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UC-3941

EXPERIMENTAL DETERMINATION OF THE NUMBER AND SIZE OF ASBESTOS FIBERS IN AMBIENT AIR

Final Report, Interagency Agreement (ARB 3-688)

Prepared by

Walter John, Axel Berner, Glenn Smith and Jerome J. Wesolowski

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January, 1976



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ABSTRACT

This investigation was designed to assess the problem which asbestos presents as a community air pollutant by quantifying asbestos fibers in ambient air near a mill. An array of samplers and wind sensors were operated during two periods with different prevailing wind directions. The Nuclepore filter samples were analyzed by transmission electron microscopy using a direct clearing method of sample preparation which was adapted to this work.

Asbestos fiber concentrations ranged up to 10^6 fibers/m^3 or 10^4 ng/m^3 with downwind concentrations approximately 100 times greater than those upwind. Large tangles of asbestos occurring due to the proximity of the source were excluded from the analysis. The size distributions indicated that fibers shorter than the pore size penetrated the filters. The principal source of the emissions appeared to be windblown material from open ore and tailing piles.

This report was submitted in fulfillment of Interagency Agreement ARB 3-688 by the Air and Industrial Hygiene Laboratory, California State Department of Health, under the sponsorship of the California Air Resources Board. Work was completed as of December 31, 1975.

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PROTOCOL FOR THE QUANTIFICATION OF ASBESTOS FIBERS IN AMBIENT AIR NEAR MINES AND MILLS

Introduction

This investigation was designed to assess the problem which asbestos presents as a community air pollutant by quantifying asbestos fibers in ambient air near a mill. The study by Davis Associates (1) estimated that 85% of all asbestos emissions comes from mines and mills. Furthermore over 60% of the U.S. asbestos production is located in California (2). Therefore emissions from asbestos mines and mills are of particular significance to California's air environment.

Inhalation of asbestos fibers has been related to a number of diseases including asbestosis, bronchogenic carcinoma, and mesothelioma. Thus it is important that data on the concentrations of asbestos in ambient air be collected for use in the assessment of adverse health effects. Since the mechanism of pathogenicity may depend on the size of the fibers, the data should include the fiber size distribution. The size distribution is also useful in estimating the transport of fibers from a source to a population center. Data on asbestos in ambient air are also needed for consideration of a possible asbestos air quality standard as well as asbestos emission control regulation. An overview of the problem of asbestos in the California environment has been published (3).

Methods for the identification of airborne asbestos have been developed in the Air and Industrial Hygiene Laboratory (AIHL) as well as elsewhere. These methods include the sizing of the fibers by electron microscopy. In the present work these techniques have been further developed and applied to the determination of the fiber concentrations and size distributions in the vicinity of an asbestos mill.

Project Plan

A basic objective of the project was to determine whether the airborne asbestos concentration downwind of an asbestos processing plant was significantly different from the concentration upwind. This was investigated by deploying an array of samplers and wind sensors in the vicinity of a plant. Sampling periods were planned to include two different prevailing wind directions.

The samples were analyzed by transmission electron microscopy to obtain fiber concentrations and fiber size distributions. It was necessary to carry out some development on methods of sample preparation. The samples were also scanned by optical microscopy to determine whether this simpler technique can yield results which correlate with electron microscopy data.

Sampling Site

The Union Carbide asbestos mill south of King City was selected as the site for field measurements. The plant is in the Salinas Valley adjacent to U.S.

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101 and the Southern Pacific Railway (see Fig. 1). Ore containing chrysotile asbestos is screened at the mine near Coalinga before it is trucked to the plant. It is stored in an open dump near the mill prior to processing. The plant operates on three shifts around the clock but outside operations are carried out only during the day shift.

The in-plant process is largely wet except for the hammer mill (4). Bag houses are used to control dust emissions. The tailings are dumped outside on the ground and subsequently transported by earth movers to another open dump in a draw to the east of the plant (see Fig. 1). The tailings still contain considerable asbestos.

The major sources of airborne asbestos from the plant are believed to be windblown material from the ore and tailings piles. Material can be blown directly from the piles, especially after being disturbed by the moving machinery. Clouds of airborne material could be observed over the piles. A water truck sprinkled the ore piles sporadically during our visits. On one occasion a "snow fall" of asbestos was observed in the pasture south of the plant near sampling station 1*. Asbestos could also be seen on the ground. Except for this occasion, no airborne asbestos could be seen at the sampling stations during the sampling periods.

Sampling Procedures

Sampling was carried out during two periods, August 29 to September 1, 1974 and November 12 to November 15, 1974. These periods were chosen to provide prevailing winds from northerly and southerly directions respectively. During the day, a sea breeze normally enters the Salinas Valley and flows southerly along the valley, the wind speed peaking in the afternoon. During the night or early morning this wind dies out and a weak counterflow starts as the air drains from the valley towards the Pacific Ocean. The counter-flow lasts over a longer period in the winter.

Mechanical wind stations (Meterology Research, Inc.) were used to measure the wind speed and direction. One instrument was positioned north of the asbestos plant and one south, i.e., normally upwind and downwind of the plant. The station locations are given in Fig. 1. Wind data are presented in Table 1. It can be seen that the wind direction was more consistent and the average wind speed higher in August than in November.

The air samples were taken with 47 mm diameter Nuclepore filters having a pore size of 0.8 μm. These were mounted in standard plastic Nuclepore filter holders, the open face horizontal and facing upward. The filters were 3.4 m (11' 3") above the ground on the end of a 1.9 cm I.D. (3/4") aluminum pipe. Flow rates were 50 to 60 l/min initially. The flow rates were measured at the beginning and end of the sampling period and the average used to determine the air volume. In order to measure the flow rates a plastic cylinder 20 cm (8") long was fitted over the filter head. A hot wire anemometer was positioned on the axis of the cylinder to measure

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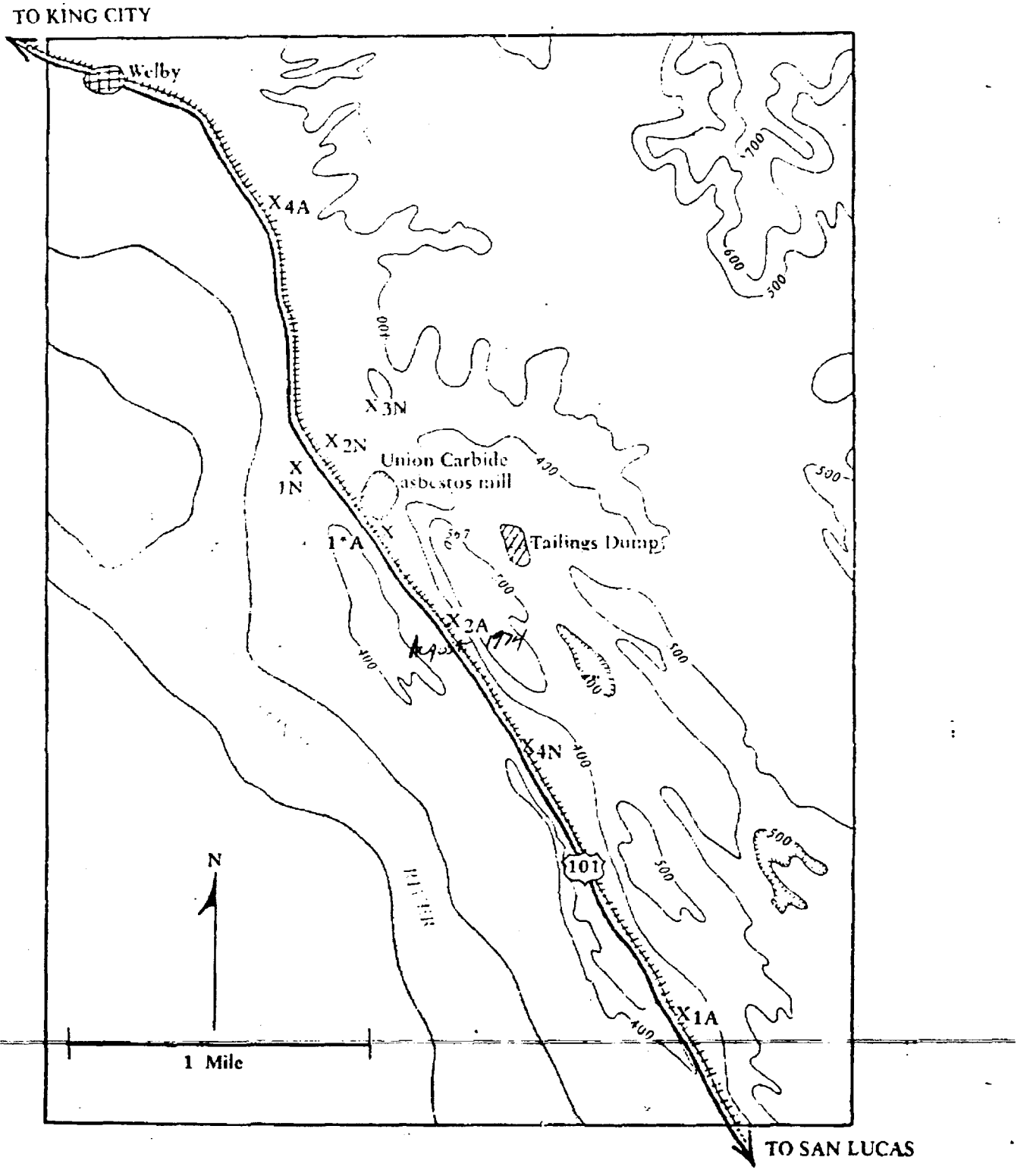


Figure 1 - Map of the sampling site. Sampling stations designated A were used in August, 1974; those designated N were used in November, 1974. Wind stations for the August sampling period were located at 1A and 3N. In November they were located at 2N and 4N.

