

State of California  
Air Resources Board

Method 427

Determination of Asbestos Emissions  
from Stationary Sources

Adopted: March 23, 1988

## Method 427 - Determination of Asbestos Emissions from Stationary Sources

### 1. PRINCIPLE AND APPLICABILITY

#### 1.1 Principle.

Particulate asbestos matter is withdrawn isokinetically from the source and collected on a polycarbonate membrane filter maintained at stack temperature. The particulate asbestos is analyzed by microscopic techniques.

#### 1.2 Applicability:

This method applies to the determination of particulate asbestos emissions from stationary sources, using stack sampling and electron microscopy. Using light microscopy, this method gives an index of airborne asbestos fibers for a known source of asbestos fibers; it will not differentiate between asbestos and other fibers. This method is not applicable to stacks that contain liquid droplets or are saturated with water vapor. In addition, this method shall not be used as written if the projected cross-sectional area of the probe extension-filter holder assembly covers more than 5 percent of the stack cross-sectional area (see Section 4.1.2). This method also applies to determining asbestos emissions from non-ducted sources when samples are taken using methods such as that described in NIOSH Method 7400, provided that the Control Agency's Authorized Representative determines that the sampling method will result in a polycarbonate membrane filter loaded in a manner that is suitable for analysis as detailed in Sections 2, 3 and 4 of this method.

Note: "Control Agency's Authorized Representative" as used in this document shall mean the Executive Officer or Air Pollution Control Officer of the state or local air pollution control agency at whose request the test is conducted, or his or her authorized representative.

### 2. APPARATUS

#### 2.1 Sampling Train.

A schematic of the sampling train used in this method is shown in Figure 427-1. Construction details for many, but not all, of the train components are given in APTD-0581 (citation 2 in Section 7); for changes from the APTD-0581 document and for allowable modifications to Figure 427-1, consult with the Control Agency's Authorized Representative. The operating and maintenance procedures for many of the sampling train components are described in APTD-0576 (Citation 3 in Section 7). Since correct usage is important in obtaining valid results, all users should read the APTD-0576 document and adopt the operating and maintenance

procedures outlined in it, unless otherwise specified herein. The sampling train consists of the following components:

2.1.1 Probe Nozzle. Stainless steel (316) or glass, with sharp, tapered leading edge. The angle of taper shall be 30° and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the buttonhook or elbow design, unless otherwise specified by the Control Agency's Authorized Representative. If made of stainless steel, the nozzle shall be constructed from seamless tubing. Other materials of construction may be used subject to the approval of the Control Agency's Authorized Representative.

A range of sizes suitable for isokinetic sampling should be available, e.g. 0.32 to 1.27 cm (1/8 to 1/2 in) - or larger if higher volume sampling trains are used - inside diameter (ID) nozzles in increments of 0.16 cm (1/16 in). Each nozzle shall be calibrated according to the procedures outlines in Section 5.1.

2.1.2 Filter Holder. The in-stack filter holder shall be constructed of borosilicate or quartz glass, or stainless steel; if a gasket is used, it shall be made of silicone rubber, Teflon or stainless steel. Other holder and gasket materials may be used subject to the approval of the Control Agency's Authorized Representative. The filter holder shall be designed to provide a positive seal against leakage from the outside or around the filter.

2.1.3 Probe Extension. Any suitable rigid probe extension may be used after the filter holder.

2.1.4 Pitot Tube. Type S, as described in Section 2.1 of Method 2 or other device approved by the Control Agency's Authorized Representative, the pitot tube shall be attached to the probe extension to allow constant monitoring of the stack gas velocity (see Figure 17-1). The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane during sampling (see Method 2, Figure 2-6b). It is recommended: (1) that the pitot tube have a known baseline coefficient, determined as outlined in Section 4 of Method 2; and (2) that this known coefficient be preserved by placing the pitot tube in an interference-free arrangement with respect to the sampling nozzle, filter holder and temperature sensor (see Figure 17-1). Note that the 1.9 cm (0.75 in) free-space between the nozzle and pitot tube shown in Figure 17-1, is based on a 1.3 cm (0.5 in) ID nozzle. If the sampling train is designed for sampling at higher flow rates than that described in APTD-0581, thus necessitating the use of larger

sized nozzles, the free space shall be 1.9 cm (0.75 in) with the largest sized nozzle in place.

Source-sampling assemblies that do not meet the minimum spacing requirements of Figure 17-1 (or the equivalent of these requirements, e.g. Figure 2-7 of Method 2) may be used; however, the pitot tube coefficients of such assemblies shall be determined by calibration, using methods subject to the approval of the Control Agency's Authorized Representative.

- 2.1.5 Differential Pressure Gauge. Inclined manometer or equivalent device (two), as described in Section 2.2 of Method 2. One manometer shall be used for velocity head (P) readings, and the other, for orifice differential pressure readings.
- 2.1.6 Condenser. It is recommended that the impinger system described in Method 5 be used to determine the moisture content of the stack gas. Alternatively, any system that allows measurement of both the water condensed and the moisture leaving the condenser, each to within 1 mL or 1 g, may be used. The moisture leaving the condenser can be measured either by: (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law of partial pressures; or (2) passing the sample gas stream through a silica gel trap with exit gases kept below 20° C (58° F) and determining the weight gain.

