. Beamon and D. M. File from Analytical Chemistry (2). *Copyright by the American Chemical Society.*)

several fibrils) will not display this characteristic because of stacking of the fibrils.

The elemental composition of microscopic grains is determined by either wavelength or energy-dispersive X-ray spectrography in conjunction with scanning or transmission electron microscopy. Extreme care must be taken in the calculation of elemental concentrations from X-ray spectral intensities because the spectral line intensities (FeKα, MgKα, CaKα, relative to SiKα) are dependent on particle diameter for small fibers (2). The particle diameter becomes a significant variable in the spectral intensity-composition relationship below 0.2 μm (fig. 34). Carbon contamination from diffusion pump oils must also be considered when analyzing small particles because the longer measuring times required to count sufficient numbers of X-ray photons allows time to build up a contamination layer. This carbon layer preferentially absorbs the lower energy X-ray photons.

Energy-dispersive X-ray spectral calibration data for each scanning or transmission electron microscope must be made using relatively pure standard minerals analyzed by accepted chemical-instrumental techniques. The analyst should be aware that other nearby grains may be contributing to the characteristic X-ray lines because of either penetration of the electron beam through the particles or secondary excitation of nearby particles from primary X-rays generated in the particle being measured. Modern electron optical instruments have electron beams diameters of approximately 0.1 μm; however, the sphere of excitation can be several micrometers in diameter as a result of scattered electrons and primary X-rays generated in this particle (26). Conversion of intensity into concentration using accepted computer programs such as "MAGIC" is limited in accuracy because these programs are designed for use with grains or particles several micrometers in diameter or larger, whereas the average mineral fiber diameter is less than 0.5 μm for amphiboles and less than 0.1 μm for chrysotile. A good example is the diameter size distribution of chrysotile fibers in ambient air samples (table 3). The important point to note is that approximately 95 percent of these chrysotile fibers are 0.12 μm or less in diameter. Therefore, quantitative correction procedures applicable to large particles will be of limited value in mineral-fiber identification because the relative X-ray spectral intensities are dependent on fiber diameter below 0.2 μm.

TABLE 3. - Frequency distribution of the width of chrysotile fibers in ambient-air samples,[1] percent

Diameter of chrysotile fibers, μm	Sample					
	1	2	3	4	5	6
0.02-<0.04	10	70	57	17	15	17
.04- <.06	47	24	28	29	33	49
.06- <.08	24	5	8	28	20	15
.08- <.10	14	1	2	12	26	6
.10- <.12	2	0	1	7	3	6
.12- <.14	0	0	2	3	1	1
.14- <.16	1	0	1	2	1	1
.16- <.18	0	0	0	1	0	1
.18- <.20	0	0	0	0	1	1
.20- <.22	1	0	0	0	0	1
.22- .24	0	0	1	0	0	1
>.24	1	0	0	1	0	1

[1] *Samples* were collected 1-2 miles from a serpentine rock quarry.

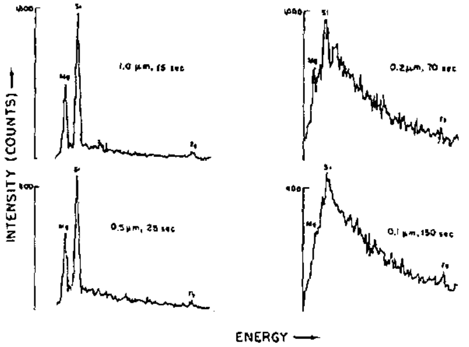

FIGURE 35. - Energy-dispersive X-ray spectra of chrysotile as a function of fiber diameter, BeO substrate (21).

Another problem with the *elemental characterization* of very small particles is the poor signal-to-background ratio. Longer counting times will help to improve the reliability of the measurement, but the best approach is to minimize the continuum background resulting from the interaction of the electron beam and the same substrate. Figures 35 and 36 show the *energy-dispersive* X-ray spectra from chrysotile fibers mounted on beryllium oxide (BeO) and beryllium (Be) substrates, respectively. The lower effective atomic number of Be compared with that of BeO results in a reduced continuum, therefore

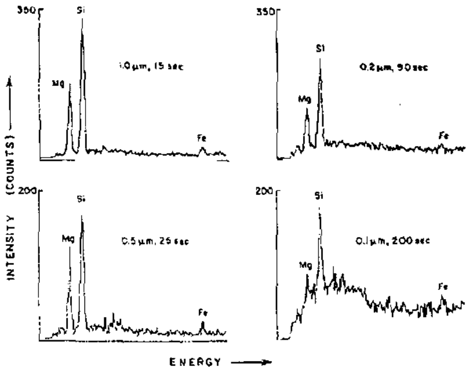

FIGURE 36. - Energy-dispersive X-ray spectra of chrysotile as a function of fiber diameter, Be substrate (21).

giving an improved signal-to-background ratio.

APPLYING MINERAL TERMINOLOGY TO THE IDENTIFICATION AND CHARACTERIZATION OF PARTICULATES

This section addresses the practical considerations and limitations encountered when applying nomenclature and identification-characterization procedures to regulatory and environmental samples.

Applying Morphological <u>Terminology</u>

One of the obvious features of minerals and their particulates is their morphology or shape. The need for precise definitions of terms such as "asbestiform," "fiber," "cleavage fragment," and "fibril" was explained earlier. *These definitions* were carefully structured to eliminate ambiguity and to be technically correct. Applying the definitions to samples requires careful thought as to what limits must be placed on interpretations resulting from the use of these terms and other mineralogical concepts. The underlying problem, recognized by both medical and regulatory personnel, *is classifying* the mineral particle as the asbestiform or nonasbestiform variety. The classification should withstand the test of mineralogical logic and proof. In a mineralogical sense, the source of the mineral particulates must be considered, as explained in the following discussion.

Particulates From a Known Asbestiform Serpentine or Amphibole Source

The definition of asbestiform minerals includes three aspects: morphology, structure, and chemistry. Morphologically, asbestiform mineral varieties separate into flexible fibers or flexible bundles of fibers. Flexible fibers bend readily and only break across the fibers into distinct pieces with some *difficulty*. Structurally, the asbestiform minerals are limited, in this report and in common practice, to the serpentine and amphibole mineral groups. Chemically, these minerals are all hydroxylated silicates; the term "hydroxylated" is preferred over "hydrated" because *these minerals* contain OH ions rather than water of crystallization. The serpentines contain approximately 13 weight-percent water; the amphiboles, approximately 2.5 weight-percent water.

For the purpose of this discussion, assume that a *hand specimen meeting* these requirements is correctly identified as an asbestiform mineral. If this sample is crushed and its fragments examined at various magnifications, its fibrous nature would be apparent, as in figures 14 and 17 to 19. These elongated fragments would be termed "fibers" and "bundles of fibers," and with the other available information would be called asbestiform. As these asbestiform particles are examined at increasing magnification, smaller particles become visible, while the image of large fibers and fiber bundles may exceed the field of the microscope. At increasingly smaller sizes, while fibers or bundles of fibers are still the predominant shape, a few of the fibers are observed to have broken into shorter and shorter segments. (Several short fiber segments are visible in figures 14 and 16.) These very short fiber segments are no longer described as fibers, but would be classified as fragments of fibers, or cleavage fragments if one or more cleavage planes govern their shape. Therefore, a known asbestiform sample would show an increase in the ratio of fiber fragments to fibers with a decrease in particle size.

Particulates From a Known Nonasbestiform Serpentine or Amphibole Source

If the hand specimen discussed previously does not separate into flexible fibers or bundles of fibers, the mineral would not be considered asbestiform. However, the specimen would be classified as serpentine or amphibole if the specific mineral is identified on the basis of optical properties, chemistry, and structure.

If crushed fragments of this known nonasbestiform mineral are examined at various magnifications, the particles would be primarily cleavage fragments, or irregularly broken fragments if cleavage does not govern breakage. However, a few elongated particles may resemble a fiber in appearance to the degree that they may be indistinguishable morphologically from fibers derived from an asbestiform mineral sample. Figures 26 and 32 for tremolite and riebeckite, respectively, show cleavage *fragments with fibrous shapes* that could be incorrectly identified as fibers.

What can be stated morphologically about particles derived from crushing a known nonasbestiform mineral is that most of the particles are cleavage fragments with nonasbestiform texture; a few are fibrous in appearance, particularly at low magnification; and all of the particles are known to be derived from a nonasbestiform source.

