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Exposure to asbestos (1332214) fibers at Dulles International Airport (SIC-4582) and at Washington National Airport were measured from January 15 to 17 1979. The survey was requested by the Federal Air Surgeon. Bulk samples of ceiling material, floor sweeping and direct insulation were analyzed for presence of asbestos fibers. Twenty five general area air samples were taken at Dulles airport and 12 at Washington National Airport. The ceiling material at Dulles contained 40 to 50 percent chrysotile (12001295) asbestos. Material at National contained 1 to 2 percent chrysotile-asbestos. No asbestos fibers in bulk samples of sweepings and no detectable concentrations of airborne asbestos fibers were found at either Dulles or National. One ventilation duct contained 40 percent chrysotile amosite (12172735) asbestos. The authors conclude there is no health hazard from exposure to asbestos fibers at either airport. They recommend monitoring air and dust at Dulles and dust at National to detect future deterioration of the ceilings. The asbestos containing duct wrapping should be covered and consideration be given to replacement.

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HAZARD EVALUATIONS AND TECHNICAL ASSISTANCE  
REPORT NO. TA 79-15

DULLES INTERNATIONAL AIRPORT  
WASHINGTON NATIONAL AIRPORT  
WASHINGTON, D.C.

April 1979

Study Requested By:	Federal Air Surgeon Federal Aviation Administration
NIOSH Project Officer:	Richard W. Gorman Industrial Hygienist Hazard Evaluations and Technical Assistance Branch Cincinnati, Ohio
Study Dates:	January 15-17, 1979



## I. SUMMARY

Atmospheric sampling was conducted to determine possible exposure to asbestos (present in ceiling material) on January 16-17, 1979 in the main terminal of the Dulles International Airport and in those areas of Washington National Airport where the ceiling material was suspected of containing asbestos.

Analysis of bulk samples of the ceiling material confirmed that the ceiling at Dulles contained approximately 40-50% chrysotile asbestos and the ceiling evaluated at Washington National contained approximately 1-2% chrysotile asbestos.

A total of 37 air samples were taken of which 25 were from the Dulles terminal and 12 were from the Washington National Airport. No detectable airborne levels of asbestos fibers were found at either airport; therefore, there is no health hazard from asbestos exposure at the present time.

## II. INTRODUCTION

NIOSH received an emergency telephone request from the Federal Aviation Administration (FAA) on January 10, 1979. The request was for assistance in evaluating employee exposure to asbestos fibers at Dulles International Airport, Washington, D.C. Recent adverse publicity in the press and on television alleged that asbestos falling from the ceiling has caused health problems, including an operation on the vocal chords of an airline employee. A written request for assistance from the Federal Air Surgeon was received on January 15, 1979. The scope of the evaluation was later expanded to include certain areas of Washington National Airport where the ceiling was suspected of containing asbestos.

NIOSH responded by sending two industrial hygienists to collect bulk samples of the ceiling to confirm the presence of asbestos material and to collect air samples to determine employee exposure. The NIOSH team arrived in Washington, D.C. on January 15, 1979 to evaluate the conditions at Dulles International Airport and selected areas at Washington National Airport.

Analysis results were transmitted to the requestor by telephone on February 9, 1979 followed by Interim Report #1 on February 15, 1979.

## III. EVALUATION

### A. Airport Description

Both Dulles International Airport and Washington National Airport are owned by the Federal Government and operated by the Federal Aviation Administration, Metropolitan Washington Airports, under the Department of Transportation.

Dulles Airport opened for business in November 1962. It is situated on 10,000 acres in Northern Virginia, 26 miles from Washington, D.C. The airport terminal has a most unique design. The terminal roof is supported by a row of columns forty feet apart on each side of the concourse, sixty-five feet high on the approach side and forty feet high on the field side. It takes the form of a catenary curve which would be the shape that a string would take if suspended from two points of unequal height. It is made of eight suspension-bridge cables between which concrete panels of the roof deck fit. The concrete piers are sloped outward to counteract the pull of the cables. Just below the roof deck is a suspended ceiling which covers the entire terminal. It conforms to the same shape as the roof and covers 2.5 acres in area. Close inspection of the ceiling revealed that it was a soft, fibrous, spongy material which was easily penetrated. It was one-half to one inch thick in the area inspected. A spray coating had been non-uniformly applied at the time of construction. There has been no major repairs or renovations to the ceiling since it was installed.

Washington National Airport opened for business in June, 1941. The terminal building had 15,000 square feet of floor area. Major expansions were accomplished in the years 1950, 1955, 1958 which brought the total area to 157,353 square feet. Only certain areas of the terminal had ceilings which were suspected of containing asbestos. The appearance and physical characteristics of these ceilings were much different than the ceiling at Dulles. The coating was uniform, thin and sandy in texture. It was not easily penetrated, but it was easily removed by scraping.

#### B. Evaluation Design

NIOSH activities during this survey were designed to determine if the ceiling materials under suspicion do contain asbestos and, if so, to determine if significant deterioration is occurring.

Bulk samples of the ceiling material were obtained and submitted for asbestos analysis.

To determine if significant deterioration was occurring, a total of 37 air samples were taken for asbestos fiber analysis. Twenty-five were taken at Dulles Airport and 12 were taken at Washington National Airport. Figures 1 and 2 show the approximate sampling locations at Dulles and Washington National Airport, respectively. All samples were general area samples except for two personal breathing zone samples (one American Airline ticket counter employee and one FAA Mobil Lounge supervisor) at Dulles Airport. To determine if there was an ambient level of asbestos fibers, a sampling apparatus was positioned outside of the main terminal at Dulles on the observation deck. Ambient sampling was not done at Washington National due to rainy weather.

Additionally, to further evaluate the degree of deterioration, floor sweepings were collected and submitted for asbestos analysis. Undisturbed dust samples collected from numerous areas in the terminal represent a composite sample and would be expected to contain asbestos fibers if the ceilings were deteriorating.

#### C. Evaluation Methods

Bulk samples of ceiling material, floor sweeping and duct insulation were collected in small vials, sealed and submitted for asbestos analysis. The analytical method used was a dispersion staining technique making use of polarized light microscopy. This method is described in Appendix A.

The majority of the air samples were collected by drawing air through a membrane filter by means of a battery powered sampling pump which was calibrated to pump air through this sampling train at the rate of 1.5 lpm. Six air samples were collected in the same manner as described above but at a flow rate of 9.0 lpm using an electrically powered vacuum pump. This increased the overall sensitivity of the method by a factor of six and maximized the chances for finding airborne asbestos fibers. Considering the sensitivity of the analytical method and the volume of air drawn through the collection filters, the limit of detection was approximately 0.01 fibers/cc for the samples run at 1.5 lpm and approximately 0.001 fibers/cc for those run at 9.0 lpm. The air samples were taken and analyzed in accordance with the procedures contained in NIOSH P&CAM analytical method No. 239, Appendix B.

The current NIOSH recommended standard for occupational exposure to asbestos is 0.1 fibers/cc. The current OSHA standard is 2.0 fibers/cc. Both standards apply to asbestos fibers greater than 5 microns in length and to 8-hour time-weighted-average exposures. NIOSH and OSHA ceiling values for a 15 minute sampling period have been set at 0.5 and 10.0 fibers/cc, respectively.

Available studies provide conclusive evidence that exposure to asbestos fibers causes cancer and asbestosis (diffuse, interstitial fibrosis of lungs) in man. There are data that show that the lower the exposure the lower the risk of developing cancer. The NIOSH recommended standard is intended to (1) protect against the noncarcinogenic effects of asbestos such as asbestosis, (2) materially reduce the risk of asbestos-induced cancer and (3) be measured by techniques that are valid, reproducible, and available to industry and official agencies.<sup>1</sup> Due to the fact that there is no evidence for a threshold for a "safe" level of asbestos exposure, every effort should be made to eliminate or positively control potential sources of exposure.

1. Revised Recommended Asbestos Standard, NIOSH Publication No. 77-169, December, 1976.

#### IV. RESULTS AND DISCUSSION

Analysis results for the bulk samples obtained during this survey are presented in Table 1. The ceiling material at Dulles was found to contain approximately 40-50% chrysotile asbestos and the ceiling evaluated at Washington National contained approximately 1-2% chrysotile asbestos.

No asbestos fibers were found upon analysis of the bulk dust samples (floor sweepings and undisturbed dust samples) taken at Dulles or Washington National Airport. No detectable airborne levels of asbestos fibers were found at either Dulles or Washington National Airport.

Based on the results of the air samples and bulk dust samples, there is no health hazard at Dulles or Washington National Airport from exposure to asbestos fibers at the present time.

Most of the ventilation ducts at Dulles were wrapped with fiber-glass type insulation; however, one duct which services the grill at the cafeteria in the center kiosk is wrapped with a material found to contain approximately 40% chrysotile/amosite asbestos. If this wrapping were to start deteriorating, the asbestos would fall through the ceiling grating and contaminate the food being prepared as well as present an inhalation hazard for those employees working in this area.

Relative humidity levels within the Dulles Airport terminal were in the 13-16% range during the day of the survey. Complaints of throat irritation may be attributed to this low humidity condition.

#### V. RECOMMENDATIONS

Based on the results of this survey, the ceilings at Dulles and those areas evaluated at Washington National Airport appear to be stable and not measurably deteriorating at the present time. However, as time goes on, the potential for deterioration will increase due to further aging of the ceiling. It is therefore recommended that a program be initiated to periodically monitor for asbestos fibers in the main terminal at Dulles Airport through air sampling and analysis of bulk dust samples from floor sweepings. This sampling should be accomplished at least annually. This could be accomplished either in-house by FAA or through a contractual effort.

Due to the type of application and low percent of asbestos composition, annual analysis of floor sweepings at Washington National should be adequate to discover whether deterioration of the ceilings is occurring.

The texture and thickness of the ceiling at Dulles suggest that the proper application of a sealant would satisfactorily guard against the generation of airborne asbestos fibers should deterioration start.

"Before-and-after" type data concerning application of sealants for control of asbestos exposure is scarce. Recent emphasis has no doubt sparked research efforts. For example, Battelle Memorial Institute, Columbus, Ohio is under contract to the Environmental Protection Agency to evaluate the potential use of sealants for control of "asbestos fallout" from the deterioration of asbestos type ceilings. Preliminary efforts indicate that butyl rubber emulsion type sealants, applied by an airless spray gun technique, would be best suited for the Dulles airport ceiling. Battelle's first report will contain their evaluation of eleven sealants and is scheduled for publication in April, 1979.

Should it be determined that the ceiling is deteriorating either through visual observation of the ceiling surface or through the monitoring program, plans for sealant application should be accelerated by contacting NIOSH and/or EPA authorities for assistance in selecting a suitable sealant.

It is recommended that the ventilation duct wrapping found to contain asbestos (grill ventilation duct, center kiosk) be covered with an additional layer of material to prevent asbestos contamination of the area below and that consideration be given to replacing this duct wrapping so that this potential source of asbestos can be eliminated.

It is recommended that throat lozengers, hard candy or chewing gum be used by those personnel complaining of throat irritation. This should help to sooth the throat irritation if it is the result of the dry atmosphere.