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TABLE 1

WIND DATA TAKEN DURING THE SAMPLING PERIODS
THE STATION LOCATIONS ARE SHOWN ON THE MAP IN FIG. 1

Month	Sampling Period	Station	Wind Direction	Wind Speed MPH
August	3	2N	N	13.0
	3	1A	N	13.0
	4	2N	N	12.4
	4	1A	N	15.8
	5	2N	N	5.8
	5	1A	N	6.8
	6	2N	N	10.6
	6	1A	N	15.0
November	1	2N	W 25%, NW 50%, NE 25%	2.5
	1	4N	N 70%, E 30%	2.0
	2	2N	SE 50%, S 50%	4.2
	2	4N	S	3.8
	3	2N	W 45%, S 30%, N 25%	3.1
	3	4N	S 70%, other 30%	2.3
	4	2N	S 80%, other 20%	3.1
	4	4N	S 80%, other 20%	3.3

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the air speed. This air speed was converted to volumetric flow rate by means of a calibration curve based on laboratory calibration with a dry test meter in the same configuration.

The filters were loaded into the filter heads prior to transporting them to the field sampling stations. Blanks were carried through the entire procedure including mounting on the pump, except that they were then immediately dismounted without pumping. During the August sampling periods the Nuclepore filters were exposed on the dull side. However, during the November sampling periods the shiny side was used. It was later found under the electron microscope that the dull side has a wavy surface which collects the shadowing material forming an objectionable background for scanning.

The samplers were deployed at the locations shown in Fig. 1. Some of the pumps were powered by portable gasoline generators and some by electricity from nearby farm buildings.

Sample Preparation

In order to examine the asbestos fibers by electron microscopy it was necessary to prepare the samples in a suitable manner. The preparation technique was chosen to meet the following requirements:

a. The fibers must be identified by transmission electron microscopy and electron diffraction.

b. A quantitative determination of the number of fibers on the filter is necessary.

c. The original size of the fibers must be preserved.

It was necessary to choose a sample preparation technique from several possibilities, and to further develop the chosen technique since no technique appears at present to be sufficiently validated. The three general methods are the rubout technique, the AIHL parlodion film method, and the direct clearing method. The rubout technique (5) involves breaking up the fibers into fibrils for counting purposes. It was not used since it is incompatible with requirement (c) above. The parlodion method (6) involves ashing the sample, ultrasonification in solution, and casting a film in parlodion. This method was not used because it was feared that the ashing and ultrasonification might break up fibers.

The direct clearing method was chosen for the present work. This method appears to involve the least sample manipulation which might break up fibers. The first step involves coating the deposit of particulate matter with silicon monoxide. The fibers are thus imbedded within the coating during the remainder of the process, minimizing fiber loss. The details of the procedure used are given in Appendix 1.

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It should be emphasized that the choice of sample preparation method was based on incomplete information since none of the methods has been sufficiently tested and documented. In addition to the considerations mentioned above, the choice of the direct clearing method was reinforced by the adoption of a similar method by McCrone Associates and Jack Murchio, University of California, Berkeley. Our method differs from theirs, however, in the technique used for clearing the filter. They employ vapor washing with a Soxhlet apparatus in which the filter is mounted on a cold finger. The filter is washed by solvent condensing on the cold surface. Our clearing technique adapted from Frank, et al., (7) involves the capillary action of a polyurethane sponge to draw the solvent into contact with the filter (see Fig. 4a, Appendix 1). We believe the sponge method is less likely to wash away fibers. A direct comparison was made between the vapor-washing method and the sponge method on samples number 16 and 18 of the November sampling period. For sample No. 16, the vapor washing technique yielded 102 fibers in 40 fields vs 160 fibers in 40 fields by the sponge method. For sample No. 18, the vapor washing technique gave 29 fibers in 60 fields, the sponge method 34 fibers in 60 fields. In addition, it was noted that on the vapor-washed samples the fibers were more concentrated in the vicinity of filter debris due to incomplete dissolution. This implies that fibers were being washed away with filter material. To summarize, the data obtained suggest that fibers were lost by the vapor washing technique, although this was not proven conclusively. There were, however, additional reasons for favoring the sponge method:

a. The yield of unbroken grid holes is considerably higher

b. More samples can be cleared at the same time

c. No hood is required and a minimum of space is used with lesser possibility of contamination.

Sample Analysis

The asbestos fibers were identified and the concentrations determined by transmission electron microscopy on a Siemens Elmiskop 1A. Following accepted techniques (8-16), the fibers were identified as chrysotile asbestos by morphology, particularly by observing the central canal in fibrils at 20,000 X. Further confirmation was obtained from selected area diffraction patterns. The appearance of a large fiber bundle is illustrated in Fig. 2. In Fig. 3, the central canals in fibrils may be seen. The electron diffraction pattern for the circular area within Fig. 3 is shown in Fig. 4.

For most samples 20 holes on each of three grids were counted at 4,000 X. In order to obtain the size distributions, 13 of the samples were reanalyzed with each fiber being sized by diameter and length by direct observation on the fluorescent screen. Large tangles of asbestos were omitted since they cannot be sized by this method. The data were tabulated and analyzed by the AIHL computer program "Fiber". The program calculates the volume

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Figure 2. Morphological appearance of a non-respirable chrysotile fiber bundle (2000X).

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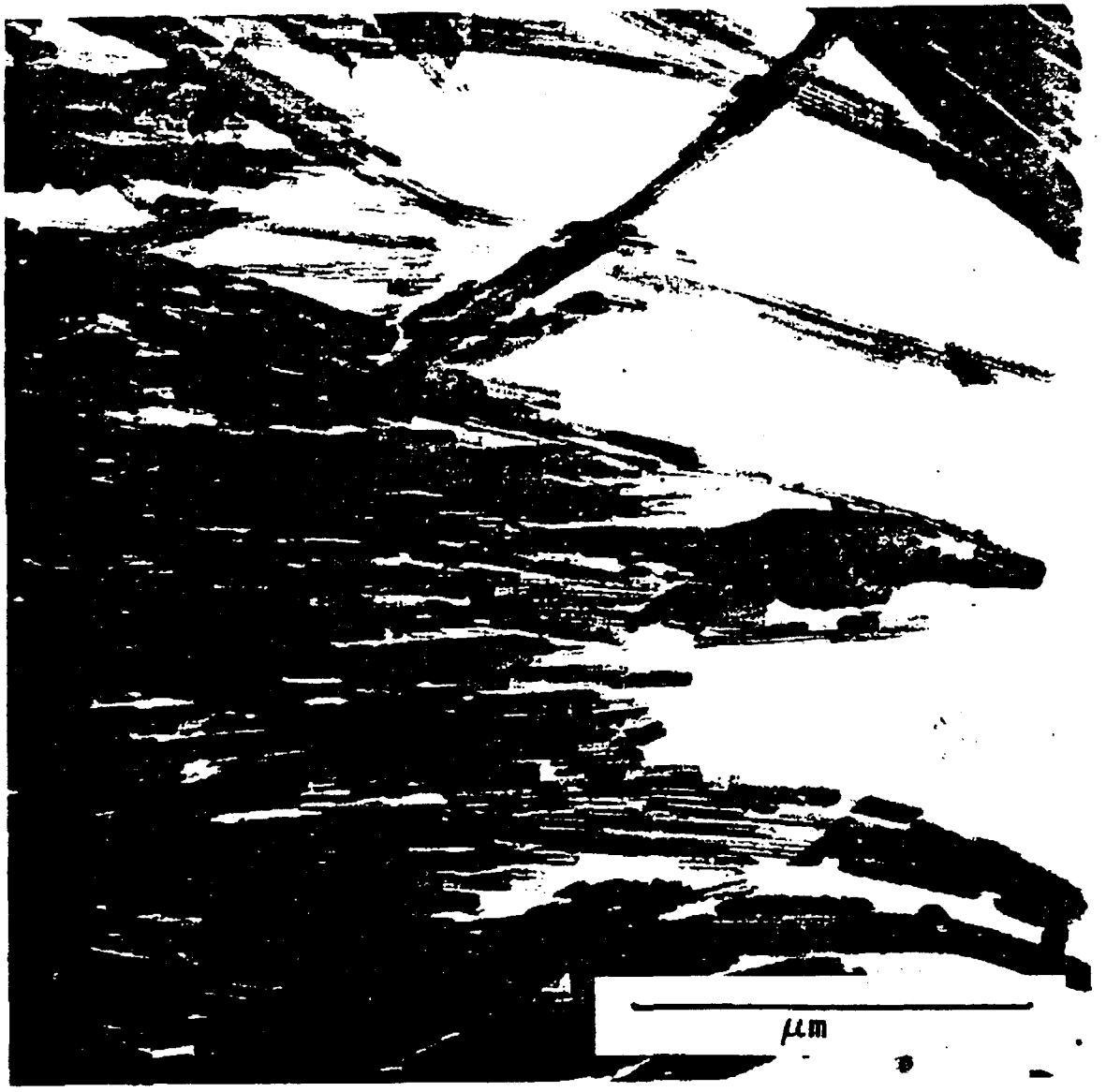


Figure 3. Morphological appearance of chrysotile fibrils with the characteristic central canals. The circle of illumination is a superimposed image of an intermediate aperture depicting the area on the crystal where the diffraction pattern was derived (20,000X).

