

Improved Techniques of Identification and Determination of Airborne Asbestos

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Collection, identification and data analyzing techniques are presented for airborne mineral fibers. The limitations of the techniques are discussed. It is shown that the adaption of a plenum 1¾ inches long for sample collection is important since it leads to a much more uniform particle collection and distribution. Separation of the asbestos particles from other solid airborne pollutants with the refractive index liquid method is shown to be accurate, convenient and useful in distinguishing asbestos from other fibrous material. For analysis of this type the image analyzing microscope is a very valuable tool.

Introduction

THE NEED TO EVALUATE THE HAZARDS from asbestos inhalation was recognized as early as 1906 but it was more than twenty years before a detailed description of asbestosis was developed. Since then, it has been documented by numerous studies that inhalation of high concentrations of asbestos or any prolonged exposure to moderate asbestos dust level can in many instances lead to the development of asbestos connected diseases of the lung.¹

In general, the available data and information are only pertinent to the high concentrations caused by occupational exposure, and not to the low asbestos fiber content found as background in air. The health problem as posed by the biologically active asbestos fibers has led to the establishment of occupational safety standards.² But, little has been done to develop reliable sample collection, data generation, and identification techniques. This paper deals with these three aspects of analyzing for airborne asbestos particles.

Sampling

Sample collection is the most important step in any analysis for air pollution. Therefore, one must be sure that the collection

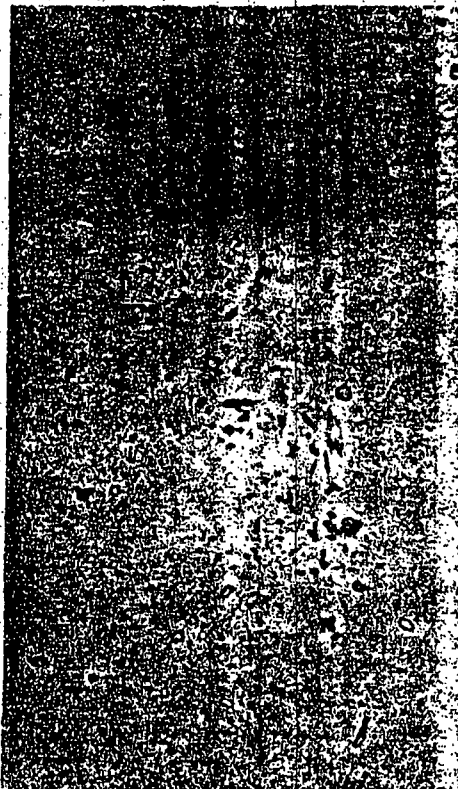


Figure 1. Non-uniform particle distribution of asbestos fibers and dust, collected with an open-face filter holder and viewed at 500X, phase contrast. (Sample A).

technique used gives an unbiased representation of those particles which are present. For this study samples were collected by the Ford Motor Industrial Hygiene Unit. Two sample collection methods were used, the approved NIOSH (National Institute for Occupational Safety and Health) and a modified NIOSH technique. All samples were simultaneously obtained at a manufacturing facility of the Ford Motor Company.

Approved NIOSH Technique

The first group of samples were taken in the mixing room of the plant using the technique approved by NIOSH which is to use a short, open-face filter holder with a 37 mm, type AA, millipore filter supported on a base pad. Two samples were collected with this device using a 2-liters/minute air sampling flow rate for 48 minutes per sample (a total of 96 liters of air was sampled). Sample A was collected in the casting room and Sample B in the mixing room. Micrographs showing the particle distribution of Samples A and B were counted with the NIOSH approved microscopy technique (phase contrast) and are shown in Figures 1 and 2. Various areas of Samples A and B were counted, and the data is presented in Table I. Samples A and B showed that dividing the entire filters into three equal parts and counting each segment separately yielded three different results on each filter. The fiber count for fibers equal to or greater than 5 μm was 5.2 fibers/ml of air sampled in one area of sample A, while the fiber count