Flexible tubing may be used between the probe extension and condenser. If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel still be used between the condenser system and pump to prevent moisture condensation in the pump and metering devices and to avoid the need to make corrections for moisture in the metered volume.

- 2.1.7 Metering System. Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3° C (5.4° F), dry gas meter capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 17-1. Other metering systems capable of maintaining sampling rates within 10 percent of isokinetic and of determining sample volumes to within 2 percent may be used, subject to the approval of the Control Agency's Authorized Representative. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

Sampling trains utilizing metering systems designed for higher flow rates than described in APTD-0581 or APTD-0576 may be used provided that the specifications of this method are met.

- 2.1.8 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.
- 2.1.9 Gas Density Determination Equipment. Temperature sensor and pressure gauge, as described in Sections 2.3 and 2.4 of Method 2, and gas analyzer, if necessary, as described in Method 3.

The temperature sensor shall be attached to either the pitot tube or to the probe extension, in a fixed configuration. If the temperature sensor is attached in the field, the sensor shall be placed in an interference-free arrangement with respect to the Type S pitot tube openings (as shown in Figure 17-1 or in Figure 2-7 of Method 2). Alternatively, the temperature sensor need not be attached to either the probe extension or pitot tube during sampling, provided that difference of not more than 1 percent in the average velocity measurement is introduced. This alternative is subject to the approval of the Control Agency's Authorized Representative.

## 2.2 Sample Recovery.

- 2.2.1 Probe Nozzle Brush. Nylon bristle brush with stainless steel wire handle. The brush shall be properly sized and shaped to brush out the probe nozzle.
- 2.2.2 Wash Bottles - Two. Glass wash bottles are recommended; polyethylene wash bottles may be used at the option of the tester. It is recommended that acetone not be stored in polyethylene bottles for longer than a month.
- 2.2.3 Glass Sample Storage Containers. Chemically resistant, borosilicate glass bottles, for acetone washes, 500 mL or 1000 mL. Screw cap liners shall either be rubber-backed Teflon or shall be constructed so as to be leak-free and resistant to chemical attack by acetone. (Narrow mouth glass bottles have been found to be

less prone to leakage.) Alternatively, polyethylene bottles may be used.

2.2.4 Petri Dishes. For filter samples; glass or polyethylene, unless otherwise specified by the Control Agency's Authorized Representative.

2.2.5 Graduated Cylinder and/or Balance. To measure condensed water to within 1 mL or 1 g. Graduated cylinders shall have subdivisions no greater than 2 mL. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. Any of these balances is suitable for use here and in Section 2.3.4.

2.2.6 Plastic Storage Containers. Air tight containers to store silica gel.

2.2.7 Funnel and Rubber Policeman. To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

2.2.8 Funnel. Glass or polyethylene, to aid in sample recovery.

### 2.3 Analysis.

2.3.1 Glass Weighing Dishes.

2.3.2 Desiccator.

2.3.3 Analytical Balance. To measure to within 0.1 mg.

2.3.4 Balance. To measure to within 0.5 mg.

2.3.5 Beakers. 250-mL.

2.3.6 Hygrometer. To measure the relative humidity of the laboratory environment.

2.3.7 Temperature Gauge. To measure the temperature of the laboratory environment.

## 3. REAGENTS

### 3.1 Sampling.

3.1.1 Filters. The in-stack filters shall be 0.2 or 0.4 um pore size polycarbonate (37 mm).

3.1.2 Silica Gel. Indicating type, 6- to 16-mesh. If previously used, dry at 175° C (350° F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Control Agency's Authorized Representative.

3.1.3 Crushed Ice.

3.1.4 Stopcock Grease. Acetone-insoluble, heat-stable silicone grease. This is not necessary if screw-on connectors with Teflon sleeves, or similar, are used. Alternatively, other types of stopcock grease may be used, subject to the approval of the Control Agency's Authorized Representative.

3.2 Sample Recovery.

Acetone, reagent grade, 0.001 percent residue, in glass bottles. Acetone from metal containers generally has a high residue blank and should not be used. Sometimes, suppliers transfer acetone to glass bottles from metal containers. Thus, acetone blanks shall be run prior to field use and only acetone with low blank values (0.001 percent) shall be used. In no case shall a blank value of greater than 0.001 percent of the weight of acetone used be subtracted from the sample weight.

3.3 Analysis.

3.3.1 Acetone. Same as 3.2.

3.3.2 Desiccant. Anhydrous calcium sulfate, indicating type. Alternatively, other types of desiccants may be used, subject to the approval of the Control Agency's Authorized Representative.