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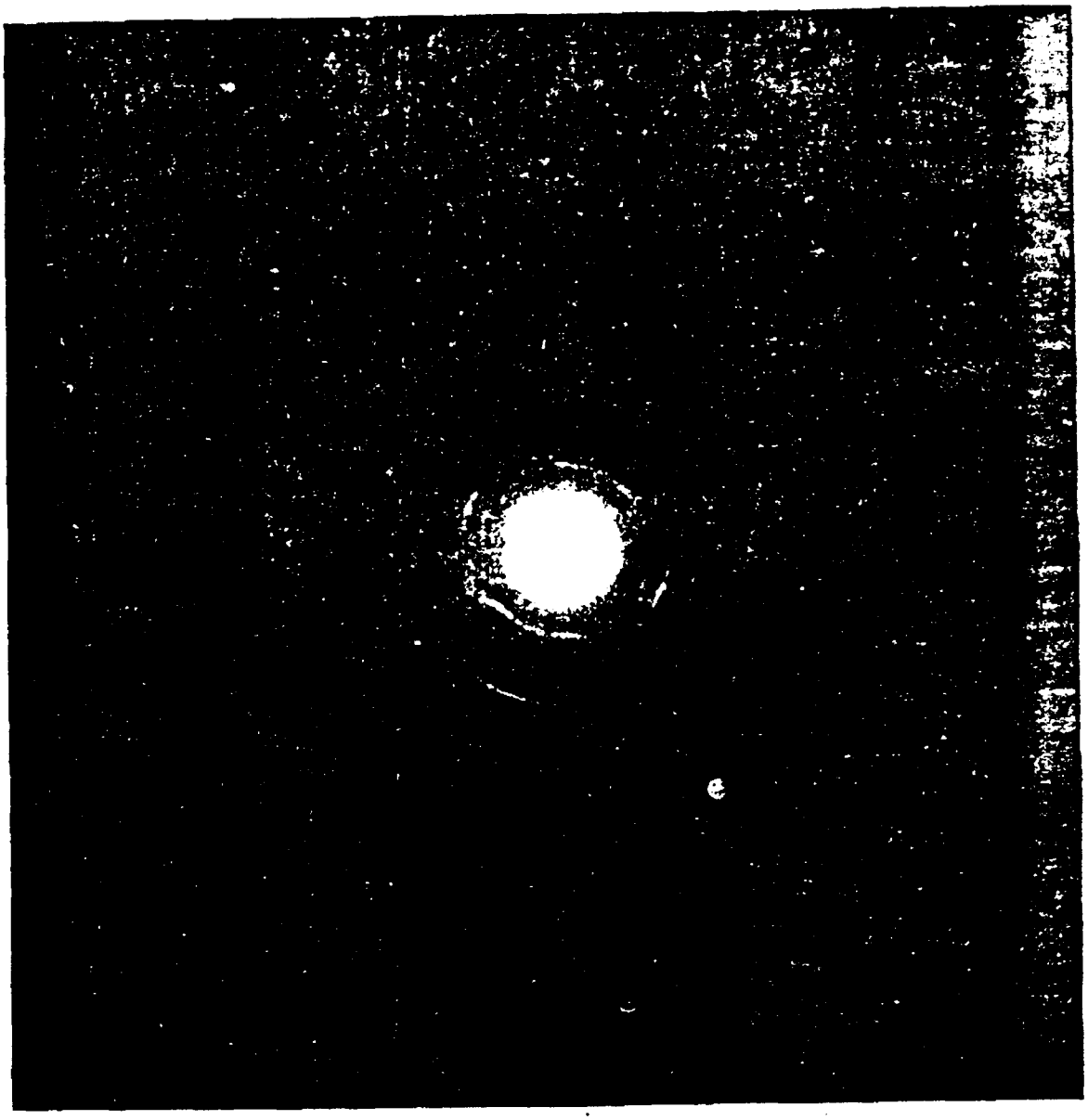


Figure 4. Characteristic selected area diffraction pattern of chrysotile obtained from the fiber bundle in figures 2 and 3.

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of a right circular cylinder of the given dimensions and computes the corresponding mass using a chrysotile density of 2.56 g/cm^3.

Results and discussion

The number of fibers/m^3 and ng/m^3 determined for the samples are listed in Tables 2 and 3. The fiber concentrations on downwind samples were of the order of 10^4 and 10^5 fibers/m^3. Referring to the August data (Table 2) it can be seen that the number of fibers/m^3 for station 4a (upwind of the asbestos plant) are approximately two orders of magnitude smaller than the numbers for downwind samples. For the November upwind samples (Table 3, Station 4N) the number of fibers was not significantly above zero. (Sampling period 1 is not included in this comparison; the wind was from the reverse direction.) The smaller upwind result for November might be due to two factors. First, the wind speed was considerably smaller in November (see Table 1). Secondly, the particulate matter loading (most of it was not asbestos) was heavier in November, amounting to about one mg per filter. This particulate matter is attributed to plowing activity in the nearby fields which took place during the November sampling period. The extra loading made it more difficult to see the asbestos fibers.

The blanks taken in November average about one fiber in 60 grid holes which is at the detection limit. This indicates that no significant contamination of the samples occurred.

As previously mentioned, the mass calculations omitted tangles of fibers. It should be pointed out that the filter samples contained large numbers of these tangles, possibly owing to the relatively short distances from the asbestos plant to the sampling stations. Of course each tangle could contain more mass than all of the other fibers on the filter. Further, the tangles could obscure individual fibers lying nearby. The tangles do not constitute respirable particles because of their large size; however, they may be a source of respirable fibers upon weathering. The fiber concentration downwind of the asbestos plant is tabulated as a function of length and diameter in Table 4 for August samples and in Table 5 for November samples. These tables show that the diameter and length tend to increase together.

The fiber concentration is plotted vs. diameter in Fig. 5 for the August samples and in Fig. 7 for the November samples. Similar plots vs. fiber length are shown in Fig. 6 and Fig. 8. The distributions vs. diameter show a peak at about 0.1 μm with the number decreasing on either side but especially rapidly towards smaller diameters. The distributions vs. length fall rapidly below 1.0 μm.

Because of the notable fall off in the number of small fibers, sample no. 16 of November was rescanned at higher magnification, namely 10,000 X for comparison to the results obtained at 4,000 X. The number of fibers found was larger, 106 vs. 62. Although the total number of fibers increased significantly, the number of small fibers did not.

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TABLE 2

Prevailing North wind

SUMMARY OF DATA FROM THE AUGUST 1974 SAMPLING PERIOD
THE ASBESTOS FIBERS WERE COUNTED AND SIZED BY
TRANSMISSION ELECTRON MICROSCOPY

Sampling Period	Sampling Station	Upwind or Downwind	Sample No.	Number of Fibers	Number Grid Holes	Total Air Volume m^3	Asbestos Concentration Fibers m^3	Asbestos Concentration ng m^3
3	1A	D	7	8	60	8.5	0.3×10^4	9.7
3	2A	D	8	312	60	7.3	12.2×10^4	310
3	4A	U	9	7	60	9.3	0.2×10^4	0.4
4	1A	D	10	17	60	7.7	0.6×10^4	-
4	2A	D	11	200	60	7.3	7.8×10^4	-
4	4A	U	12	0	60	6.7	< 400	-
5	1*A	D	13	670	30	7.9	48×10^4	-
5	2A	D	14	145	60	7.1	5.8×10^4	-
5	4A	U	15	1	60	7.9	0.04×10^4	-
6	1*A	D	16	-	-	11.2	-	-
6	2A	D	17	346	30	11.0	17.9×10^4	7700
6	4A	U	18	3	60	7.9	0.1×10^4	171

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Prevailing Southwind

TABLE 3

SUMMARY OF DATA FROM THE NOVEMBER 1974 SAMPLING PERIOD
THE ASBESTOS FIBERS WERE COUNTED AND SIZED BY
TRANSMISSION ELECTRON MICROSCOPY

Sampling Period	Sampling Station	Downwind or Upwind	Sample No.	Number of Fibers	Number Grid Holes	Total Air Volume m^3	Asbestos Concentration Fibers $/m^3$	Asbestos Concentration ng $/m^3$
1	1N	U	1	51	60	13.5	1.1×10^4	48
1	2N	U	2	2	60	11.7	0.02×10^4	0.3
1	3N	U	3	1	60	10.8	< 300	0.03
1	4N	D	4	290	3	10.3	160×10^4	2680
2	1N	D	5	26	30	12.6	1.1×10^4	166
2	2N	D	6	160	40	12.3	5.5×10^4	-
2	3N	D	7	197	60	15.0	3.7×10^4	225
2	4N	U	8	1	60	16.1	< 200	-
Blanks	-	-	9	2	60	-	-	(0.9 ng)
"	-	-	10	0	60	-	-	-
"	-	-	11	3	60	-	-	(1.2 ng)
"	-	-	12	0	60	-	-	-
3	1N	D	13	56	60	8.8	1.8×10^4	801
3	2N	D	14	101	60	8.4	3.4×10^4	-
3	3N	D	15	62	60	8.8	2.0×10^4	264
3	4N	U	16	0	60	7.4	< 400	-
4	1N	D	17	51	60	10.9	1.3×10^4	-
4	2N	D	18	34	60	10.5	0.9×10^4	-
4	3N	D	19	35	60	10.5	0.9×10^4	-
4	4N	U	20	1	60	9.2	< 300	-