Comparison of Particulates From Known Serpentine and Amphibole Minerals and Their Asbestiform Varieties

The appearance of particles generated by milling known serpentine and amphibole minerals and their asbestiform varieties is shown in figures 37 to 40. The samples shown in figures 37 to 39 were photographed using light optical microscopy at three magnifications to show that, at decreasing size (*depicted by increasing magnification*), the *original habit* generally persists. For the nonasbestiform amphibole minerals, there were a few elongated particles from the riebeckite and tremolite. Elongated particles of this type

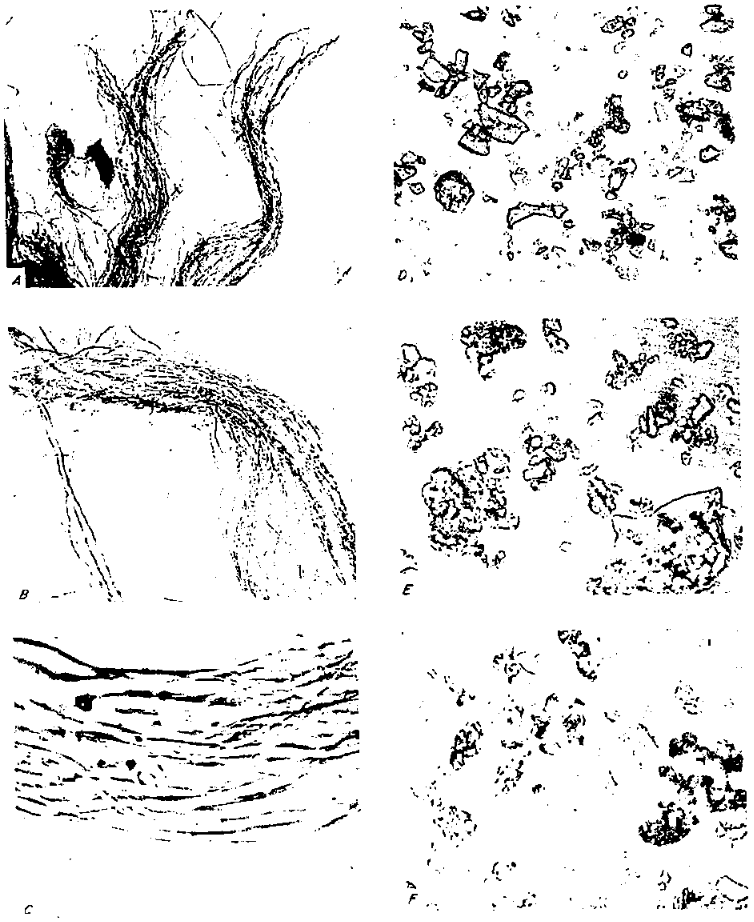

FIGURE 37. - Light optical photomicrographs of chrysotile and antigorite-lizardite at three magnifications. Chrysotile (left) at *A*, X 100; *B*, X 500; and *C*, X 950. Antigorite-lizardite (right) at *D*, X 100; *E*, X 500; and *F*, X 950.

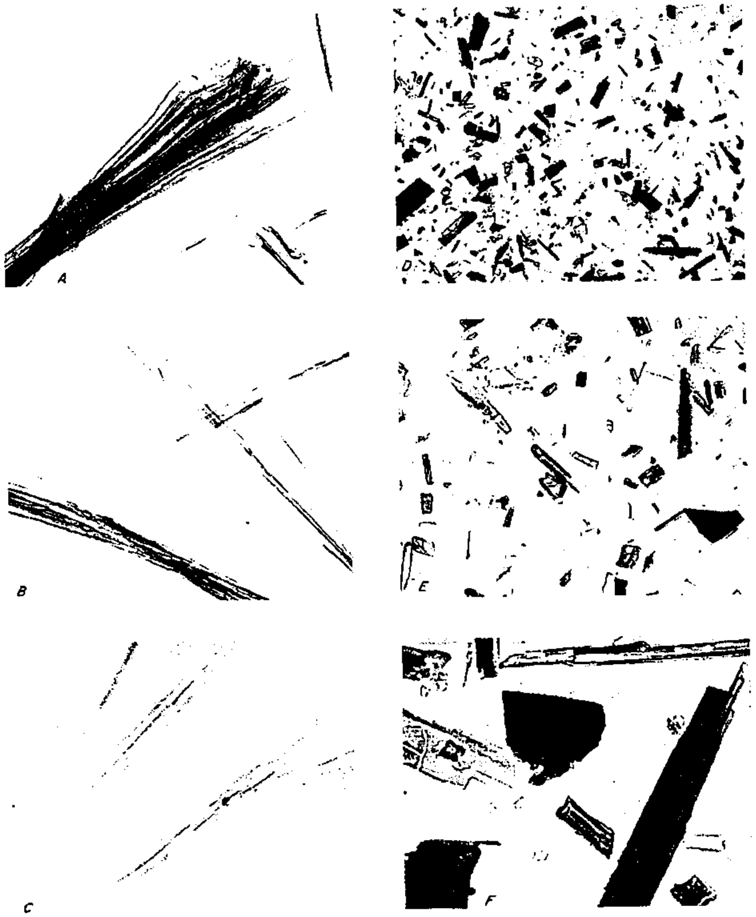

FIGURE 38. - Light optical photomicrographs of crocidolite and riebeckite at three magnifications; Crocidolite (left) at *A*, X 100; *B*, X 500; and *C*, X 950. Riebeckite (right) at *D*, X 100; *E*, X 500; and *F*, X 950.

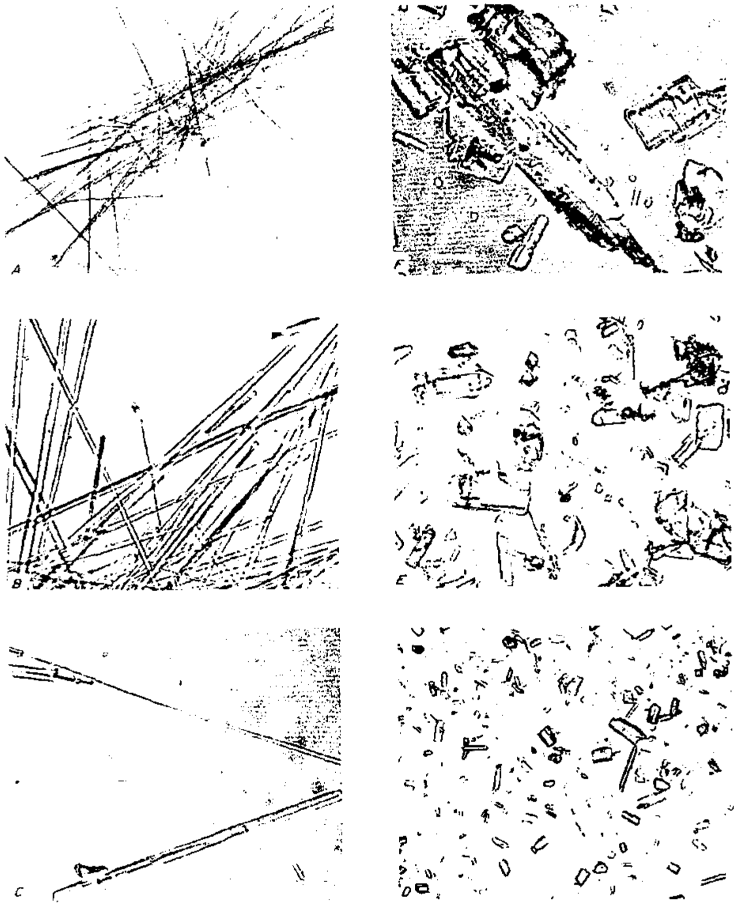

FIGURE 39. - Light optical photomicrographs of tremolite asbestos and tremolite at three magnifications. Tremolite asbestos (left) at *A*, X 100; *B*, X 500; and *C*, X 950. Tremolite (right) at *D*, X 100; *E*, X 500; and *F*, X 950.