4. PROCEDURE.

4.1 Sampling.

The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

4.1.1 Pretest Preparation. All components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified herein.

Weigh several 200 to 300 g portions of silica gel in air-tight containers to the nearest 0.5 g. Record the total weight of the silica gel plus container, on each container. As an alternative, the silica

gel need not be preweighed, but may be weighed directly in its impinger or sampling holder just prior to train assembly.

Check filters visually against light for irregularities and flaws or pinhole leaks. Label filters of the proper size on the back side near the edge using numbering machine ink. As an alternative, label the shipping containers (glass or plastic petri dishes) and keep the filters in these containers at all times except during sampling and weighing.

Desiccate the filters at  $20 \pm 5.6^\circ \text{C}$  ( $68 \pm 10^\circ \text{F}$ ) and ambient pressure for at least 24 hours and weigh at intervals of at least 6 hours to a constant weight, i.e., 0.5 mg change from previous weighing; record results to the nearest 0.1 mg. During each weighing the filter must not be exposed to the laboratory atmosphere for a period greater than 2 minutes and a relative humidity above 50 percent. Alternatively (unless otherwise specified by the Control Agency's Authorized Representative), the filters may be oven dried at  $105^\circ \text{C}$  ( $220^\circ \text{F}$ ) for 2 to 3 hours, desiccated for 2 hours, and weighed. Procedures other than those described, which account for relative humidity effects, may be used, subject to the approval of the Control Agency's Authorized Representative.

- 4.1.2 Preliminary Determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Control Agency's Authorized Representative. Make a projected-area model of the probe extension-filter holder assembly, with the pitot tube face openings positioned along the centerline of the stack, as shown in Figure 17-2. Calculate the estimated cross-section blockage, as shown in Figure 17-2. If the blockage exceeds 5 percent of the duct cross sectional area, the tester has the following options: (1) a suitable out-of-stack filtration method may be used instead of in-stack filtration; or (2) a special in-stack arrangement, in which the sampling and velocity measurement sites are separate, may be used; for details concerning this approach, consult with the Control Agency's Authorized Representative. (See Citation 10 in Section 7.) Determine the stack pressure, temperature, and the range of velocity heads using Method 2; it is recommended that a leak-check of the pitot lines (see Method 2, Section 3.1) be performed. Determine the moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate settings. Determine the stack gas dry molecular weight, as described in Method 2, Section 3.6; if integrated Method 3 sampling is used for molecular weight determination, the integrated

bag sample shall be taken simultaneously with, and for the same total length of time as, the particular sample run.

Select a nozzle size based on the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.2 of Method 2).

Select a probe extension length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of probes.

The sample flow rate should be limited to 0.75 CFM or less and total particulate loading on the filter should be one milligram or less. (At 0.75 CFM and 0.01 grains per standard cubic foot, this would give a sampling time of two minutes.) As the analysis is sensitive to filter loading, samples should be taken in groups of three, with a range of filter loadings such that the highest loading is about twice that of the lowest.

It is recommended that the number of minutes sampled at each point be an integer or an integer plus one-half minute, in order to avoid timekeeping errors.

In some circumstances, e.g., batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Control Agency's Authorized Representative's approval must first be obtained.

- 4.1.3 Preparation of Collection Train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

If impingers are used to condense stack gas moisture, prepare them as follows: place 100 mL of water in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

If some means other than impingers is used to condense moisture, prepare the condenser (and, if appropriate, silica gel for condenser outlet) for use.

Using a tweezer or clean disposable surgical gloves, place a labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as not to allow the sample gas stream to circumvent the filter. Check filter for tears after assembly is completed. Mark the probe extension with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Assemble the train as in Figure 17-1, using a very light coat of silicone grease on all ground glass joints and greasing only the outer portion to avoid possibility of contamination by the silicone grease. Place crushed ice around the impingers.

#### 4.1.4 Leak-Check Procedures

4.1.4.1 Pretest Leak-Check. A pretest leak-check is required. The following procedure shall be used.

After the sampling train has been assembled, plug the inlet to the probe nozzle with a material that will be able to withstand the stack temperature. Insert the filter holder into the stack and wait approximately 5 minutes (or longer, if necessary) to allow the system to come to equilibrium with the temperature of the stack gas stream. Turn on the pump and draw a vacuum of at least 380 mm Hg (15 in. Hg); note that a lower vacuum may be used, provided that it is not exceeded during the test. Determine the leakage rate. A leakage rate in excess of 4 percent of the average sampling rate or 0.00057 m<sup>3</sup> per min. (0.02 cfm), whichever is less, is unacceptable.

The following leak-check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with by-pass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the by-pass valve until the desired vacuum is reached. Do not reverse direction of by-pass valve. If the desired vacuum is exceeded, either leak-check

at this higher vacuum or end the leak-check as shown below and start over.

When the leak-check is completed, first slowly remove the plug from the inlet to the probe nozzle and immediately turn off the vacuum pump. This prevents water from being forced backward and keeps silica gel from being entrained backward.

