



Research

MEMORANDUM

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Subject CRITIQUE OF ELECTRON MICROSCOPE MEASUREMENT OF AIRBORNE
ASBESTOS CONCENTRATIONS--A PROVISIONAL METHODOLOGY MANUAL
by A.V. Samudra, C.F. Harwood and J.D. Stockham, IIT Research
Institute, Chicago, Illinois. EPA 600/2-77-178, August 1977.
Project Officer, J. Wagman, Emissions Measurement and Charac-
terization Division, Environmental Sciences Research Laboratory,
Research Triangle Park, North Carolina

Summary

A review of the subject Provisional Methodology Manual for ambient air sampling indicates that the methods described are valid for monitoring commercial asbestos emissions from highly localized sources. However, we have serious reservations about the validity of the sample collection methods, particulate identification methods, data analysis, and the estimates of minimum detectable limits when monitoring ambient air quality or emissions from extended sources. Specific criticisms of a number of the proposed procedures are detailed and recommended changes presented.

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ELECTRON MICROSCOPE MEASUREMENT OF AIRBORNE ASBESTOS
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J. D. Stockham, IIT Research Institute, Chicago,
Illinois. EPA 600/2-77-178, August 1977. Project
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Summary

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Discussion

For several years much attention has been focused on possible health hazards resulting from exposure to particulates present in low concentrations in ambient air, food, or water. Despite the absence of direct medical data, concern for the public health has been aroused by certain mineralogical analyses and an extrapolation of established effects due to occupational exposure to commercial asbestos. In response to wide variations in the results of sample analyses reported in various laboratories the EPA awarded a contract to the IIT Research Institute in Chicago to develop methods of identifying and counting fibers as discussed in the Foreword to the report (reproduced below). Unfortunately, detailed examination of the Provisional Methodology Manual has revealed that many unanswered questions remain and that we have a number of specific objections to the proposed methods, detailed below. The format used is to reproduce the Manual's recommendation, comment on the recommendation, and, where appropriate, make an alternative recommendation.

Asbestos

Asbestos or asbestiform minerals include several types or groups of fibrous crystalline substances with special physical and electrical properties that have long encouraged their use in the manufacture of such products as roofing, insulation, brake linings, fireproof curtains, etc. Their presence as pollutants in the ambient air and in supplies of food and drinking water has caused considerable concern because occupational exposures to asbestos have been found to induce mesothelioma of the pleura and peritoneum, as well as cancer of the lung, esophagus, and stomach, after latent periods of about 10 to 40 years.

Electron microscopy is currently the principal method used to identify and characterize asbestos fibers in air and water samples. Because of the poor sensitivity and specificity of conventional bulk analytical methods, electron microscopy is also being used for routine measurement of asbestos concentrations. The accuracy of such analyses generally have been poor, however, and interlaboratory comparisons have not been satisfactory. Therefore, the EPA is currently conducting a study to develop a more reliable method for the measurement of asbestos concentrations.

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This manual describes a provisional optimum electron microscopical procedure for measuring the concentration of asbestos in air. It results from a study, carried out under EPA Contract No. 68-02-2251, to evaluate the various methods available in use in the various laboratories. Statistical techniques were used to evaluate the effects of the many interacting variables and arrive at an optimum composite procedure.

This manual does not provide the vast amount of data which supports the provisional methodology. These data are contained in the final report on EPA Contract No. 68-02-2251.

1.1. Foreword

The Foreword clearly indicates the methods described in the Provisional Methodology Manual were developed for the measurement of airborne asbestos. However, the definitions of asbestos identification methods described in the Manual effectively broaden the definition of asbestos from that given in the Foreword to include non-asbestos amphibole minerals and a large number of other minerals.

1.2. Asbestos-Fiber Definition

Record the length and breadth of all fibers that are at least twice of greater than 3:1 and have substantially rectangular sides. Observe the morphology of each fiber through the electron microscope and note whether a tubular structure characteristic of chrysotile asbestos is present. Switch into SAED mode and observe the diffraction pattern. Note whether the pattern is typical of chrysotile or amphibole, or whether it is typical of neither chrysotile nor amphibole.

1.3. Comments

This is the only section in which the term "fiber" is defined. The definition is apparently taken to be a particle with an aspect ratio of 3:1 and nearly parallel sides. The authors' choice of choosing a 3:1 aspect ratio as the lower limit for calling a particle an asbestos fiber is not clear; it could well have been 2:1, 5:1, 10:1, 20:1, or even 40:1. This last number is far more representative of the typical aspect ratios found when counting airborne commercial asbestos particles. The authors' definition, for example, would not count the fibers of asbestos chrysotile particles in the air which are typically over 100:1 aspect ratio. In a study

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of waterborne chrysotile, found the average aspect ratio to be 200:1.²⁾ In a recent round-robin study conducted by an ASTM committee, the average aspect ratio of chrysotile fibrils was on the order of 30:1.³⁾ Similar data have been developed in our laboratory for airborne amphibole particles. In one study, the aspect ratios of airborne amphibole particles fell into two well separated categories.⁴⁾ One group of amphibole particles generally had very high aspect ratios on the order of 40 or more to 1. These particles are often observed to have defect properties such as those described for amphibole asbestos by Hutchinson, and others.^{5,6,7)} The second group of particles having aspect ratios on the order of 5 to 7:1 were generally well crystallized and bear little resemblance to the type of faulted amphibole asbestos particle observed in commercial asbestos samples. Finally, the authors appear to be extending the definition of asbestos from that given in the Foreword to include not only chrysotile and amphibole asbestos, but all amphibole particles in all size ranges.