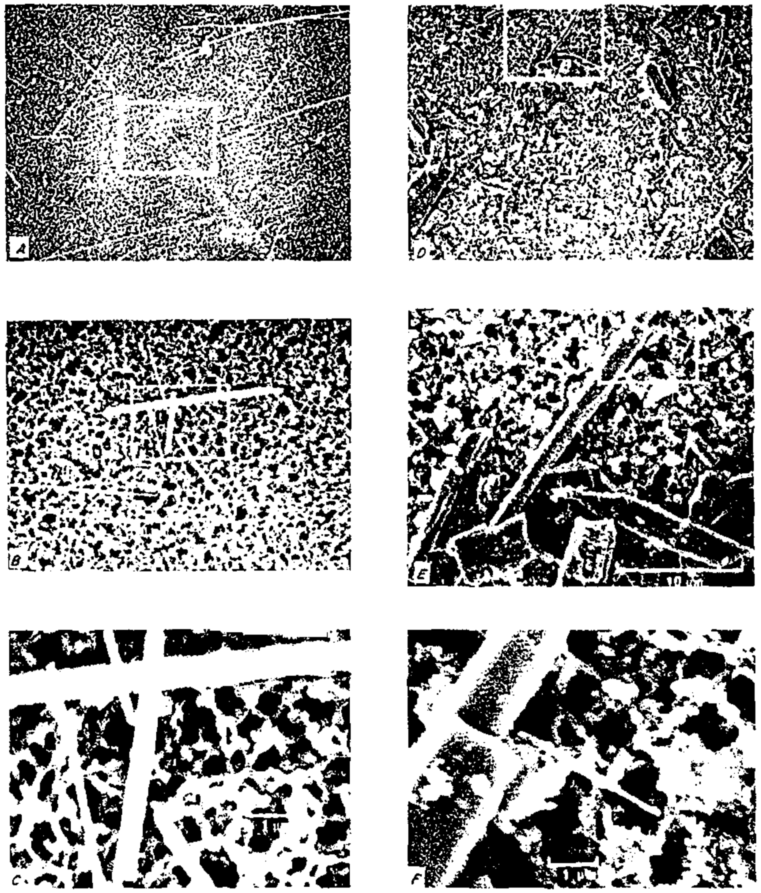

FIGURE 40. - SEM photomicrographs of crocidolite and riebeckite at three magnifications: Crocidolite (left) at *A*, X 500; *B*, X 2,500; and *C*, X 10,000. Riebeckite (right) at *D*, X 500; *E*, X 2,500; and *F*, X 10,000. Rectangles indicate the area shown at the next higher magnification.

are typical of the prismatic cleavage of amphiboles. To increase optical contrast, the serpentine group samples were dispersed in an immersion oil considerably below the refractive indices for the serpentine.

Riebeckite and crocidolite particles are compared at higher magnifications in figure 40. The outlined areas in the scanning electron micrographs indicate the area displayed at the next higher magnification. Again, note the presence of a few elongated cleavage fragments of riebeckite visible at the higher magnification. In contrast, the aspect ratio of the crocidolite will decrease with decreasing particle size because the individual fibers cannot cleave further along the fiber axis; they can only break into shorter segments.

Aspect Ratio

Existing regulatory standards are based on counting specific mineral particulates with aspect ratios of 3 to 1 or greater. This report emphasizes that the aspect ratio has little mineralogical significance for individual particulates but is applicable to a large number of particles. A few relatively long thin particles are produced as cleavage fragments from the crushing and grinding of many nonasbestiform minerals. Conversely, similar milling treatment will result in a few short segments of true fibers from the asbestiform varieties. However, statistically, the length-to-width characteristics of the milled amphiboles and serpentine and their asbestiform varieties are significantly distinct, as shown by the data in figures 41-44.

Figures 41, 42, and 43 show the frequency polygons of the aspect ratio distribution for milled samples of the normal nonasbestiform variety of three amphiboles--anthophyllite, tremolite, and hornblende, respectively. Note that in all three examples, approximately 70 percent of the particles have an aspect ratio of less than 3 to 1, and 95 percent of the particles have a length-to-width ratio of less than 10 to 1. The frequency distribution maximums of the aspect ratios for milled anthophyllite asbestos and tremolite asbestos are significantly higher than those for the normal, nonasbestiform variety. Thirty to forty percent of the asbestiform particulates are in the 10-to-1-or-longer class, with a significant number of particles having an aspect ratio greater than 20 to 1.

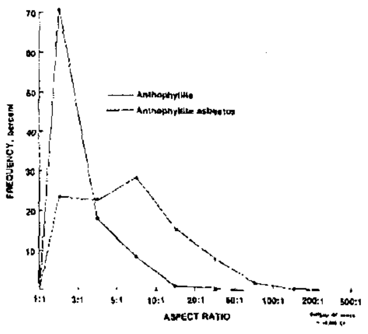

FIGURE 41. - Frequency polygons for the aspect ratios of anthophyllite and anthophyllite asbestos.

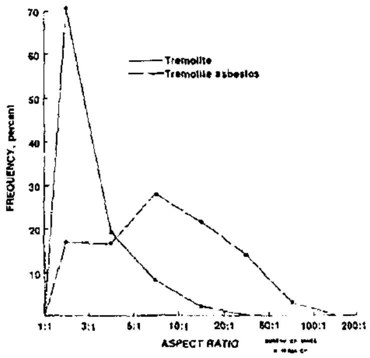

FIGURE 42. - Frequency polygons for the aspect ratios of tremolite and tremolite asbestos.

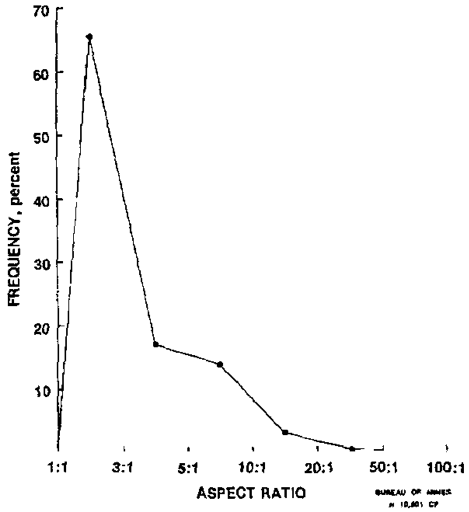

FIGURE 43. - Frequency polygons for the aspect ratio of hornblende.

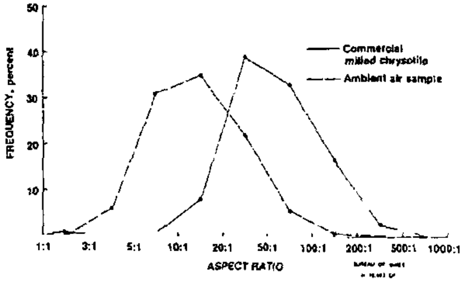

FIGURE 44. - Frequency polygons for the aspect ratios of commercial-grade chrysotile and chrysotile in ambient air.

Figure 44 shows the distribution frequencies for a milled commercial grade of chrysotile asbestos and for chrysotile particulates collected on ambient air filters in the vicinity of a serpentine rock quarry. For the commercial-grade chrysotile, over 50 percent of the particles have an aspect ratio greater than 50 to 1, whereas the frequency distribution for the ambient air sample has a maximum between 10 to 1 and 20 to 1. These results are anticipated because the higher aspect ratios for the commercial-grade chrysotile are characteristic of the significantly longer starting material.

All of the aforementioned samples except the ambient air were milled, then dispersed in water for collection on a suitable substrate. The samples were then measured using electron microscopy at magnifications of 5,000 to 10,000. The ambient air sample, collected near a serpentine rock quarry, was measured using a TEM with magnifications of X 5,000 to X 32,000.

Based on these data, one test for distinguishing the presence or absence of the *asbestiform variety of a mineral* could be an examination of the frequency distribution of the aspect ratio for that mineral. Assuming positive identification of the mineral type, then the designation of variety would be based both on particle morphology and the frequency maximum of the aspect ratio. Cleavage fragments will generally have a frequency maximum less than 3 to 1, whereas the asbestiform varieties will fall between 10 to 1 and 20 to 1 or higher, depending on the characteristics of the mineral and the history of the sample, particularly the type and degree of milling. *If any shape or* size limits are placed on characterizing mineral particulates, such limits should be based on *medical evidence or on some* limitation of the characterizing technique and so stated.

Particulates From Unknown Sources

Samples such as environmental airborne or waterborne mineral particulates collected at a considerable distance from a possible source are examples of particulates from an unknown source. The samples could have been collected at a location so distant from a known source that other mineral particulates originating from other sources compose most of the sample.

The *source of the particulates* in an environmental sample may be located by taking additional samples at selected intervals in the direction of, and *closer to*, the suspected source. However, several factors must be considered: The direction of air and water currents with respect to the suspected source, and the proximity to and direction of other sources with regard to the suspected source. One study found very low concentration of airborne chrysotile upwind from a source compared with a concentration two orders of magnitude greater downwind (14). Another important consideration is the *level of natural* or human disturbances of particulates; for example, strong versus weak winds, or heavy versus light vehicle traffic. *In some instances*, it may be possible to identify the source if the mineral particulates of interest have *unique trace elements or combinations* of elements that are specific to the probable mining or milling operation emitting the particulates. Detailed elemental analysis using the X-ray spectral capabilities of an SEM or TEM is required on both the suspected source and the particulates.