- 4.1.4.2 Leak-Checks During Sample Run. If, during the sampling run, a component (e.g., filter assembly or impinger) change becomes necessary, a leak-check shall be conducted immediately before the change is made. The leak-check shall be done according to the procedure outlined in Section 4.1.4.1 above, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than  $0.00057 \text{ m}^3/\text{min}$  (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered; if, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and plan to correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run.

Immediately after component changes, leak-checks are to be done per procedure outlined in Section 4.1.4.1 above.

- 4.1.4.3 Post-Test Leak-Check. A leak-check is mandatory at the conclusion of each sampling run. The leak-check shall be done in accordance with the procedures outlined in Section 4.1.4.1 except that it shall be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is found to be no greater than  $0.00057 \text{ m}^3/\text{min}$  (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run.

- 4.1.5 Particulate Train Operation. During the sampling run, maintain a sampling rate such that sampling is within 10 percent of true isokinetic, unless otherwise specified by the Control Agency's Authorized Representative.

For each run, record the data required on the example data sheet shown in Figure 17-3. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak-check, and when sampling is halted. Take other readings required by Figure 17-3 at least once at each sample point during each time increment and additional readings when significant changes (20 percent variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

Clean the portholes prior to the test run to minimize the chance of sampling the deposited material. To begin sampling, remove the nozzle cap and verify that the pitot tube and probe extension are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available, which aid in the rapid adjustment to the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type S pitot tube coefficient is  $0.85 \pm 0.02$ , and the stack gas equivalent density (dry molecular weight) is equal to  $29 \pm 4$ . APTD-0576 details the procedure for using the nomographs. If  $C_p$  and  $M_d$  are outside the above stated ranges, do not use the nomographs unless appropriate steps (see Citation 7 in Section 7) are taken to compensate for the deviations.

When the stack is under significant negative pressure (height of impinger stem), take care to close the coarse adjust valve before inserting the probe extension assembly into the stack to prevent water from being forced backward. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

Traverse the stack cross section, as required by Method 1 or as specified by the Control Agency's Authorized Representative, being

careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe extension through the portholes, to minimize chance of extracting deposited material.

During the test run, take appropriate steps (e.g., adding crushed ice to the impinger ice bath) to maintain a temperature of less than 20° C (68° F) at the condenser outlet; this will prevent excessive moisture losses. Also, periodically check the level and zero of the manometer.

If the pressure drop across the filter becomes too high, making isokinetic sampling difficult to maintain, the filter may be replaced in the midst of a sample run. It is recommended that another complete filter holder assembly be used rather than attempting to change the filter itself. Before a new filter holder is installed, conduct a leak-check, as outlined in Section 4.1.4.2. The total particulate weight shall include the summation of all filter assembly catches.

A single train shall be used for the entire sample run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or, in cases where equipment failure necessitates a change of trains. In all other situations, the use of two or more trains will be subject to the approval of the Control Agency's Authorized Representative. Not that when two or more trains are used, a separate analysis of the collected particulate from each train shall be performed, unless identical nozzle sizes were used on all trains, in which case the particulate catches from the individual trains may be combined and a single analysis performed.

At the end of the sample run, turn off the pump, remove the probe extension assembly from the stack, and record the final dry gas meter reading. Perform a leak-check, as outlined in Section 4.1.4.3. Also, leak-check the pitot lines as described in Section 3.1 of Method 2; the lines must pass this leak-check, in order to validate the velocity head data.

- 4.1.6 Calculation of Percent Isokinetic. Calculate percent isokinetic (see Section 6.11) to determine whether another wet run should be made. If there is difficulty in maintaining isokinetic rates due to source conditions, consult with the Control Agency's Authorized Representative for possible variance on the isokinetic rates.

## 4.2 Sample Recovery.

Proper cleanup procedure begins as soon as the probe extension is removed from the stack at the end of the sampling period. Allow the assembly to cool.

When the assembly can be safely handled, wipe off all external particulate asbestos matter near the tip of the probe nozzle and place a cap over it to prevent losing or gaining particulate asbestos matter. Do not cap off the probe tip tightly while the sampling train is cooling down, as this would create a vacuum in the filter holder, forcing condenser water backward.

Before moving the sample train to the cleanup site, disconnect the filter holder-probe nozzle assembly from the probe extension; cap the open inlet of the probe extension. Be careful not to lose any condensate, if present. Remove the umbilical cord from the condenser outlet and cap the outlet. If a flexible line is used between the first impinger (or condenser) and the probe extension, disconnect the line at the probe extension and let any condensed water or liquid drain into the impingers or condenser. Disconnect the probe extension from the condenser; cap the probe extension outlet. After wiping off the silicone grease, cap off the condenser inlet. Ground glass stoppers, plastic caps, or serum caps (whichever are appropriate) may be used to close these openings.

Transfer both the filter holder-probe nozzle assembly and the condenser to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Save a portion of the acetone used for cleanup as a blank. Take 200 mL of this acetone directly from the wash bottle being used and place it in a glass sample container labeled "acetone blank".