Given the significant number of studies which show no (or at least reduced) hazard from small particles,⁸⁾ this extension does not appear warranted. Hence, the value in adopting a method for the sole purpose of resolving submicrometer particles is debatable.

NBS Recommendation

If ambient asbestos levels are to be analyzed, the definition of fiber should be such that the particles counted are similar in aspect ratios, composition, and structure to commercial asbestos. Since it is pointed out in the Foreword that only occupational exposures to commercial asbestos minerals are known to induce biological response, it would seem prudent for any monitoring program to separate those amphibole particles with physical properties similar to commercial asbestos from those amphibole particles which would not be considered commercial asbestos.⁸⁾

EPA Recommendation-Sample Collection

(1) Take an air sample on a polycarbonate membrane filter, 0.4 μ m, using a high-volume or personal sampler.

NBS Comment

The use of a polycarbonate membrane filter to collect samples is regarded as undesirable. At the recent NBS Workshop held July 18 through 20, 1977, a number of

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objections to the use of polycarbonate filters for field sampling were raised. These objections were reinforced at a meeting held by the EPA at Research Triangle Park on November 1, 2, and 3, 1977, which was attended by Dr. Samudra, the author of the EPA manual. One of the problems with the polycarbonate filter for use in field sampling is collection efficiency; for example, Merchio, et al found that 39 percent of the chrysotile fibers less than or equal to 0.1 micron in diameter by 4 microns in length passed through an 0.8 μ m polycarbonate filter, and 15 percent of the chrysotile fibers less than 0.1 micron in diameter by 15 microns in length were passed. Their data did not show the extent of the variability of the collection efficiency for different filters of nominally the same diameter. Other factors which would seem to preclude the use of polycarbonate filters for field sampling are the difficulties in handling during transport and the potential loss of material from the filters during transport and sample preparation.

USS Recommendation

Until problems with particle retention and filter handling are solved, field collection of ambient air samples should be carried out using Millipore filters, with the material transferred to polycarbonate filters for laboratory analysis.

EPA Comment

Neither the morphology, nor the electron diffraction pattern, nor both, can give irrefutable proof that a given fiber is asbestos. Positive results from the tests indicate only that asbestiform fibers are present.

USS Comment

This again implies "asbestiform fibers" based on their aspect ratio and the general appearance of the selected area diffraction pattern. These criteria encompass a much larger group of minerals than those described as asbestos in the Foreword. As stated by the authors, the methods described in the Manual will not permit positive identification of a given particle. As a result, only in the case of highly-localized sources emitting asbestos can the methods described be considered an acceptable measure of the concentration of fibers. They are not acceptable for ambient air samples where many unknown minerals may be present.

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EPA Recommendation-Fiber Classification

The following rules should be followed when classifying a fiber:

- (a) Observe a fiber at a TEM screen magnification of about 20,000X through a binocular with a magnification of about 70X. At a screen magnification of 20,000X, the tubular structure of chrysotile asbestos is usually apparent (compare with standard specimens). Fibers showing the tubular structure may be classified as chrysotile asbestos with confidence. There are only rare exceptions; amphibole asbestos usually have a lath shape; but sometimes appear similar in form to chrysotile fibers without lumina.
- (b) Electron diffraction patterns from particles with fibrous morphology fall into distinct groups. Chrysotile asbestos has a characteristic streaked layer line through the central spot and also a triple set of double spots on the second layer line. Amphibole asbestos gives a layer pattern, generally with little or no streaking.
- (c) Transmission electron micrographs and selected area electron diffraction patterns obtained with standard samples should be used as guides to fiber identification [4,5].

USS Comment

In (a), the authors indicate that a tubular morphology is characteristic of chrysotile and no other minerals. However, as pointed out in Grimms' book on Clay Mineralogy, halosite is tubular and the possibility exists that some kaolinite may exist in a tubular structure.¹¹⁾ Evidence for elongate forms of illite, atapulgite, alophane, and montmorillonite with diameters $\sim 10^2$ - 10^3 Å was also discussed by Grimm. As Beaman has pointed out, further complications arise when the chrysotile has been subject to thermal or chemical treatments.¹²⁾ He finds the tubular structure is often not apparent when the chrysotile has undergone extensive physical processing. Beaman also reported that only about 10 percent of the single-fibril-chrysotile particles give good diffraction patterns, while about 40 percent can be recognized as crystalline. This presents a serious problem in identification as the very small particles which do not give well defined electron-diffraction patterns may be chrysotile or they may be some other mineral particle.

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...IN CONTROL OF TESTING
...TO DETERMINE ...
...STANDARD ...

In (b), the authors indicate that electron-diffraction patterns from particles with fibrous morphology fall into distinct groups. If their definition of fibrous is simply that of 3:1 particles, this statement is incorrect. As shown in Figures 1 through 3, taken from a paper by Lee, Lally and Fisher, which was presented at the NBS Asbestos Workshop, (3) a variety of diffraction patterns can be obtained for individual particles, and thus the existence or absence of a "layer pattern" is not sufficient to identify particles as amphibole or nonamphibole. Here we have taken the authors' meaning of "layer pattern" as any diffraction pattern having a row of spots, with a projected separation of $\sim 5\text{\AA}$. This is not the conventional use of the term "layer" pattern, a term normally reserved for the diffraction pattern from textured materials having an axis of azimuthal symmetry.