APPLICATIONS

The following examples illustrate the *application of mineral terminology* and identification-characterization procedures to three types of problems: (1) *chrysotile determination in ambient-air* samples collected near a serpentine rock quarry, (2) identification of asbestiform minerals in ceilings and walls of public buildings, and (3) characterization of a mineral product. These examples illustrate, in order, the need for higher magnification than available with the light optical microscope, the use of various characterization techniques to screen and identify asbestiform minerals, and the judgment of the analyst in distinguishing cleavage fragments and asbestiform particles.

Ambient-Air Samples Collected Near Serpentinite Rock Quarry

The Bureau of Mines is working with State and Federal officials to measure mineral particulates in ambient-air samples collected in the vicinity of a serpentine rock quarry. Optical microscopic procedures at about X 500 are limited to the *identification of* mineral particulates longer than 5 μm with an aspect ratio of 3 to 1 or larger (criteria set by the Mining Enforcement and Safety Administration and the Occupational Safety and Health Administration). The mineralogist can further identify the particles as belonging to the serpentine, amphibole, or other mineral group with index oils (table 2).

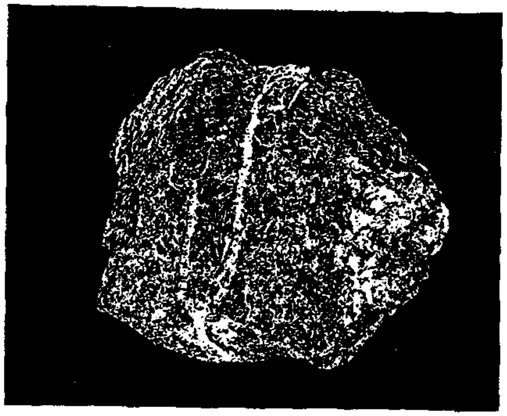

FIGURE 45. - Macrophotograph showing chrysotile veins in serpentine rock (X 1).

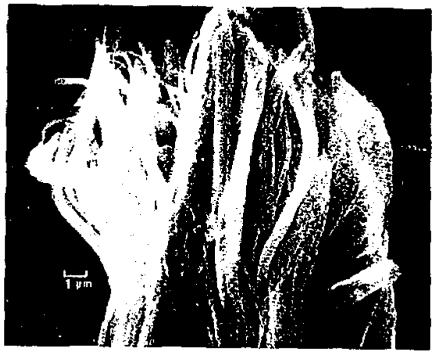

FIGURE 46. - Chrysotile bundle (SEM photomicrograph at X 5,000).

The serpentine rock in the quarry is interlaced with small veins of chrysotile (fig. 45). Optical microscopic procedures used for industrial hygiene are adequate for the detection of large chrysotile fiber bundles. These fiber bundles of commercial-grade chrysotile can be several micrometers or larger in diameter (fig. 46). In contrast, the mining and crushing operations in the quarry plus transport of particulates *over a distance* breaks bundles of fibers down to fibers or fibrils with diameters of 250 to 1,000 A (table 3).

Figure 47 is a series of SEM photomicrographs of a mixture of chrysotile and nonasbestiform serpentine handpicked from a small vein in the serpentine rock quarry. Note that at X 450 (corresponding to the optical microscope magnification), only one or two bundles of chrysotile are faintly visible; the predominant particles are the nonasbestiform serpentine. As the magnification is increased, the high concentration of chrysotile fibers becomes readily visible. The fiber diameter size data in table 3 indicate that more than 95 percent of the chrysotile fibers in these ambient air samples are below the limit of resolution of the optical microscope. Although many other scientists have pointed out the limitation of the optical procedures for chrysotile in ambient air, there is need for continuous emphasis that higher magnification techniques are necessary for environmental and regulatory samples.

Asbestos in Ceiling and Wall Materials

A possible environmental hazard is the release of asbestos from ceilings and walls in homes, churches, schools, and various other public and commercial buildings. Because of the very high number of potential samples to be examined by various State or Federal agencies, a rapid and reliable screening procedure is necessary to identify those samples that warrant further tests. Three complementary analytical methods for screening, identification, and semiquantitative estimate of the asbestiform mineral concentration are X-ray diffractometry, differential thermal analysis, and microscopy (light optical and scanning electron).

The screening identification procedures can be relatively simple because chrysotile is the principal asbestos mineral used for building insulation materials, with Amosite used to a much lesser extent. In 18 samples from a midwestern municipal health department, chrysotile was a major constituent (>50 weight-percent) in 2 samples, a minor constituent (1 to 10 weight-percent) in 12 samples, and not detected in 4 samples. Other minerals present in various concentrations in these samples were calcite, quartz, gypsum, and mica. Amosite was found as a major constituent in the ceiling of an older building located on a university campus.

The presence of either serpentine or amphibole minerals in the insulation materials can be used as a probable indication of asbestos. Therefore, screening tests are based on the presence or absence of characteristic differential thermal analysis or X-ray diffraction peaks of either serpentine or amphibole minerals. For the positive samples, confirmation of the presence of the asbestiform variety requires some type of microscopic examination because the thermal and X-ray diffraction methods do not identify the mineral variety.

Differential thermal analysis provides a detectable signal from chrysotile at 0.5 to 1.0 weight-percent, as indicated by the curves shown in figure 48. Note the increase in the endothermic (A) and exothermic peaks (B) upon addition of about 5 weight-percent chrysotile to a sample taken from a school ceiling. The sensitivity of differential thermal analysis for the amphibole minerals is significantly poorer because the H_2O content of amphiboles is approximately 2.5 percent compared with about 13 percent for chrysotile. The sensitivity of the X-ray diffraction method also ranges from 0.5 to

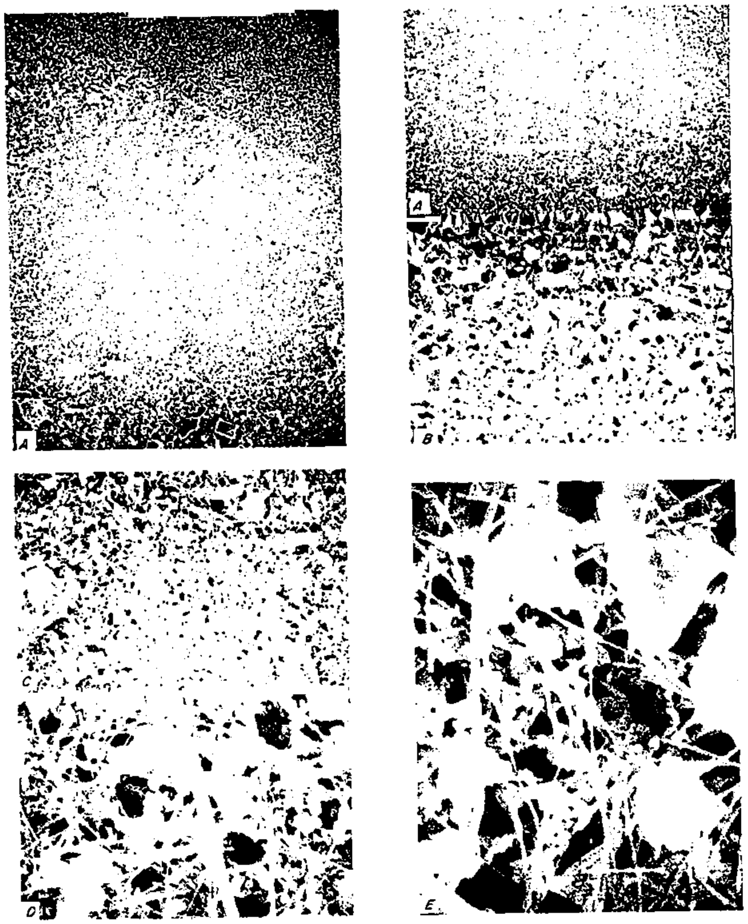

FIGURE 47. - Mixture of nonasbestiform serpentine and chrysotile at five magnifications: *A*, X 450; *B*, 2,250; *C*, X 9,000; *D*, 1,800; and *E*, X 18,000. Rectangles indicate the area shown in the next panel.

