

MEASUREMENT OF
AIRBORNE ASBESTOS FIBER
BY THE
MEMBRANE FILTER METHOD

ASBESTOS TEXTILE INSTITUTE
(FOUNDED 1944)

PLAINTIFF'S EXHIBIT

ASA-766

CONTENTS

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SCOPE	
DEFINITION	
OUTLINE OF METHOD	
APPARATUS AND MATERIALS	
CLEANLINESS	
PREPARATION OF MOUNTING SOLUTION	
AEROSOL FIELD MONITOR AND FILTERS	
CALIBRATION OF EQUIPMENT	
SAMPLING	
COUNTING	
CALCULATION OF RESULTS	
INDUSTRIAL HYGIENE WORK SHEET	
BIBLIOGRAPHY	

CONTENTS

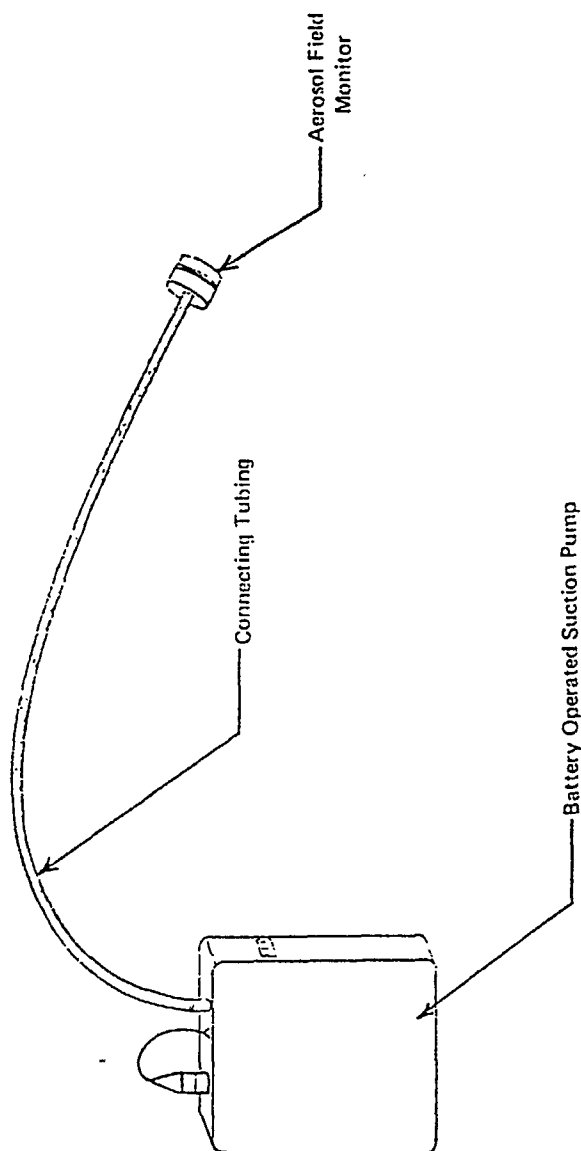
	Page
SCOPE	5
DEFINITION	5
OUTLINE OF METHOD	5
APPARATUS AND MATERIALS	5
CLEANLINESS	7
PREPARATION OF MOUNTING SOLUTION	7
AEROSOL FIELD MONITOR AND FILTERS	7
CALIBRATION OF EQUIPMENT	7
SAMPLING	10
COUNTING	11
CALCULATION OF RESULTS	14
INDUSTRIAL HYGIENE WORK SHEET	15
BIBLIOGRAPHY	16

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SAMPLING EQUIPMENT
USED IN THE
MEMBRANE FILTER METHOD



1. SCOPE:

- 1.1 This method covers a procedure for the determination of fiber concentration in air. The procedure is used for the determination of concentration using phase contrast microscopy.

This method is also useful for the determination of fiber concentration for fibers less than 5 microns, the minimum length by the inherent limitations of ordinary phase contrast optics. Further, this method is used for determinations only in areas where the procedure is predominantly or entirely asbestos. All fibers detected by this procedure are reported as such, unless otherwise warranted by other assumptions.

This method can be used to assess fiber concentrations to standards as set forth in various statutory requirements for Asbestos.