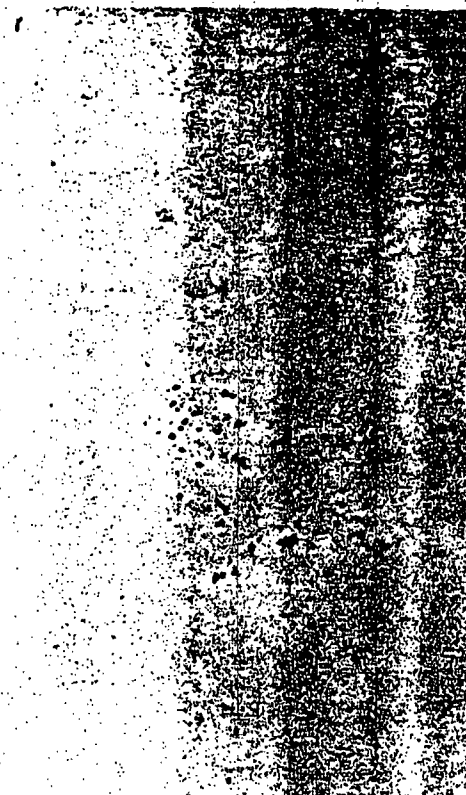


Figure 2. Phase contrast micrograph at 500X, showing the unevenly distributed asbestos-dust particles as collected with an open-face filter holder. (Sample B).

was 2.5 and 4.1 fibers/ml in the other randomly selected areas.

The values obtained for particles equal to or greater than 5 μm for Sample B were 1.1 fibers/ml in the first segment, 3.5 fibers/ml

TABLE I
Particle Size Analysis of Samples A and B

Sample	A	B
Where Obtained	Casting Room	Mixing Room
Plenum Length	Short, Open Face	Short, Open Face
Technique Used	NIOSH Standard	NIOSH Standard
Total Air Volume Sampled	96 liters	96 liters
Analyzed Filter Segments	3	3
Segment Number 1	5.2 f/ml*	1.1 f/ml
Segment Number 2	2.5 f/ml	3.5 f/ml
Segment Number 3	4.1 f/ml	6.8 f/ml

*Fiber count per ml; fibers 5 μm or greater.

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in the second, and 6.8 fibers/ml in the third section. It can be seen from Figures 1 and 2 and the data in Table I that the particles are not uniformly distributed on the millipore filters and, therefore, these samples cannot be assumed to be representative of the sampled working area. For this reason it is almost impossible to avoid a biased area selection for the particle counting. From these typical results, it is deduced that the samples collected with an open-face filter holder do not present a reliable or reproducible base for the analysis. Other aspects of reliability of this method will be discussed later.

Modified NIOSH Technique

The collection method for the second group of samples was modified so that the filter holders were fitted with a plenum $1\frac{3}{4}$ inches high over the base of the filters. The air inlet on the top of the plenum was the standard size 0.150 inch opening and all other parameters were kept the same. Figure 3 shows this modified sample collector with the $1\frac{3}{4}$ -inch long plenum, and the $\frac{7}{8}$ -inch long lower chamber behind the filter assembly.

Preliminary calculations predicted that the $1\frac{3}{4}$ -inch long plenum with the $\frac{7}{8}$ -inch long

lower chamber would create a condition that would satisfy the desired air flow properties of this small system. Not only does the plenum lengthen the particle path but it creates a low grade air turbulence which helps to uniformly distribute particles over the entire filter surface. The air stream is accelerated (pressure higher) through the orifice (air inlet) but behind the orifice inside the plenum the pressure drops immediately and the air flow decelerates. This diminution of the air velocity is the result of the sudden increase in the cross sectional dimension of the plenum. The particles, upon entering the plenum, become turbulent but the turbulence decays within a distance of 2 to 4R of the plenum. Uniform collection over the entire filter areas is accomplished by placing the filter assembly at least a distance $d = 2R$ upstream⁴ from the air inlet where R and d are respectively the radius and the diameter of the plenum.

The elevated orifice pressure with the consequently higher air velocity is necessary to overcome the gravitational force of the larger particles in order to collect them on the filter. Therefore, representative collection of the large particles with the "open-face" filter is not reliable. Translating into practical terms, the complicated aeromechanics of the modified sample collector, the following consideration must be made in order to design a properly functioning system:

The filter assembly should be located at a distance that is at least one plenum diameter from the air inlet, and one plenum radius from the air outlet. The sample collection improvement with this modified collector is twofold:

- (1) uniform collection over the whole filter area, and
- (2) representative collection of large particles.