USS Recommendation

The identification of particles in ambient air samples is most efficiently accomplished by grouping the particles into classes on the basis of their X-ray spectra using either transmission electron microscopy (TEM) or scanning electron microscopy (SEM) equipped with an energy-dispersive spectrometer. The homogeneity of each class can be established by using quantitative selected area diffraction (SAD) techniques on randomly selected particles from each class. This will also permit the analyst to establish confidence levels on the certainty of identification for particles within each class.

EPA Recommendation-Fiber Classification

From the examination of the electron-diffraction patterns, fibers are classified as belonging to one of the following categories: chrysotile, amphibole, ambiguous, non-asbestos, unknown (no pattern).

It should be noted that other particles with fibrous morphology also give layer patterns; for example, hornblende. The complete quantitative indexing and deriving interplanar d-spacings from diffraction patterns is a time consuming and complex undertaking and is not feasible for routine analysis.

USS Comment

Identification by inspection is not reliable for multi-component samples. While the authors apparently do not intend to include hornblende in the amphibole-asbestos category, it is an amphibole mineral. Separation of this amphibole from

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other non-asbestos amphibole particles is very difficult with some types of elemental analysis.

It should be noted that USS Research was not consulted with respect to the quantitative interpretation of electron diffraction patterns or as to the suitability of our methods for routine analysis, even though these techniques have been presented at several meetings attended by IITRI personnel.

Finally, an abundance of data suggests that significant physical differences exist which differentiate the asbestos and non-asbestos amphibole varieties.^{5,6,7,12,13} A potential means for distinguishing these minerals was offered at the NBS Workshop. This was based on differing crystal habits reflected in the orientation of particles in the TEM. This difference and its effect on SAD patterns are illustrated in Figures 4 and 5. Further study in this area is required to determine whether these differences have general applicability in the identification of asbestos.

USS Recommendation

If electron optical methods are chosen, our methods or their equivalent should be used. Amphibole particles with very large aspect ratios ($>40:1$) should be separated from those with moderate aspect ratios ($<10:1$).

EPA Comment-Fiber Identification

It is not possible to inspect electron-diffraction patterns for some fibers even when their identity as asbestos fibers is known. There are several reasons for the absence of a pattern. These include contamination of the fiber, interference from nearby particles, too small a fiber, too thick a fiber, and non-suitable orientation of the fiber. Some chrysotile fibers are destroyed in the electron beam, resulting in patterns that fade away within seconds of being formed.

USS Comment

Here the authors describe numerous reasons for the absence of well defined diffraction patterns. While their statements here are in essence correct, they fail to give estimates of the percentage of particles of a given mineral type for which they are unable to obtain diffraction patterns. In a similar vein, there is no estimate of the percentage of misidentified particles; this becomes very important when analyzing ambient air samples or any sample in which a variety of mineral types can occur.

The absence of any examples of electron-diffraction patterns from chrysotile, amphibole, and other mineral particulates is particularly troublesome. If, indeed, the diffraction patterns from fibrous materials were diagnostic, any methodology making use of electron diffraction should include specific diffraction patterns to be used in identifying each mineral species.

EPA Recommendation-Particle Counting

Counting at Low Loading Level

When fewer than 50 fibers per grid opening are encountered, the preferred counting method is to scan the entire grid opening and define the full grid opening as one field. With the microscope magnification at 20,000X, a series of parallel scans across the grid square are made starting with the top corner of the square and ending at the bottom (see Figure 2(c)). (With the tilting section of the fluorescent screen used as a single field of view, approximately 300-400 fields will be observed if the entire grid opening is scanned.) Fibers noted in each full grid opening (or single field) are classified in accordance with the procedure described above.

Additional grid openings are selected, scanned, and counted until the total number of fibers counted exceeds 100, or a minimum of 10 grid openings have been scanned, whichever occurs first.

Counting at Medium Loading Level

When the loading on the filter is in the range of 50 to 300 fibers per grid opening, counting is done on randomly selected fields of view. At a screen magnification of 20,000X, fields are randomly selected within a grid opening until a total of 20 fibers have been counted, sized, and classified. (Generally 20-40 fields of view are observed per grid opening.) After about 20 fibers have been counted, another grid opening is selected and an additional 20 fibers (approx.) are counted. This procedure is repeated for 5 grid openings until a minimum of 100 fibers are counted. (When estimating fibers of a particular type of asbestos, counting is continued until 50-100 fibers of that type are counted.)

UCS Comment

The authors describe three levels of counting. The ranges are from 0 to 50 fibers, from 50 to 300, and greater

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than 300. In our experience, acquired while analyzing ambient-air samples for the purpose of developing satisfactory methods, we have found that the occurrence of 300 fibers per grid opening in an ambient-air sample which was not taken very near a point source is unlikely. In fact, the average number of particles per grid opening generally ranges from 0.1-10. These loadings are most suitable for analysis. When counting particles in the range from 0.1 particles per grid opening to 1 particle per grid opening, the use of 10 grid openings as a limiting value necessarily implies extremely poor accuracy in the resulting statistics. In addition, in this range, the background levels of 3:1 particles on the filters takes on an important role. In a study by Cook, et al, of Duluth tap water, they reported from one to five chrysotile and from 1-5 ambiguous particles per grid opening on nominally blank grids.¹⁴⁾ Computation of a concentration, therefore, based on the analysis of a very small number of particles, could suggest the existence of higher than ambient levels of chrysotile amphibole in the sample, when, in fact, the particles observed were on the original filter.