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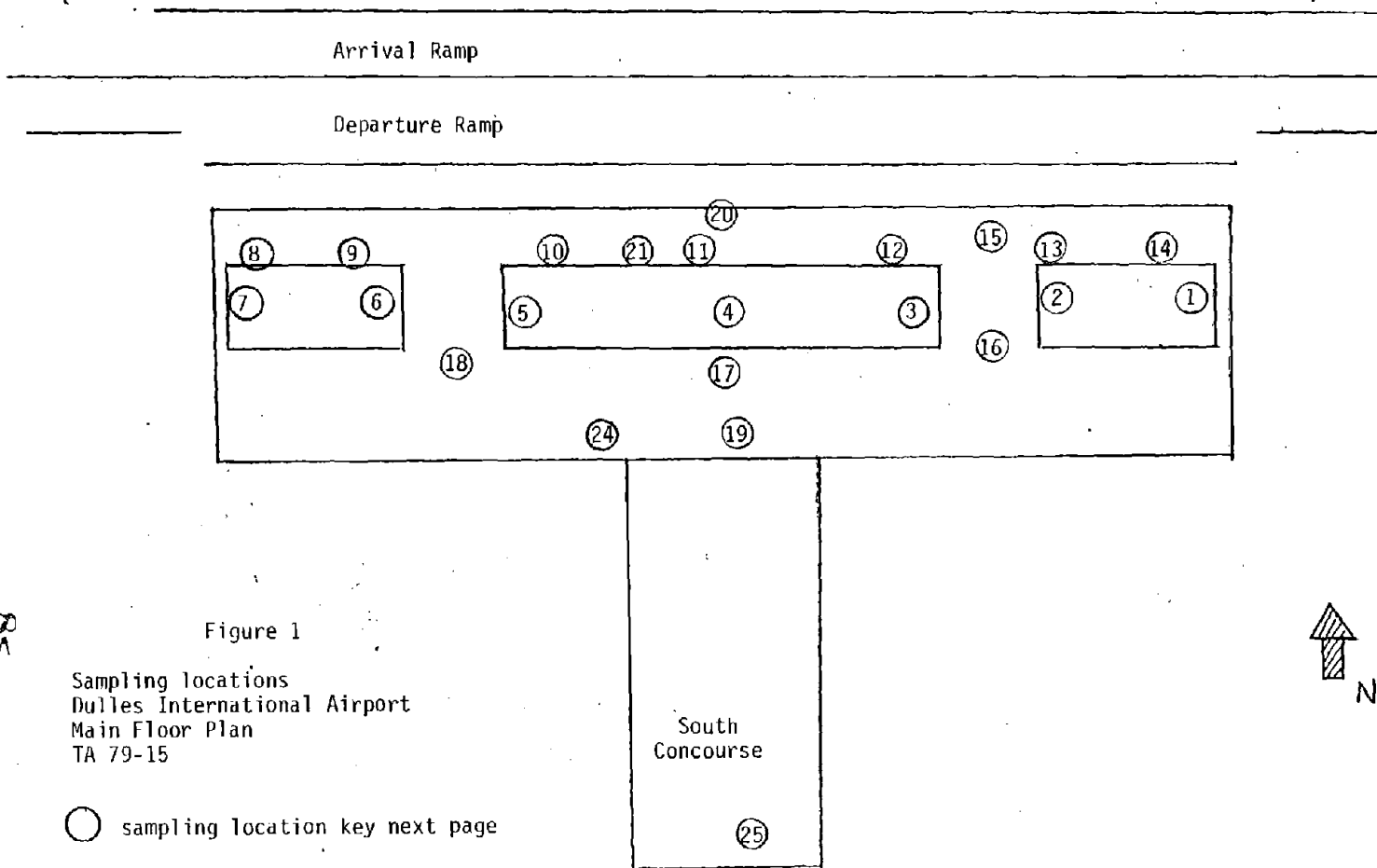


Figure 1

Sampling locations  
Dulles International Airport  
Main Floor Plan  
TA 79-15

○ sampling location key next page

Sampling Location Key  
Figure 1  
Dulles Airport

1. Top of east side of east kiosk
2. Top of west side of east kiosk
3. Top of east side of center kiosk
4. Top of center of center kiosk
5. Top of west side of center kiosk
6. Top of east side of west kiosk
7. Top of west side of west kiosk
8. Braniff ticket counter
9. North-West ticket counter
10. Eastern/British ticket counter
11. Piedmont/American ticket counter
12. United ticket counter
13. Trans World ticket counter
14. Pan American ticket counter
15. NE Insurance station
16. Eastern baggage check point
17. Newstand
18. Western baggage check point
19. Top of entry canopy to south concourse
20. Top of exit canopy
21. Personnel sample, American Airline employee
22. Secretary's desk, airport manager's office (ground floor)
23. Customs office area (ground floor)
24. Personnel sample, FAA Mobil Lounge supervisor
25. Outside, observation deck

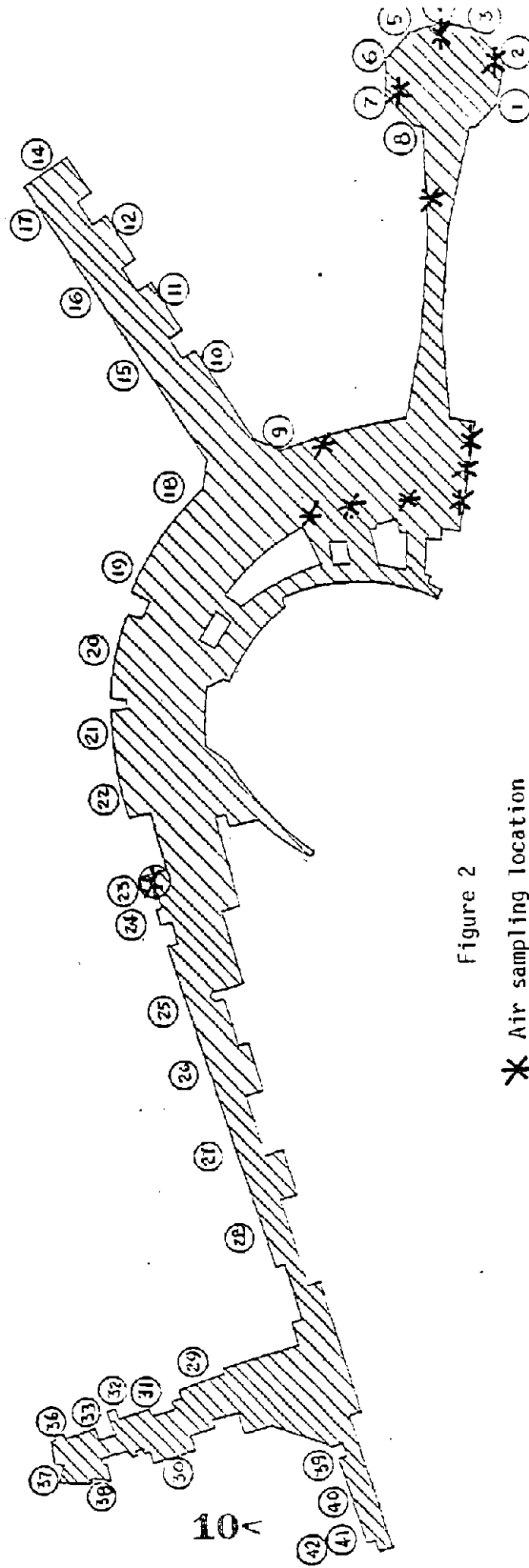


Figure 2  
 \* Air sampling location  
 Washington National Airport  
 TA 79-15

GATE POSITIONS  
 WASHINGTON NATIONAL A

Table 1  
Asbestos Analysis  
Bulk Samples  
Dulles and Washington National Airports  
TA 79-15

Bulk Sample No.	Location	Substances Identified	Approximate % Asbestos
1	duct insulation center kiosk, Dulles	cellulose, binder, amosite and chrysotile asbestos	40
2	ceiling scraping above eastern checkpoint, Dulles	rockwool, cellulose, chrysotile asbestos	40-50
3	undisturbed dust, western checkpoint	cellulose	—
4	undisturbed dust, light fixtures, top of center kiosk and floor sweepings, Dulles	cellulose	—
5	ceiling scrapings, newstand area on way to gates 1-8, Washington National	binder, some cellulose, chrysotile asbestos	1-2
6	dust, floor sweepings, Washington National	cellulose	—
7 (note 1)	ceiling material, air route traffic control center, Leesburg, Va.	rockwool, cellulose, chrysotile and amosite asbestos	50
8 (note 1)	ceiling material, AF SFO, radar site, Oakdale, Pa.	binder, rockwool, chrysotile and amosite asbestos	30

Note 1: Bulk samples #7 and 8 were not from Dulles or Washington National, but submitted by FAA from two other facilities for asbestos analysis.

# the microscop<sup>e</sup>

Incorporating "Crystal Front". Founded 1937

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## Identification of Asbestos Fibers by Microscopical Dispersion Staining\*

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### Abstract

The Cherkasov focal screening dispersion staining procedure has been successfully applied to the identification of asbestos. The various types of asbestos can be differentiated by noting the refractive index of the Cargille liquid giving matching wavelengths in the region near 550 nm. It is not necessary to use polarized light although the much more definitive data obtained with polarized light may eventually permit identification of the mine from which each asbestos came.

Although too few samples of amosite and crocidolite have been studied, it appears that these two types can be differentiated by dispersion staining. In Cargille liquid  $n_D^{20}$  C I-680 the central stop without polars will show colors in the blue magenta region ( $\lambda_0 = 550-650$  nm) for amosite and in the golden yellow region ( $\lambda_0 = 400-500$  nm) for crocidolite. Furthermore, with polarized light crocidolite will show lower birefringence than amosite; in terms of  $\lambda_0$  difference in a given liquid amosite will show 160-190 nm and crocidolite 120-140 nm. Finally, the higher value of  $\lambda_0$  is observed for the vibration direction parallel to the length for crocidolite (three samples) but perpendicular to the length for amosite (two samples).

\* Presented at INTER/MICRO-69, London, England.

Through publications<sup>1-3</sup> and current research, the pneumoconiotic (lung hardening) and frequently cancer-producing hazards of asbestos are finally fully acknowledged. Hence, there is need of a method of determining qualitatively and quantitatively environmental pollution by respirable asbestos dust (1-7  $\mu$ m).

Asbestos is a fibrous form of silicate rock. There are six types: chrysotile, amosite, crocidolite, actinolite, tremolite and anthophyllite; only the first three are of commercial importance. All are amphiboles (minerals with chains of silica tetrahedra as their basic structure) except chrysotile which is a serpentine (mineral made up of layers of silica tetrahedra).

The property of asbestos that best lends itself to identification is refractive index. The fibers are so fine that electron microscopy would be necessary if morphology were to be used. There are no dependable differences in absorption color (except possibly for crocidolite) nor specific gravity (other than chrysotile) (Table I). If we wish to use chemical composition for identification of single asbestos fibers, we require highly sophisticated instruments and techniques—the electron microscope or an electron (or ion) microprobe. X-ray diffraction requires considerable sample and is not sensitive to small percentages in any sample (i.e., < 5-10%). Very small asbestos fibers can, however, be identified simply and quickly using dispersion staining.

#### Experimental

The McCrone dispersion staining objective, based on the focal screening method of Cherkasov<sup>4</sup>, was used in this study of asbestos (Figure 2).

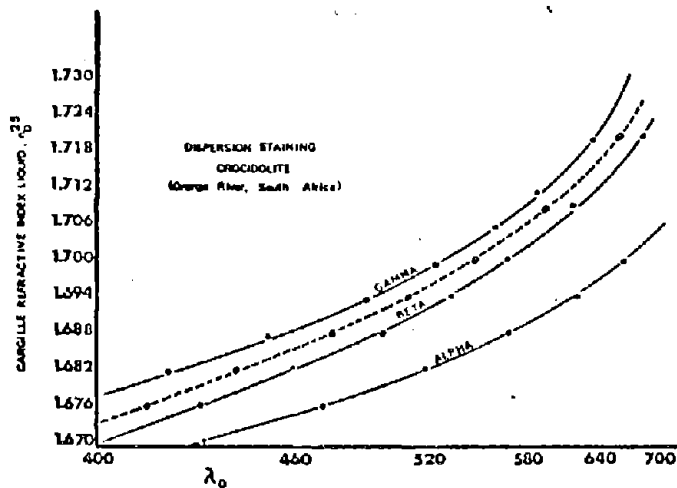


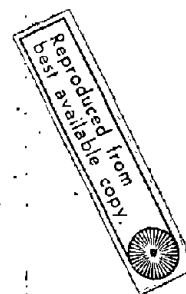
Figure 1. Dispersion staining data for a typical sample of crocidolite.

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TABLE I  
ASBESTOS

	AMPHIBOLES				
	Actinolite	Tremolite	Anthophyllite	Amosite	Crocidolite
Composition	2CaO.4MgO. FeO.85SiO <sub>2</sub> .H <sub>2</sub> O	2CaO.5MgO. 8SiO <sub>2</sub> .H <sub>2</sub> O	7MgO.8SiO <sub>2</sub> . H <sub>2</sub> O	5.5FeO. 1.5MgO. 8SiO <sub>2</sub> .H <sub>2</sub> O	NaO.FeO. 3FeO.8SiO <sub>2</sub> . H <sub>2</sub> O
Spec. Grav.	3.03-3.5	2.9-3.2	2.85-3.4	2.6-3.0	3.0-3.45
Cryst. Syst.	Monoclinic	Monoclinic	Orthorhombic	Monoclinic	Monoclinic
Extinction	$\gamma \wedge L = 10-15^\circ$	$\gamma \wedge L = 10-21^\circ$	$\gamma \wedge L = 0^\circ$	$\gamma \wedge L = 14-21^\circ$	$\alpha \wedge L = 3-15^\circ$
Sign. (elong.)	+	+	+	+	—

\*L = long direction of fibers



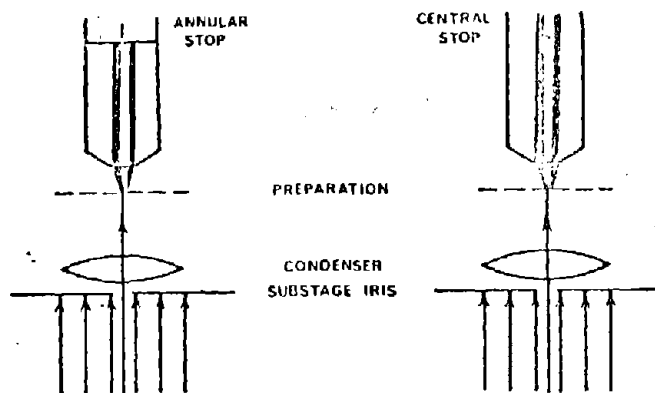


Figure 2. The arrangement for focal screening; the annular stop permits passage of the matching wavelength and the central stop, white light minus the matching wavelength.