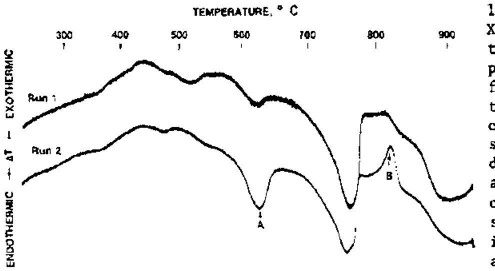

FIGURE 48. - Differential thermal analysis of sample from school ceiling, showing endothermic (A) and exothermic (B) peaks of serpentine. Run 1 is the sample as received; run 2 is a mixture of 95 pct of the as-received sample and 5 pct chrysotile.

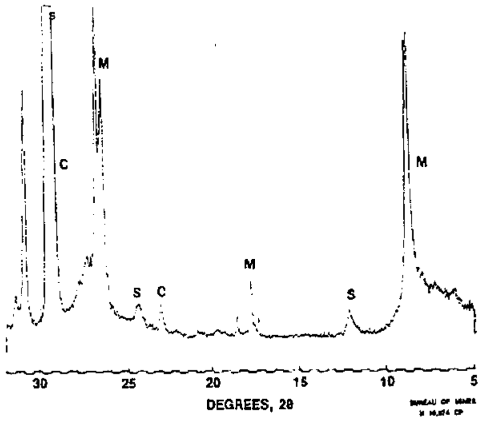

FIGURE 49. - X-ray diffractometer scan of sample from school ceiling, showing the presence of calcite (C), mica (M), and serpentine (S).

1.0 weight-percent. An X-ray diffractometer scan of the 2θ range for major serpentine peaks is shown in figure 49. The magnitude of the characteristic peaks for chrysotile are a function of several factors, including degree of fiber orientation and the type of milling or crushing used to process the sample. Also, the sensitivity of both methods is affected by the presence of other minerals that have characteristic thermal or diffraction peaks in the same region as those of the minerals of interest.

Some samples will be composed of a mixture of synthetic and natural fibers, such as the mixture of fiberglass and chrysotile shown in figure 50. Generally, it is not difficult to identify the synthetic fibers based on their larger diameter and the more uniform appearance.

Amphiboles and Talc

Asbestos-related health regulations are having a significant impact on the domestic talc industry from occupational exposure at the mines and mills and at various manufacturing plants that use talcs in their operations. Certification that the talc does or does not contain asbestiform minerals is important because the occupational health requirements are much more restrictive if the talc is designated as containing asbestiform serpentine or amphibole minerals.

FIGURE 50. - Sample from university building, showing a mixture of chrysotile and fiberglass (X 140).

Talc is both the name of a specific mineral, $Mg_3Si_4O_{10}(OH)_2$, and a commercial term for a mixture of minerals ranging from essentially 100 percent talc to blends where the mineral talc is a minor constituent (12, 23). Semiquantitative estimation of the serpentine and/or amphibole mineral concentration, if present, can be obtained by X-ray diffraction and differential thermal analysis. Several talc deposits contain a variable amount of tremolite. Therefore, the essential question faced by the analyst is whether or not the tremolite is fibrous. Figure 51 shows the typical platy morphology of talc; no tremolite (amphibole) was detected in this sample by X-ray diffraction. Figure 52 illustrates the type of particles obtained from a mixture of tremolite and platy talc. The cleavage fragments of tremolite are typical of the nonasbestiform variety. Better judgment is required of the analyst with the type of sample illustrated in figure 53. This sample consists of platy talc, cleavage fragments of tremolite, and minor to trace amounts of fibrous tremolite. For this latter sample, the 3-to-1 aspect-ratio criteria would greatly overestimate the number of fibrous tremolite particles collected on air filters or other monitors.

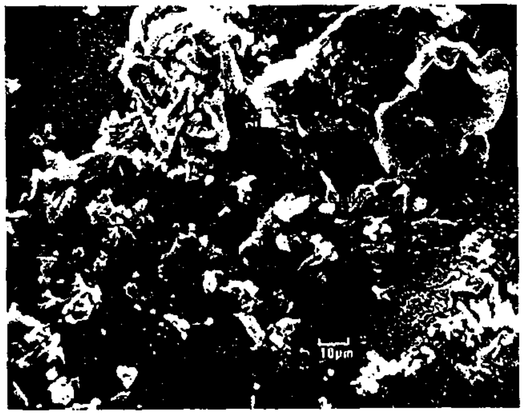

FIGURE 51. - Typical platy morphology of talc (X 600).

RESEARCH NEEDS

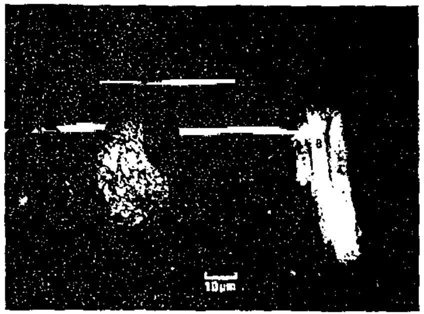

FIGURE 52. - Platy talc (A) and tremolite cleavage fragment (B) (X 640).

FIGURE 53. - Platy talc, tremolite cleavage fragments, and a fibrous tremolite particle (A) (X 400).

There are several areas in particulate identification-characterization where further research is warranted. Areas of research that are immediately applicable to asbestos are briefly summarized.

Commercially available electron optical instruments are generally limited to morphological characterization for mineral particles with diameters less than 0.2 µm. As pointed out in the identification-characterization section, both the signal-to-background ratio for energy *dispersive X-ray* spectra and the SAED pattern are significantly degraded for elongated particles less than 0.2 µm in diameter. Field emission electron optical microscopes with their higher vacuums and smaller beam *diameter may* have some advantages over conventional SEM instruments. Also, other microprobe techniques, in particular ion microprobe *mass spectrographs* and laser Raman microprobes, should be evaluated for particulate characterization.

Although electron microscopic methods can generally positively identify chrysotile in air and water samples, the quantitative aspects of the measurements need substantial improvement. Sample treatment and measurement errors need to be isolated from sampling variance. Because of the ambiguity of fiber counts, results should be reported both in mass equivalents and in fibers per unit volume.

Fundamental studies should be conducted to determine if there are unique chemical and physical characteristics of a mineral fiber as compared to elongated cleavage fragments. For example, the surface properties of chrysotile are similar to those of magnesium hydroxide, whereas the nonasbestiform varieties of serpentine have the surface characteristics of a silicate (30). Variations in surface properties, if any, between asbestiform particles and cleavage fragments of amphiboles should be investigated. Surface characterization techniques to be considered should include Auger electron spectrography and low-energy X-ray spectrography. Research at the University of Minnesota indicates that asbestos fibers have an extensive surface charge over the whole surface, whereas cleavage fragments have a significantly lower surface charge (40). Extinction angle measurements are another possible approach to distinguishing asbestiform from the nonasbestiform varieties of amphiboles (37).

There is a critical need to reexamine the 3-to-1 aspect ratio as a criterion for a mineral fiber. The aspect ratio for *fibers from asbestiform minerals* were as much as 200 to 1 or higher, whereas the ratio for cleavage fragments is about 3 to 1, as illustrated by the data in this report. The 3-to-1 aspect ratio may be valid *for the industrial* hygiene control of asbestos-processing plants; however, its applicability to existing nonasbestos mining and ore processing plants requires critical evaluation. There *is also need* to evaluate the restriction of the mineral particulate measurments to light optical microscopy because many particulates of interest, especially chrysotile fibrils, are not visible by this technique. Low-cost scanning electron microscopes are in the same price range as research-grade petrographic microscopes, and the skill requirements for the operator are comparable for both instruments.

Particulate measurements by *microscopic procedures* are time consuming and expensive. Two possible approaches to reducing the time and cost are (1) automation of the particulate identification-characterization measurements using computerized image analyzers and (2) development of chemical reagents that give a specific response with either chrysotile or the various asbestiform amphibole minerals. *Using the chemical-reagent approach*, mass-concentration values could be obtained by measurement of some response such as color, ultraviolet fluorescence, X-ray spectral intensity, etc.

Health studies related to inhalation and ingestion of fibers have been essentially limited to *well-defined commercial types* of asbestos. The fundamental question to be resolved is the biological effects of cleavage fragments compared with those of true mineral fibers. If shape and size are the *critical parameters*, the analyst could establish and measure suitable analytical parameters to monitor the particulates of interest. Likewise, if the health scientists find a correlation between health and the amount of trace metals, adsorbed organics, surface area, etc., the analyst can respond accordingly. Therefore, there is a primary need for an adequate quantity of well-*characterized amphiboles* and serpentine of both the normal and asbestiform varieties for use in health-related studies.