Another problem occurs when analyzing larger numbers of particles. The "stop after counting 100 fibers" rule will often dictate that the final field not be completely inspected, and unless the portion of the field inspected is used in the estimate of the area, the computed fiber density will be biased. Since the variance of the estimate is inversely proportional to the number of the fields sampled, the effect of this fixed-count sampling rule is to lower the precision of the estimates of large concentrations.

USS Recommendation

Counting particles using electron optical methods should aim at establishing the concentration at a predetermined minimum level of precision. The unit field to be counted should be adjusted so the expected number of fibers per field is between 1 and 10. A minimum should be set on the number of particles to be identified before an estimate of the concentration is made. Whatever the definition of field used, complete fields should be analyzed. If the data obtained does not satisfy the Poisson distribution, the analysis should be rejected.

As an alternative to the manual's stopping rule we would propose a technique called sequential testing. This technique was developed for use in quality-control situations where the number of samples to be taken is not predetermined. The approach is to try to classify the value of the parameter

of the population being sampled as belonging to one of two groups of values. In effect, two hypotheses are proposed and sampling takes place until one hypothesis is accepted. For example, let λ be the total number of fibers of a particular type on a filter. The two hypotheses proposed are

$$H_0: \lambda \leq \lambda_0$$

$$H_1: \lambda > \lambda_1$$

where $\lambda_0 < \lambda_1$.

Sequential sampling directs that one field at a time is sampled and a cumulative count of the fibers of interest is kept. After each field is sampled and counted tables are consulted; and if the total count is above a critical value, H_1 is accepted; if it is below another critical value, H_0 is accepted; and if the current total is between the two table values, sampling continues. For filter data λ_0 and λ_1 could be chosen to be symmetrically placed about the maximum allowable count of specific fibers on a filter. In this way the acceptance of H_0 or H_1 would signify whether or not certain air-quality standards are met.

EPA Recommendation-Precision of TEM Estimates

When more than one TEM grid is used, it is possible to obtain the mean values and 95 percent confidence levels on the means. This is done for each important parameter. The method consists of obtaining the mean, $\bar{x}$, the standard error of the mean, SEm , and t -value [6] (0.025, $n - 1$) for $n - 1$ degrees of freedom, where n = number of TEM grids examined and hence n replicates available. The 95 percent confidence limits are given by $\bar{x} \pm t \cdot (SEm)$.

USS Comment

The Manual's authors use the "T" statistic to estimate the precision of their mean TEM concentration levels. An underlying assumption of the "T" statistic is that the distribution of the sample mean concentration is normal, and a powerful statistical theorem, the Central Limit Theorem, proves that regardless of the underlying distribution of the data, as long as the data had a mean μ and a variance σ^2 , the distribution of the sample means calculated from that data is asymptotically normally distributed with parameters μ and σ^2/n , where n is the number of samples used to calculate each sample mean. (Ex.....)

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Asymptotically normally it is meant that the distribution becomes more closely normal as n gets larger.) Of course, if the underlying distribution of the samples is normal the distribution of sample means is exactly normal. If it is not, then for any fixed n , the distribution of sample means is only approximately normal, and hence the precision derived from the use of the "T" statistic is affected.

As shown by Merchio in 1972 the distribution of count data is often a Poisson with parameter λ_2 . This assumption can be confirmed by using contingency table tests which are given in many statistical methods books (Snedcor and Cochran's Statistical Methods, pp. 236-7). The test statistic for these tests is a calculated χ^2 value which can be compared to a table value. If the table value is not exceeded by the calculated one, the data can be assumed to have an underlying Poisson distribution. With this information the precision of the mean concentration estimates can be increased. An example of the application of the Poisson distribution is given in Tables I and II using the data presented in the manual (Tables D2-D5, p. 19). Table I shows the data for chrysotile fiber count. The null hypothesis was that data from both grids was distributed as a Poisson with parameter $\lambda_2 = 1.1625$. The calculated χ^2 value of 4.662 was less than the critical value of 9.490 for the $\alpha = 0.05$ level of significance; therefore the data could be assumed to be distributed as a Poisson. The estimate of concentration was then calculated along with the bounds for a 95 percent confidence interval. Note that this interval falls considerably inside the corresponding limits in the manual obtained using the "T" statistic. This procedure is repeated in Table II for all fibers with comparable results. If the data are not distributed as a Poisson, then the appeal to the Central Limit Theorem and estimates of the mean and confidence intervals calculated using "T" statistics can be made.

It should be noted that the half-fiber counting rule makes the basic unit of count in terms of a Poisson distribution a half fiber, and these data were analyzed on that basis. The half-fiber rule presents a problem since, when counting whole grid openings, presumably a long fiber could be counted as a half fiber several times. In addition, each time it is counted it must be moved to the center of the field to perform the selected area-diffracton analysis, which results in noncertainty in the repositioning of the sample.

2.1. Recommendation

We would suggest that the analysis should include investigation of the applicability of using the Poisson distribution. Estimates made from this distribution will yield improved reliability and evaluation of the concentration over those based on a "T" statistic.