2. DEFINITION:

- 2.1 A fiber is any particle having a length greater than 5 microns.

3. OUTLINE OF METHOD:

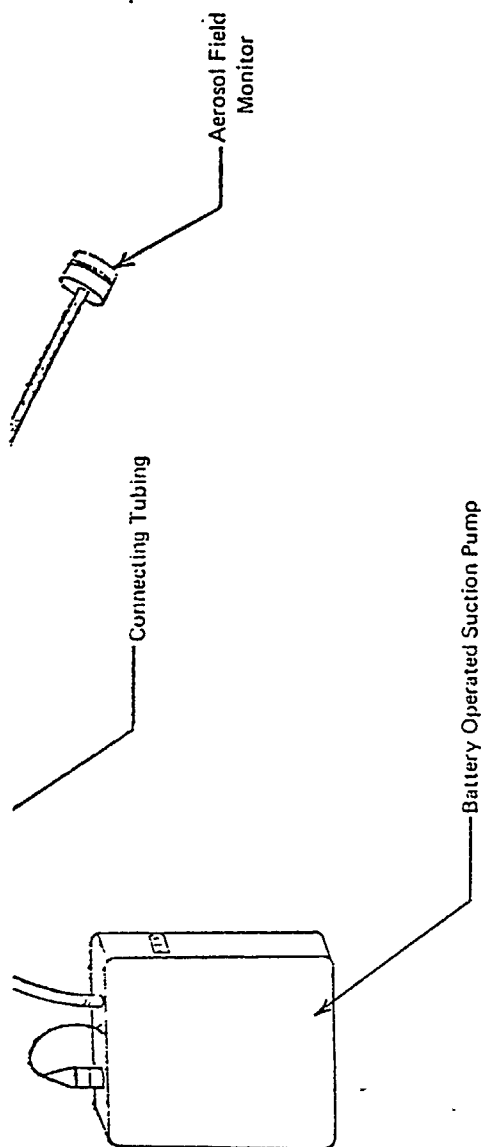
- 3.1 Airborne asbestos fibers are collected from air through a membrane filter by which fibers are deposited on the filter.

A representative section of the filter is placed on a microscope slide, and examined at 400x (or 400x) using phase contrast optics. The number of fibers observed in a given area under this figure is used to calculate the number of fibers per cubic meter (cc.) of air sampled.

Fiber concentrations are categorized in the following manner; total number of fibers, and greater than 10 microns per cubic meter.

4. APPARATUS AND MATERIALS:

- 4.1 Sample Collector:
The samples shall be collected on a filter.



1. SCOPE:

- 1.1 This method covers a procedure for the determination of asbestos fiber concentration in air. The procedure is designed specifically for the determination of concentrations of fibers 5 microns or longer using phase contrast microscopy.

This method is also useful for the determination of fibers shorter than 5 microns, the minimum length detectable being limited only by the inherent limitations of ordinary light microscopy using phase contrast optics. Further, this method is meant to be used for fiber determinations only in areas where airborne fibers, if present, are predominantly or entirely asbestos fibers; it does not specify procedures for distinguishing between asbestos and other types of fibers. All fibers detected by this procedure are assumed to be asbestos fibers and are reported as such, unless independent evidence is at hand to warrant other assumptions.

This method can be used to assess conformance of asbestos fiber concentrations to standards as set forth by Federal, State, or Provincial statutory requirements for Asbestos Threshold Limit Value.

2. DEFINITION:

- 2.1 A fiber is any particle having a length to diameter ratio of 3:1 or greater.

3. OUTLINE OF METHOD:

- 3.1 Airborne asbestos fibers are collected by drawing a specified volume of air through a membrane filter by use of a suction pump. The fibers are deposited on the filter.