Number of Samples

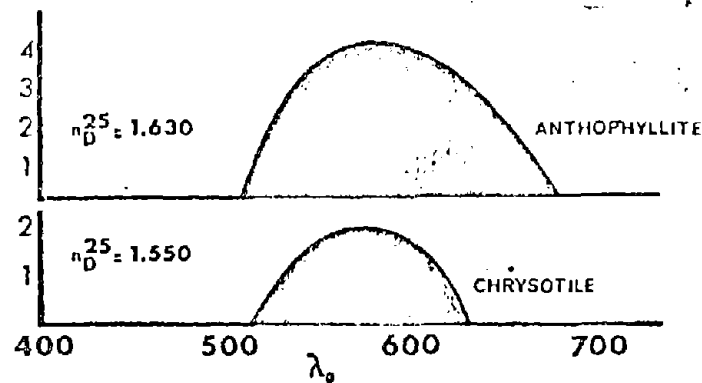
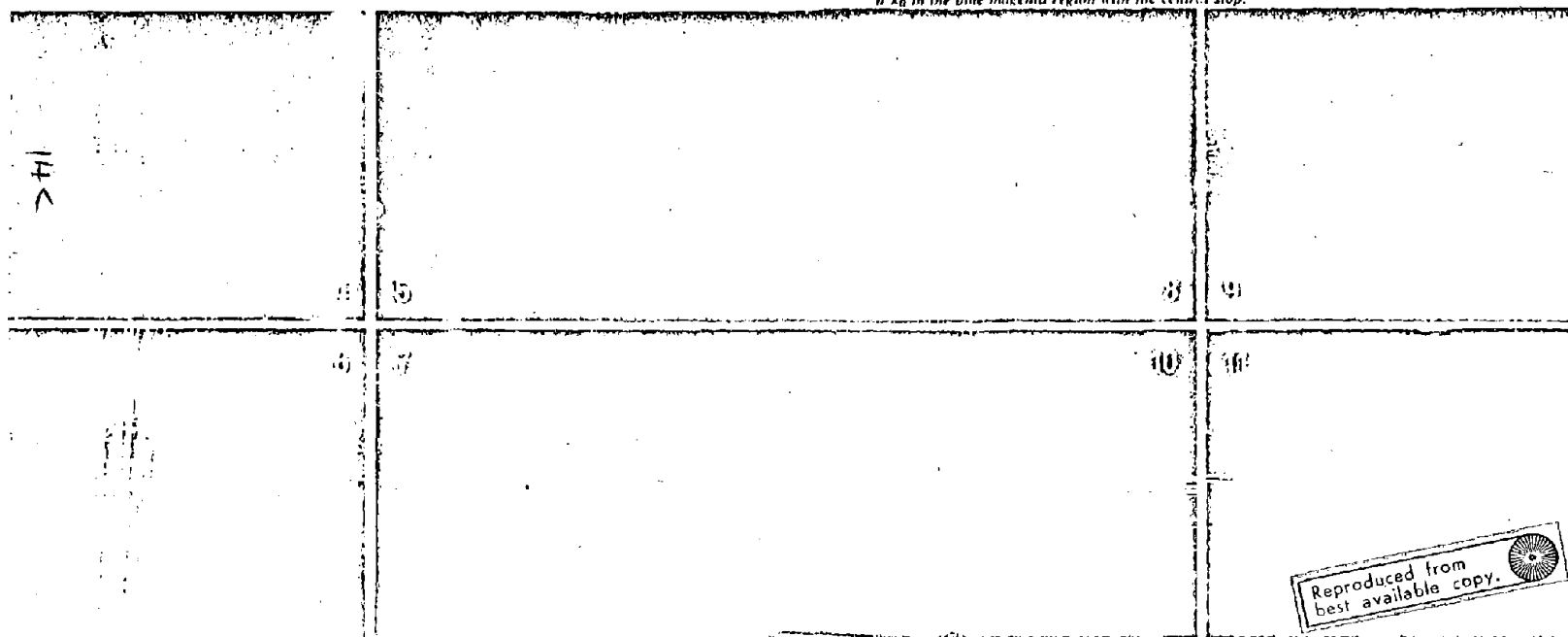


Figure 3. The range of matching wavelengths shown by anthophyllite and chrysotile with unpolarized light. The Cargille liquid was chosen so that most samples of each asbestos show a  $\lambda_0$  in the blue magenta region with the central stop.



Figures 4-7. Central stop colors for chrysotile (Figure 4), anthophyllite (5), amosite (6) and crocidolite (7) each with the appropriate Cargille refractive index liquid, 1.560 (Figure 4), 1.610 (Figure 5), 1.670 (Figure 6), 1.700 (Figure 7). The vibration direction of the polar is east-west in each figure.

Figures 8-11. Central stop colors for chrysotile (Figure 8), anthophyllite (9), amosite (10) and crocidolite (11) in the same Cargille liquids as Figures 4-7 respectively. Unpolarized light was used for this series.



TABLE II

Wavelengths Corresponding to Observed Dispersion Staining Colors

Matching Wavelength $\lambda_0$ , nm	Colors observed	
	Central stop	Annular stop
< 420	light yellow	dark-blue
420-440	yellow	blue-violet
440-470	golden yellow	blue
470-500	golden magenta	blue-green
500-540	reddish-magenta	green
540-580	magenta	yellow-green
580-610	blue-magenta	yellow
610-640	blue	orange
640-680	blue-green	orange-red
> 680	blue-white	brownish-red

Though phase contrast and dark-ground illumination are often used for dispersion staining, the data obtained by these methods are empirical. Pure colors are not obtained and the mixture of colors observed is dependent upon the particular optical system used. With the Cherkasov procedures, axial illumination is utilized and hence essentially pure reproducible colors are obtained.

A graph of matching wavelength,  $\lambda_0$ , was determined as a function of immersion liquid refractive index for each of 26 asbestos samples. The central stop in conjunction with Table II was found to be most useful in estimating  $\lambda_0$  because it provides better resolution and contrast; hence higher sensitivity for small single fibers. The procedure involved:

1. mounting each asbestos successively in those Cargille refractive index liquids imparting dispersion colors to it.
2. noting  $\lambda_0$  for the sample using both polarized and unpolarized light.
  - (a) fibers oriented parallel to the vibration direction of the polar show  $\alpha$  (crocidolite) or  $\gamma$  (all other asbestos fibers).
  - (b) fibers oriented crosswise show  $\beta$  and  $\gamma$  (crocidolite) or  $\alpha$  and  $\beta$  (all other asbestos fibers). Random orientations under these conditions show crocidolite in  $\beta \leq n \leq \gamma$  positions and all other asbestos fibers in  $\beta \geq n \geq \alpha$  positions. The two extreme colors noted for crosswise vibrations are designated  $\alpha$  (highest  $\lambda_0$ ) and  $\beta$  (lowest  $\lambda_0$ ) for all asbestos fibers other than crocidolite or  $\beta$  (highest  $\lambda_0$ ) and  $\alpha$  (lowest  $\lambda_0$ ) for crocidolite.

The small oblique extinction angle often observed with asbestos causes no significant variation in  $\lambda_0$ . Figure 1 shows the data for one sample of crocidolite determined in this way.

When the polarizer is removed, asbestos fibers show nominally a single color that is a mixture of all the wavelengths observed during rotation of the polarizer. In practice, due to polarization by reflection

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from the microscope mirror, there is a slight change in color when the stage is rotated. All our data were taken with the fibers normal to the mirror reflection vibration direction.

Figures 4-7 show the dispersion staining colors of representative samples of chrysotile, anthophyllite, amosite and crocidolite respectively. Each is shown in the various orientations (relative to the polarizer vibration direction) necessary for determination of  $\alpha$ ,  $\beta$  and  $\gamma$ . Careful study of these figures may indicate the simplified procedure used to orient the fibers at precise 90 degree angles to each other.

To determine if the simpler application of dispersion staining without a polarizer is adequate for identifying asbestos, the fields of view of Figures 4-7 were also taken with no polar (Figures 8-11). The  $\lambda_0$  curves obtained without polar were plotted in a single graph (Figure 12). The bunching of the curves of each different type of asbestos indicates that for identification purposes a liquid can be chosen that will impart a characteristic color to one species of asbestos only. A polarizing microscope is not, therefore, required. However, we can anticipate that with additional samples of crocidolite and amosite there might be some overlap. For this reason, the distinction between the signs of elongation (Figures 6 and 7) and the crocidolite blue absorption color are mentioned as special aids for differentiation. In this case a polarizer is useful.

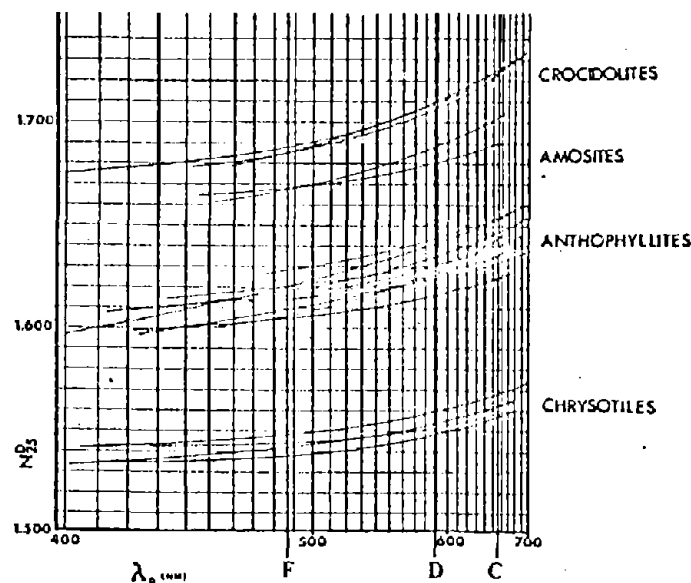


Figure 12. Matching wavelengths shown by different samples of four types of asbestos when mounted in the Cargille refractive index liquids shown and observed with unpolarized light.

## APPENDIX

Dispersion Staining Data,  $n_D^{20}$  of Cargille refractive index liquid

## Chrysotiles

Sample	Orientation	Matching wavelength, $\lambda_m$ , nm				
		450	486(F)	520	590(D)	656(C)
Thetford Mines, Quebec	$\gamma$	1.539	1.542	1.546	1.553	1.559
	$\alpha$	1.535	1.539	1.543	1.550	1.556
	$\beta$	1.533	1.539	1.543	1.547	1.556
	$\alpha$	1.528	1.531	1.534	1.541	1.546
Eden, Vermont E19048**	$\gamma$	1.549	1.553	1.558	1.566	1.574
	$\alpha$	1.543	1.547	1.551	1.559	1.566
	$\beta$	1.543	1.547	1.551	1.559	1.566
	$\alpha$	1.534	1.539	1.543	1.550	1.557
Thetford Mines, Quebec E20436	$\gamma$	1.545	1.549	1.552	1.559	1.565
	$\alpha$	1.540	1.546	1.547	1.553	1.558
	$\beta$	1.538	1.541	1.544	1.550	1.554
	$\alpha$	1.536	1.539	1.542	1.548	1.552
Thetford Mines, Quebec E12185	$\gamma$	1.543	1.547	1.550	1.556	1.563
	$\alpha$	1.540	1.543	1.546	1.552	1.558
	$\beta$	1.536	1.540	1.543	1.549	1.555
	$\alpha$	1.531	1.534	1.537	1.543	1.548
King's Mine, Quebec	$\gamma$	1.543	1.547	1.551	1.559	1.566
	$\alpha$	1.539	1.543	1.547	1.554	1.561
	$\beta$	1.535	1.539	1.543	1.550	1.557
	$\alpha$	1.529	1.532	1.535	1.542	1.547
Barquisimeto, Venezuela	$\gamma$	1.536	1.541	1.546	1.556	1.566
	$\alpha$	1.530	1.537	1.541	1.550	1.559
	$\beta$	1.530	1.534	1.539	1.547	1.555
	$\alpha$	1.524	1.528	1.532	1.540	1.548

\* no points were used.