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TABLE 4

NUMBER OF FIBERS IN 90 GRID HOLES VS LENGTH AND DIAMETER
FOR SAMPLES NO. 8 AND 17, AUGUST 1974
(STATION 2A, DOWNWIND)

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DIAMETER, μm

LENGTH, μm	.05-.10	.10-.15	.15-.30	.30-.60	.60-.80	≥ .80
0.5-1.0	12	5	7	-	-	1
1.0-2.0	11	48	45	14	1	1
2.0-3.0	1	18	24	30	5	3
3.0-5.0	1	9	33	34	18	17
5.0-8.0	1	5	18	17	24	37
> 8.0	1	2	13	34	22	97

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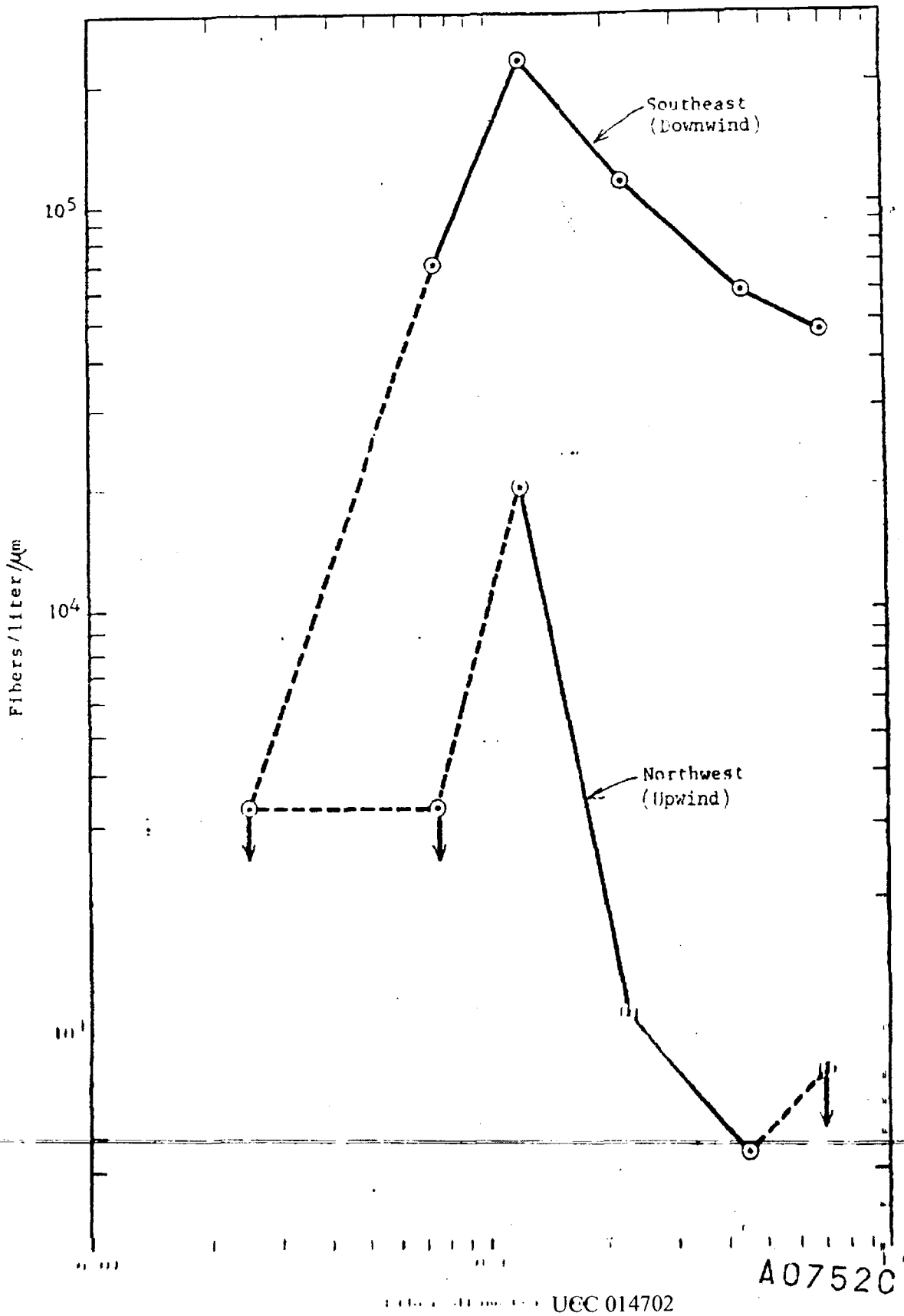
TABLE 5

NUMBER OF FIBERS IN 210 GRID HOLES VS LENGTH AND DIAMETER
FOR SAMPLES NO. 5, 7, 13 AND 15, NOVEMBER 1974
(STATIONS 1N AND 3N, DOWNWIND)

LENGTH μm	DIAMETER, μm					
	.05-.10	.10-.15	.15-.30	.30-.60	.60-.80	> .80
0.5-1.0	1	1	-	-	-	-
1.0-2.0	2	9	15	1	-	-
2.0-3.0	2	8	11	9	-	1
3.0-5.0	-	19	29	22	3	4
5.0-8.0	-	7	21	34	6	5
> 8.0	1	3	8	31	13	14

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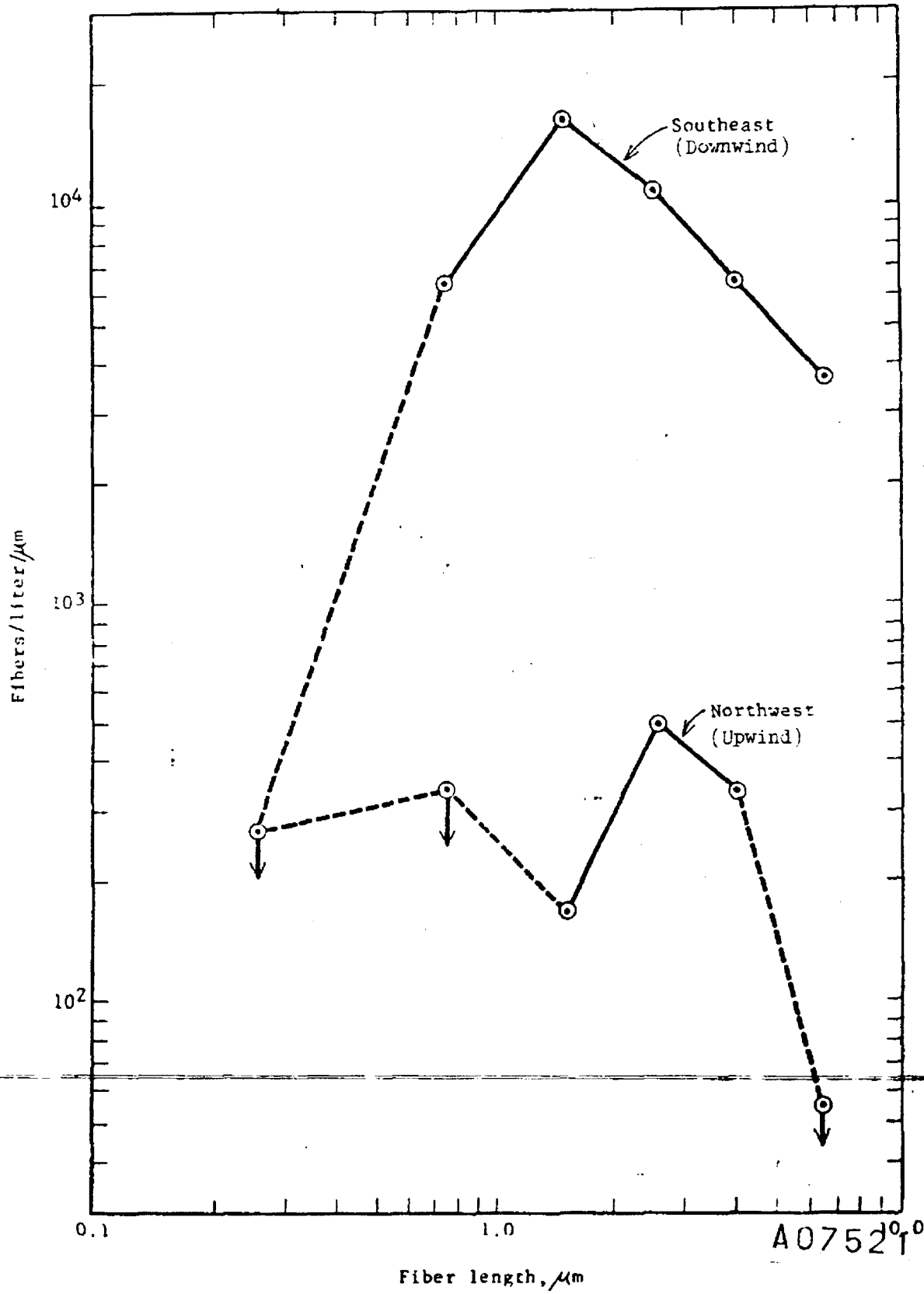
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Figure 6 - Fiber concentration vs length for samples taken in