Experience has shown that a sampling rate of 2 liters per minute for either 48 minutes as was the case for Sample C, or the 38 minutes collection time used for Sample

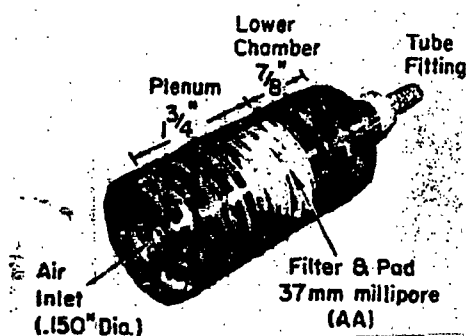


Figure 3. Overall view of modified sample collector.

TABLE II
Particle Size Analysis of Samples C and D

Sample	C	D
Where Obtained	Casting Room	Mixing Room
Plenum Length	13 $\frac{1}{4}$ inches long	13 $\frac{1}{4}$ inches long
Technique Used	Modified	Modified
Total Air Volume Sampled	96 Liters	76 liters
Analyzed Filter Segments	3	3
Segment Number 1	5.6 f/ml*	10.5 f/ml
Segment Number 2	4.9 f/ml	11.2 f/ml
Segment Number 3	5.8 f/ml	9.8 f/ml

*Fiber count per ml; fibers 5 μ m or greater.

D will produce an adequate, moderately dense and very uniform particle distribution, as shown in Figures 4 and 5 and in Table II.

An analysis corresponding to that used in Sample A and B was made on these speci-

mens. It was found that the particle count of these samples with the evaluation method as outlined by the NIOSH, yielded results with much less scatter between the filter segments.

An evaluation of Sample C taken in the



Figure 4. Uniform distribution of asbestos fibers and dust particles as collected with the modified filter holder (13 $\frac{1}{4}$ " long plenum) and viewed at 500X, phase contrast. (Sample C).

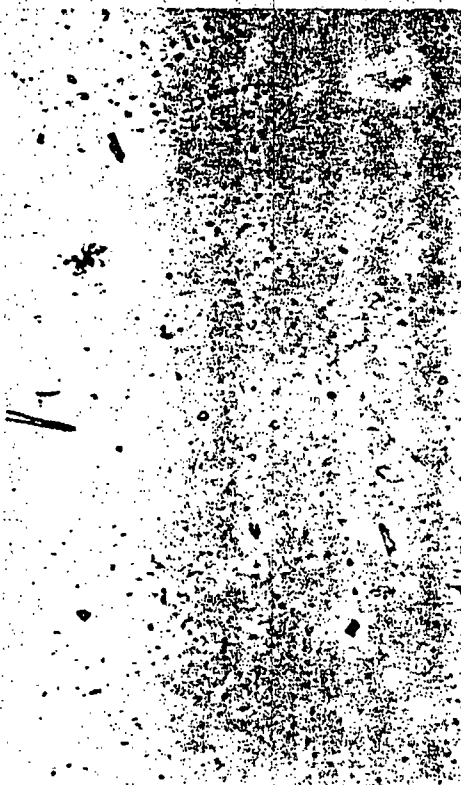


Figure 5. Even distribution of asbestos and non-asbestos particles as collected with the modified filter holder (13 $\frac{1}{4}$ " long plenum) and viewed at 500X, phase contrast. (Sample D).

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casting room, revealed that there were 5.6 fibers/ml present for fibers 5 μ m or greater in length in the first segment of the filter while 4.9 fibers/ml were found in the second and 5.8 fibers/ml were counted in the third segment. Sample D, taken in the mixing room, shows an equally good distribution between the three segments of the filter. In the first filter segment 10.5 fibers/ml were counted for fibers 5 μ m or longer in length, while 11.2 fibers/ml were found in the second, and 9.8 fibers/ml were found in the third segment.

Comparing the results of the analysis of Sample C with Sample A, the superiority of the long plenum collection method is apparent. The difference between the high and the low fiber count of Sample C is only 0.9 fiber, while Sample A shows a three times higher discrepancy or 2.7 fibers. The relationship between Sample D and B is even worse. Sample D shows only a 1.4 fiber count difference between the individual filter segments, while a 5.7 fiber count difference exists between the three filter segments of Sample B. The fiber count discrepancy of Sample B is slightly more than four times that of Sample D.