\*\* sample identification number of the Field Museum of Natural History, Chicago, Illinois, U.S.A.

## Anthophyllites

Minas Gerais, Brazil E16696	$\gamma$	1.604	1.613	1.620	1.633	1.645
	$\alpha$	1.602	1.608	1.614	1.628	1.638
	$\beta$	1.599	1.606	1.612	1.625	1.636
	$\alpha$	1.582	1.588	1.594	1.606	1.616
Maryland E3713	$\gamma$	1.617	1.623	1.630	1.642	1.658
	$\alpha$	1.613	1.618	1.624	1.636	1.649
	$\beta$	1.609	1.614	1.619	1.630	1.642
	$\alpha$	1.599	1.604	1.609	1.620	1.630
New Mexico E3709	$\gamma$	1.620	1.625	1.629	1.638	1.648
	$\alpha$	1.616	1.621	1.625	1.634	1.642
	$\beta$	1.609	1.612	1.616	1.620	1.628
	$\alpha$	1.601	1.604	1.607	1.613	1.618
Macon Co., N. Carolina E3700	$\gamma$	1.614	1.618	1.623	1.632	1.641
	$\alpha$	1.610	1.614	1.619	1.627	1.635
	$\beta$	1.606	1.610	1.614	1.622	1.629
	$\alpha$	1.594	1.597	1.604	1.607	1.613

The characteristic color will vary somewhat for different samples of the same type of asbestos. So, the problem arose of determining the ranges of colors for each asbestos in the chosen liquid (the liquid in which most samples of any asbestos type give a color of wavelength 580-620 nanometers). To determine this range of colors for any type of asbestos, the matching wavelengths observed in the chosen liquid are plotted against the number of different samples showing that  $\lambda_m$ . The area under the resulting curve (colored in Figure 3 as per Table II) shows all the colors shown by our samples of anthophyllite and chrysotile. Too few samples of amosite or crocidolite have been studied to justify their inclusion in Figure 3.

## Conclusion

The limited results obtained indicate that asbestos can be identified by dispersion staining. More work on many more samples will establish the full range of  $\lambda_m$  values for each asbestos. Relating  $\lambda_m$  data to asbestos source may then also pinpoint the mine source of unknown asbestos samples.

## Acknowledgement

The authors are grateful to Dr. Bertram G. Woodland, Curator of Igneous and Metamorphic Petrology of the Field Museum of Natural History in Chicago for the samples of asbestos from various sources.

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APPENDIX (Continued)

Lincoln Co., Nevada E1101	$\gamma$	1.609	1.611	1.616	1.623	1.610
	$\beta$	1.602	1.605	1.609	1.615	1.621
	$\alpha$	1.596	1.599	1.602	1.609	1.615
	$\alpha$	1.589	1.592	1.595	1.600	1.606

Rio Grande do Sul, Brazil E14494	$\gamma$	1.610	1.615	1.620	1.630	1.641
	$\beta$	1.605	1.610	1.615	1.624	1.635
	$\alpha$	1.603	1.607	1.611	1.620	1.628
	$\alpha$	1.596	1.600	1.604	1.611	1.618

California E14464	$\gamma$	1.619	1.624	1.630	1.642	1.656
	$\beta$	1.614	1.619	1.624	1.635	1.646
	$\alpha$	1.612	1.616	1.622	1.632	1.642
	$\alpha$	1.601	1.605	1.610	1.618	1.627

Pine Mt., Georgia	$\gamma$	1.607	1.613	1.619	1.632	1.647
	$\beta$	1.602	1.608	1.614	1.625	1.638
	$\alpha$	1.597	1.602	1.607	1.617	1.628
	$\alpha$	1.589	1.593	1.597	1.606	1.614

Encampment, Wyoming	$\gamma$	1.619	1.620	1.626	1.630	1.638
	$\beta$	1.613	1.616	1.619	1.625	1.630
	$\alpha$	1.609	1.612	1.615	1.621	1.626
	$\alpha$	1.598	1.600	1.601	1.607	1.611

Bedford, Virginia E12790	$\gamma$	1.614	1.619	1.622	1.634	1.644
	$\beta$	1.609	1.613	1.618	1.628	1.638
	$\alpha$	1.606	1.610	1.615	1.624	1.633
	$\alpha$	1.599	1.603	1.607	1.614	1.622

Cruciololites

Orange River, South Africa	$\gamma$	1.688	1.694	1.700	1.712	1.726
	$\beta$	1.684	1.690	1.696	1.707	1.721
	$\alpha$	1.680	1.686	1.692	1.702	1.713
	$\alpha$	1.674	1.678	1.682	1.690	1.698

Westerburg, South Africa	$\gamma$	1.686	1.692	1.697	1.708	1.718
	$\beta$	1.682	1.687	1.692	1.701	1.710
	$\alpha$	1.679	1.683	1.688	1.697	1.706
	$\alpha$	1.674	1.678	1.683	1.691	1.699

South Africa	$\gamma$	1.691	1.696	1.701	1.712	1.723
	$\beta$	1.685	1.690	1.695	1.705	1.714
	$\alpha$	1.680	1.685	1.690	1.700	1.710
	$\alpha$	1.674	1.679	1.683	1.692	1.700

Amosites

Lydenburg Dist., Transvaal	$\gamma$	1.676	1.682	1.687	1.699	1.712
	$\beta$	1.665	1.670	1.676	1.687	1.698
	$\alpha$	1.658	1.663	1.668	1.678	1.689
	$\alpha$	1.652	1.657	1.662	1.672	1.682

Penge, Transvaal	$\gamma$	1.672	1.677	1.682	1.692	1.701
	$\beta$	1.665	1.669	1.674	1.682	1.691
	$\alpha$	1.660	1.664	1.668	1.676	1.684
	$\alpha$	1.653	1.657	1.661	1.669	1.676

## APPENDIX B

### ASBESTOS FIBERS IN AIR

#### National Institute for Occupational Safety and Health Analytical Method

<b>Analyte:</b>	Asbestos fibers	<b>Method No.:</b>	P&CAM 239
<b>Matrix:</b>	Air	<b>Range:</b>	0.1-60 fibers/cm <sup>3</sup>
<b>Procedure:</b>	Filter collection, microscopic count	<b>Precision (CV<sub>T</sub>):</b>	0.24 to 0.38
<b>Date Issued:</b>	3/30/77	<b>Classification:</b>	D (Operational)
<b>Date Revised:</b>			

#### 1. Principle of the Method

- 1.1 This method describes the equipment and procedures for collecting, mounting, and counting asbestos fibers on cellulose ester membrane filters in the evaluation of personal samples of airborne asbestos fibers. The purpose of the method is to determine an employee's index of exposure to airborne asbestos fibers. The method is primarily a personal monitoring technique, but can be used for area monitoring.
- 1.2 The sample is collected by drawing air through a membrane filter by means of a battery powered personal sampling pump. The filter is transformed from an opaque solid membrane to a transparent optically homogeneous gel. The fibers are sized and counted using a phase-contrast microscope at 400-450X magnification.
- 1.3 Definitions. Asbestos fiber, for counting purposes, means a particulate which has a physical dimension longer than 5 micrometers and with a length to diameter ratio of 3 to 1 or greater. Asbestos includes chrysotile, cummingtonite-grunerite (amosite), crocidolite, fibrous tremolite, fibrous anthophyllite, and fibrous actinolite.
- 1.4 Any laboratory attempting to use this procedure should have at least one counter attend a training course conducted by an experienced, proficient laboratory. Novice, untutored counters, using only published instructions, can easily obtain counts of half those performed by experienced, proficient counters. Large differences between laboratories can be caused by: 1) differences in technique and observing ability among counters and 2) small, but significant, differences between microscopes meeting the basic specifications of Section 6.2. The following procedures are recommended:
  - 1.4.1 All microscopists who perform asbestos counting should meet together for an "asbestos counting workshop" at least quarterly. This is best accomplished with counters from several laboratories using their own microscopes.
  - 1.4.2 Each microscopist should count the same series of slides and with the results being compared.
  - 1.4.3 Differences between counters should be resolved with side-by-side counting of the fields by the different counters.
  - 1.4.4 Individuals who are found to be persistent outliers over several sessions should be encouraged to seek other tasks in their respective laboratories.

## 2. Range and Sensitivity

- 2.1 The usable range is primarily a function of sample volume, microscope count field area, and background airborne particulates. The influence of these variables is discussed in 8.1.3. For a microscope count field area of  $0.003 \text{ mm}^2$  (see Figure 1) and a pump flow rate of 1.7 lpm, the optimal fiber densities would be produced over the range of 0.4 fiber/cm<sup>3</sup> (8-hour sample) to about 60 fibers/cm<sup>3</sup> (15-minute sample). For a field area of  $0.006 \text{ mm}^2$  (see Figure 2) and a pump flow rate of 1.7 lpm, the optimal range is 0.2 fiber/cm<sup>3</sup> (8-hour sample) to about 30 fibers/cm<sup>3</sup> (15-minute sample). In each case, the optimal detection limits are inversely proportional to pump flow rate.

The upper detection limit can be extended by using sample times less than 15 minutes or using lower flow rates. The lower detection limit can be extended by increasing the flow rate up to about 2.5 lpm. Filter surface fiber densities less than optimal (less than about 0.5 to 1.0 fiber per count field) are still adequate, but will lead to decreased precision for the method (increased coefficient of variation, see Section 4).

The minimum total fiber count in 100 fields considered adequate for reliable quantitation is 10 fibers. Thus, the lower limit of reliable quantitation is 0.1 fiber/cm<sup>3</sup> (100,000 fibers/m<sup>3</sup>). For this level, a flow rate of about 2.5 lpm is recommended. For a field area of  $0.003 \text{ mm}^2$ , the minimum sample time would be about 2 hours. For a field area of  $0.006 \text{ mm}^2$ , the minimum sample time would be about 1 hour.

- 2.2 This method considers only fibers with a length to diameter ratio of 3 to 1 or greater and a length greater than 5 micrometers.

## 3. Interferences

In an atmosphere known to contain asbestos, all particulates with a length to diameter ratio of 3 to 1 or greater, and a length greater than 5 micrometers should, in the absence of other information, be considered to be asbestos fibers and counted as such.

## 4. Precision and Accuracy

- 4.1 In the past decade, there have appeared a number of articles examining sources of variation in the asbestos sampling and counting procedure. These include: Lynch et al. (11.1), Weidner and Ayer (11.2), Conway and Holland (11.3), Leidel and Busch (11.4), Beckett and Attfield (11.5), and Rajhans and Bragg (11.6). The sources of variation will be discussed by stages in the membrane filter evaluation procedure.

- 4.2 Sources of Variation in the Sampling Process. These include variations in pump flow rate, proximity of the filter to the employee's body, and filter location (left to right) in the employee's breathing zone.

4.2.1 Section 9.1 requires that the personal sampling pump be calibrated with sufficient accuracy such that the 95% confidence limits on the flow rate are  $\pm 10\%$ . This is equivalent to a coefficient of variation (CV) of about 5%. However, this CV makes a negligible contribution to the total CV for the method due to the relatively large CV of the counting procedure.

4.2.2 Conway and Holland (11.3) concluded that positioning of the filter cassette on the wearer (regarding the angular portions of the filter and their proximity to the wearer) is not a significant factor in determining the fiber distribution on filters.

4.2.3 Weidner and Ayer (11.2) concluded that there is no appreciable difference between samples collected on either the right or left sides of a breathing zone or between samples collected side-by-side, especially for samples with concentrations less than 2.5 fibers/cm<sup>3</sup>.

#### 4.3 Sources of Variation in the Counting Procedure

4.3.1 Random variations exist in the fiber distribution on a filter wedge (intra-wedge variability). The industrial hygiene literature has seen considerable debate in the last 20 years concerning whether or not the distribution of mineral dust or asbestos fibers on a filter surface is adequately described by a Poisson distribution probability density function. Leidel and Busch (11.4) found excellent agreement between empirical error variance and theoretical variance calculated from the assumption of Poisson distributed true counts. They concluded that there was not excessive variation among count fields for a filter wedge and that clumping of fibers (non-random coalescence) did not occur.