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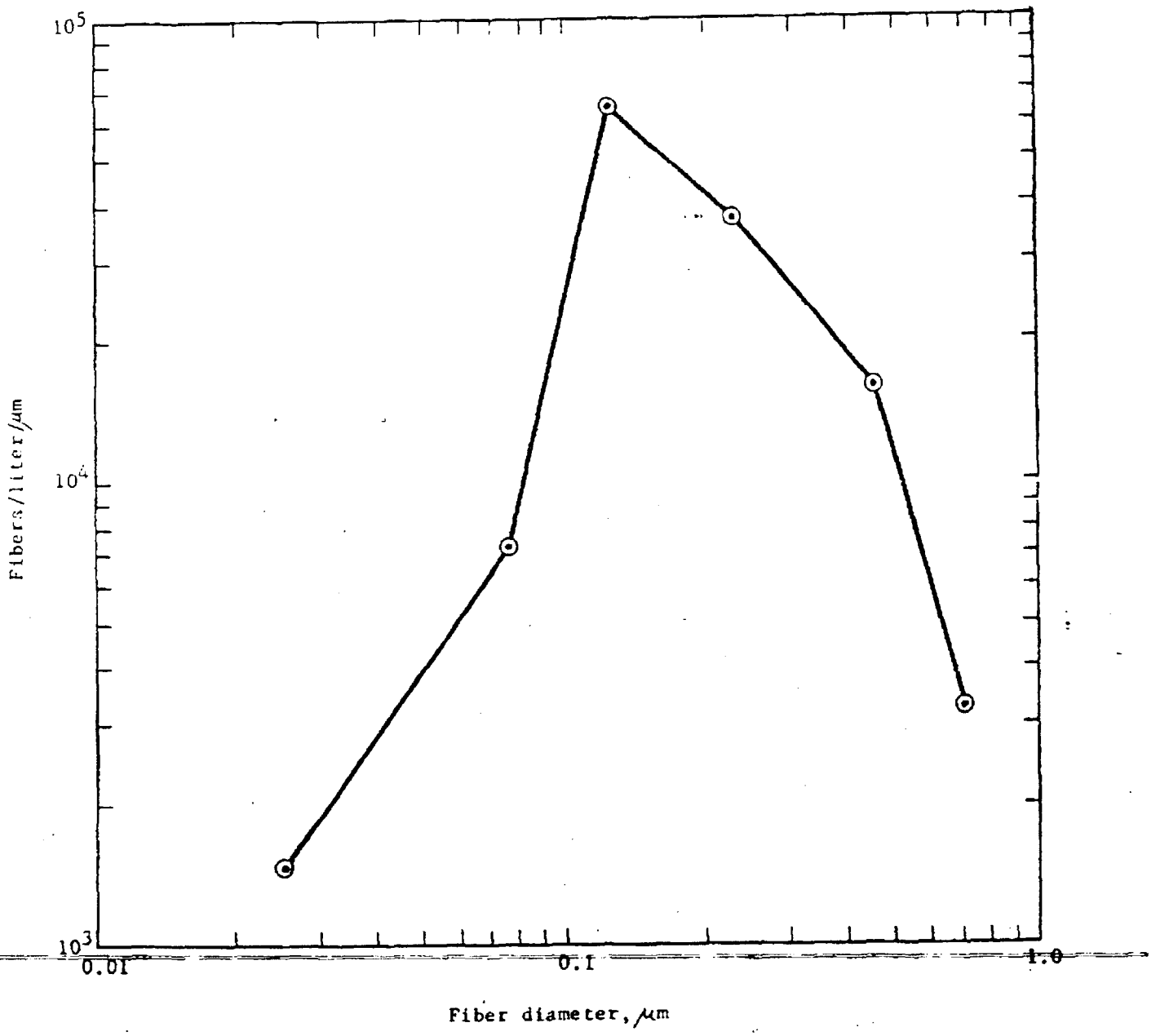


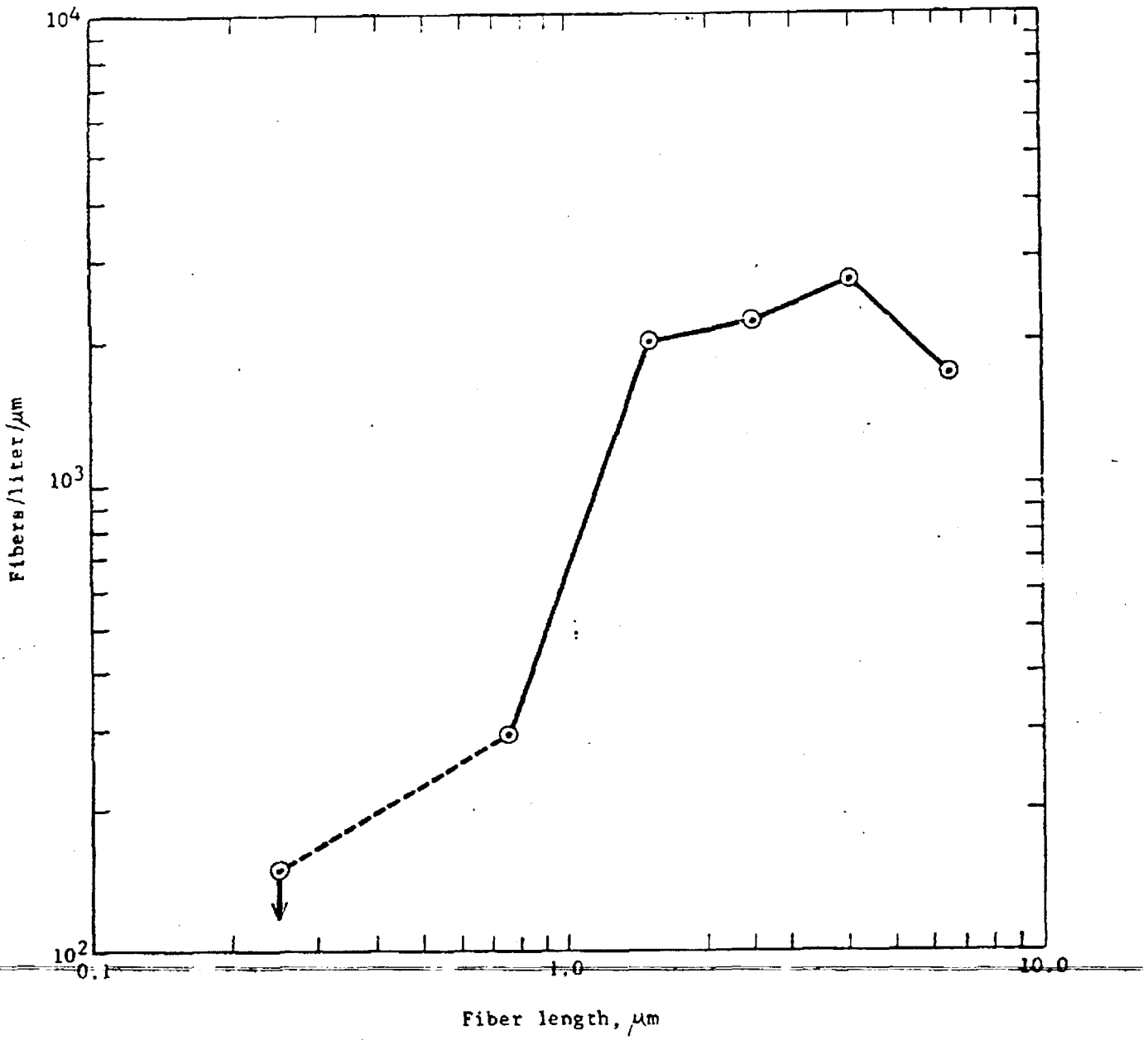
Figure 7 - Fiber concentration vs diameter for samples taken in November, 1974. Data are shown for stations downwind of the asbestos plant: almost no fibers were found on the upwind samples.

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Figure 8 - Fiber concentration vs length for November samples. Data are shown for stations downwind of the asbestos plant.

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It is believed that the absence of small fibers may be attributed to the use of 0.8 μm pore size Nuclepore filters. Unlike other filters which present tortuous paths to the aerosol, the Nuclepore holes are uniform circular holes directed in a straight path from front to back. Therefore the Nuclepore filter can be penetrated by particles smaller than the pore size. This would account for the rapid decrease in fibers shorter than 0.8 μm. Since the diameters decrease with the length, the absence of short fibers will result in an absence of fibers with small diameters also. An interesting question concerns the possibility that fibers could become aligned with their lengths parallel to the streamlines and thus readily penetrate the pores. Such alignment could be produced by the shear flow in the vicinity of a pore or by electrostatic fields. At present there is insufficient knowledge of these phenomena to answer the question.

In Fig. 9 the fiber concentrations are plotted against the wind speed. Although the scatter is large, there is a rough correlation between fiber concentration and wind speed (correlation coefficient = 0.59). The observed increase of concentration with wind speed implies that the fibers are wind-generated since otherwise the concentration would decrease because of dilution. The data are in agreement with our expectation that windblown material from the ore and tailings piles are the main source of emissions, based on visual observations.