2.2. Recommendation-Limits of Detection

The minimum detection limit of the electron microscope method for the enumeration of airborne asbestos fibers is variable and depends upon the amount of total extraneous particulate matter in the sample and the contamination level in the laboratory environment. This limit also depends on the sampling parameters, loading level, and the electron microscope parameters used.

In the provisional method proposed, 100 fields, each field with an area $0.18 \times 10^{-15} \text{ cm}^2$ are scanned. Assuming that a fiber count has an accuracy of ± 1 fiber then the detection limit is

$$\text{Detection Limit} = \frac{1}{100} \cdot \frac{\text{Area of Filter (cm}^2\text{)}}{0.18 \times 10^{-15} \text{ (cm}^2\text{)}} \\ \cdot \frac{1}{\text{Vol. of Air (m}^3\text{)}}$$

2.3. Comments

The authors suggest that the minimum detectable limit is one fiber per sample. However, the use of this number does not account for the probability of contamination, nor does it allow any estimate of the certainty of the limit. A more reasonable rule proposed by Rickards¹⁵⁾ is that the limit of detection be determined on the basis of a given number of fibers per unit field. However, we know of no satisfactory estimate for the limit of detection.

2.4. Recommendation

The number referred to by the authors as the minimum detectable limit is actually the unit change in concentration for the area analyzed. It is used as an upper bound on the detection when no fibers are detected in the area analyzed, and as a lower bound when a small number of fibers are detected,

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only a comparison of the number with the results of analysis of blank samples will give the analyst an indication of whether the concentration measured on the sample is significantly above background.

Conclusions

The methods and definitions proposed in the EPA Manual are not suitable for measuring the concentration of airborne asbestos without extensive modification. As presented, the techniques do not offer a clear advantage over the current optical methods except in the superior resolution of the electron microscope. Since adoption of the electron microscope as a routine monitoring device will increase the time and expense associated with such analyses, the quality of the data generated should reflect this expenditure and make full use of the information available about crystal structure and composition.

Detailed comments concerning many aspects of the provisional methodology manual have been made. In areas where it seemed appropriate specific suggestions for improved methods are described. In summary, we would suggest the following portions of the Manual or Method should be revised.

- A. Sample collection procedure.
- B. Counting rules.
- C. Particle identification.
- D. Estimate of precision.
- E. Estimate of the minimum detectable limit.

We would recommend that particle counting using electron optical methods incorporate the following requirements:

- 1) The establishment of concentrations at a predetermined precision and level of confidence; or the use of sequential testing procedures which permit upper or lower bounds to be placed on the concentration.
- 2) The estimate of uncertainty in the identification of particles.
- 3) A minimum number of particles to be identified for a given species before an estimate of the concentration is made.

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4) A minimum size and aspect ratio for particles
consistent with physical data on asbestos, and reliable identifi-
cation using electron optical methods.

5) Selection of field size based on the requirement
of 1-10 particles per field and analysis of data on the basis
of Poisson Statistics.

6) Rejection of samples not satisfying Poisson
distribution.

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TABLE 1
CHRYNOTILE FIBERS ⁺

Sample 1

<u>Field</u>	<u>Fiber Count</u> <u>Grid 1</u>	<u>Fiber Count</u> <u>Grid 2</u>
1	1	1
2	4	.5
3	3	1
4	1	2
5	.5	1
6	2	1
7	1	1
8	0	0
9	.5	1
10	1.5	.5
11	2	1
12	1	1.5
13	1	.5
14	1	1
15	1	1.5
16	0	.5
17	1.5	1
18	2.5	.5
19	1	1
20	1	2.5
Total	26.5	20.0

	<u>Average</u>	<u>Std. Dev.</u>	<u>Variance</u>	<u>Est. Conc. ($10^6/m^3$)</u>
Grid 1	1.325	0.977	0.955	$292.4 \times 10^6/m^3$
Grid 2	1.000	0.502	0.250	$242.4 \times 10^6/m^3$
Combined	1.1625	0.804	0.646	$268.8 \times 10^6/m^3$
Transformed*	2.325	1.607	2.584	

Has: Combined data is distributed Poisson ($\lambda = 1.1625$)

Method: Use contingency table "goodness-of-fit" test

$\chi^2 = 4.002$ - cannot reject H_0 at $\alpha = 0.05$ ($\chi^2_{.05} = 2.401$)

Confidence interval for "combined concentration": $2\alpha = 0.05$

Upper limit: 357.61×10^6 particles/ m^3

Estimate: 268.8×10^6 " "

Lower limit: 190.82×10^6 " "

* See Table 1, from Table 1-2, 9 of the EPA
Method 100.1 Manual.

* Count taken as half-fiber.