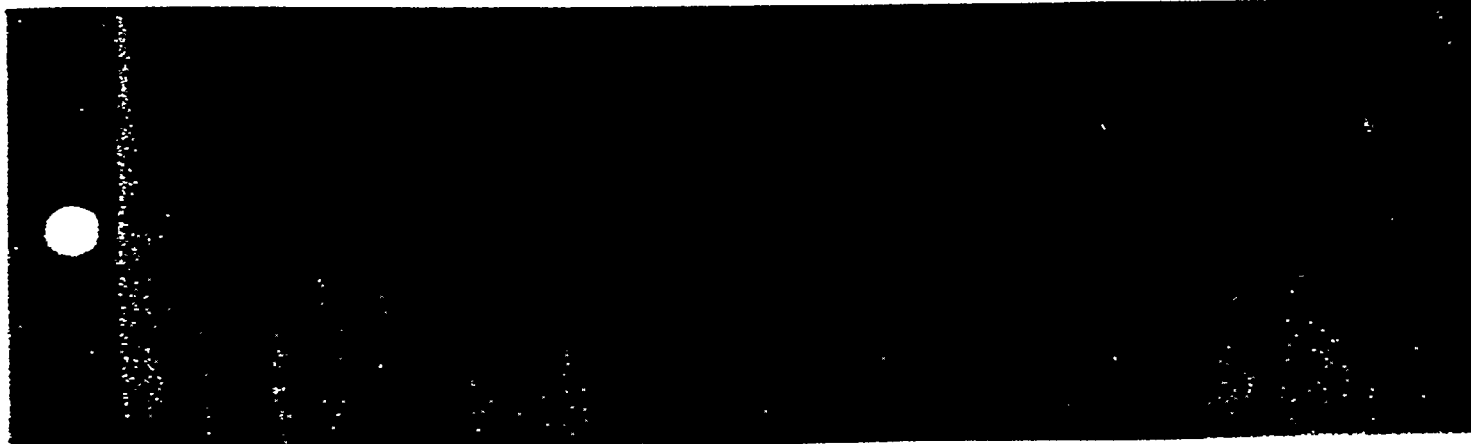
A representative section of the filter is removed, properly mounted on a microscope slide, and examined at a magnification of 430x (or 400x) using phase contrast optics. The number and length range of fibers observed in a given area under the microscope is noted and this figure is used to calculate the number of fibers per cubic centimeter (cc.) of air sampled.

Fiber concentrations are categorized with respect to length in the following manner; total number of fibers seen, greater than 5 microns, and greater than 10 microns per cubic centimeter of air.

4. APPARATUS AND MATERIALS:

- 4.1 Sample Collector:

The samples shall be collected on a filter having a pore size of $0.8 \pm$



0.05 micrometers (microns), supported on a thick pad to control air distribution and mounted in a segmented enclosure (filter holder, field monitor) to prevent contamination of the filter prior to exposure.(1)

4.2 Cellulose Bands:

The segments of the filter holder (field monitor) containing the filter and pad shall remain sealed against contamination until ready for examination.(2)

4.3 Tubing:

Rubber or plastic tubing of suitable length is connected to the inlet side of the pump and to the filter holder assembly. One quarter inch inside diameter, non-collapsible tubing is recommended.

4.4 The suction pump shall be capable of providing a constant air flow rate of 2 liters per minute for the duration of a test when connected to the filter assembly through a required length of tubing. It is desirable, although not necessary, that the pump have an explosion-proof motor.(3)

4.5 Microscope and Accessories:

A binocular microscope fitted with phase contrast optics should be used for counting the asbestos fibers. Any reticle, such as Porton or Patterson Globe and Circle, which projects a constant counting area and has provision for sizing, is suitable. This procedure is written using the Bausch and Lomb phase-equipped microscope and the Porton reticle. All set up procedures correspond with this equipment. However, any good microscope with phase contrast accessories is acceptable.

The reticle is mounted in the non-adjustable eyepiece and a stage micrometer is used to calibrate the dimensions of the counting area.

Microscope slides size 3 x 1 inch and cover glasses No. 1½, 18mm. must be used.

(1) A suitable filter assembly is a Type AA, 37mm., 0.8 micron cellulose ester filter, pad and plastic "field monitor" - obtainable from the Millipore Corporation, Bedford, Mass. 01730.

(2) Cellulose bands for this purpose may be obtained from the W. H. Jelly and Co., Inc., Franklin Park, Illinois 60131.

(3) Suitable pumps may be obtained from: a) Mine Safety Appliance Co., Pittsburgh, Penna. 15208; b) Willson Products, Div., Reading, Penna. 19603; c) Unico Environmental Instruments Inc., Fall River, Mass. 02720.