4.3.2 Variations exist in the fiber distribution on the total filter surface (inter-wedge variability) due to the random or non-random distribution of fibers across the total surface of the filter. This type of variation is easily confused with intra-wedge variations. The count procedure does not require counting of multiple sectors of the filter. There may be significant differences between average counts for different wedges, or the fiber distribution variations for the total filter surface may be greater than the variations of the Poisson distribution. If either of these occur experimentally, one must use the experimental variations to estimate the minimum precision of the count procedure. The minimum precision is governed by the variations of the fiber distribution on the total surface of the filter.

Conway and Holland (11.3) concluded the distribution of fibers on filters is not uniform and the distribution of fiber counts is more disperse than Poisson. For their filters which had significant variations in fiber concentrations between sectors (as much as 50-60% of the total filter mean), they described the following relation for the standard deviation of the total number of fibers counted on a wedge (N)

$$\text{empirical } s(N) = 1.6 (N)^{1/2}$$

where N is about 100. The Poisson standard deviation would be:

$$\text{Poisson } \sigma(N) = (N)^{1/2}$$

Rajhans and Bragg (11.6) in Series I of their study found significant variation between filter segments and rejected the Poisson distribution for the total filter surface. However, in Series II of their study, utilizing various experimental modifications, they found no significant variation between filter segments and no reason to reject the assumption of Poisson distributed fiber counts.

4.3.3 Systematic variations due to differences between microscopes were studied by Leidel and Busch (11.4). In their study using five different brands of microscopes, they found no significant differences among four, but the fifth gave counts approximately 45% higher on the average than the other four.

4.3.4 Variations due to differences between counters should be examined at three levels: experienced counters occasionally counting, experienced counters routinely counting, and inexperienced (new or untutored) counters. Leidel and Busch (11.4) studied five experienced counters, with one counting only occasionally. There were no significant differences among three of the counters, but a fourth was 16% lower than the first three. The fifth, who occasionally counted, averaged 27% higher than the first three. Conway and Holland (11.3) studied three experienced counters and three inexperienced counters. They found statistically significant differences between the means of both the experienced and inexperienced counters that typically were in the range plus or minus 5 to 15%. They concluded that experience as a fiber counter is not a significant parameter affecting intercounter variations.

Rajhans and Bragg (11.6) found no significant differences among means of five experienced counters in Series I of their study. But in their carefully controlled Series II, an analysis of variance showed significant variations between counters that were plus or minus 1 to 15%.

4.3.5 Variations between laboratories are most likely due to systematic biases and are not a significant additional source of random variations. Any additional variations are most likely due to differences in counting technique. Beckett and Atfield (11.5) observed that standard counters improved greatly after personal instruction; also new counters, after instruction, tended to overcompensate and get exceedingly high counts. Additionally, they found that counts from an experienced laboratory that had not had contact with other laboratories performing the same analysis were as far from the standard values as were the counts by new counters.

4.4 Sources of variations between samples taken at different times on one employee during one work shift can affect the exposure estimate for that employee. These are primarily due to a) differences in exposure concentrations during the day, b) differences in location of the employee within the plant, and c) differences in work operation performed by the employee during the day. These sources of variation can be controlled by proper choice of sampling strategy. Refer to Leidel and Busch (11.7) and Leidel, Busch, and Lynch (11.8) for an extended discussion of sampling strategies. Interday temporal variations can affect the exposure estimates obtained on different days. Refer to Leidel, Busch, and Crouse (11.9) for a discussion of this type of variation.

4.5 Until recently, the total coefficient of variation ( $CV_T$ ) for the sampling and counting procedure was best estimated from the work of Conway and Holland (11.3). The conclusions of their study included:

4.5.1 The precision of their procedure for filters not containing an abundance of fine fibers can be estimated by a coefficient of variation of 16.2%. This value includes variation among counters and observed interaction effects.

4.5.2 The accuracy of the procedure for similar filters may be estimated for a 100-fiber count by a coefficient of variation of 21.4%. This assumes that the contribution of the overall variance from the nonuniform fiber distribution is additive.

4.5.3 A high percentage of very fine fibers on the filter can significantly affect the standard deviation and confidence limits for counts by different counters. After combining variations in fiber concentrations over the entire filter with those for different counters, it was concluded:

a. For filters with a low concentration of fine fibers, the coefficient of variation is estimated at 21% and the 95% confidence interval is  $\pm 43\%$ .

b. For filters with a high concentration of fine fibers, the coefficient of variation is estimated at 25% and the 95% confidence interval is  $\pm 50\%$ .

Lynch, Kronoveter, and Leidel (11.1) have also reported on variations of the method. Their intralaboratory study utilized the data from a large number of dust counts made by different methods by experienced counters over a period of years in an epidemiologic study of the asbestos products industry. They concluded that the standard deviation of counts of fibers longer than 5 micrometers on membrane filters could be estimated from the relation  $\sigma = (N)^{0.301}$ . Thus for counts of about 100 fibers, the coefficient of variation could be estimated at about 15.2% and the 95% confidence limits at  $\pm 30.4\%$ . These values are lower than the values reported by Conway and Holland (11.3).

Recently, the Johns-Manville Corporation conducted an in-house investigation of the asbestos count method (11.10). The study data contained total fiber counts for over

100 filters with each filter counted by two to five counters. From the Johns-Manville data, NIOSH calculated over 100 estimates of the count CV for the method (11.11). The NIOSH CV estimates included random intrafilter variations and intercounter variations, but did not include random pump flow rate variations. It was found that the count coefficient of variation (all random variations except for pump variations) was a function of the total fiber count. NIOSH then included a CV of 0.05 for random pump variations (see Section 9.1) in the CV-estimator equation to obtain a  $CV_T$ -estimator. The  $CV_T$ -estimator line is plotted on Figure 3 for total fiber counts in the range 10 to 100 fibers. Or the following equation can be used:

$$CV_T = [\text{antilog}_{10}(-0.215 - 0.203 (\log_{10} FB)) + 0.0025]^2$$

where FB is total fiber count as discussed in Section 10.

Figure 3 demonstrates that for a total fiber count of 100, the best  $CV_T$  is attainable with the appropriate sampling times given in 8.1.3 and the count rules in 8.3.9. When making decisions regarding compliance with the OSHA asbestos exposure standards in 29 CFR 1910.1001, the statistical procedures given in Leidel et al. (11.11) should be followed. The procedures are based on statistical theory and assumptions given in References 11.12, 11.13.

Because of the possibility of systematic biases due to differences between microscopes, counters, and laboratories as discussed above, it is strongly recommended that any laboratory counting asbestos should participate in an interlaboratory quality control program that includes the counting of standard reference filters. These standard filters are available from NIOSH through the Proficiency Analytical Testing (PAT) Program. The PAT Program is used by the American Industrial Hygiene Association (AIHA) as part of its Laboratory Accreditation Program. Each laboratory's quality control program must include protocols for routinely adjusting and calibrating sampling and counting equipment plus training and evaluation programs for counters.

## 5. Advantages and Disadvantages of the Method

- 5.1 The method is intended to give an index of employee exposure to airborne asbestos fibers of specified dimensional characteristics.
- 5.2 It is not meant to count all asbestos fibers in all size ranges or to differentiate asbestos from other fibrous particulates.

## 6. Apparatus

### 6.1 Sampling Equipment

The personal sampling equipment train consists of 1) personal sampling pump, 2) tubing, 3) clothing spring clip, 4) tubing-to-field monitor metal adaptor, and 5) field monitor (filter and holder).

- 6.1.1 Personal Sampling Pump. The pump must be capable of sampling at 1.0 to 2.5 liters per minute (lpm) against a flow resistance of 7.5 inches of water (1.4 cm Hg) for continuous hours on a fully charged battery.
- 6.1.2 Tubing. Laboratory tubing such as rubber or plastic with 6-mm bore and about 10 cm length.
- 6.1.3 Clothing Spring Clip. The clip attaches the rubber tubing to the lapel or shirt of the individual being monitored.
- 6.1.4 Tubing-to-field Monitor Adaptor. A short metal adaptor with ridges on one end to grip the inside of the tubing. The other end is designed for a pressure fit into the field monitor.
- 6.1.5 Field Monitor (Filter and Holder). The only field monitor currently considered acceptable by NIOSH is manufactured by the Millipore Corporation. The unit



sists of 1) a three section styrene plastic case designated Millipore Aerosol Monitor Case, 2) a 37-mm diameter plain white cellulose ester membrane filter designated Millipore AA (pore size of 0.8 micrometer), 3) a support pad, and 4) two plastic sealing caps. If a large number of samples are to be taken, it may be less expensive to reuse the plastic cases. Great care must be taken in the cleaning and reassembly process. The outside mating surfaces of the field monitors may be covered with a "shrink-fit" band to provide proper sealing and a writing surface for filter identification.

## 6.2 Optical Equipment and Microscope Features

- 6.2.1 Microscope body with binocular head.
- 6.2.2 10X Huygenian eyepieces are recommended. Other eyepieces can be substituted if necessary. Wide field eyepieces can be used; however, wide field eyepieces may yield a count field area less than 0.003 mm<sup>2</sup> with the Porton reticle. This is not always desirable from the standpoint of obtaining optimum sampling times (see Section 8.1.3). If wide field eyepieces are used, it is preferable to use the Patterson Globe and Circle reticle to obtain a larger count field area.
- 6.2.3 Koehler illumination (preferably built-in with provisions for adjusting light intensity).
- 6.2.4 A Porton reticle is recommended. Others such as the Patterson Globe and Circle can be substituted.
- 6.2.5 Mechanical stage.
- 6.2.6 Phase-Contrast condenser with a numerical aperture (N.A.) equal to or greater than the N.A. of the objective.
- 6.2.7 40-45X phase contrast achromatic objective (N.A. 0.65 to 0.75).
- 6.2.8 Phase-ring centering telescope or Bertrand lens.
- 6.2.9 Green or blue filter, if recommended by microscope manufacturer.
- 6.2.10 Stage micrometer with 0.01 mm subdivisions.
- 6.2.11 For general guidance on phase contrast microscopy, consult Needham (11.12), Clark (11.15) and McCrone (11.14).

## 6.3 Filter Mounting Equipment. Experience has shown that certain equipment is useful for efficient sample mounting. The following items are recommended for extracting and mounting a portion of the filter for counting.

- 6.3.1 Microscope slides. 2.5 by 7.5 cm glass slides are most commonly used. Sample number, data, initials, etc., can be conveniently written on a frosted end slide.
- 6.3.2 Cover Slips. Cover slips are a necessary part of the slide mount and optical system. The shape should be appropriate for the size of the filter wedge. The appropriate cover slip depends upon the objective to be used. Ordinarily, objectives are optically corrected for a #1½ (0.17 millimeter) thickness cover slip. Improper cover glass thickness will detract from the final image quality.
- 6.3.3 Scalpel. A scalpel is needed to cut out a portion of the filter to be examined. A number-ten curved blade scalpel is recommended.
- 6.3.4 Tweezers. A pair of fine-tipped tweezers is used to remove the membrane filter slice from the field monitor and place it upon the slide.
- 6.3.5 Lens Tissue. To insure cleanliness, a lint-free tissue is recommended. This tissue should also be used for wiping mounting tools and for cleaning slides and cover slips.
- 6.3.6 Glass Rod. A fire-polished glass rod may be used to spread the mounting solution on the slide.

- 6.3.7 Wheaton Balsani Bottle. This special glass container has a glass top which prevents contamination of the mounting solution. A glass rod is included for dispensing the solution.

## 7. Reagents

Chemicals should be reagent grade, free from particles and color, conforming to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

### 7.1 Dimethyl phthalate

### 7.2 Diethyl oxalate

Avoid getting the mounting solution on the skin. Wash skin promptly with soap and water if skin contact occurs.

## 8. Procedure

### 8.1 Sampling

#### 8.1.1 General Information

Guidelines for the monitoring of employee exposures to industrial atmospheres are given in Reference 11.8. The Federal requirements for monitoring employee exposure to airborne asbestos are found in 29 CFR 1910.1001.

#### 8.1.2 Mounting the Sampling Pump on the Worker

Fasten the sampling pump to the worker's belt and fasten the field monitor to the lapel or shirt front (as close to the breathing zone as is practical). Remove the top cover of the plastic monitor, then invert the monitor making certain the exposed filter is facing downward. Turn the pump on and adjust to the calibrated flow rate (1.0 to 2.5 lpm). Record the following information in a logbook.