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

Container No. 1. Carefully remove the filter from the filter holder and place it in its identified petri dish container. Use a pair of tweezers and/or clean disposable surgical gloves to handle the filter. If it is necessary to fold the filter, do so such that the particulate asbestos cake is inside the fold. Carefully transfer to the petri dish any particulate asbestos matter and/or filter fibers which adhere to the filter holder gasket, by using a dry Nylon bristle brush and/or sharp-edged blade. Seal the container.

Container No. 2. Taking care to see that dust on the outside of the probe nozzle or other exterior surfaces does not get into the sample, quantitatively recover particulate asbestos matter or any

condensate from the probe nozzle, fitting, and front half of the filter holder by washing these compounds with acetone and placing the wash in a glass container. Distilled water may be used instead of acetone when approved by the Control Agency's Authorized Representative and shall be used when specified by the Control Agency's Authorized Representative, in these cases, save a water blank and follow Control Agency's Authorized Representative's directions on analysis. Perform the acetone rinses as follows:

Carefully remove the probe nozzle and clean the inside surface by rinsing with acetone from a wash bottle and brushing with a Nylon bristle brush. Brush until acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone.

Brush and rinse with acetone the inside parts of the fitting in a similar way until no visible particles remain. A funnel (glass or polyethylene) may be used to aid in transferring liquid washes to the container. Rinse the brush with acetone and quantitatively collect these washings in the sample container. Between sampling runs, keep brushes clean and protected from contamination.

After ensuring that all joints are wiped clean of silicone grease (if applicable), clean the inside of the front half of the filter holder by rubbing the surfaces with a Nylon bristle brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make final rinse of the brush and filter holder. After all acetone washings and particulate matter are collected in the sample container, tighten the lid on the sample container so the acetone will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label the container to clearly identify its contents.

Container No. 3. If silica gel is used in the condenser system for moisture content determination, note the color of the gel to determine if it has been completely spent; make a notation of its condition. Transfer the silica gel back to its original container and seal. A funnel may make it easier to pour the silica gel without spilling, and a rubber policeman may be used as an aid in removing the silica gel. It is not necessary to remove the small amount of dust particles that may adhere to the walls and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for Container No.3 under "Analysis."

Condenser water. Treat the condenser or impinger water as follows: make a notation of any color or film in the liquid catch. Measure the liquid volume to within  $\pm 1$  mL by using a graduated cylinder or, if a balance is available, determine the liquid weight to within  $\pm 0.5$  g. Record the total volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas. Discard the liquid after measuring and recording the volume or weight.

## 5. ANALYSIS

NIOSH Method 7400 as issued February 15, 1984, an analytical method using light microscopy, is attached. It may be used subject to the limitations given in Section 1.2 "Applicability".

5.1 Sampling was discussed earlier in this method. It is assumed that the sample will have been acquired on a 0.2 or 0.4 pore size polycarbonate filter (37 mm diam.) and has been transported to the laboratory in the polypropylene (dichotomous sampler) filter cassette enclosed in a plastic petri dish.

This method covers all aspects of sample preparation, analysis, computation, and reporting of results. Precision based on comparisons of counts in different grid windows is discussed. However, accuracy is more difficult to access. It is expected that interlaboratory comparisons will be carried out on a subset of all samples.

### 5.2 Limit Defining Parameters

Throughout this protocol, the analyte air concentrations listed are based on minimum and maximum air volumes of  $1.8 \text{ m}^3$  (flowrate = 15 L/min, 2 hour sampling time) and  $9.6 \text{ m}^3$  (flowrate = 20 L/min, 8 hour sampling time), filter area of  $855 \text{ mm}^2$  (37 mm filter holder), and 300 mesh electron microscope grid (grid opening area approx.  $0.0032 \text{ mm}^2$ ). Other mesh size grids can be substituted, as long as the total area analyzed is kept approximately the same.

5.2.1 Detection limit of the analytical procedure. The detection limit of the analytical procedure is 5 fibers (structures) in all grid windows counted. This is the number at which the lower 95% confidence limit due simply to Poisson counting statistics is equal to one fiber.

5.2.2 Detection limit of the overall procedure. The detection limits of the overall procedure are approximately  $7 \times 10^3$  asbestos fibers (or free fibers or asbestos structures)/ $\text{m}^3$  and  $3 \times 10^2$  NIOSH equivalent fibers/ $\text{m}^3$ . (See Section 7.2.2.7 for definitions). These are the

values equivalent to the detection limit of the analytical procedure assuming a maximum air volume of 9.6 m<sup>3</sup>. The mass concentration detection limit is approximately 0.008 ng/m<sup>3</sup>.

This assumes, in addition to the above assumptions, that all fibers are chrysotile, having a diameter of 0.04 μm (a typical chrysotile fibril diameter) and a length of 0.4 μm.

5.2.3 Reliable quantitation limit. The reliable quantitation limit is unknown because the intralaboratory precision is unknown. However, if the precision due simply to Poisson counting statistics is required to give CV = 12.5%, then 64 fibers must be counted. Assuming the maximum air volume, the reliable quantitation limit based solely on counting precision is 9 x 10<sup>4</sup> asbestos fibers (free fibers, structures)/m<sup>3</sup> and 3.6 x 10<sup>3</sup> NIOSH equivalent fibers/m<sup>3</sup>.