4.6 Miscellaneous:

A 1:1 mixture of dimethyl phthalate for preparing the sample mounting medium

Tweezers, scalpel, lens tissue, and a cleaning solution for the cleanliness of the microscope and

5. CLEANLINESS:

5.1 All equipment used in fiber counting only for this purpose. Fiber counting part of the laboratory; and, if possible, microscope and accessories should be cleaned according to the procedures for care of the microscope as recommended by the manufacturer's recommendations.

6. PREPARATION OF MOUNTING SOLUTION

6.1 The mounting solution used in this procedure is a mixture of dimethyl phthalate and diethyl oxalate when the sample is to be counted with a microscope. If samples are to stand for longer periods, a fixative should be used. This solution may be prepared by dissolving 1 gram of membrane filter material per 100 ml of the dissolved material in a solvent of low viscosity as possible without being too viscous. A viscous solution delays the migration of the sample to the center of the mount. The sample may show an apparent loss in concentration for application.

7. AEROSOL FIELD MONITOR AND FILTER

7.1 The monitors may be reused after being cleaned thoroughly in warm detergent water and rinsed.

8. CALIBRATION OF EQUIPMENT:

8.1 Pump:

The frequency of calibration is dependent on the handling of the instrument. The calibration is carried out with a filter in place. It is recommended that an intermediate standard be used in the calibration.

8.1.1 Suction pumps equipped with a flowmeter calibrated by a primary standard procedure is shown in Fig. 1. A direct measure of air volume is

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-llson Products, Div., Reading, Penna. 19603;
truments Inc., Fall River, Mass. 02720.

4.6 Miscellaneous:

A 1:1 mixture of dimethyl phthalate and diethyl oxalate is needed
for preparing the sample mounting medium.

Tweezers, scalpel, lens tissue, and a camel's hair brush for maintain-
ing cleanliness of the microscope and accessories are needed.

5. CLEANLINESS:

5.1 All equipment used in fiber counting should be isolated and used
only for this purpose. Fiber counting should be done in the cleanest
part of the laboratory; and, if possible, in a separate room. The
microscope and accessories should be kept clean at all times. Pro-
cedures for care of the microscope and accessories should follow the
manufacturer's recommendations.

6. PREPARATION OF MOUNTING SOLUTION:

6.1 The mounting solution used in this method is a 1:1 mixture of
dimethyl phthalate and diethyl oxalate. This solution may be used
when the sample is to be counted within 24 hours after preparation.
If samples are to stand for longer periods of time an alternate solution
should be used. This solution may be prepared by dissolving 0.05
grams of membrane filter material per milliliter of solution. The pur-
pose of the dissolved material is to provide a solution with as high a
viscosity as possible without being difficult to handle. The highly
viscous solution delays the migration of particles outward from
the center of the mount. The samples will remain stable with no
apparent loss in concentration for approximately 30 days.

7. AEROSOL FIELD MONITOR AND FILTERS:

7.1 The monitors may be reused after each test by cleaning them thor-
oughly in warm detergent water and rinsing with distilled water.

8. CALIBRATION OF EQUIPMENT:

8.1 Pump:

The frequency of calibration is dependent on the use, care, and
handling of the instrument. The calibration for the suction pump
is carried out with a filter in place. It is suggested that a primary or
intermediate standard be used in the calibration.

8.1.1 Suction pumps equipped with a floating-ball gauge may be
calibrated by a primary standard. The set up for this pro-
cedure is shown in Fig. 1. A primary standard provides a
direct measure of air volume displaced per unit time.

Before starting the calibration, be sure the pump battery is fully charged. Fill a 250 ml. graduate with water and invert in a beaker of water. Note the starting level in the graduate. Let the water come to room temperature. Adjust the ball float to one of the lines on the gauge scale. Insert the tubing from the discharge side of the pump into the graduate. Start the pump and timer simultaneously. As the liquid level in the graduate approaches the liquid level in the beaker, simultaneously stop the timer and lift the graduate from the discharge tube. The pump is still operating. The mouth of the graduate should remain below the level of water in the beaker during this step. Stop the pump and note the final level in the graduate.