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A Critical Review

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Fiber analysis by SEM/ADXS

The analytical technique used to perform identification of the fibers is of serious concern. The Scanning Electron Microscope method is very limited in the information that it can provide for accurate identification of minerals. No crystal structural information can be deduced from SEM, so there is no means of differentiating minerals of similar chemical composition but different crystal structure. Quantitative chemical analysis using Energy Dispersive x-ray Spectrometry in SEM is only possible if the material under analysis is presented as an flat surface perpendicular to the electron beam. The computer algorithms used for converting crude element x-ray counts to chemical analysis can be applied successfully if the material is an infinitely thick plate (or effectively so) or if it is a thin film. In any other situation, such as in this method, with irregular particles scattered on a plastic substrate, the best that can be achieved is an approximation to the chemical composition. This is because of the lack of information about mass absorption coefficients, secondary fluorescence effects and the geometry of the particle surfaces with respect to the x-ray detector. In addition, if the particles are 0.2 μm thick or less the x-ray yield is very much reduced and the statistical reliability of x-ray yields from different elements in the mineral is very much reduced. The result is that small particles (0.2 μm) of minerals such as talc, anthophyllite, chrysotile, cummingtonite, forsterite and enstatite which all contain magnesium and silicon in slightly different proportions are effectively indistinguishable. The same would be true of the minerals tremolite, actinolite, diopside, pigeonite, and monticellite, all of which contain calcium magnesium and silicon. Furthermore, in my experience, the x-ray yield from fibers with diameters of 0.1 μm or less is so low as to make it impossible to identify fibers with any confidence. It is disconcerting then to observe that the geometric mean widths of mineral fibers identified in the mesothelioma cases as talc, anthophyllite and chrysotile are in the range from 0.05 to 0.20 µm. The same minerals in the non-mesothelioma cases range from geometric mean widths of 0.08 to 0.34 µm., and the minimum widths of fibers, cited as identified, are as low as 0.03 µm.

The authors provide no information as to how these identifications were achieved, what reference materials were used, or what the statistical reliability of the x-ray counts were. In view of this lack of information the identifications can only be considered as unreliable.

Fiber lung burdens

The paper provides Tables with lists of the lung burdens in terms of fiber numbers, asbestos bodies and mineral particles. These are expressed as fibers per gram of dry weight tissue (fibers and asbestos bodies) or as numbers of particles per millilitre of lung (talc and silica particles). There is no way of correlating these figures so the relationship between particles and fibers is unknown.

The lung fiber burdens are provided separately for the two mesothelioma cases and the eight non-mesothelioma cases, but the fiber dimensions, upon which much of the argument is based consists of the geometric mean widths, lengths and aspect ratios for all fibers of the different types in the two groups of cases. The statistical validity of making that calculation is questionable since the authors are actually trying to demonstrate whether any of these fiber populations are the same or similar. At this point they appear to have already decided that by grouping all the fiber types in the two sets of cases together. As a result, at first sight, there is little that can be done to assess the quality of the results.

The total numbers of fibers of each type found in the analyses (Hull et al, Tables 4 and 5) must relate in some way to the lung burden results (Hull et al, Table 3). The lung burden results report tremolite/actinolite concentrations in lung while the dimensional analysis of Table 4 and 5 report tremolite and actinolite separately, so there are still some difficulties.

Since the actinolite numbers are comparatively small, for this analysis the Table 4 and 5 fiber numbers of tremolite and actinolite will be grouped together.

Doing so produces Table 1 shown below. For the mesothelioma cases only, this shows the fiber burdens in each lung and the actual numbers of fibers found in both lungs together. The analysis is too complicated to apply to the non-mesothelioma cases

Table 1

Mineral	M1 F/g dwl	M2 F/g dwl	Number found (M1 + M2)
Anthophyllite	5	0	6
Tremolite/Act	2	1110	39
Chrysotile	5	21	5
Talc fibers	46	96	54

Understanding how these numbers must relate is complex but can be deduced from the analytical processes used in arriving at them. The fundamental number is the number of fibers actually found in the course of the SEM search which is shown in column 3 as the sum for the two mesothelioma cases. The lung concentration is calculated from the fibers found by simple arithmetic using the formula; $F_{lung} = n . V/v . A/a . 1/W$, involving the weight of tissue, volumes of initial suspension of residue and any aliquots taken from it, the effective area of the filter and the area searched by SEM. Irrespective of the exact values of these factors, for any one case, the final factors must be exactly the same for each of the fiber types, effectively a constant, as in $F_{lung} = n . K_1$.

From Table 1 we know that for the anthophyllite in case M1, $F_{lung} = 5 = 6 . K_1$. The value of K_1, 0.833 must be the same for all of the other minerals in that sample since they were found in the same residue during the same search.

Similarly, for case M2, for the tremolite, $F_{lung} = n . K_2$, so $1110 = (39 - X) \times K_2$ where X is the number of tremolite fibers found in case M1.

But we also know that for case M1, $2 = X . K_1$, and $K1 = 0.833$, so the actual number of tremolite fibers found in the search of case M1 was $X = 2/0.833 = 2.40$. Assuming some arithmetical rounding of the numbers the number of fibers observed in M1 must have been 2.5 since only whole or half integer numbers can ever be found in such a search, and using 2.5 for X in the equation $F_{lung} = X \times 0.833$ gives 2.08 fibers in lung.

So now we know that for tremolite in Case 2, $1110 = (39 - 2.5) \times K_2$, therefore

K2 = 30.4.

Using these factors for the other fiber numbers in Table 1 of this document allows an iterative series of tests for the internal consistency of the data, remembering that these must be constant factors for all of the fibers in any one lung sample. Unfortunately there are no two constants that can produce the results as given by Hull et al, for all of the four fiber types listed. The data are not internally consistent, and this raises serious questions about their validity. A more detailed evaluation of these factors is given in the appendix.

Fiber dimensions

The authors consider that the similarity in the geometric mean dimensions of the fibers found in the lungs of the mesothelioma and non-mesothelioma cases to be very significant in ascribing the mesothelioma causation to the exposures in the talc mining industry. They specifically mention the similarity between the talc fibers and the tremolite fibers in the two sets of cases. These are shown below together with the anthophyllite dimensions in Table 2 below.

Table 2 Fiber dimensions as given in Hull et al (2002)

		Width (µm)				Length (µm)			
	Fiber type	n	GM	Min	Max	GM	Min	Max	AR
Meso	Tremolite	38	0.22	0.1	0.4	4.5	1.7	10.6	26
Non-Meso	Tremolite	51	0.34	0.04	1.00	5.3	1.5	17	24
Meso	Talc	54	0.20	0.05	1.00	5.3	1.4	53	43
Non-Meso	Talc	248	0.33	0.06	2.8	6.4	1.3	219.0	30
Meso	Anthophyllite	6	0.15	0.06	0.3	10.6	3.9	30.2	90
Non-Meso	Anthophyllite	85	0.24	0.05	1.6	7.7	1.6	146.0	56

Ignoring the fact that the grouping of these fibers may not be statistically valid for fibers found in 10 different lung residues with no known association, other than that the cases worked for some time in one of what may have been a number different talc mines in New York State, the figures cited do not appear to support the conclusions. The authors state that there is no significant difference between the size distributions of the mineral types in the two sets of cases based upon Student's t-test, with significance determined as $P < 0.05$. This assertion is made with no reference to what was actually tested and there is no way that the data presented can be used to confirm it. As the table above shows, the geometric mean widths of fibers of each type in the non-mesothelioma cases are consistently 50% or more greater than those in the mesothelioma cases. As fiber width is a critical characteristic of asbestos fibers it would seem that this difference should be examined more closely before reaching a judgement about the 'similar dimensions' of the dusts to which the miners were exposed. In addition, it seems unreasonable to make such assertions on the basis of a relatively small number of fibers in each class (with the exception of the talc fibers in the non-mesothelioma cases). A serious comparison of fiber size distributions ought to be made on the basis of at least 100 fibers, preferably more, and a Kolmogorov-Smirnov statistical test would be the preferred method of comparison. Asbestos fiber populations are bivariate log normally distributed and such a test is the only real way to test population similarities. Unfortunately the length and width of each fiber found is needed for this type of test and the authors do not provide this data.

