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**PLAINTIFF'S
EXHIBIT**

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E. I. DU PONT DE NEMOURS AND COMPANY
TEXTILE FIBERS DEPARTMENT

MANUAL OF ENVIRONMENTAL ANALYTICAL METHODS

METHOD NO. 3537

REVISION NO. _____

TITLE: ASBESTOS IN AIR-AREA AND PERSONAL MONITORING

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NOTE: Effective adoption date Departmentally is the date the method is approved by the Department EAM Coordinator.

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EAM 9537
ASBESTOS IN AIR
AREA AND PERSONAL MONITORING

TECHNICAL SECTION

I. PRINCIPLE

Asbestos in air is collected and determined by drawing air through a membrane filter by means of a battery powered personal sampling pump. The filter is then transformed, by chemical treatment, 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 magnifications.

II. PRECISION AND TIME REQUIRED

- A. Coefficient of variation determined in reference is 16.2, which includes experienced and non-experienced counters.
- B. Total analysis time required for one sample (does not include sample collection) is 1.2 hours, and for eight samples is 8.0 hours.

III. ECONOMICS

- A. Standard operator time to make a single determination has not been set.
- B. Cost of material for a single determination is \$0.90.

IV. REFERENCE

USPH/NIOSH membrane filter method for evaluating airborne asbestos fibers, DIVISION OF TRAINING, U. S. DEPT. OF HEW, CINCINNATI, OHIO (1974).

V. EQUIPMENT AND MATERIAL SPECIFICATION

A. Sampling Equipment

1. MSA portable personal vacuum pump or equivalent - Mine Safety Appliance Company, Pittsburg, Pa.

Specifications - Able to operate up to 8 hours at a constant flow rate of at least 1.7 liters per minute against a resistance of 15.2 centimeters of water measured at the pump inlet.

2. Laboratory tubing, plastic or equivalent with 6 millimeter bore and 90 centimeters long.
3. Clothing clip spring to monitor personal, John T. McGuire Company, Pawtucket, R.I.
4. Three piece filter monitor cassette, Millipore Corporation; Cat. No. PG37AD, preloaded cassettes.
5. Cellulose sealing band, Mines Safety Appliance Company.

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V. EQUIPMENT AND MATERIAL SPECIFICATION (CONTD.)

A. Sampling Equipment (Contd.)

5. 0.30u 37MM plain white cellulose ester membrane filter, Millipore Corp.,
Cat. No. SCWP03700.

B. Pump Calibration Equipment

1. Tubing-co-filter monitor cassette adaptor, Millipore Corp.
2. 1000-ml. bubble burette, Cat. No. LG4130, Lab Glass, Inc.
3. Stopwatch, graduated in 0.5 seconds, A. H. Thomas Company.
4. 500-ml glass beaker
5. Soap solution - 2 ml Ivory dishwashing liquid or equivalent in 400 ml. water.

C. Optical Equipment

1. Bausch and Lomb dynoptic microscope with binocular head or equivalent.
2. 10 X Huygenian eye pieces or equivalent.
3. Bausch and Lomb PR-27 ribbon-filament illuminator (preferably built in and having provisions for adjusting light intensity) or equivalent. Specifications - Must obtain Koehler illumination, in Appendix B.
4. A porton reticle or equivalent.
5. Mechanical stage.
6. Abbe or Zernike condenser fitted with phase ring (or Heine) with a numerical aperture (N.A.) equal to or greater than the N.A. of the objective or equivalent.
7. 40-45X (N.A. 0.65 + 0.75) positive phase contrast achromatic objective.
8. Phase-ring centering telescope or bertrand lens.
9. Stage micrometer with 0.01MM subdivisions.

D. Filter Mounting Equipment

1. 2.5 x 7.5 cm glass microscope slides.
2. #1 1/2 (0.17MM) thickness cover slip.
3. #10 curved blade scalper or equipment.
4. Tweezers

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7. EQUIPMENT AND MATERIAL SPECIFICATION (CONTD.)

- D. 5. Lens tissue
6. Glass rod or equivalent
7. Wheaton Balsam Bottle

E. Reagents

1. Dimethyl Phthalate (Reagent Grade)
2. Diethyl oxalate (Reagent Grade)

- Mounting Solution Preparation

Add 5 ml dimethyl phthalate to 5 ml diethyl oxalate in a Wheaton Balsam Bottle. Add 0.5 grams of cut up membrane filter material. The solution is ready for use after 24 hours. Stir solution the first hour. The normal shelf life is 6 months.