Calculate the flow rate in standard liters/min. from the formula,

$$Q = \frac{V_i - V_f}{T} \times \frac{60}{1000} \times K$$

where:

- Q = flow rate in standard liters/min.
- V_i = initial volume in ml. from graduate.
- V_f = final volume in ml. from graduate.
- T = time in seconds of calibration.
- 60 = conversion from seconds to minutes when T is in seconds.
- 1000 = conversion from ml. to liters.
- K = factor taking into consideration — temperature and pressure corrections.

To calculate the value of K, use the following formula,

$$K = \frac{P_a - P_v}{760} \times \frac{530}{460 + F}$$

where:

- P_a = atmospheric pressure in mmHg.
- P_v = vapor pressure of water at room temperature in mmHg.
- F = room temperature in °F

Duplicate tests should be run at each major scale division on the flowmeter gauge. On a plot of flowmeter setting

versus air flow rate (Q), draw the points. From this line, then go to two liters/min. and adjust to this setting.

An alternate method for calibrating the test meter, which is an indirect method, the pump's suction displacement of water in the meter is indicated by the meter. After the meter is leveled and the apparatus is assembled as a watch, time the wet test meter. If the meter does not indicate proper setting, the actual air flow times a conversion factor (K). The calculation of the value of K is above in the primary calibration.

8.2 Microscope:

Before using the microscope for counting, the microscope must be in proper adjustment. This procedure is as follows:

- 8.2.1 Screw the 10x and 43x phase nosepiece and insert the 10x objective.
- 8.2.2 Position the 10x objective slide on the stage, turn on the diaphragm on the condenser and set the phase ring to the 10x position.
- 8.2.3 Set the phase ring to the 10x position.
- 8.2.4 Close the iris diaphragm on the point where only a small bright spot is visible. Adjust the iris set screws on the condenser so that the spot is centered in the field.
- 8.2.5 Focus sides of Decagon with the 10x objective. Sides will appear blue when focused down. The proper focus is reached when the phase ring to cover entire field of view.
- 8.2.6 With the phase ring setting still on the 10x position, insert the centering

the calibration, be sure the pump battery is full a 250 ml. graduate with water and invert water. Note the starting level in the graduate. Note room temperature. Adjust the ball valve on the gauge scale. Insert the tubing large side of the pump into the graduate. Start pump and timer simultaneously. As the liquid level in the graduate approaches the liquid level in the tube, stop the timer and lift the graduate. The pump is still operating. The graduate should remain below the level of water during this step. Stop the pump and read the level in the graduate.

Flow rate in standard liters/min. from the

$$\frac{V_i - V_f}{T} \times \frac{60}{1000} \times K$$

Flow rate in standard liters/min.

Initial volume in ml. from graduate.

Final volume in ml. from graduate.

Time in seconds of calibration.

Conversion from seconds to minutes when

T is in seconds.

Conversion from ml. to liters.

Factor taking into consideration — temperature and pressure corrections.

Value of K, use the following formula,

$$\frac{P_a - P_v}{760} \times \frac{530}{460 + F}$$

Atmospheric pressure in mmHg.

Vapor pressure of water at room temperature

in mmHg.

Room temperature in °F

Should be run at each major scale division on gauge. On a plot of flowmeter setting

versus air flow rate (Q), draw the best straight line through the points. From this line, the flowmeter setting corresponding to two liters/min. is determined. The pump should be adjusted to this setting when taking samples.

An alternate method for calibrating pumps is by using a wet test meter, which is an intermediate standard. In this method, the pump's suction is determined by the displacement of water in the meter by the gas being measured. This displacement is indicated by a dial on the face of the meter. After the meter is leveled and the sight gauge properly filled, the apparatus is assembled as shown in Fig. 2. Using a stopwatch, time the wet test meter displacement for one minute. If the meter does not indicate two LPM at 6" H₂O, then an adjustment should be made on the pump to obtain the proper setting. The actual air flow rate is the calibrated rate times a conversion factor (K) for temperature and pressure. The calculation of the value of K is the same as described above in the primary calibration procedure.

8.2 Microscope:

Before using the microscope for counting, its phase contrast optics must be in proper adjustment. This procedure is described below.