5.2.4 Data from the analyses of seven air samples from Alviso, CA were used to evaluate the precision of the method. Total fiber counts ranged from 13 to 164, and asbestos structure counts ranged from 6 to 63. Based upon the standard deviation of counts among n grid windows, the 67% (1) confidence limits as a percentage of the average grid window count N was calculated. This coefficient of variation, CV, was estimated as

$$(1) \quad CV = \frac{t}{z n/M} \times 100\%$$

where t is the student's t statistic for the number n. This value approaches 2 for large n.

Based upon an assumed Poisson distribution, the coefficient of variation due simply to counting statistics (CV<sub>1</sub>) should be related to nN, the total number of fibers counted, by

$$(2) \quad CV_1 = \frac{nN}{nN} \times 100\% = \frac{100\%}{nN}$$

In Figure 1, CV is plotted as a function of nN (on a log log plot) for this theoretical counting variation. The CV ranges from 45% at a total count of five fibers to 10% for 100 fibers. Also shown in the figure are the seven estimates of CV for the actual asbestos structure counts, as well as the estimated CV for a count from a reference filter material obtained from the National Bureau of Standards, and from a proposed standard reference filter material, also from the National Bureau of Standards.

These data were fit with a straight line as shown in the figure. This line corresponds fairly well with that for the theoretical counting errors at low counts, but diverges slightly at high counts. By 100 structures counted, the line fit to the data predicts a CV of approximately 11%. This result is to be expected if other errors less dependent on total fiber count are present, since the counting error should predominate at low counts. An alternative interpretation is that the line fit to the data points is not significantly different from the theoretical line, especially in view of the fact that the fit line dips below the theoretical one for low counts. In any case, for counts less than 200, counting errors will clearly dominate the method's precision.

The data points for the two reference material counts do not appear to be consistent with the structure counts. Again, counting statistics are expected to dominate the method's precision.

The estimated coefficients of variation for the total count data are plotted in Figure 2, along with the theoretical curve for Poisson counting statistics. Here the least-squares fit predicts a CV ranging from 65% for 5 total fibers to 24% for 100 fibers. This suggests another significant source of imprecision. Since many of the fibers counted were attached to air particles, the variation in the number of fibers attached to each particle is expected to be responsible for the increase in the CV for these counts. The results for the reference material sample total fiber counts support this assumption, since they are composed principally of free fibers or fiber bundles, and their CV's lie close to the theoretical line. The precision for total fiber concentrations is thus likely to improve as the proportion of free fibers increases.

5.2.5 Accuracy. The accuracy of the method is unknown, although a negative bias is expected due to obscuring of fibers by particles. Interlaboratory comparisons have yet to be carried out using this method.

### 5.3 General Comments

5.3.1 Estimated EM times of 600 and 120 minutes for high and low magnification analyses respectively appear to be reasonable. Times for high magnification analysis can be significantly longer if the loading of particles with many fibers attached is high. Additional costs are incurred in sample preparation, analyzing count data, and reporting of results.

- 5.3.2 EM analysis for asbestos is tedious work, and places great demand on the microscopist. As a result, the microscopist should be accorded latitude in the scheduling of breaks to coincide with natural stopping places in the analysis (such as finishing a grid opening). Lack of consideration of operator discomfort or fatigue will result in less reliable analyses.
- 5.3.3 Although this methodology has been developed for asbestos in ambient air, it can be used for analysis of asbestos in other media such as water or soil with suitable modifications. The most important criterion is that the sample be finely divided and filtered uniformly through a polycarbonate membrane filter.
- 5.3.4 The order of analysis is field blanks, then laboratory blanks (if applicable), and then field samples.
- 5.3.5 The counting rule of 100 asbestos structures or 20 300 mesh grid openings, (or 40 400 mesh grid openings) whichever comes first is a minimum to keep costs down. More extensive counts are desirable, especially when structure counts are low.
- 5.3.6 Mass concentrations are the least reliable results reported, since the total mass is often dominated by a few large fibers, for which the precision is poor, and whose volume is often difficult to determine accurately. Although mass concentration should be reported, estimating confidence intervals is virtually impossible.
- 5.3.7 The order of analysis as specified in this method is
- 1) High magnification analysis (Sect. 7.2.2.2), and
  - 2) Low magnification analysis (Sect. 7.2.2.10).

This order may be reversed at the operator's discretion. In fact the low magnification analysis can be used as a screening tool to select particular samples for high magnification analysis.

## 6. LABORATORY REQUIREMENTS

### 6.1 Apparatus

- 6.1.1 A modern 100 kV transmission electron microscope (TEM) with energy dispersive x-ray spectrometer (EDS) is needed for the analysis. It is desirable for the microscope to have a scanning attachment for secondary electron imaging (SEI) so that particle surfaces can be examined. It should be capable of obtaining

selected area diffraction patterns on areas 100 nm in diameter, or preferably it should be capable of obtaining "microdiffraction" patterns from the same size area. For fiber length and width measurements, the microscope should be equipped with a fluorescent viewing screen which is inscribed with graduated circles of known radii, or the eyepiece of the binoculars should contain a reticule with a scale.