F. Projectors

The microscope image may be further projected to aid counting. Numerous projection devices are available, however, Old Hickory uses a television method having a trinocular microscope combined with a Millipore 77 MC television camera and monitor. The unit is manufactured by Millipore Corporation, Bedford, Massachusetts 01730.

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LABORATORY SECTION

VI. APPLICABILITY

This method has been successfully applied to asbestos in air using a 17MM millipore (0.8 micron) filter and a small battery operated personal sampling pump at a flow rate of 1.0 to 2.5 liters per minute for time periods of 15 to 150 minutes at concentrations of 1 to 20 fibers per cubic centimeter.

The optimum sampling time is reached when the sample has 1 to 5 fibers per counting field.

VII. UNUSUAL SAFETY PRECAUTIONS

- A. Wear rubber gloves and splash proof goggles while mixing dimethyl phthalate and diethyl oxalate.
- B. Keep reagents away from heat and open flame. They are flammable.
- C. Avoid contact of reagents with the skin. Wash skin with soap and water if contact occurs.

SAMPLING TIME

- o The optimum sample time is determined from:
 - a. Optimum fiber concentration of 2 to 10 fibers per cm^3 or 1-5 fibers per field.
 - b. Optimum flow rate of 1 to 2.5 liter per minute.

VIII. PROCEDURE

A. Filter Installation and Sampling

1. Calibrate the portable personal vacuum pump (Appendix A).
2. Separate the filter cassette and install one filter pad and 0.3 micron filter at the base of the pump inlet side of cassette.
3. Assemble the cassette and place a damp cellulose band around the cassette, insuring that the two sections are joined without leakage.
4. Allow the cellulose band to dry.
5. Connect a charged calibrated pump (change the pump battery for 16 hours on "High" one day prior to use) to the filter side of the cassette with tubing.
6. Remove the top section of the cassette to expose the filter.
7.
 - a. For area sampling, connect the pump and filter to a ring stand or buggy. Position the cassette so that the open end is facing down.
 - b. For personal sampling, fasten the pump to the worker's belt and fasten the cassette to the lapel or shirt front (as close to the face as practical). Position the cassette so that the open end is facing down.

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VIII. PROCEDURE (CONTD.)

A. 8. Start the pump and record:

- a. Start time
 - b. Flow rate
 - c. The workers name for personal sampling
 - d. Area location
 - e. Type of work being done
 - f. Filter number
 - g. Type of sample (asbestos on air, etc.)
9. Check the pump periodically during sampling for proper operation and flow rate.
10. Stop the pump at the end of the test period and record the stop time.
11. Replace the cassette top and plug.

B. Sample Mounting

1. Clean all mounting equipment.
2. Apply a small drop of mounting solution, using a glass rod, onto the center of a clean microscope slide.
3. Spread the mounting media into a triangular shape, the size of which will coincide with the dimensions (one-eighth of the sample filter surface) of the filter wedge to be cut from the sample filter.
4. Separate the middle and bottom sections of the filter cassette to expose the sample filter.
5. Cut a triangular wedge from the sample filter center to the outer edge with a scalpel. (The size of the wedge should be 1/8 of the filter surface.)
6. Grasp the filter wedge, with tweezers, at the outer edge portion and place the wedge, sampled side up, upon the mounting medium on the microscope slide.
7. Place a clean cover slip, using tweezers, on the sample filter wedge (Once this contact has been made, do not reposition the cover slip).
8. The sample wedge will become transparent after 15 minutes. If cloudy, it may be necessary to press gently on the cover slip.
9. Read the sample within one day after mounting.

C. Sample Counting and Sizing

1. Calibrate the microscope (Appendix B and C)
2. Place the microscope slide on the mechanical stage and position the center of the wedge under the 10 X objective lens. Focus.

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VIII. PROCEDURE (CONTD.)