1. Filter number
  2. Pump start time and date
  3. Flow rate
  4. Subject's name and job title
  5. Type of operation or process
  6. Ventilation controls and is the worker wearing a respirator approved for asbestos?
- The pump should be checked periodically during the sampling period for proper operation and flow rate.

#### 8.1.3 Optimum Sampling Times

The requirement for the minimum count of 100 fibers or 20 fields in 8.3.9 was determined to be the best compromise to achieve adequate precision for the airborne fiber estimate and reasonable counting times. An optimum fiber density of about 1 to 5 fibers per microscope count field is recommended. To estimate appropriate sampling times for feasible counting and optimal counting, one must consider the following constraints:

1. microscope count field area (generally 0.003 to 0.006 mm<sup>2</sup>)
2. pump flow rate (typically 2.5 lpm maximum)
3. average airborne fiber concentrations
4. counting rule range of 20 to 100 fields
5. adequate fiber density to obtain a minimum count of 10 fibers in 100 fields, which is the least total fiber count that yields an acceptable count precision
6. background airborne particulate levels that can reduce the count precision due to an obscuring of fibers on the filter surface

The preceding constraints were considered in drawing Figures 1 and 2. These figures were developed from the following relationship:

$$\text{sampling time} = \frac{(\text{FB/FL})(\text{ECA/MFA})}{(\text{FR})(\text{AC})(1000)} \text{ minutes}$$

where:

FB/FL = 1 to 5 fibers/field

ECA = effective collecting area of filters (855 mm<sup>2</sup> for 37-mm filter with effective diameter of 33 mm)

MFA = microscope field area (generally 0.003 to 0.006 mm<sup>2</sup>)

FR = Pump flow rate (generally 1.0 to 2.5 lpm)

AC = Air concentration of fibers in fibers/cm<sup>3</sup>.

Figure 1 (microscope field area = 0.003 mm<sup>2</sup>) and Figure 2 (microscope field area = 0.006 mm<sup>2</sup>) show optimum and feasible sampling times for a pump flow rate of 1.7 lpm. Each individual responsible for sampling asbestos should prepare a similar chart for his particular pump flow rate and microscope field area before sampling is performed to aid in estimating proper sampling times. On Figures 1 and 2, the areas with solid shading lines are generally the optimum conditions for counting. The broken shading lines are for conditions very close to optimal.

However, feasible counting conditions may extend down to about 0.1 fiber/field and and above 5 fibers/field. Recommended sampling times are most strongly influenced by background airborne particulate levels, once all the other constraints have been estimated. For heavy particulate levels, it may be necessary to limit each filter to about 60 to 180 minutes sampling duration. Each individual responsible for sampling should work closely with the microscopist to attain as high as possible filter surface fiber densities (up to about 5 fibers/field), while avoiding filter surface background particulate levels that create very difficult or impossible counting conditions. If one has very little idea of airborne fiber and particulate levels, the best procedure is to take several long samples (as one 8-hour or two consecutive 4-hour samples) in conjunction with several short samples (as four consecutive 2-hour or eight consecutive 1-hour samples). If the longer samples prove very difficult to count, the microscopist will have the shorter samples to fall back on.

From Figures 1 and 2, it can be seen that there are certain sampling times which will yield optimum fiber densities on the filter for almost all airborne fiber concentrations from 1 to 10 fibers/cm<sup>3</sup>. These optimum times have been calculated and are presented in Figure 4. Note that the optimum times given by Figure 4 are approximate and can be varied by as much as  $\pm 25\%$ . The nomogram is intended as a guide to be used where no prior knowledge of the air concentration is available.

#### 8.1.4 End of Sampling Period

Remove the field monitor, replace the plastic top cover and the small end caps, and store the monitor. Always shut off the pump when changing monitors to avoid contaminating or damaging the pump. Record the pump shutoff time and flow rate in the logbook.

#### 8.1.5 Blanks

With each batch (25 to 50 filters) of samples sent for analysis, submit two unopened field monitors which have been subjected to the same treatment as the samples except that they were not exposed to the sampling environment. Label these as blanks. If the blanks yield fiber counts greater than 5 fibers/100 fields, then the entire sampling procedure should be examined carefully for the cause of contamination. The

mounting solution of Section 8.2.1 should also be examined for contamination and/or crystal growth.

#### 8.1.6 Shipping

The field monitors in which the samples are collected should be shipped in a rigid container with sufficient packing material to prevent crushing.

#### 8.1.7 Numbers of Samples

When sampling for the Federal ceiling standard of 10 fibers ( $>5\mu\text{m}$ )/ $\text{cm}^3$ , [29 CFR 1910.1001(b) (3), effective July 7, 1972], only one sample (15 minutes maximum duration) is necessary, theoretically. However, several samples should be taken during expected periods of peak air concentrations to allow for detection of gross sampling or counting errors.

When sampling for determination of noncompliance with the Federal 8-hour TWA standard of 2 fibers ( $>5\mu\text{m}$ )/ $\text{cm}^3$ , [29 CFR 1910.1001(b) (2)], one should continuously sample as large a portion of the work day as is feasible for airborne concentrations of about 2 to 10 fibers/ $\text{cm}^3$ . However, for a lower airborne concentration such as 0.5 fiber/ $\text{cm}^3$ , one sample might require 4 to 8 hours sampling time in order to get the proper filter fiber density (Section 8.1.3). For this situation, the 8-hour TWA exposure would be determined from one 8-hour or two 4-hour samples as appropriate.

### 8.2 Sample Preparation

#### 8.2.1 Preparation of Mounting Solution

A very important part of the sample evaluation is the mounting process. This process involves a special mounting medium of prescribed viscosity. The proper viscosity is important in order to expedite filter dissolving and still minimize particle migration. After the sample has been mounted, an elapsed time of approximately sixty minutes is needed before the sample is ready for evaluation.

Combine the dimethyl phthalate and diethyl oxalate in a one to one ratio by volume and pour into a Wheaton balsam bottle. Add approximately 0.05 ( $\pm 0.005$ ) grams of new membrane filter per milliliter of solution to reach the necessary viscosity. The mixture must be stirred periodically until the filters have dissolved and a homogeneous mixture is formed. The normal shelf life of the mounting solution is about three months. Twenty milliliters of mounting solution will prepare approximately 300 samples.

#### 8.2.2 Sample Mounting

Cleanliness is important! A dirty working area may result in sample contamination and erroneous counts. The following steps should be followed when mounting a sample.

1. Clean the slides and cover slips with lens tissue. Lay each slide down on a clean surface with the frosted end up. It is a good practice to rest one edge of the cover slip on the slide and the other edge on the working surface. By doing this, you keep the bottom surface (the one which contacts the filter) from becoming contaminated.
2. Wipe all the mounting tools clean with lens tissue and place them on a clean surface (such as lens tissue). All tools should be wiped clean prior to mounting each sample.
3. Using the glass rod supplied with the Wheaton balsam bottle, apply a drop of mounting solution onto the center of the slide. It may be necessary to adjust the quantity of solution so that after the cover slip has been placed on top, the solution extends only slightly beyond the filter boundary. If the quantity is greater than this, particle migration may occur.

4. Using another glass rod, spread the mounting media into a triangular shape. The size of this triangle should coincide with the dimension of the filter wedge.
5. Separate the middle and bottom sections of the field monitor case to expose the filter. Cut a triangular wedge from the center to the edge of the filter using the scalpel. The size of the wedge should approximate one-eighth of the filter surface. The filter can be very carefully removed from the cassette for cutting, but this should only be done with great care.
6. Grasp the filter wedge with the tweezers on the perimeter of the filter which was clamped between the monitor case sections. Do not touch the filter with your fingers. Place the wedge, sample side up, upon the mounting medium.
7. Pick up a clean cover slip with tweezers and carefully place it on the filter wedge. Once this contact has been made, do not reposition the cover slip.
8. Label the slide with the sample number and current date before proceeding to the next filter. On the bottom (backside) of the slide, trace the perimeter of the filter wedge with a felt tip marking pen. This will enable the counter, after the filter has become transparent, to stay within the filter perimeter when counting.
9. The sample should become transparent within fifteen minutes. If the filter appears cloudy, it may be necessary to press very lightly on the cover slip. This is rarely necessary; however, counting should not be started until an hour after the mounting. This allows the microscopic texture of the filter to become invisible to microscope viewing.
10. Discard the sample mount after two days if it has not been counted. Crystals appearing similar to asbestos fibers may begin to grow at the mounting media/air interfaces. They seldom present any problems if the slide is examined before two days. In any case, stay away from the filter's edges when counting and sizing.

### 8.3 Counting of Fibers

- 8.3.1 Place the slide on the mechanical stage of the microscope and position the center of the wedge under the objective lens and focus upon the sample. Start counting from one end of the wedge and progress along a radial line to the other end (count in either direction from perimeter to wedge tip). Random fields are selected, without looking into the eyepieces, by slightly advancing the slide in one direction with the mechanical stage control.
- 8.3.2 It is essential to continually scan over a range of focal planes (generally the upper 10 to 15 micrometers of the filter surface) with the fine focus control during each field count. This is especially necessary for asbestos fibers due to their impaction into the filter matrix.
- 8.3.3 On most airborne samples, asbestos fibers will generally have fiber diameters less than one micrometer. Therefore, it is necessary to look carefully for faint fiber images.
- 8.3.4 Regularly check phase ring alignment.
- 8.3.5 When an agglomerate (mass of material) covers a significant portion of the field of view (approx 1/6 or greater) reject the field and select another. (Do not include it in the number of fields counted.) However, report the fact as it may have meaning on other data collection.
- 8.3.6 Bundles of fibers are counted as one fiber unless both ends of the fiber can be clearly resolved.
- 8.3.7 Count only fibers with a length to width ratio greater than or equal to 3:1.
- 8.3.8 Count only fibers greater than 5 micrometers in length. (Be as accurate as possible in accepting fibers near this length.) Measure curved fibers along the curve to estimate the total length.

- 8.3.9 Count as many fields as necessary to yield a total count of at least 100 fibers. Exceptions: a) count at least 20 fields even if you count more than 100 fibers, and b) stop at 100 fields even if you haven't reached 100 fibers.
- 8.3.10 For fibers that cross either one or two sides of the counting field, the following procedure is used to obtain a representative count.  
 COUNT any fiber greater than 5 micrometers in length, that lies entirely within the counting area. COUNT as "½ fiber" any fiber with only one end lying within the counting area. DO NOT COUNT any fiber crossing any two sides.  
 Reject and do not count all other fibers. Refer to Figures 5 through 10. Note that the fibers in Figures 5 through 10 are not representative of the appearance of most asbestos fibers. Most fibers have a very faint image.

## 9. Calibration and Standards

### 9.1 Sampling Train Calibration

The accurate calibration of the sampling pump is essential to the correct calculation of the air volume sampled. The frequency of calibration is dependent on the use, care, and handling to which the pump is subjected. Pumps must be recalibrated if they have just been repaired, misused, or received from the manufacturer. If the pump receives hard usage, more frequent calibration may be necessary. Ordinarily, pumps should be calibrated in the laboratory both before they are used in the field and after they have been used to collect a large number of field samples.

The accuracy of calibration is dependent upon the type of instrument used as a reference. The choice of a calibration instrument will depend largely on where the calibration is performed. For laboratory testing, a 1-liter buret used as a soap bubble flow meter or wet-test meter is recommended. Other standard calibrating instruments, such as a spirometer, Mariott's bottle, or dry gas meter can be used. The calibration should be of sufficient precision that the 95% confidence limits on the flow rate are  $\pm 10\%$  (95% of the flow rates will fall within  $\pm 10\%$  of the calibrated value).

Instructions for calibration with the soap bubble flow meter follow. The sampling train used (pump, hose, filter cassette) in the pump calibration should be the same as the one used in the field.