8.2.1 Screw the 10x and 43x phase contrast objectives into the nosepiece and insert the 10x eyepieces.

8.2.2 Position the 10x objective for viewing, place a specimen slide on the stage, turn on the illuminator, and set the iris diaphragm on the condenser about half open.

8.2.3 Set the phase ring to the 0 position and focus on the specimen.

8.2.4 Close the iris diaphragm on the illuminator down to the point where only a small bright area is visible in the field. Adjust the iris set screws on the illuminator until this spot is centered in the field.

8.2.5 Focus sides of Decagon with phase contrast control knob. Sides will appear blue when focused up and red when focused down. The proper focus is mid-point. Open the iris diaphragm to cover entire field of view.

8.2.6 With the phase ring setting still at 0, remove the right eyepiece and insert the centering telescope. Adjust the tele-

scope until the dark circle comes into sharp focus and lock it in position with the set screw provided.

8.2.7 Turn the phase ring to the 10 setting. By using the adjustment screws on the side of the condenser, move the rings until they are perfectly concentric.

8.2.8 Rotate the 43x objective into viewing position and turn the phase ring to the 43 setting. Align the two rings as described in 8.2.7.

8.2.9 Remove the telescope and replace the 10x eyepiece. Use fine adjustment for proper focus. The microscope is now adjusted for phase contrast viewing at 430x magnification.

8.3 Reticle:

The microscope must contain a reticle. Calibration of the reticle is done by means of a stage micrometer. The Porton reticle is a rectangle divided into two squares. The counting area is divided into six rectangles which constitutes the left square. A series of circles in which every other circle doubles in diameter is located on the top and bottom of the large rectangle. The diameter, D , of each is given by the formula: $D = L\sqrt{2^N}$ where L is a unit of length and N represents the circle number. The length of the large reticle is 200 L units. By measuring the actual length of the rectangle using the stage micrometer and dividing this value by 200, the value of L at 430x magnification is obtained. With this value for L , the actual diameter of each of the circles can be determined.

The calibration of the Porton reticle need be done only once, unless the eyepieces or objectives are changed, the magnification is altered in any way, or the microscope had been disassembled for cleaning prior to use.

9. SAMPLING:

9.1 The suction pump battery must be in a fully charged condition before each test. Recommended battery life for constant pumping rate is furnished by the manufacturer and must not be exceeded for any one test (see paragraph 4.4).

9.2 After inserting pad and filter into each of the plastic holders (Aerosol Field Monitors) they are immediately capped and plugged. The junctures of the holders are then sealed with cellulose bands.

All loaded monitors must be carried to the test sites in a closed carrying case. An extra monitor must be taken along to serve as a blank. All monitors must be marked for identification.

9.3 At the test site, the top section of the "filter" testing. The outlet plus an appropriate length of 1/4 inch into the pump intake. Sampling can be either in taking general area samples it assembly be mounted on a tripod floor level. For a personal sample the bly is worn by the person being to close to the person's breathing zone

The sampling time should be gauged the filter for satisfactory counting. E avoided since no dilution is possible flow rate of two liters per minute. field monitor must be re-sealed.

10. COUNTING:

10.1 Remove the cover of the field monitor be examined. Cut out a pie shaped section with a scalpel. The section to be about 1 cm.

10.2 Place a drop of mounting solution on the drop into a shape corresponding to

10.3 Remove the sample section from the tweezers and deposit it. sample side up and cover with a clean cover glass. Press with the tweezers until it is in intimate contact with the cover glass. Mounts may take 30 minutes. The solution is completely dissolved.

10.4 Place a sample slide on the stage and focus because dust is retained only in the top section. The total fiber count should be at least whichever is less.

If a fiber crosses the limits of the counting area, two adjacent sides are counted. The sides of the counter, but they are always the same. The fibers counted are estimated as to size either the top or bottom of the reticle three size groups: all fibers seen, all fibers longer than ten microns.