C. Sample Counting and Sizing (Contd.)

1. Change to the 40-45 X objective and focus.

Achieve comparable results:

- a. Count random fields from one end of the wedge and progress along a radial line to the other end.
- b. Select the field of view without looking through the microscope eyepiece to eliminate unconsciously selecting "Heavy" or "Light" areas.
- c. Size only fibers with a length to width ratio greater than or equal to 3:1.
- d. Count only fibers greater than 5 micrometers in length.
- e. 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).
- f. Count bundles of fibers as one fiber unless both ends of a fiber crossing another can be clearly resolved.
- g. In fibers that cross either one or two sides of the counting field, the following procedure is used to obtain a representative count.
First select:
 - The left and bottom sides and
 - The upper and lower left corners are used as decision aids.
- h. Then count any fiber greater than 5 micrometers in length, but only if the fibers
 - Lie entirely within the counting area or
 - Crosses the left or bottom sides, or
 - Crosses the upper or lower left corners, or
 - Crosses both the top and bottom sides.
- i. Reject and do not count all other fibers.
 - Refer to Figures 1-6 in Appendix D.
- j. Count as many fields as necessary to yield a total of at least 100 fibers.

Exception

- Count at least 20 fields even if you count more than 100 fibers.
- Stop at 100 fields even if you have not reached 100 fibers.

VIII. PROCEDURE (CONTD.)

Blank Sample

k. With each new batch (100 per package) of filters, read 4 unused filters.

IX. CALCULATION

Airborne asbestos fiber concentration is calculated from the formula:

$$C = \frac{(F-B) (W)}{(A) (V_S)}$$

Where:

C = Airborne fiber concentration in fibers $> 5 \mu\text{m}$ per cm^3

F = Average fiber count in fibers $> 5 \mu\text{m}$ per field

B = Average fiber count of blank in fibers $> 5 \mu\text{m}$ per field

W = 855 mm^2 for 37mm diameter filter

A = Area of the counting field of a calibrated reticle in mm^2 per field

V_S = Total air volume collected through filter expressed in milliliters at standard conditions of temperature and pressure.

Where:

$$V_S = \frac{V \times P}{760} \times \frac{298}{T + 273}$$

and

V = Volume of air sampled in milliliters

P = Barometric pressure in millimeters mercury at sample time.

T = Temperature of air sampled, $^{\circ}\text{C}$

APPENDIX A

CALIBRATION OF PORTABLE VACUUM PUMP

PROCEDURE

A pump should be calibrated before each use.

1. Charge the pumps' battery for 16 hours on "high" prior to calibration.
2. Pour 200-ml of soap solution in a 50 ml beaker.
3. Wet the 100 ml bubble burette inside with soap solution and leave wet.
4. Invert the 1000 ml burette and clamp to a ring stand.
5. Remove the plugs from a prepared (has filter and pad installed) filter cassette, and attach the pump side of the cassette to a hose which is connected to the personal pump.
6. Attach the free end of the cassette to the top of the inverted burette, with tubing (approx. 4 inches in length). Turn pump on.
7. Raise the beaker with soap solution to touch the mouth of the burette. A bubble should form at burette mouth and rise toward the top.
8. Determine the time required for the bubble to rise from zero to one thousand milliliters, using a stopwatch.
9. For the desired 1.7 liters per minute air flow, the bubble will take 25.5 seconds to reach the 1000 ml mark.
10. Adjust the pump setting until the flow rate is exact.

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APPENDIX 3

MICROSCOPE SETUP

1. The porton reticle is a round disc made of glass and about the same size and thickness as a dime. The reticle has a photographically produced scales and is mounted in the eyepiece.
2. Koehler method of illumination requirements:
 - a. An image of the light source must be in focus at the plane of the microscope condenser iris.
 - b. The image of the field iris must be in focus at the plane of the specimen.
 - c. The field iris opening is slightly greater than the microscope's field of view.
3. Set up procedure with Bausch and Lomb Dynoptic Microscope and PR-27 illuminator. (The same principles that apply to this particular system will apply to any similar system.)
 - a. The porton reticle must be mounted in eye piece.
 - b. Clip the stage micrometer into the mechanical stage.
 - c. Turn on the illuminator to the high position.
4. Place the front of the filter holder seven inches from the mirror fork.
5. Close the field iris.
6. Center the band of illumination (the projected image of filament) across the mirror fork.
7. Using the microscope's condenser focus knob, move the condenser upward until it nearly touches the slide.
8. Move the lens fully forward using the illuminator's condenser focus knob.
9. Position yours so that you can see the reflection of the bottom of the condenser in the mirror.
10. Close the condenser iris.
11. Sharply focus the image of the filament using either the illuminator condenser focus knob or the focus trimming knob at the rear base of the illuminator.
12. Open the microscope condenser iris full.
13. Open the field iris half way.
14. Select the 10X objective.
15. Select the 40-45X annealers.

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APPENDIX 3 (CONTD.)