It is instructive to compare the present results with those obtained in 1972 by Smith, et al (16). The plots in Fig. 10 and 11 show corresponding data. The comparison shows that the fiber concentrations were fairly consistent in magnitude above 0.1 μm diameter and 1.0 μm length. The earlier work utilized Millipore filters; the strikingly higher concentrations for small fibers is strong evidence that these fibers penetrate the 0.8 μm pores of Nuclepore. The plots of the 1974 results indicate somewhat higher concentrations of large fibers than the 1972 measurements. This could be due to breakdown of large fibers by high temperatures and/or ultrasonification in the 1972 sample preparations by the parlodion method.

Light microscopy

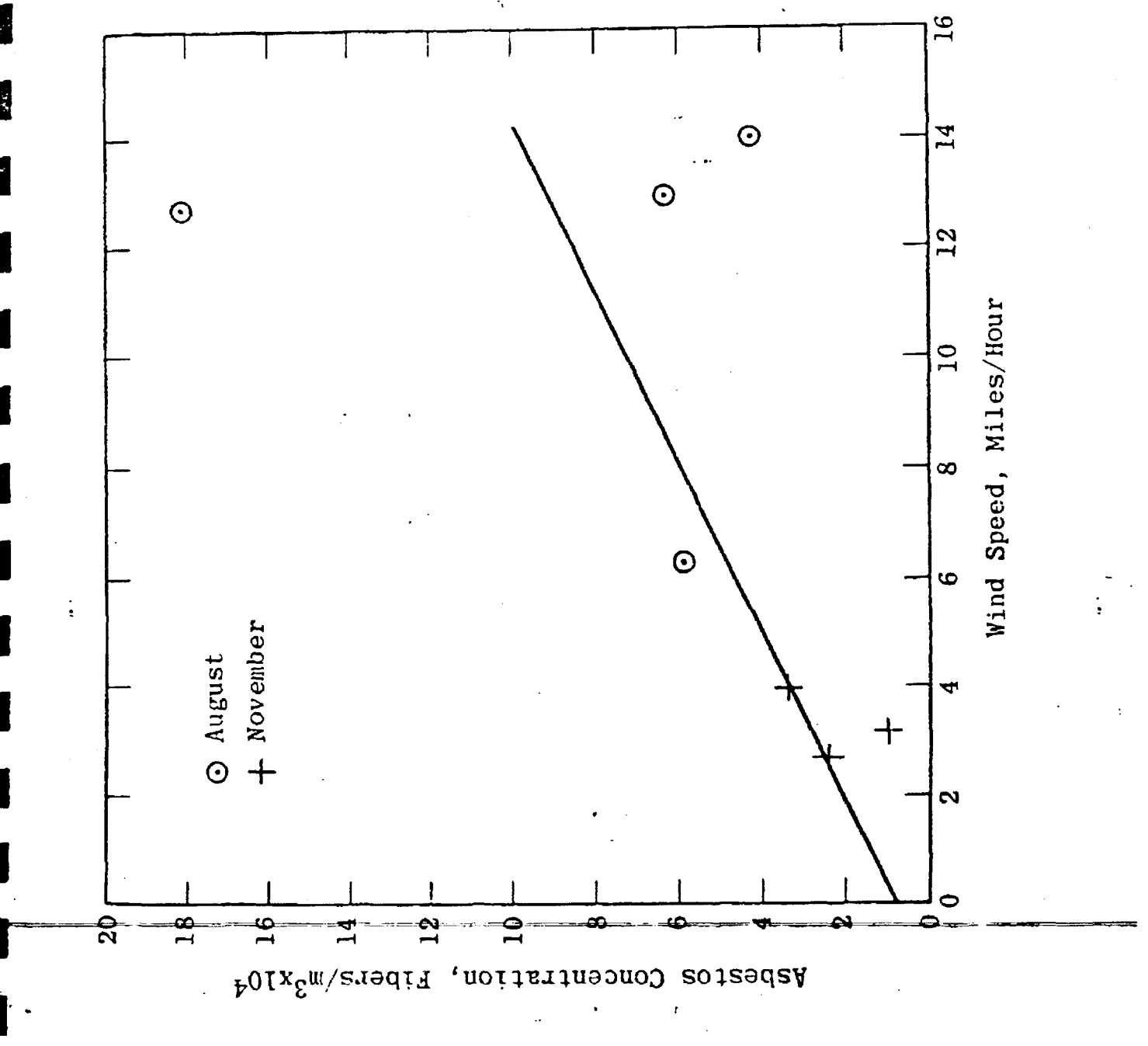
The samples were also analyzed by light microscopy because it is a relatively rapid, inexpensive technique which is well standardized. If light microscopy could be shown to yield results sufficiently quantitative, this would be important for future work.

The standard NIOSH (17) method was used except for the employment of a different clearing agent, 1, 1, 2, 2 tetrachlorethane which is effective for Nuclepore. (Precautions should be taken against the noxious fumes.) Briefly, the NIOSH method involves counting all fibers greater than 5 μm in length which have a length-to-diameter ratio greater than 3. For each sample 100 fields are counted at 400X.

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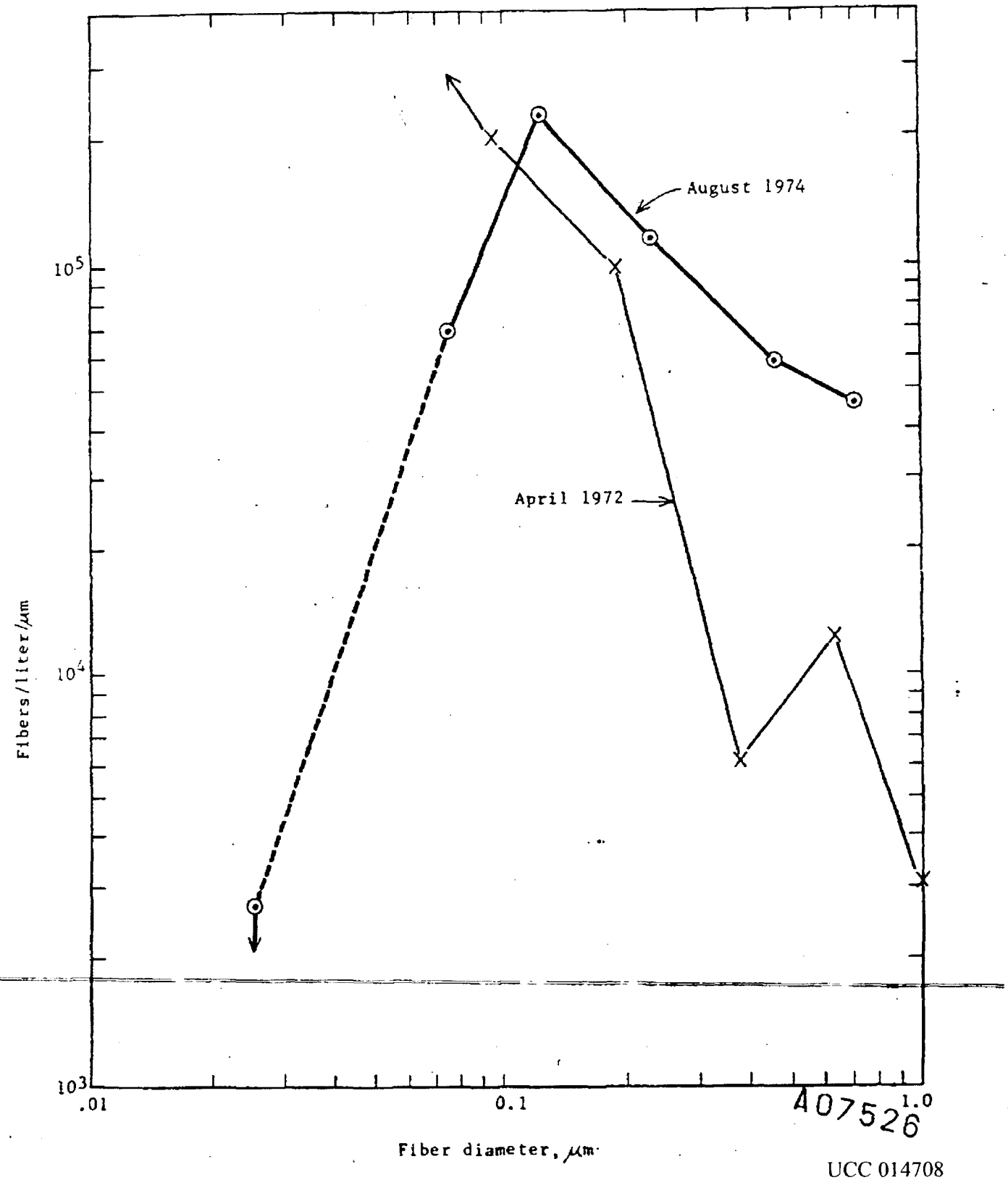
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Figure 9 - Fiber concentration vs wind speed. The line is a least-squares fit to the data points.