The size distributions are unusual in other ways in comparison to asbestos and other mineral samples. The geometric mean lengths are considerably longer than most other asbestos samples; most asbestos samples measured by SEM or TEM have mean lengths of the order of 2 - 3 μm (for all fibers longer than 1 μm), rather than the 3 – 10 μm found here. Aspect ratios for whole populations of asbestos fibers are normally in the range of 7 – 10 rather than the 20 – 30 range found in most of these samples, and never near the extraordinary geometric mean aspect ratios of 90 to 133 found for the anthophyllite of the mesothelioma cases and the chrysotile of the non-mesothelioma cases. Such dimensional departure from the normal asbestos shapes and sizes lead to doubt about the credibility of the measurements. Finally, the dimensions of the fibers are not consistent with those found for NY talc as described by Siegrist and Wylie (1980) and as such it is suggested that other sources of mineral dust may have contributed significantly to the lifetime lung burdens of both the mesothelioma and non-mesothelioma cases.

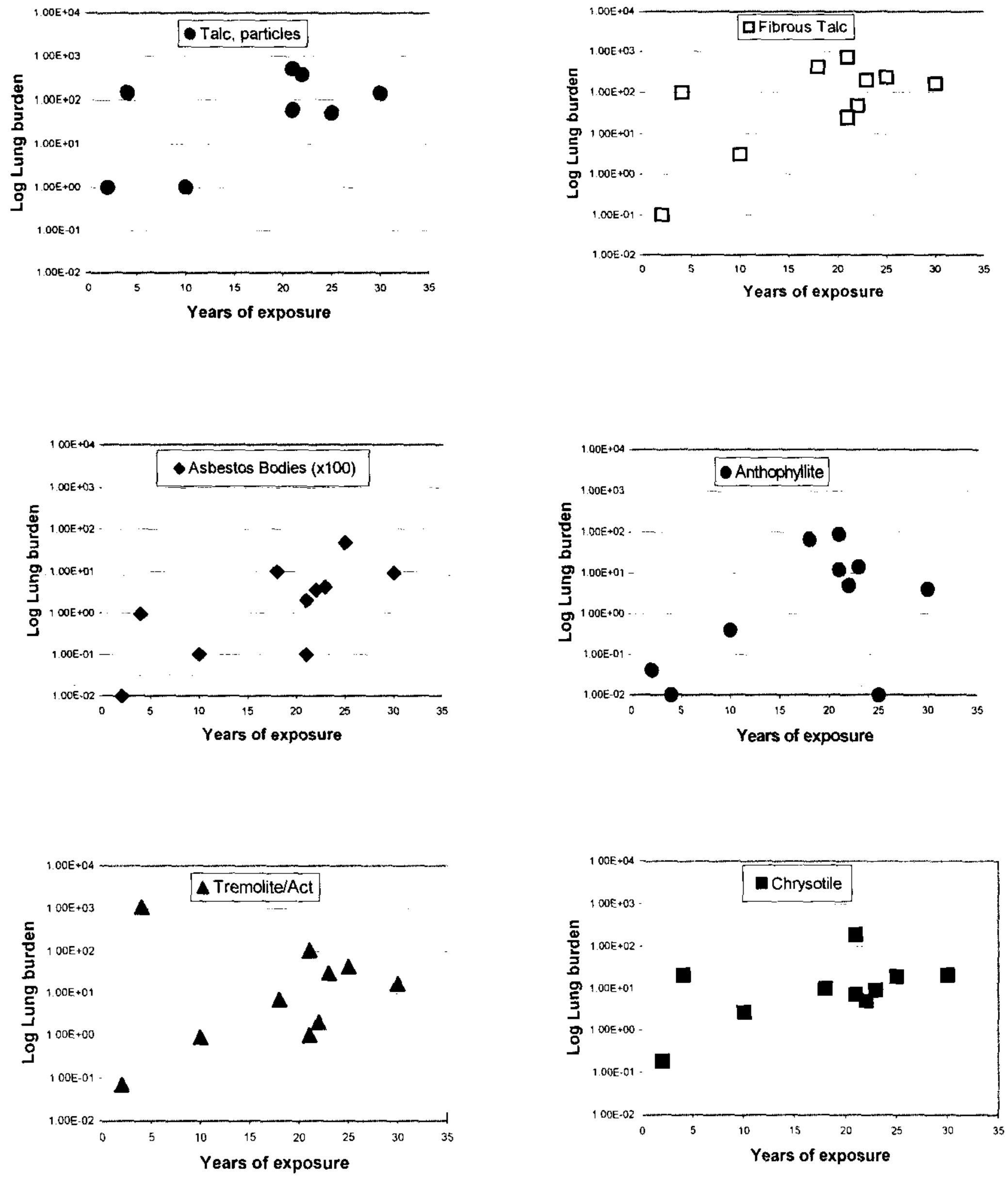
Lung burden and exposure

More evidence for this is found when the lung burdens are compared to the years of exposure in talc mining as shown in Figure 1.

It is apparent at first sight that whatever the relationship between lung burden and duration of employment is, it is not simple and it appears to differ between particle types and individuals. There is some general trend towards increasing lung burden with duration of employment but this is largely because the shortest duration burdens are , as expected, very low. Without this single point in each graph the trends are much less clear. Asbestos bodies and talc particles both appear to increase as expected with years of exposure. Tremolite fibers show no increasing trend; it is very low in the shortest exposure case and extremely high in the mesothelioma case (with the second lowest exposure period); for longer exposures it shows no increase at all. Anthophyllite is generally low, absent in two cases, including one of the mesothelioma cases, and very much higher in one case only. The same case also has the highest lung concentration of talc fibers and chrysotile as well as the second highest tremolite/actinolite.

Figure 1a -f. Showing the relationship between lung burdens of all types of particle and the duration of employment in the talc mines. The two mesothelioma cases are shown with blue symbols.

All of the data are from Table 3 of Hull et al (2002). The asbestos bodies are expressed as thousands per gram of dry lung (divided by 100 in the figure). The fibers are expressed as millions of fibers per gram of dry lung. The talc particles are expressed as millions per millilitre of lung.



An equally good case could be made for the tremolite/actinolite fibers being the result of environmental exposure; as shown in Figure 2 they appear to some extent to be a function of the age at death of the cases. Cases G and H, two of the oldest three cases at death, have very much lower lung burdens than any of the others and without any more information about their life or work histories it is difficult to draw any conclusions about their exposures. The remaining lung burdens do appear to show a progression with age.

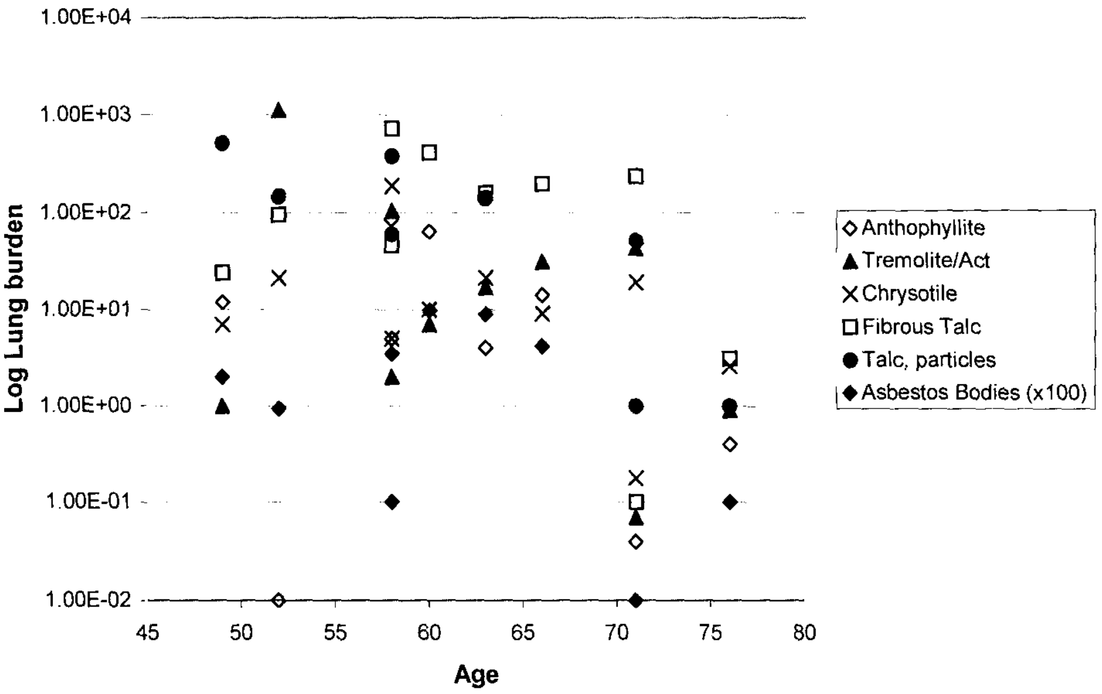


Figure 2. The relationship between lung burdens of all types of particles and the age of the case at death. (Same data as for Fig 1) Mesothelioma cases are shown in blue.