TABLE II

ALL FIBERS⁺

Sample 1

Field	Fiber Count Grid 1	Fiber Count Grid 2
1	2	4
2	6	2.5
3	5	3
4	3	2
5	2.5	2
6	2	3
7	1	1
8	1	1
9	1.5	2
10	3.5	2.5
11	3.5	3
12	3	2.5
13	2	1.5
14	1	2
15	4	4.5
16	2	2.5
17	3.5	3
18	3.5	.5
19	3	2
20	3.5	2.5
Total	58.5	47.0

	Average	Std. Dev.	Variance	Est. Conc. (9.2m^3 Air)
Grid 1	2.925	1.321	1.744	$623.5 \times 10^6/\text{m}^3$
Grid 2	2.350	0.961	0.924	$570.5 \times 10^6/\text{m}^3$
Combined	2.588	1.165	1.357	$598.3 \times 10^6/\text{m}^3$
Transformation*	5.175	2.330	5.427	

* Combined data is distributed Poisson ($\lambda = 2.588$)

$\chi^2 = 7.143$ (D.F. = $n = 5$) cannot reject at $\alpha = 0.05$ ($\chi^2 = 11.1$)

95% Confidence Interval for "combined concentration": $2\alpha = 0.05$

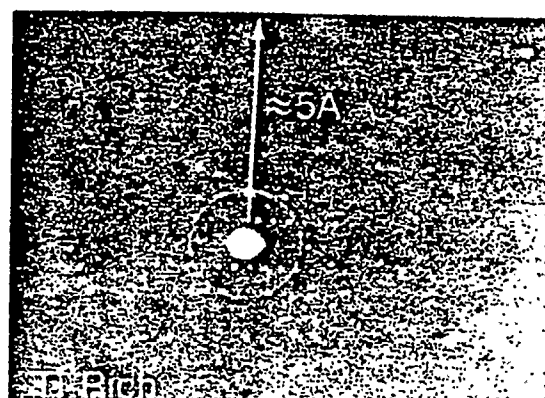
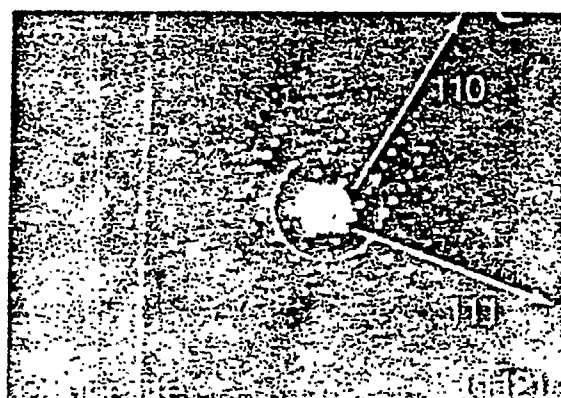
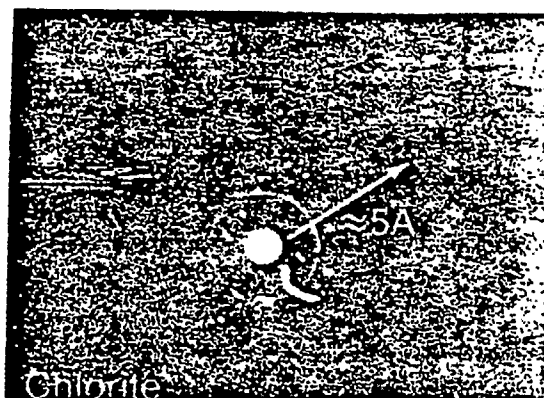
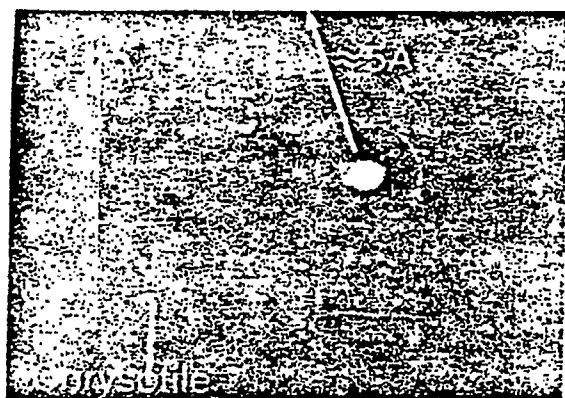
Upper limit: 716.91×10^6 particles/ m^3

Lower limit: 594.11×10^6 " "

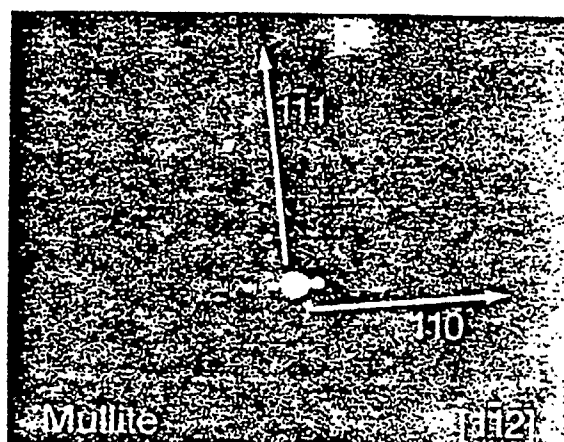
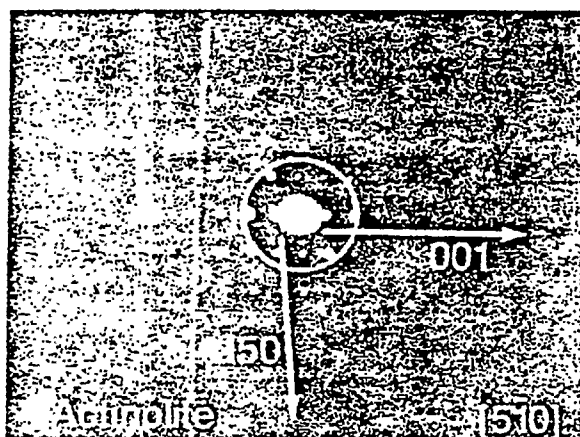
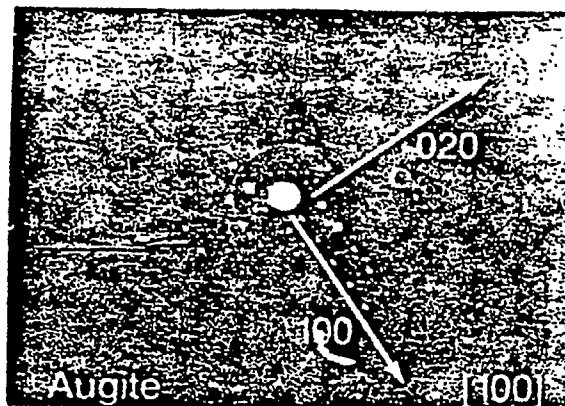
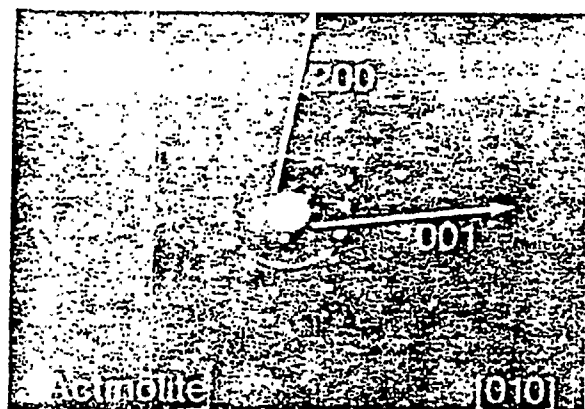
Lower limit: 493.74×10^6 " "