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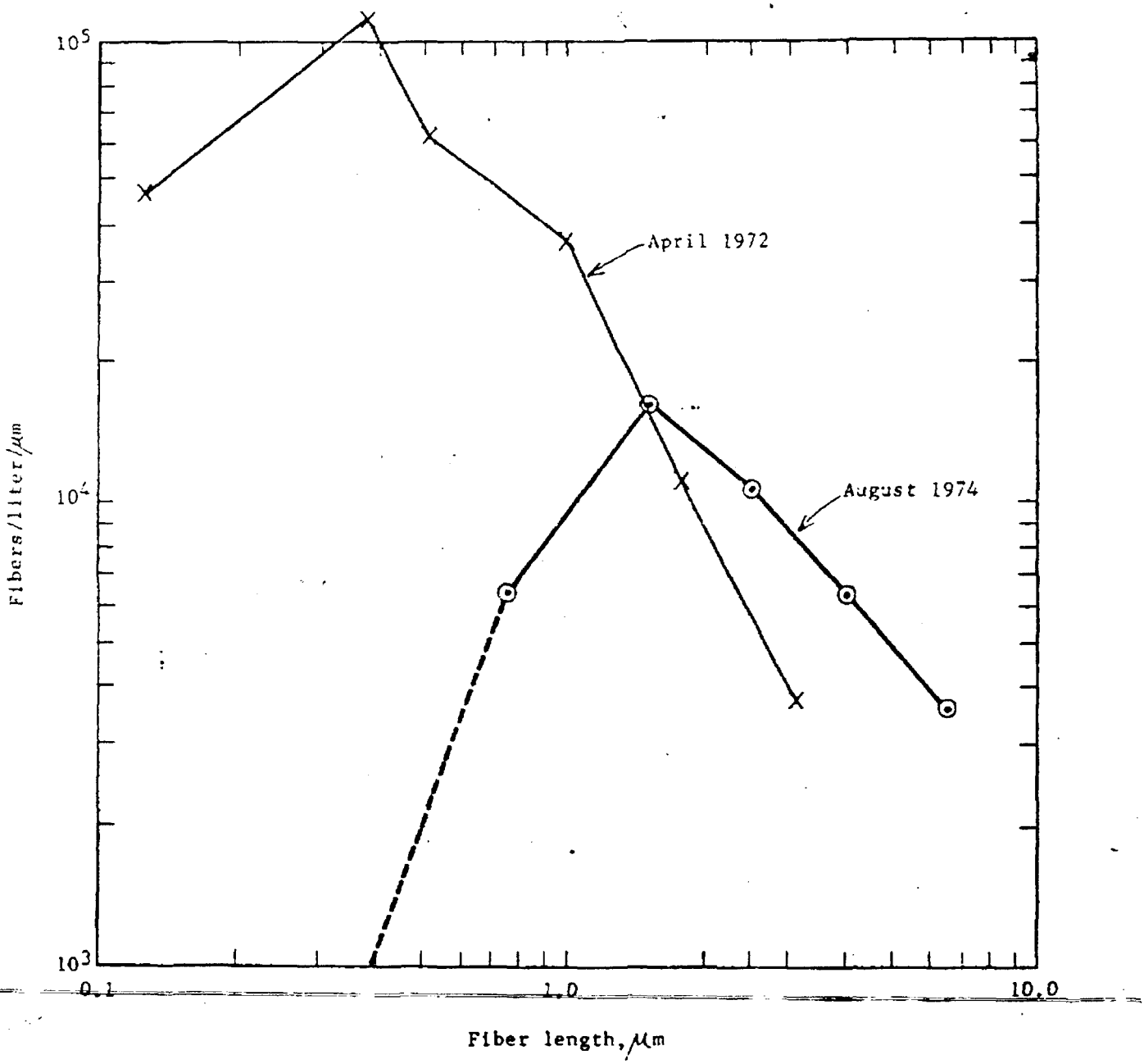


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Figure 10 - Comparison of the fiber concentration vs diameter found in August, 1974 (downwind stations) with that reported by Miller, et al., for April, 1972.

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Figure 11 [illegible] UCC 014709
in August, 1974 (theoretical plot based on data reported
by Mueller, et al., for April, 1972.

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The observed concentrations are listed in Table 6. The numbers are seen to be very low. The downwind - upwind difference is small except in a few cases. Furthermore, there is very poor correlation with the electron microscope results (Table 2 and 3).

There are a number of reasons which could explain why the NIOSH method is apparently ineffective for this work. One is that most of the fibers are smaller so that only the end of the size distribution is counted. The relatively heavy loading of particulate matter made it difficult to see asbestos. The tangled masses were not counted since they did not satisfy the NIOSH counting criteria. In general, the NIOSH method is non-discriminating, no attempt being made to identify the fibers. All particles which have the prescribed dimensions are counted as fibers.

Conclusions

1. The asbestos fiber concentration near an asbestos plant has been found to range up to 10^6 fibers/m^3 or 10^4 ng/m^3.

2. Fiber concentrations downwind of the asbestos plant were approximately two orders of magnitude greater than those upwind; upwind concentrations were barely detectable.

3. The principal source of the emissions appears to be windblown material from open ore and tailings piles.

4. Sampling within one mile of the source resulted in the deposition of large tangles of asbestos on the filters. These were simply excluded from the present analysis since the technique used could not accommodate such tangles. Although this means that the calculated masses are low, the excluded tangles are not respirable. In future work if size distributions are desired it might be better to sample at greater distances from the source. If only total mass were desired, the rubout technique could be used.

[illegible]

6. The direct shearing method of sample preparation for electron microscopy was further developed and applied to this work. However, it cannot be said that this method is sufficiently validated for routine work. The same conclusion applies to all other sample preparation methods which attempt to preserve the fiber size distribution.

7. Light microscopy utilizing the NIOSH method was found to be ineffective for evaluating asbestos fiber concentrations in ambient air.

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TABLE 6

FIBER CONCENTRATIONS DETERMINED BY LIGHT MICROSCOPY

August		November	
Sample Number	Fibers m^3	Sample Number	Fibers m^3
7	16	1	0
8	26	2	3
9	12	3	0
		4	3
10	19		
11	108	5	3
12	42	6	8
		7	6
13	415	8	0
14	92		
15	34	13	17
		14	159
16	330	15	29
17	77	16	67
18	17		
		17	49
		18	50
		19	86
		20	80

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We appreciate the support of the Air Resources Board and the encouragement by Jack Suder. Arndt Lorenzen supplied wind data.

It is a pleasure to acknowledge the friendly cooperation of John Maloney and Edward Munson of the Monterey Bay Unified Air Pollution Control District.

We are grateful to John Layous for permission to operate samplers on his property.

Les Carpenter assisted with the field sampling. Margarita Dorland assisted with sample preparation and carried out the light microscopy. SuzAnne Twiss supplied computer support.

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APPENDIX

AIHL Method

DETERMINATION OF ASBESTOS IN AMBIENT AIR UTILIZING NUCLEPORE FILTERS AND TRANSMISSION ELECTRON MICROSCOPY

Analyte:	Asbestos	Method No.:	68
Matrix:	Ambient air	Lower limit of detection:	$3X10^3$ fibers per filter
Procedure:	Transmission Electron Microscopy	Precision:	Not determined
Date:	8/1/75	Collaboratively Tested:	No

1. Summary of Method

In order to perform asbestos analysis on air samples in a simple and rapid manner by transmission electron microscopy, a direct membrane clearing method of sample preparation has been developed.

The sample is collected on 47 mm diameter Nuclepore membrane filters at a rate of 10 liters per minute for three to twenty-four hours.

A one-eighth area sector is cut from the filter with a cutting tool, shadowed with silicon monoxide, and 3 squares 3 mm per side are cut from the shadowed filter sector.

Prepared by G. R. Smith, Air and Industrial Hygiene Laboratory, State of California Department of Health, Berkeley, California 94704, August 1975

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Each is placed face down on a bare 200 mesh electron microscope grid, and the filter dissolved by placing the grid, filter up, on a polyurethane sponge immersed in chloroform for 72 hours.

The grids are dried in a dust-free chamber prior to electron microscopic examination. Each of the three grids is examined at 4,000X in the electron microscope. Twenty grid holes per grid are examined and each asbestos fiber sized on the calibrated fluorescent viewing screen. Typical fibers are identified by morphology and selected area diffraction at 20,000X. (9.1)

2. Range and Sensitivity

2.1 The lower detectable limit is one fiber per 60 grid holes.

2.2 The range is 3×10^3 fibers to 4×10^6 fibers per sample.

3. Interferences

3.1 Fibrous clay minerals (9.2), i.e., the tubular fibers-kaolin and halloysite, and solid fibrous attaclay; gypsum (9.3) and fibrous talc (9.3).

4. Precision and Accuracy

4.1 The precision and accuracy has not been determined.

4.2 The precision is dependent on the number of fibers counted, the number of fields counted, and the number of fibers per field. This is also true of the NIOSH procedure for light microscopic counting of asbestos fibers, which claims an interlaboratory

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variation of about ±50% when the same sample is counted by different analysts.