- 9.1.1 Check the voltage of the pump battery with a voltmeter both with the pump off and while it is operating to assure adequate voltage for calibration. If necessary, charge the battery to manufacturer's specifications.
- 9.1.2 Fill a beaker with 10 ml of soap solution.
- 9.1.3 Connect the filter cassette inlet to the top of the buret with a length of hose.
- 9.1.4 Turn the pump on and moisten the inside of the soap bubble meter by immersing the open end of the buret into the soap solution and drawing bubbles up the inside of the buret. Perform this task until the bubbles are able to travel the entire length of the buret without breaking.
- 9.1.5 Adjust the pump rotameter to provide a flow between 1.5 to 2.5 lpm.
- 9.1.6 With a water manometer, check that the pressure drop across the filter is less than 13 inches of water (about 1 inch of mercury).
- 9.1.7 Start a soap bubble up the buret and measure the time it takes for the bubble to travel a minimum volume of 1 liter.
- 9.1.8 Repeat the procedure in 9.1.7 at least three times, average the results, and calculate the calibrated flow rate by dividing the volume traveled by the soap bubble by the elapsed time. If the range between the highest and lowest of the three flow rates is greater than about 0.33 lpm, then the calibration should be repeated since it is likely that the precision is not adequate.

- 9.1.9 Data required for the calibration include the volume measured, elapsed time, pressure drop, air temperature, atmospheric pressure (or elevation), pump serial number, date, and name of person performing the calibration.
- 9.1.10 Corrections to the flow rate for pumps with rotameters may be necessary if the pressure (elevation) or temperature where the samples are collected (actual flow rate) differs significantly from that where the calibration was performed (indicated flow rate). Actual flow rates at time of sampling may be calculated for a linear scale rotameter by using the following correction formula:

$$Q_{\text{actual}} = Q_{\text{indicated}} \sqrt{\frac{P_{\text{cal}}}{P_{\text{actual}}} \cdot \frac{T_{\text{actual}}}{T_{\text{cal}}}}$$

where both pressure (P) and temperature (T) are in absolute units such as:

psia = psig + 14.7  
deg Rankin = deg Fahrenheit + 460  
deg Kelvin = deg Celsius + 273

## 9.2 Microscope Setup

### 9.2.1 Porton Reticle and the Counting Field

The asbestos fiber count procedure consists of comparing fiber length to the diameters of calibrated circles of a Porton reticle, and counting all fibers greater than 5 micrometers in length lying within a given counting field area. The Porton reticle is a glass plate inscribed with a series of circles and rectangles. The left half of the reticle is divided into six rectangles constituting the counting field. The counting field is illustrated in Figures 5 through 10.

### 9.2.2 Placement in Eyepiece

The Porton reticle is placed inside the Huygenian eyepiece where it rests on the field-limiting diaphragm. If other types of eyepieces are used, it may be necessary to insert a counting collar for retaining the reticle. The reticle should always be kept clean, since dirt on the reticle is in focus and could complicate the counting and sizing process.

### 9.2.3 Stage Micrometer

The Porton reticle cannot be used for counting until it has been properly calibrated with a stage micrometer. Most stage micrometer scales are approximately two millimeters long and are divided into units of one-hundredth of a millimeter (ten micrometers).

### 9.2.4 Microscope Adjustment

When adjusting the microscope, follow the manufacturer's instructions while observing the following guidelines.

1. The light source image must be in focus and centered on the condenser iris or annular diaphragm.
2. The particulate material to be examined must be in focus.
3. The illuminator field iris must be in focus, centered on the sample, and opened only to the point where the field of view is illuminated.
4. The phase rings (annular diaphragm and phase-shifting elements) must be concentric.

### 9.2.5 Porton Reticle Calibration Procedure

Each eyepiece-objective-reticle combination on the microscope must be calibrated. Should any of the three be changed (disassembly, replacement, zoom adjustment, etc.), the combination must be recalibrated. Calibration may change if interpupillary dis-

tance is changed. For proper calibration, the following procedure should be followed closely.

With a 10X objective in place, place the stage micrometer on the mechanical stage, focus the millimeter scale, and center the image. Change to the 40-45X objective and adjust the first millimeter scale division to coincide with the left boundary of the Porton rectangle. Measure the distance between the left and extreme right boundaries of the Porton rectangle, estimating any portion of the final division. This measurement represents 200 L units. The rectangle is 100 L units on the short vertical dimension. The calculated "L" is inserted into the formula  $D = L(2^N)^{1/2}$  where "N" is the circle number (indicated on the reticle) and "D" is the circle diameter. Since the circle diameters vary logarithmically, every other circle doubles in diameter. For example, circle number three is twice the diameter of number one; number four is twice the diameter of number two. When the circle sizes have been determined, the counting field area which consists of the left six smaller rectangles can be calculated from the relation  $10,000 L^2$ . This completes the reticle calibration for this specific objective-eyepiece-reticle combination.

#### Example for Porton Reticle

The following calibration was obtained for a pair of 10X Huygenian eyepieces and a 43X objective:

200 L = 0.148 mm = 148 micrometers

100 L = 0.074 mm = 74 micrometers

One L-unit = 0.74 micrometers

Thus Circle #1 has a diameter  $D = L(2^1)^{1/2} = 0.74(2^1)^{1/2} = 0.74 (1.414) = 1.05$  micrometers.

Then our circle diameter calibration table looks like:

Diameter of Circle #1 = 1.05 micrometers

#2 = 1.48

#3 = 2.09

#4 = 2.96

#5 = 4.19

#6 = 5.92

Field area =  $(10,000) (L^2) = (100 L) (100 L) = (0.074) (0.074) = 0.0055$  mm<sup>2</sup>

Thus fibers with a length greater than a distance halfway between the diameters of the #5 and #6 circles would be counted.

If a Patterson Globe and Circle reticle is used, a different calculation procedure is required. The circle diameters are related as follows. The #25 circle diameter is (0.1) (reticle length).

The circle diameters are proportional to the ratio of their numbers. Thus the #20 circle diameter is (20/25) or 0.8 times the #25 circle diameter.

## 10. Calculations

10.1 The average airborne asbestos fiber concentration estimated by the filter sample may be calculated from the following formula:

$$AC = \frac{[(FB/FL) - (BFB/BFL)] (ECA)}{(1000) (FR) (T) (MFA)}$$



where:

AC = Airborne fiber concentration in (fibers > 5  $\mu$ m)/cm<sup>3</sup>.  
 BFB = Total number of fibers counted in the BFL fields of the blank or control filters in fibers > 5  $\mu$ m.  
 BFL = Total number of fields counted on the blank or control filters.  
 ECA = Effective collecting area of filter (855 mm<sup>2</sup> for a 37-mm filter with effective diameter of 33 mm).  
 FR = Pump flow rate in liters/min (lpm).  
 FB = Total number of fibers counted in the FL fields in fibers > 5  $\mu$ m.  
 FL = Total number of fields counted on the filter.  
 MFA = Microscope count field area in mm<sup>2</sup> (generally 0.003 to 0.006).  
 T = Sample collection time in minutes.

- 10.2 Recount criteria. It is very desirable for a counter to conduct a "blind recount" for about 1 in every 10 filter wedges (slides) counted. Alternatively, a second counter could perform the blind recount. In training sessions for novice counters, the trainee should conduct a blind recount for filter wedges counted by an experienced, proficient counter. In all cases, we will observe differences between the first and second counts of the same filter wedge. Most of these differences will be due to chance alone, that is, due to the random variability (precision) of the count method. Statistical recount criteria enable us to decide whether observed differences can reasonably be explained due to chance alone or are probably due to systematic differences between counters or microscopes or due to some other biasing factor. The following recount criterion is for a pair of counts that estimate some airborne fiber concentration (AC) in fibers/cm<sup>3</sup>. The criterion is given at the type-I error level. That is, there is a 5% maximum risk that we will reject a pair of counts for the reason that one might be biased, when the large observed difference is really due to chance. Reject a pair of counts because one might be biased if:

$$(AC_2 - AC_1) \text{ exceeds } 2.77(\overline{AC})(CV_{FB})$$

where:

AC<sub>1</sub> = lower estimated airborne fiber concentration  
 AC<sub>2</sub> = higher estimated airborne fiber concentration  
 $\overline{AC}$  = average of the two airborne concentration estimates  
 CV<sub>FB</sub> = average CV for the two concentration estimates which are a function of the total fiber count (FB) in each case. Use the relation in Section 4 or Figure 3.

For a pair of counts on the same filter, reject the pair because one might be biased if:

$$(FB_2 - FB_1) \text{ exceeds } 2.77(\overline{FB})(CV_{FB})$$

where:

FB<sub>1</sub> = lower fiber count on the filter (total fibers)  
 FB<sub>2</sub> = higher fiber count on the filter (total fibers)  
 $\overline{FB}$  = average of the two total fiber counts  
 CV<sub>FB</sub> = CV<sub>T</sub> for the value FB. Use the relation in Section 4 or Figure 3.

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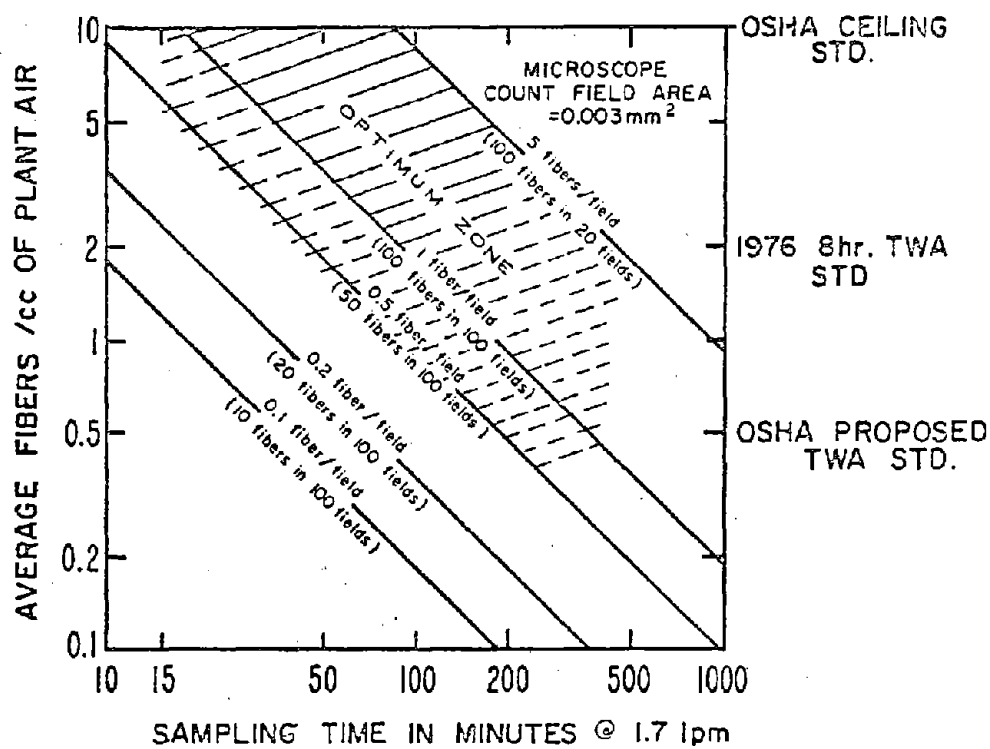


FIGURE 1. Optimum Sampling Times for airborne asbestos where microscopic field area = 0.003 mm<sup>2</sup>

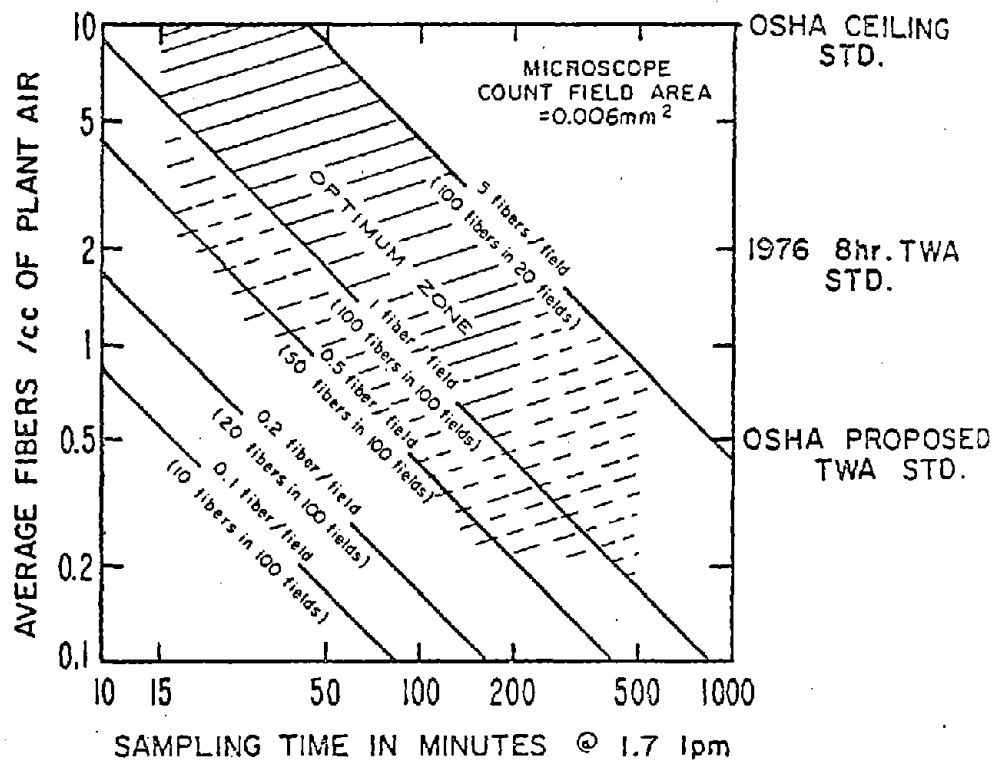


FIGURE 2. Optimum sampling times for airborne asbestos where microscopic field area = 0.006 mm<sup>2</sup>