A different way of viewing the relative lung burdens with respect to duration of employment is to rank the lung burdens by assigning a value of 10 to the highest, 9 for the next highest and so on as shown in Table 3.

Table 3 Ranking of lung contents for each case; cases are ordered in terms of duration of work in the industry.

Case No	**G**	**M2**	**H**	**C**	**A**	**B**	**M1**	**E**	**F**	**D**
Actual Years	2	4	10	18	21	21	22	23	25	30
Rank years	1	2	3	4	5	5	7	8	9	10
Rank AB	1	4	2	9	5	2	6	7	10	8
Rank Anth	3	1	4	9	7	10	6	8	1	5
Rank T/A	1	10	2	5	3	9	4	7	8	6
Rank Chr	1	8	2	6	4	10	3	5	7	8
Rank Talc F	1	5	2	9	3	10	4	7	8	6
Rank Talc NF	1	8	1	Na	10	6	9	Na	5	7
Average Rank	1.3	6.0	2.2	7.6	5.3	7.8	5.3	6.9	6.5	6.7

Na – not analysed

If the only source of fibers was occupational exposure in the talc mining then a regular increase in rank should appear with age rank. Other than for cases G and H , the average ranks are scarcely different over the whole set, and indeed the highest ranks are found for the cases with intermediate duration of employment. While the conclusion that non-occupational exposures are significant is not possible, because there is no information about the severity of the exposures of each case, nevertheless the hypothesis that some other source of exposure must be playing a part is suggested and should not be excluded.

A similar approach can be taken with the order of concentration of the different fibrous minerals in each of the lung burdens as shown in Table 4.

Table 4 Ranking of fibrous minerals in the lung of each case. 1 is the lowest of the four minerals in the lung; 4 is the highest of the four minerals in the lung. All are the numbers of fibers per gram of dry lung as in Table 3 of Hull et al (2002)

	M 1	M 2	A	B	C	D	E	F	G	H	Average
Anthophyllite	2	1	3	1	3	1	2	1	1	1	1.6
Trem/actinolite	1	4	1	2	1	2	3	3	2	2	2.1
Chrysotile	2	2	2	3	2	3	1	2	4	3	2.4
Talc fibrous	4	3	4	4	4	4	4	4	3	4	3.8

If the four minerals were all the result of a consistent industrial exposure the ranking should be the same in each lung, and indeed three of the cases, B, D, and H have the same pattern of fiber burdens. However this pattern is not followed by any other cases and only two others, A and C have the same pattern. The talc fibers are the highest in 8 out of the ten cases and second highest in 2. Anthophyllite is the lowest in 6 out of ten, but second highest in 2. The tremolite and chrysotile are both very variable in their rankings, ranging from highest to lowest, and their average ranks are very similar. That the lung contents are inconsistent lends support to the hypothesis that at least some of the lung burdens are not related to occupational exposure in the talc mines. Other explanations for this could be that the miners worked at different mines with different mineralogy or that the mineralogy of the mines varied over time, leading to variation in the quality of the mineral exposures. There is no evidence for or against any of these propositions. Whatever the explanation the suggestion that the men all experienced the same mineral exposure is not strictly tenable.

It is speculative to suggest possible environmental exposures for any of the minerals found in the lung burdens. However, the geology of Northern New York State is complex, the Adirondaks being part of the Appalachian metamorphic belt, and outcrops of rocks bearing these minerals are probably not uncommon. So it is to be expected that many activities in agriculture or even gardening could result in environmental exposures that would not be negligible in the long term.

Conclusions

It is my opinion that the SEM analytical procedures used leave some doubt as to the validity of the mineral identifications in the paper. The fiber lung burdens can not be consistently derived from the observed fiber numbers given in the paper. The size distributions of the fibers lead to further questions about the quality of the data and do not support the conclusions drawn. The conclusion that the lung burdens represent a picture of a coherent pattern of occupational exposure is not justified by close examination of the lung burdens.

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August 2004

Appendix

It is difficult to check the validity of the final lung burden result given by Hull et al because the numbers of fibers found in the individual lung samples are not given, only the total numbers of fibers in each group that were used to calculate the geometric mean dimensions. However, if we assume that the fiber numbers given in Table 5 of Hull et al represent combinations of those used to calculate the lung burdens the mesothelioma cases at least can be checked for internal consistency.

The formula for calculation of lung burden from the numbers of fibers found during a systematic search of a sample prepared on a filter and examined by microscopy is usually given as follows: $F_{lung} = n \times \mathbf{V/v \times A/a \times 1/W}$

n – number of fibers found;
W - Dry weight of lung tissue digested, in grams;
V - total volume of solvent used in digestion of tissue, millilitre;
v - volume of aliquot filtered, millilitre;
A - Total area of filter deposit, square millimetres;
A - area of filter searched, square millimetres.

Irrespective of the exact values of these individual factors, for any one case, they can be grouped into a single constant which must be exactly the same for each of the fiber types in any one sample. This can be called x for Mesothelioma Case 1, and y for Mesothelioma Case 2. For any mineral the total number of fibers found in the search can be called T, and if the number found in Case 1 was n, then the number found in Case 2 was T – n. The lung burdens F1 and F2 calculated from these are

$F1 = n.x,$ (1)

$F2 = (T-n).y$ (2)

Equation 1 can be written to allow the term for n in equation (2) to be substituted as $n = F1/x$, giving:- $F2 = (T - F1/x).y$ (3)

This equation can be rewritten as $0 = F2.x - T.xy + F1.y$

This is a simple equation that contains only terms that are known or can be calculated from values provided by Hull et al.

The figures from Hull et al are:

Fiber Type	Mesothelioma Case 1 Lung burden F/g dwl	Mesothelioma Case 2 Lung burden F/g dwl	Number found (M1 + M2)
Anthophyllite	5	0	6
Tremolite/Act	2	1110	39
Chrysotile	5	21	5
Talc fibers	46	96	54

So the calculations must be as in the following table;

	Mesothelioma Case 1	Mesothelioma Case 2
Fiber Type	Lung burden,	Lung burden,
Anthophyllite	$5 = n_{ant*}\, x$	$0 = (6 - n_{ant})\, y$
Tremolite/act,	$2 = n_{/at}\, x$	$1110 = (39 - n_{t/a})\, y$
Chrysotile	$5 = n_{chr}\, x$	$21 = (5 - n_{chr})\, y$
Talc fibers	$46 = n_{talc}\, x$	$96 = (54 - n_{talc})\, y$

* n is the number of each fiber type found in the SEM search of Case 1; the number for Case 2 is the total number found minus the number for found in Case 1

The algebra used for equations 1, 2, and 3 above allows these to be recast as the following equations

For the Anthophyllite; $x = 5/6 = 0.833$

For the Tremolite/act, $1110x - 39xy + 3y = 0$

For the Chrysotile $21x - 5xy + 5y = 0$

For theTalc fibers $96x - 54xy + 46y = 0$

Case 2 contains no anthophyllite, so it is possible to calculate x from $F_{ant} = n_{ant}\, x$; 6 fibers found gives the result of 5 fibers per gram of lung tissue; therefore $x = 5/6$,

i.e. $x = 0.8333$.

Of course, because the analyst may have rounded numbers up or down after the calculation, the nmber 5 in the first of these equations could have been anything between 4.5 and 5.5 and the resulting value of x between 0.75 and 0.916.

Table 5 shows the best solutions for *x* and *y* in the equations for tremolite, talc and chrysotile found by iteration.

Table 5

	x	*y*
Tremolite.	0.915	30.15
Talc	0.905	30.3
Chrysotile	1.16	30.40

Theoretically these factors should be identical, and while those for tremolite and talc fibers are close, the factor for chrysotile in Case 1 can not be reconciled with the reported lung burdens and numbers of fibers found in the search.

It is possible to test these values by applying them back into the original equations to determine the actual numbers of fibers found in the Case 1, 'n'. For Case 2, 'n' will be the total numbers of fibers found minus Case 1 'n'. These should be whole or half integers if the factors *x* and *y* are correct.

	Case 1	Case 2
Anthophyllite	6.00	0
Tremolite	2.19	36.81
Chrysotile	4.31	0.69
Talc fibers	50.83	3.17

These are obviously not whole or half integers.

Unfortunately, no two constants have been found that can produce the results as given by Hull et al, for all of the four fiber types listed. The data do not appear to be internally consistent, and this raises serious questions about their validity.