5. Apparatus

5.1 Nuclepore filters 47 mm. (9.4)

5.2 Nuclepore filter holders 47 mm. (9.4)

5.3 Air pump with capacity of 40 liters per minute, Gast (9.5).

5.4 Portable, gasoline powered electric generators with 2200 watts capacity (9.6).

5.5 Tungsten wire baskets, Ladd (9.7).

5.6 Vacuum evaporator with rotating stage, Mikros (9.8).

5.7 Vented grid drying chamber, 2-3 liter capacity desiccator. (See illustration - Fig. 3).

5.8 White porcelain chip.

5.9 Diffusion pump oil, Diffelen.

5.10 Clean air bench with laminar air flow. (9.9)

5.11 Electron microscope grids, 2.3 mm diameter, 200 mesh, copper with square holes, Pelco. (9.10)

5.12 Transmission electron microscope 40 to 100 kv, Siemens. (9.11)

6. Chemicals

6.1 Chloroform; analytical reagent. (9.12)

6.2 Glass-distilled water.

6.3 Silicon monoxide chunks, Ladd. (9.7)

7. Procedure

7.1 Collection of Samples

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7.1.1 Assemble the filters in the filter holders with the smooth surface showing.

7.1.2 Connect the sample holders to the air pump and collect a 1200 to 7200 liter sample.

7.1.3 After sampling remove the filter and store it in a plastic Petri dish.

7.2 Analysis

7.2.1 Excise a one-eighth sector from the sample filter with a scalpel and tape down all sides to a microscope slide using adhesive transparent tape. See figure 1, step 1 and a.

7.2.2 Place sample slide in a vacuum evaporator and rotary shadow at vertical incidence with 10 nm of silicon monoxide, figure 1, step 2.

7.2.3 Remove the shadowed filter with a scalpel, excise three squares each 3 mm per side, figure 2, step 3.

7.2.4 Put three polyurethane blocks in a 50 mm glass Petri dish and place a bare grid on top of each sponge, figure 2, step 4.

7.2.5 With forceps, place one shadowed square on top of each grid, keeping the coated particle side down (membrane up), figure 2, step 4.

7.2.6 Add chloroform to the Petri dish and fill to within 2 mm of the top surface of the sponges, figure 2, step 4a.

7.2.7 Cover the dish and place the entire Petri dish assembly in a vented chamber (desiccator) and let stand for 72 hours to dissolve the filter material.

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7.2.8 Remove the grids from the Petri dishes and place upon a Whatman 25 mm filter paper contained within a 50 mm plastic Petri dish. Then cover.

7.2.9 Place the specimen grids in a drying chamber and let them air dry one hour after partially removing the covers.

7.2.10 Examine completely 20 random grid holes on each of three grids at 100 kv and a magnification of 4,000X with TEM.

7.2.11 Count and determine the dimensions of all asbestos fibers (length and width) having an aspect ratio of 3:1, or greater length to diameter.

7.2.12 Identify chrysotile asbestos by morphology (observation of the characteristic canal) and by selected area diffraction when possible at 20,000X.

7.2.13 Identify amphibole asbestos by morphology and by selected diffraction (pattern recognition) at 20,000X.

8. Calculations

8.1.1 Determine the number of fibers per filter.

$$N = \frac{An}{ag}$$ where

N is the number of fibers per filter.

A is the area in mm^2 of the filter on which the sample was collected (usually about 1400 mm^2 for a 47 mm (Nuclepore filter).

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n is the total number of fibers counted in g.

g is the number of grid holes examined.

a is the area in mm^2 of a grid hole (usually determined by measuring a photograph of known enlargment).

8.1.2 Determine the number of fibers per milliliter of air.

$$C = \frac{N}{V}$$

Where;

N = Number of fibers per filter

V = Volume of air sampled in milliliters at standard conditions

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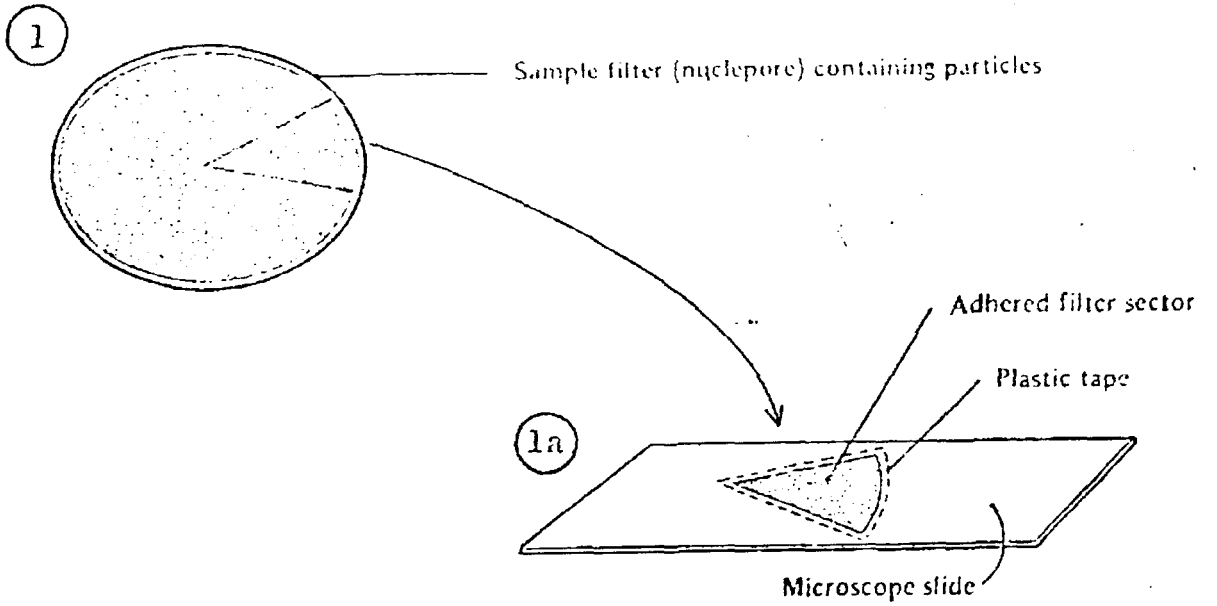
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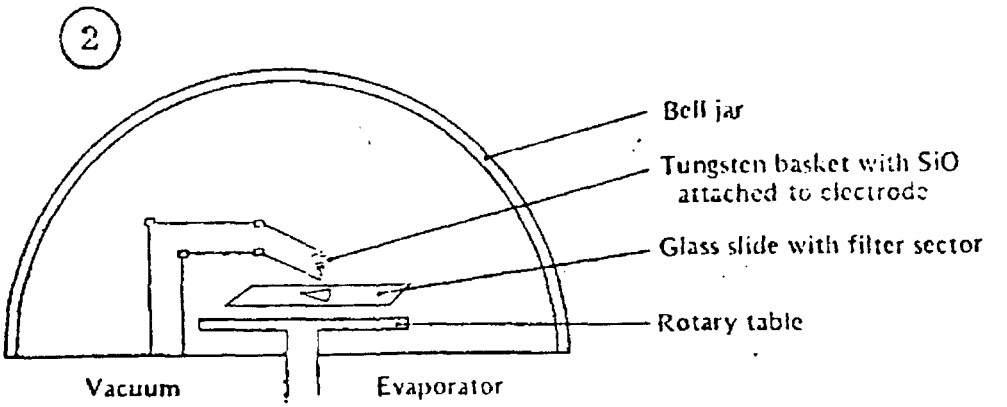
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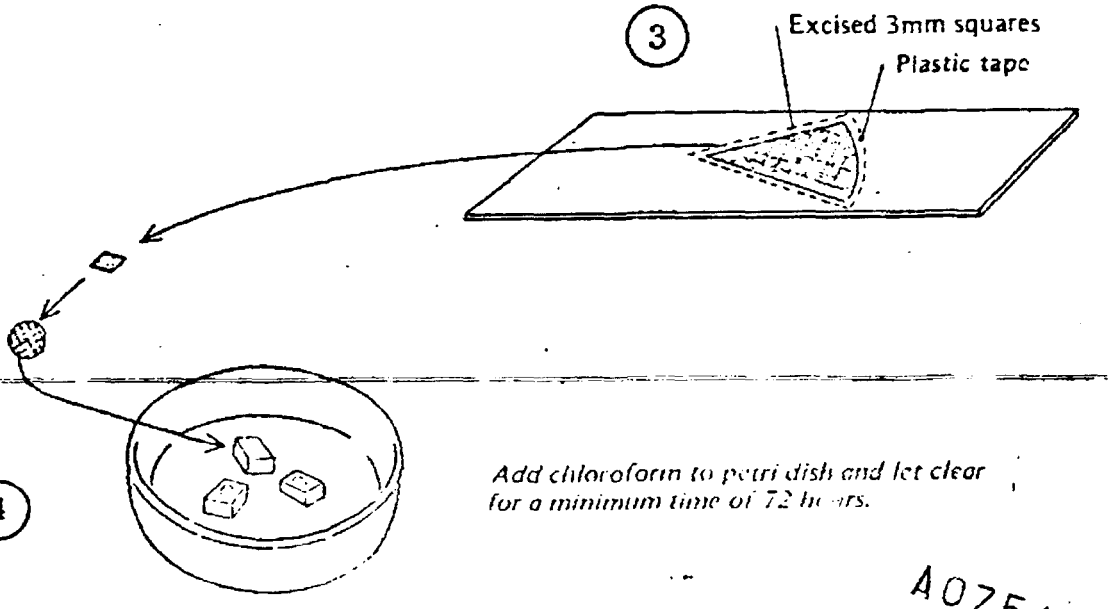
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(4a)

Add chloroform to petri dish and let react for 72 hours.

(5)

Dry grids in drying chamber one hour before examination.

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