The previously described modified sample collector with the 0.150-inch size air inlet was found acceptable for all other flow rates as specified by the NIOSH standards. Since the recommended flow rate of air is between 1 and 2.5 liters per minute and the calculations for the lengthened plenum were made for 2 liters per minute of air flow, no difference is expected at either end of the flow rate range. It should be pointed out that the uniform distribution of heavier particles as seen here (*i.e.* > 5 μ m) insures the uniform distribution of smaller particles (*i.e.* < 5 μ m).

Sample Preparation for Identification

Since the index of refraction (ND) of most asbestos fibers is near to the refractive index of the chemically cleared membrane filter, it becomes necessary to eliminate the pres-

ence of the filter. This can best be accomplished using a low temperature asher. This method uses a very slow oxidizing process, that will neither harm nor disturb even the finest particles of asbestos or other non-oxidizable material. This instrument allows the oxidation reaction to occur at a low temperature by converting approximately 20% of the molecular oxygen to atomic oxygen⁵ with the use of a radio frequency field. The atomic oxygen reacts with the oxidizable materials in the specimen chamber. A mechanical vacuum pump then removes the volatile oxidation products. This method also eliminates many other oxidizable airborne particles commonly found in industrial operations. Approximately four hours are required to "ash" the filter. Samples C and D were prepared by this technique. The pressure of the specimen chamber was maintained at a pressure of 0.6 mm of mercury and 300 watts of power was used. The flow rate of the oxygen was 150 cc/minute.

Identification and Measurements of Asbestos Fibers

An extremely convenient and easy method of measuring particles is with an image analyzing microscope (IAM). With the aid of an IAM it is not difficult to measure 50 to 100 fields in a relatively short time. The particles can be bracketed in various size groups, and the measuring of a previously mapped out area can be accomplished. Data collected from 50 fields requires approximately 15 minutes.

In order to determine the number and size of asbestos particles present on a filter, using the IAM, a multi-process procedure is used. The specimen is first ashed (as has been outlined) to eliminate the filter and any oxidizable particles such as lint. The area (or areas) of interest are mapped out, measured, and all the particles present are counted. This will give the total number of particles (asbestos and non-asbestos). A second measurement of the exact same area is

made after a refractive index liquid having the same refractive index as chrysotile⁶ is placed on the sample. This has the effect of making all the asbestos particles invisible and allows us to determine the number of non-asbestos particles present in the sample. By this means, an accurate measure of only the asbestos particles present can be made.

Some care must be exercised when using the refractive index liquid, since the threshold sensitivity of the instrument's detection system for proper functioning requires approximately 10% difference in contrast. Minute shade variations due to the close refractive index similarities cannot be detected. Consequently, the absolute matching of the various refractive indexes among the different asbestos types is not necessary. For this experiment a refractive index liquid (ND = 1.546) was used that very well coincides with the index of refraction of chrysotile (the most widely used form of asbestos for industrial application) and also acceptable for serpentine since the contrast produced is still a shade below the detection limit of the instrument. On the average any perceptible shade difference produced by a refractive index difference of ND = 0.010 is acceptable to make proper measurements. It is also recognized that substances other than asbestos with refractive indexes within this range will be measured as asbestos. This source of error cannot be totally eliminated from knowing the total material involvement of the locality to be tested, and the type of asbestos used, the proper choice of the refractive index liquid will minimize this problem. The refractive index liquid used for this study is available from the Cargille Corporation. It contains aliphatic, halogenated and chlorinated hydrocarbons.

Correlation of the data is very simple. Since the measurements accumulated in the first part of the analysis encompass all particles present, the calculated values do not reflect accurately the presence of the asbestos plus alone. Separating the asbestos particles from all other detectable features by

suppressing their visibility with the refractive index liquid in the second portion of the analysis enables the data accumulation of the non-asbestos content of the sample.