- 6.1.2 A vacuum evaporator with a turntable for rotating specimens is necessary for carbon-coating samples. It can also be used for preparing carbon films, and for gold-coating of EM grids.
- 6.1.3 An EM preparation room adjacent to the room containing the electron microscope should either be a clean room or contain a laminar flow class 100 clean bench to minimize contamination during sample preparation.
- 6.1.4 A microbalance accurate to a few micrograms should be located in a temperature and humidity controlled chamber for gravimetric analysis if it is desired.

## 6.2 Supplies

- 6.2.1 Modified Jaffe Wick Washers are required for dissolving polycarbonate membrane filters (see Ref. 4.5 or 4.6).
- 6.2.2 Miscellaneous EM supplies and chemicals needed are 300 or 400 mesh copper grids, plastic petri dishes, scalpels, tweezers, carbon rods, carbon grating replica, chloroform and EM grid storage boxes.

## 7. ANALYTICAL PROCEDURE

### 7.1 Sample Preparation

- 7.1.1 If gravimetric analysis is required, post-weighing of the sample filters should be carried out following receipt in the laboratory. (Pre-weighing is discussed in Appendix A - Sampling). Filters should be equilibrated overnight in the environmental chamber, and neutralized with a polonium source before weighing.
- 7.1.2 The weighed polycarbonate sample filters and suitable blanks should be coated with carbon as soon as possible. The filter is placed in the bottom half of a 100 mm plastic petri dish, and taped down around the edges with transparent tape. The petri dish is then placed on the turntable of the vacuum evaporator, the

evaporator is evacuated and approximately 40 nm thickness of carbon is evaporated (according to the manufacturer's instructions), while the turntable is rotated. The evaporation should be carried out in short bursts to prevent overheating and consequent degradation of the filter material. Such degradation makes dissolution of the filter virtually impossible.

After carbon-coating, the vacuum chamber is slowly returned to atmospheric pressure, a clear cover is put on the plastic petri dish, and the marked dish is stored in the clean bench.

7.1.3 The particles on the filter surface along with their embedding carbon coat are then transferred to 300 or 400 mesh electron microscope grids in the clean room or clean bench by means of the modified Jaffe Wick Washer (see Ref. 4.5 or 4.6 for details). Four or five sections should be cut from widely separate portions of the filter so that an assessment can be made of the uniformity of the filter deposit. Dissolution should be carried out for 72 hours so that it goes to completion. At temperatures lower than 68° F, complete dissolution may take longer than 72 hours. After the grids have been removed from the washer, they should be placed on clean filter paper inside clean, 50 mm plastic petri dishes, or inside a grid storage box. The locations of the grids should be noted in the logbook.

## 7.2 TEM Analysis

### 7.2.1 TEM Analysis Guidelines

7.2.1.1 The magnification at the fluorescent screen must be determined by calibration with a diffraction grating replica in the specimen holder. If sizing of fibers is carried out using binocular viewing (to facilitate diffraction pattern viewings), the calibration should be carried out (with fluorescent screen tilted) in both horizontal and vertical directions because of vertical stretching due to the screen tilt. In this case, fiber lengths should be measured in both vertical and horizontal components, the magnification corrected separately on the two components, and the two components combined by the Pythagorean Theorem to yield the total length. Measured widths should also be corrected based on the angle of the fiber from the vertical  $\theta$ , and the two magnifications  $M_h$  and  $M_v$  according to the formula

$$W_T = W^2 \frac{\cos^2\theta}{M_v^2} + \frac{\sin^2\theta}{M_h^2}$$

(3)

Where  $W_T$  is the true fiber width, and  $W$  is the apparent width on the screen.

- 7.2.1.2 The operator should define a "gate" through which the window is scanned. This can be the rectangular central portion of the fluorescent screen. If binocular viewing is used, the area will be much smaller, and may be defined by an enclosed figure scribed on a reticule.
- 7.2.1.3 Selection of the grid opening should be on a semi-random basis, that is to say, several widely dispersed areas of the grid should first be systematically selected, and then one opening should be selected randomly within each area. This helps to ensure detection of uneven distributions across large areas of the grid.
- 7.2.1.4 The average grid opening for a batch of grids should be determined by low magnification micrography of several openings. For this measurement, light, scanning, electron, or transmission electron microscopy can be used, but the measurement must be done with an instrument for which the magnification used has been independently calibrated. Account should be taken of the rounding of the corners of the openings. Finally, during the actual asbestos analysis, only grid openings of the standard (square with round corners) shape should be selected for detailed analysis. The analyst should be aware of selecting openings of irregular shape due to gross grid imperfections.
- 7.2.1.5 Suggested instrument settings are:
- a) Accelerating voltage: 100 kV
  - b) Magnification: 200 - 1000x for assessing the quality of the grid and the particulate loading, as well as for selecting grid openings to be analyzed, 400 - 4000x for low magnification analysis, and 20,000 - 50,000x (total with or

without binocular viewing) for detailed analysis (high magnification scanning).