PRIMARY CALIBRATION

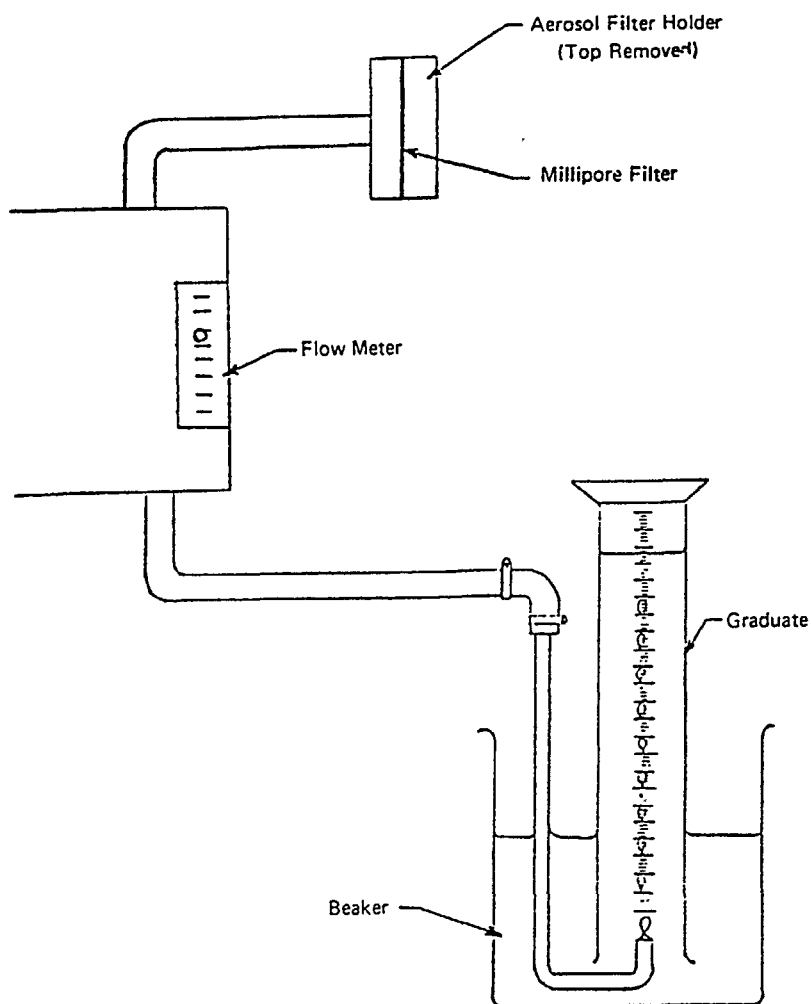
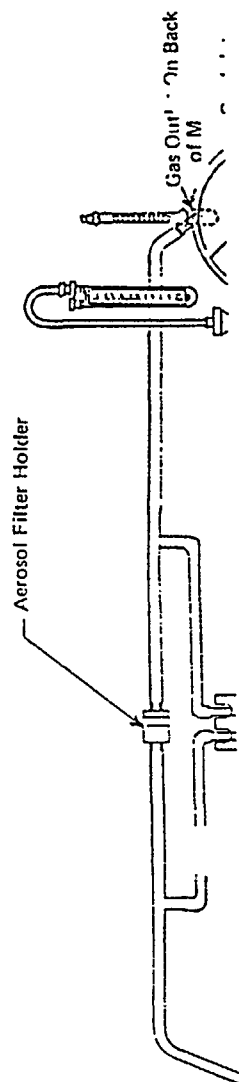
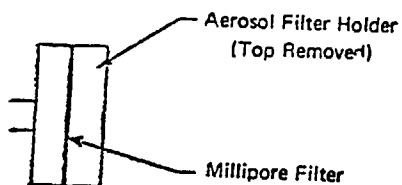