Sample C and D were analyzed with the IAM. Calculations for the asbestos fiber content of the sample were carried out using the formulae published in the appendix of the "Criteria Document."²

$$\frac{\text{Filter Area}}{\text{Field Area}} = K \quad (1)$$

$$\frac{\text{Average Net Count} \times K}{\text{Air Volume Sampled}} = \text{Fibers/ml} \quad (2)$$

Measurements of Sample C indicated that only 0.2% of the particles were non-asbestos, therefore, the sample was viewed as being 100% asbestos. The particles were measured in six different size groups that ranged from 2 μm to 15 μm . Only 163 particles or 12.7% were counted as being 5 μm or larger in the total particle population of 1,280. Most particles were found in the 2 μm to 3 μm range and the calculated average particle size was 2.9 μm . The total fiber count of all sizes was 5.6 fibers per milliliter, and 12.7% of this or 0.6 fiber/ml were 5 μm or larger.

The analysis of Sample D was carried out identically to Sample C. Micrographs of Sample D are shown in Figures 6 and 7 before and after application of the refractive index liquid. It was found that Sample D contained 31.9% of non-asbestos particles. There was a total of 53.8 particles counted per milliliter, but the 31.9% or 16.8 particles of non-asbestos origin reduced this count to 37 fibers/ml. The size distribution measurements indicated that 3.3% of the particles were 5 μm or larger. Consequently, for these sizes the fiber count per milliliter was 1.1. The largest number of particles were again found to be in the 2 μm to 3 μm group, and the average particle size measured 2.3 μm . These data are presented in Table III.

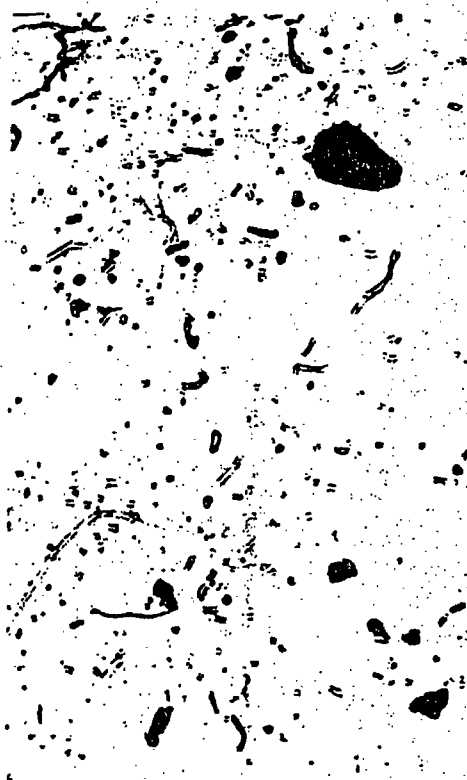


Figure 6. Asbestos and non-asbestos particles before the application of refractive index liquid. 500X. (Sample D).

TABLE III
Data from IAM Analysis

Sample	C	D
% of Non-Asbestos	0	31.9
% of Particles 5 μ m or >	12.7	3.3
Fiber Count. ml 5 μ m or >	0.6	1.1
Average Particle Size	2.9 μ m	2.3 μ m

Discussion

The present use of the "total fiber count" system as recommended by the NIOSH for overall evaluation of asbestos concentrations coupled with the "open-face" filter collection technique can be a source of error in the measurement of exposure to asbestos. The low number of total particle count and an approximately 1/9000 analyzed area of the



Figure 7. Same area as shown in Figure 6 after application of the refractive index liquid. 500X. (Sample D).

total filter surface (as specified by NIOSH) along with the non-uniform particle distribution cannot produce a reliable analysis. The presence of asbestos resembling fibers also adds to the problems.

The accuracy of the IAM method relies on two important facts, the large number of particles counted, and the large number of areas covered. It is also important that within these areas *all* particles are counted. These factors will minimize the uncontrollable errors. There is no correlation between the results obtained by IAM and the microscope counts of Samples C and D (Tables II and III). This is due to the fact that Sample D is composed to almost one-third of non-asbestos particles.

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Acknowledgements

The author is grateful to Dr. E. Eichen for the critical review of the manuscript, and to Dr. C. D. Melvin for the fruitful discussions. I also wish to thank Mr. J. W. Sprys and Mr. R. C. McCune for their help with the sample preparation using the low temperature asher, and Mr. H. B. Lick of the Industrial Hygiene Unit for fabricating the sample holder and collecting the samples.

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Received July 26, 1972

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