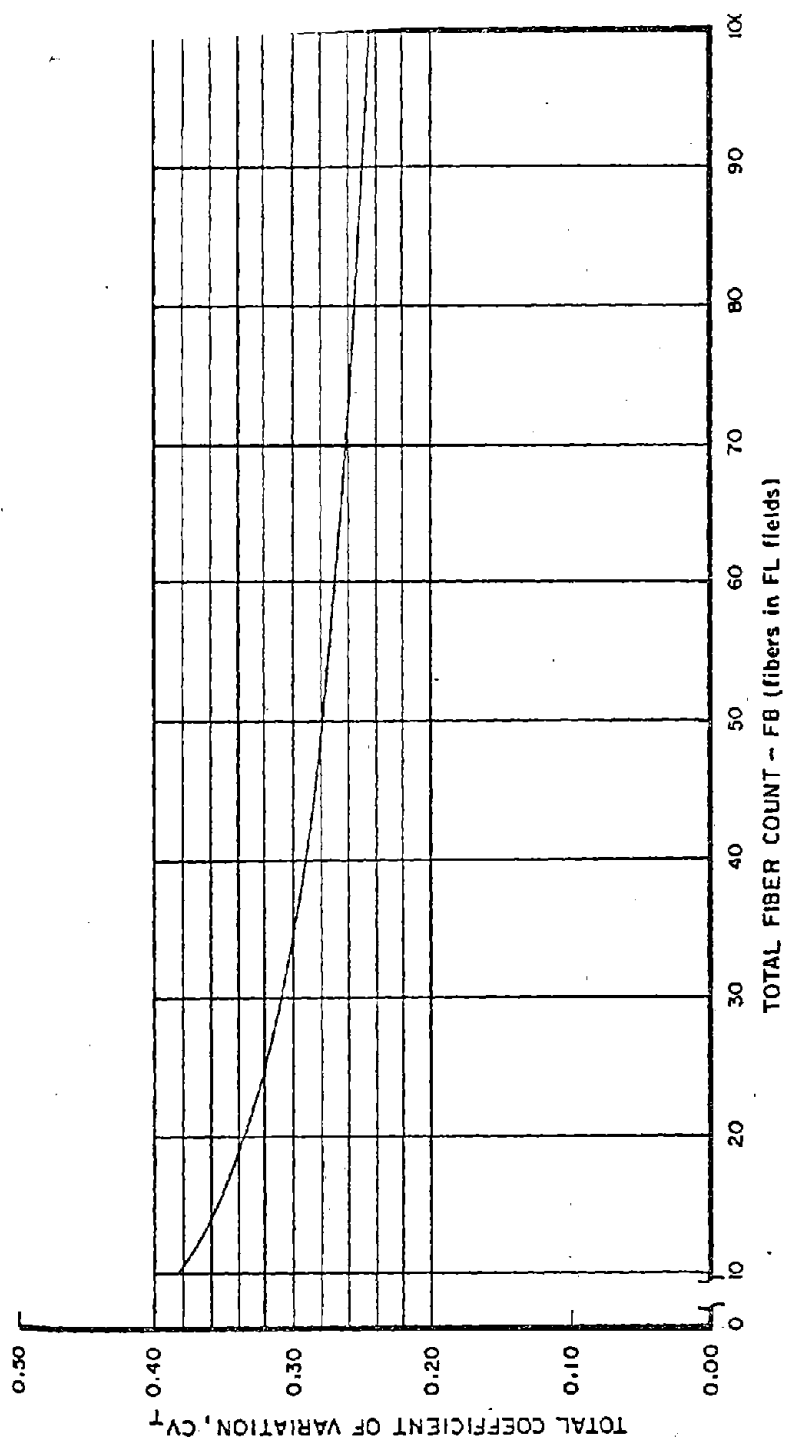


FIGURE 3. Total coefficient of variation as a function of total fiber count

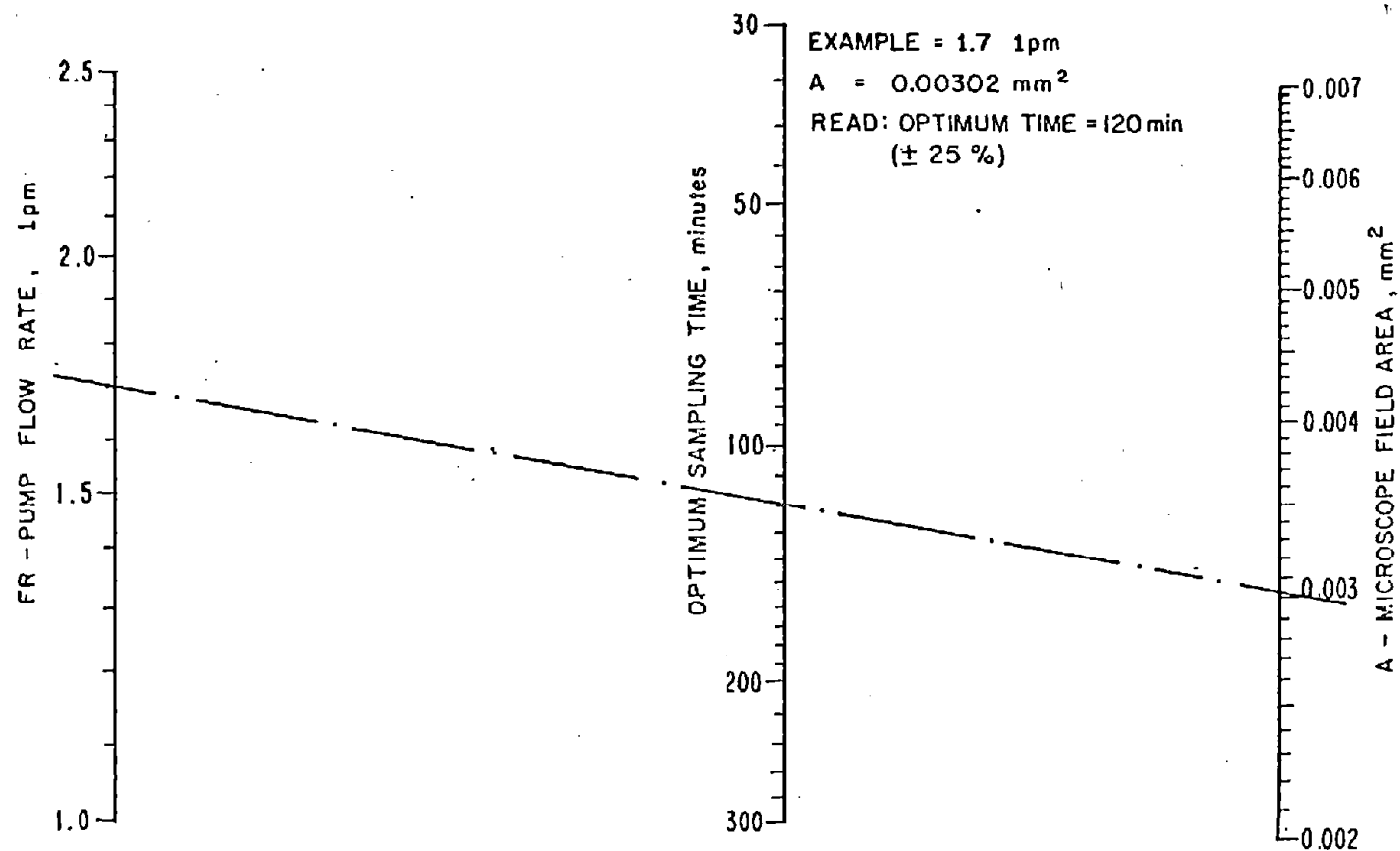


FIGURE 4. Nomogram of optimum sampling times for airborne asbestos fibers in concentrations of 1 to 10 fibers/cm<sup>3</sup>

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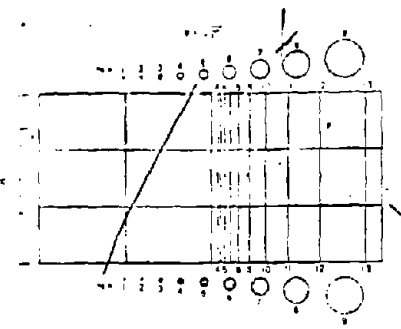


FIGURE 5

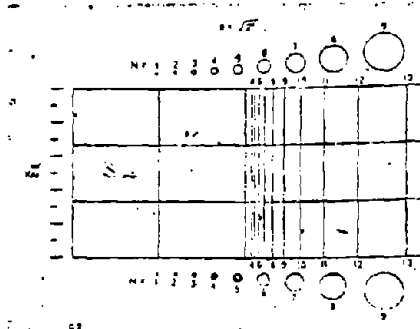


FIGURE 6

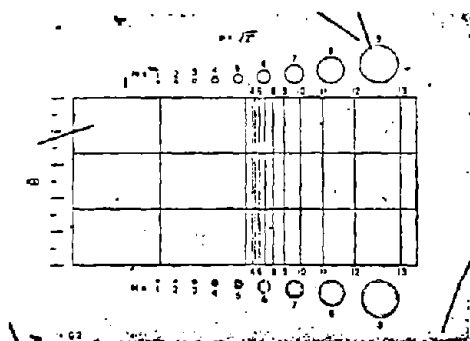


FIGURE 7

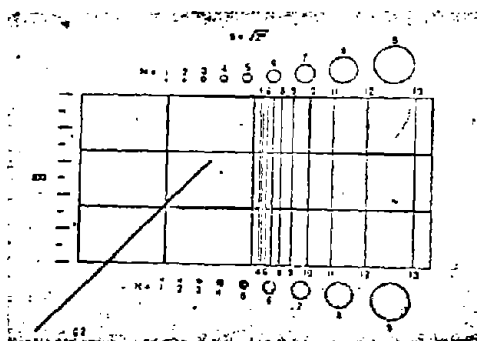


FIGURE 8

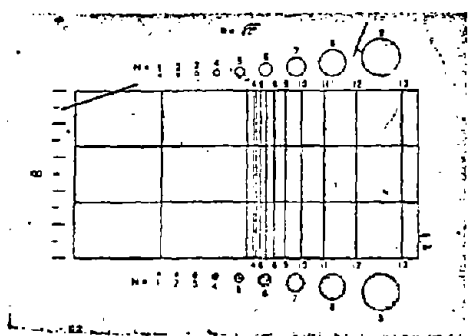


FIGURE 9

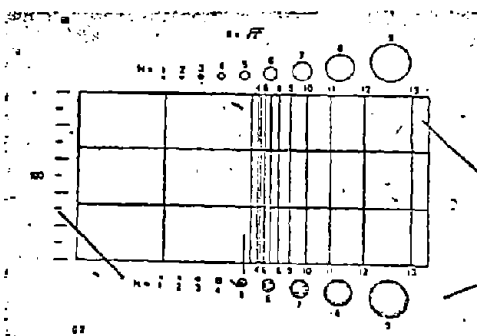


FIGURE 10

## LIST OF FIGURES

(5 through 10)

FIGURE 5. DO NOT COUNT. Fiber crosses top and bottom sides.

FIGURE 6. COUNT. One fiber.

FIGURE 7. COUNT. One-half fiber. Fiber crosses left side and one end lies within count area.

FIGURE 8. COUNT. One-half fiber. Fiber crosses bottom side and one end lies within count area.

FIGURE 9. DO NOT COUNT. Fiber crosses two sides.

FIGURE 10. DO NOT COUNT. Fiber crosses two sides (bottom left corner).

COUNT. One-half fiber. Fiber crosses bottom side and one end lies within count area.

COUNT. One fiber (top right corner).