Fig. No. 1
12

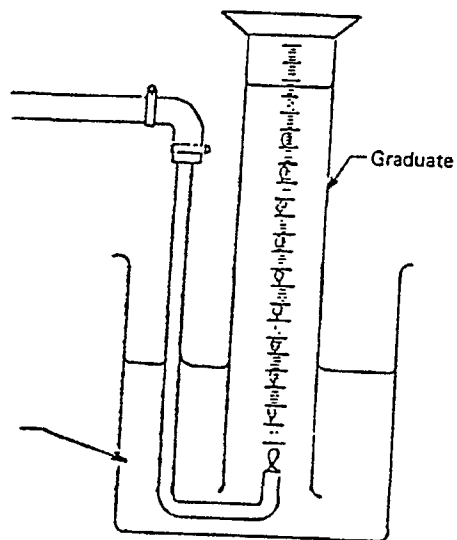
INTERMEDIATE CALIBRATION



TON



meter



INTERMEDIATE CALIBRATION

1. No. 1
2

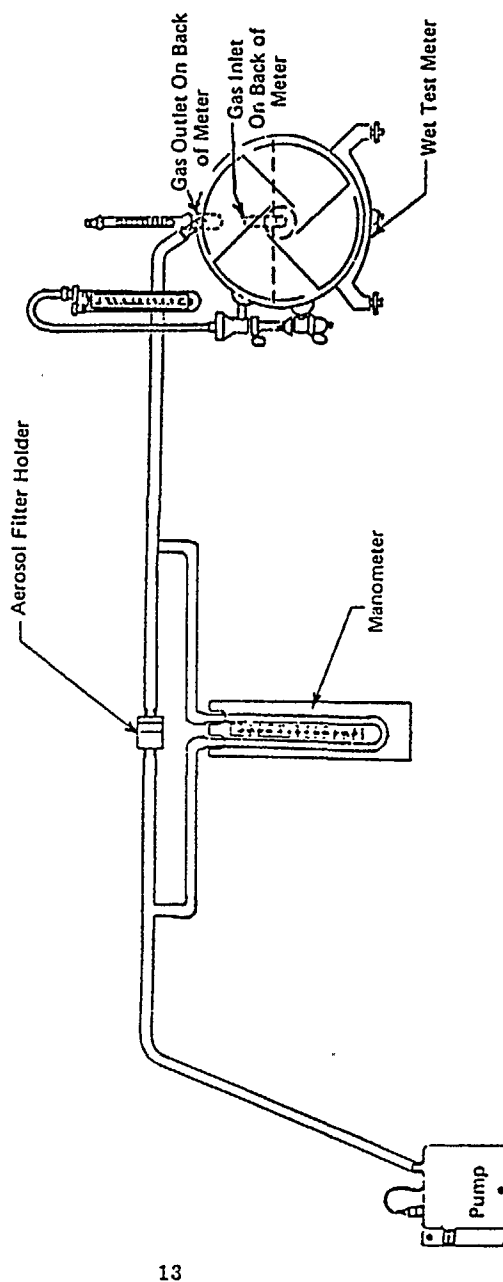


Fig. No. 2

10.5 In shifting from one area of examination to another, the choice must be random. Once chosen, the field must be counted regardless of the number of fibers observed. In shifting from one field to another the operator should take his eyes away from the eyepieces and concentrate them on moving the stage to a new location. The choice of the new location should be arbitrary, except that areas near the edge of the sample should be avoided. During the count it is very important to constantly adjust the fine focus back and forth. This brings into focus fibers that might not be seen otherwise.

11. CALCULATION OF RESULTS:

The concentration of fibers shall be expressed as the number of fibers per cubic milliliter of air reported to two (2) significant figures only.

$$\text{Concentration} = \frac{(\text{Av. Fiber Count}) (\text{Filter Area})}{(\text{Field Area}) (\text{Sample Volume}) (1000)}$$

where:

Av. Fiber Count: Average Fiber Count Per Size Range.
 Filter Area: 855mm² for 37mm. dia. membrane filters.
 Field Area: Area of counting field in mm².
 Sample Volume: Total Time x Flow Rate, Express in liters.
 1000: Converts liters to milliliters.

If blanks are used, subtract the average blank count of the same size range from the average fiber count.

INDUSTRIAL HYGIENE V

1. STATION NUMBER _____ PU
 DEPARTMENT _____
 PLANT _____ TI:
 DIVISION _____ TI
 DATE SAMPLED _____ ET
 DATE ANALYZED _____ V'

2. STATION LOCATION _____

3. SPECIFIC PRODUCT AND MATERIALS _____

4. OPERATORS AND PROTECTION _____

5. VENTILATING CONTROL _____

6. DRAFTS _____ TOTAL

7. REMARKS _____

ANALYSIS: _____ F/cc

TLV: _____ F/cc

SAMPLED BY _____

TOT. _____

AV. _____

15

A

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