* Transformation obtained from Table 1-3.9 of the EPA
Federal Methodology Manual.

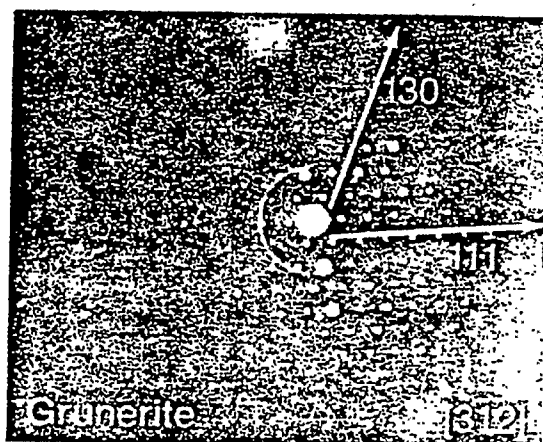
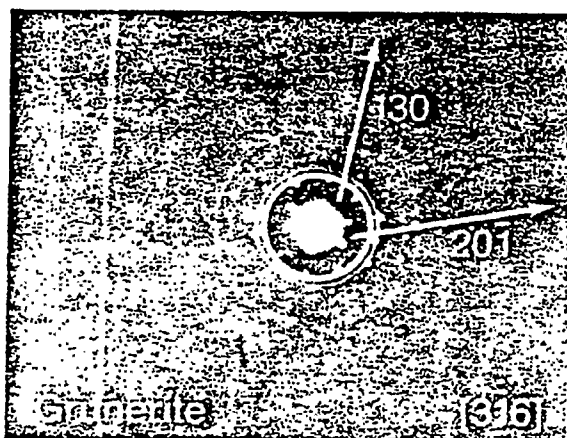
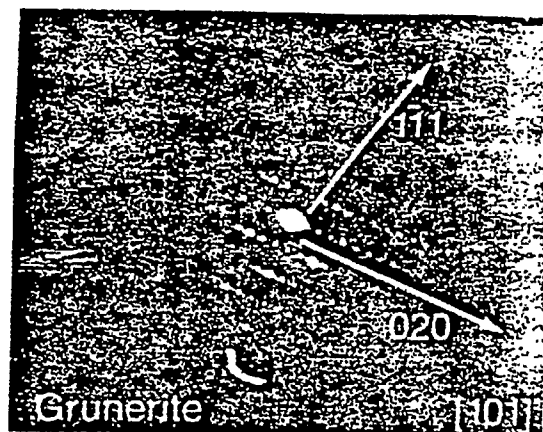
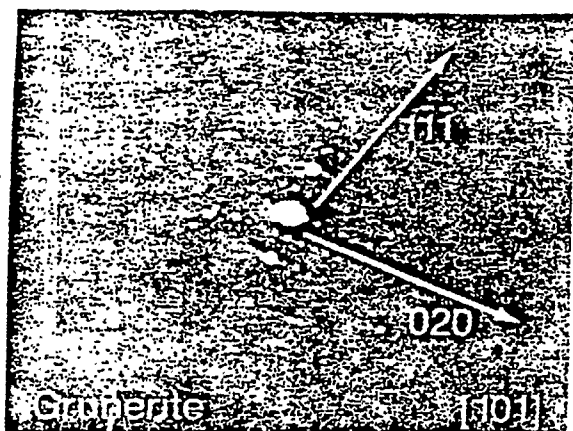
* Count taken as half-fiber.