16. Find correct focus by looking from the side of the microscope while moving the objective to the coverslip until they nearly touch.
17. Look through the eye pieces and widen the objective-coverslip distance with the coarse-focus knob until the image of the micrometer comes into sharp focus.
18. Select the 40-45X objective and sharply focus the image.
19. Close the field iris fully.
20. On the field of view there will be a bright spot of light:
 - a. Be sure you are in focus on the stage micrometer.
 - b. Focus the spot using the microscope condenser knob.
 - c. While looking through the microscope, open the field iris until it just clears the field of view.
 - d. After the illumination system is finished, there is one more thing that must be done before you calibrate the microscope, this is centering the cone of illumination with the objective's phase shifting element. Here are the necessary steps for completion.
 - Insert either the phase telescope (as in the case of Bausch and Lomb) or the Bertrand Lens (as in the case of Leitz Dialux) into the optical system. This enables the microscopist to see both rings. One is bright and comes from the condenser, the other is dark and is in the objective.
 - Make the rings concentric using the centering knobs on the condenser.
 - Remove the telescope or Bertrand lens. (The microscope is now properly set up for phase-contrast viewing and can be used for counting and sizing.)

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APPENDIX C

CALIBRATION OF PORTON AND PATTERSON GLOBE RETICLE

Recalibration is required only when a specific objective - eye piece - reticle combination is changed.

1. Install the 10X eye piece.
2. Put the 10X objective in place.
3. Place the stage micrometer on the mechanical stage, focus, and center the image.
4. Change to the 40-45X objective and focus.
5. Adjust (lineup) the first scale division of the micrometer to the left boundary of the reticle.
6. Count the number of divisions between the left and right boundaries of the long horizontal dimension of the largest rectangle, estimating any portion of the final division. (This measurement is 200 L.)
7. Determine L from the micrometer reading by dividing the measurement by 200.
8. Calculate the height of the verticle dimension which is 100 L.
9. Insert the calculated L value into the formula $D = L (2^n)^{1/2}$ where "n" is the circle number and "D" is the diameter of the circles N = 1 through 9. This is the reference to use when counting and sizing fibers.
10. Calculate the area of the counting field by multiplying 100 L times 100 L to give MM^2 .

This reticle calibration is valid only for a specific objective - eyepiece - reticle combination.

/skj
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APPENDIX D

MSFHS/NIOSH Membrane Filter Method

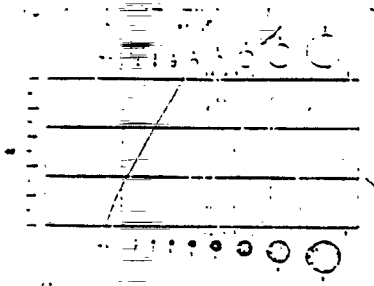


Figure 1. COUNT, fiber crosses both top and bottom sides.

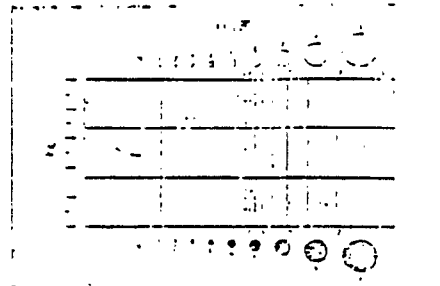


Figure 2. COUNT as one fiber.

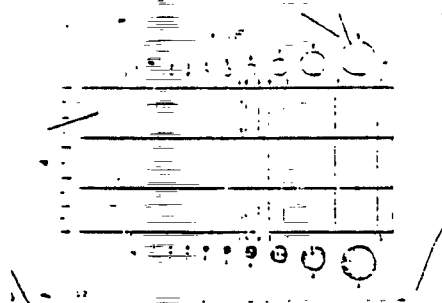


Figure 3. COUNT, fiber crosses left side.



Figure 4. COUNT, fiber crosses bottom side.

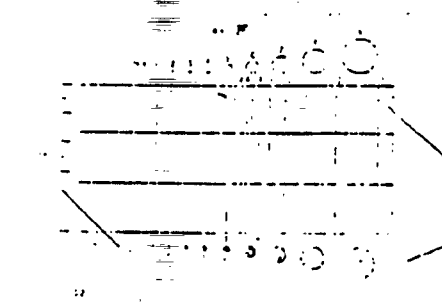


Figure 5. COUNT, fiber crosses lower left corner.



Figure 6. COUNT, fiber crosses upper left corner.