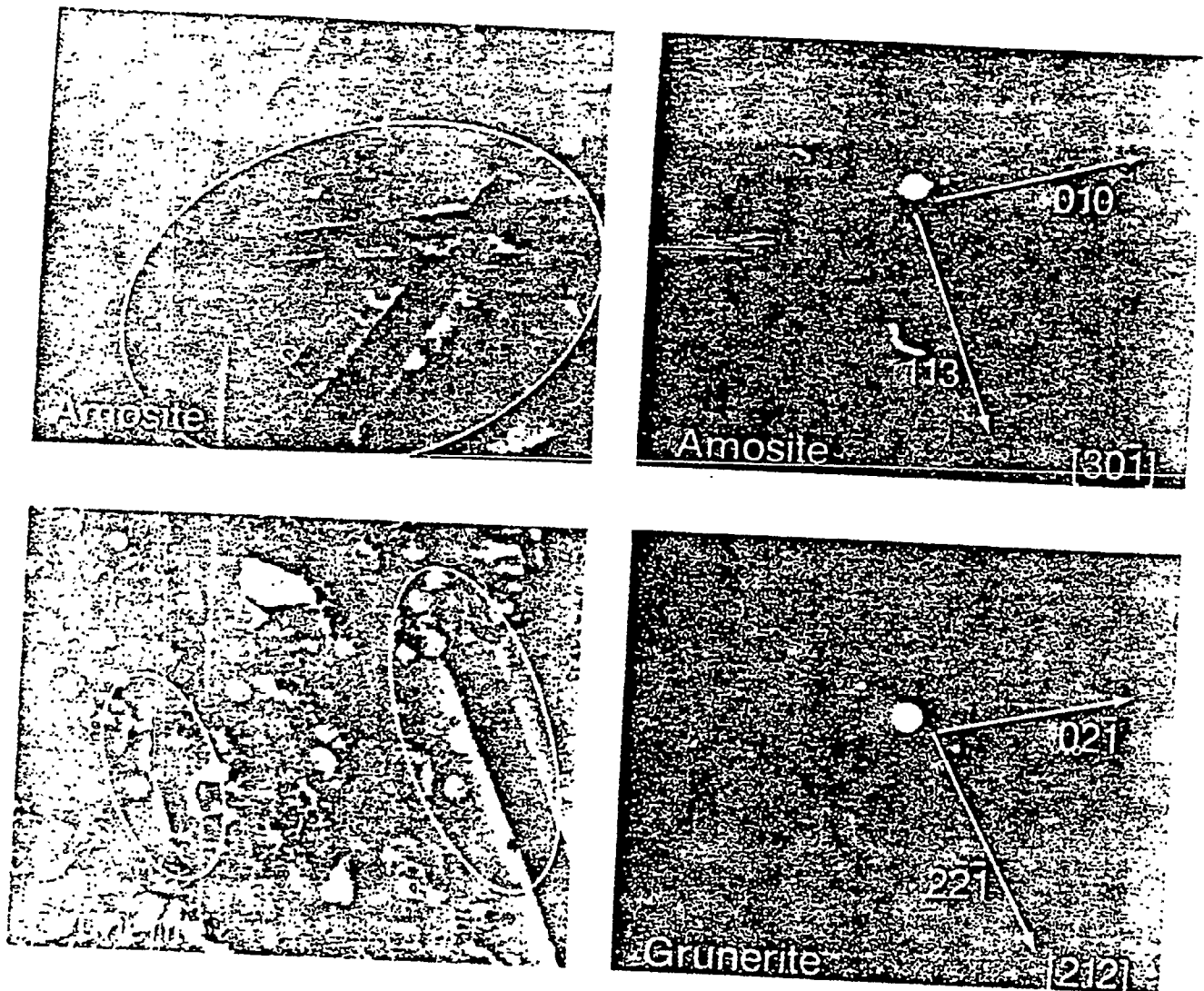
Selected area electron diffraction patterns of mineral particles which all show a d -spacing of about 5 Å.



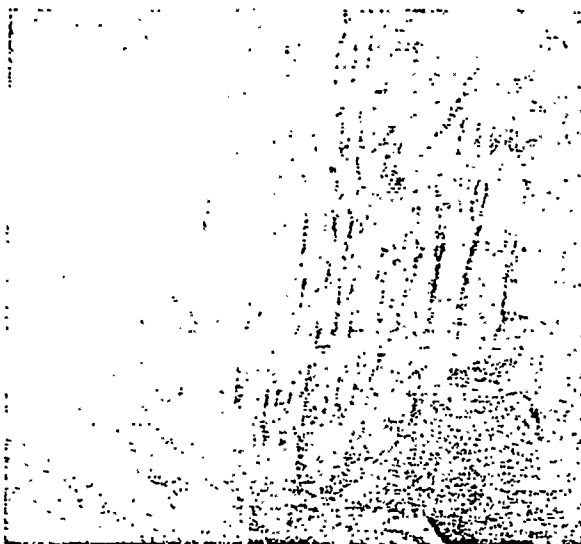
Indexed electron diffraction patterns of representative amphiboles and non amphiboles showing essentially similar appearance.



Indexed electron diffraction patterns of different orientations of grunerite achieved by tilting specimens in the electron microscope.



Electron micrographs and diffraction patterns of amosite and grunerite particles, which indicate that the asbestos and non-asbestos varieties can be distinguished if the diffraction patterns are interpreted properly.



a

b

10,000X

Grain-sections of grunerite and amosite looking down the c-axis.
 (a) Parallel, cleavage traces and (100) parting are all
 in evidence.
 (b) The formation of thin lamellae produced by fine-scale
 twinning (100), along (100) is shown. This leads to the
 development of amosite particles with large (100) faces.