- c) **Brightness:** Beam brightness should be adjusted during the scanning procedure such that features are just clearly visible. High brightness is undesirable for two reasons. First, brightness (electron flux) should be minimized to prevent radiation damage to chrysotile fibers, and second, viewing a high brightness screen reduces operator dark adaptation, compromising the ability to see weak electron diffraction patterns.
  
- d) **Diffraction:** If possible, microdiffraction should be used rather than selected area diffraction. Microdiffraction is carried out by exciting all lenses below the specimen in the same way as for selected area diffraction. The condenser lens system is adjusted so that a narrow, coherent beam is incident on the sample. This can be accomplished by highly exciting the first condenser lens, and using a small (10-60  $\mu\text{m}$ ) second condenser aperture. The extent of the beam determines the diffraction area, and can be made as small as a fibril diameter if desired. An image can be viewed in this mode by expanding the beam slightly by underexciting the second condenser lens, and slightly overexciting the intermediate (diffraction) lens. Once the beam is centered on the desired fiber, the second condenser lens can be used to converge the beam onto the fiber. The diffraction pattern will then be visible.

If selected area diffraction must be used, care must be taken to adjust the objective lens current for proper focus.

Microdiffraction is preferred over selected area diffraction because the focus is not critical for visibility of the pattern. Furthermore, contamination of the pattern with spots from closely adjacent material can be prevented by careful location of the beam.

## 7.2.2 TEM Procedure

- 7.2.2.1 Low magnification sample assessment. At a magnification of 200 - 1000x, assess the quality of the carbon film, the clearing of the filter and the uniformity of the particle loading. Obviously non-uniform grids with many broken or folded windows should be rejected.
- 7.2.2.2 High magnification analysis. At a total magnification of 20,000 - 50,000x, a series of parallel traverses should be made across the grid opening. Starting at one corner, traverse the opening by moving it through the "gate" already established. Movement through the gate is stop-and-go, rather than continuous, to allow the operator time to visually inspect each area of the opening as it enters the gate. At the end of each traverse, the sample is moved one gate width, and a parallel traverse is made in the opposite direction. The process is continued until the entire grid opening has been scanned.
- 7.2.2.3 Fiber identification. When a suspected asbestos-containing particle is detected, scanning is stopped. The presence of asbestos is confirmed by electron diffraction (see 7.2.2.5 below). If the possible asbestos is suspected to be an amphibole, or if it is suspected chrysotile for which diffraction gives an ambiguous result, energy dispersive x-ray analysis should be employed for confirmation (see 7.2.2.6 below).
- 7.2.2.4 Particle sizing. Confirmed asbestos structures are categorized as fibers, bundles, or mats, and their length and width is measured and recorded. (If measurement is done through the binoculars, vertical and horizontal components of the length are measured.) If the asbestos is attached to other particles, the size of these particles should also be measured and recorded. Specific counting rules are detailed in 7.2.2.7 and 7.2.2.8 below.
- 7.2.2.5 Electron diffraction. Electron diffraction is used for positive identification of chrysotile asbestos. Once chrysotile has been tentatively identified by

morphology (tubular appearance, diameter approximately 35 to 50 nm), a diffraction pattern should be obtained. For training purposes, it is useful to have micrographs of typical electron diffraction patterns of chrysotile. These should include patterns of both helical and right-cylindrical clino- and ortho-chrysotile (see Ref. 4.9 and 4.10 and Figures 3 and 4.) For positive identification, the following characteristics of the diffraction pattern should be observed:

- 1) The pattern should be streaky in appearance, with mirror-symmetric layer lines perpendicular to the fiber axis at the (silicate) spacing of 1/5.32 Å. (See Figure 3.)
- 2) Odd-numbered layer lines should contain streaks (hko reflections, h, k odd) whose intensity varies with radius as cylindrical Bessel functions symmetrically placed about the line parallel to the fiber axis. There should be no diffraction intensity at this center point, since this would be h00, and k must be odd. For helical polytypes, these streaks are split into two streaks above and below the layer lines. (See Figure 4). The vertical splitting increases with increasing k.
- 3) The zero layer contains spots at a spacing of 1/7.3Å (00 reflections) together with streaks corresponding to the same lattice as is seen on the odd layer lines (0k0 reflections with k even) at a spacing of a 1/4.6Å. For helical fibers, the 0k0 reflections will also be split.
- 4) The even-numbered layer lines contain either pairs of split spots for the clinochrysotile polytype (h0, and h0, h = even, = odd) or single spots spaced 1/14.6Å a part for orthochrysotile (h0, h = even). The central spots on the second layer line are often missing, due to slight tilt of the fiber axis from perpendicular to the electron beam.

7.2.2.6 Energy Dispersive X-ray Spectroscopy. Although suspected amphiboles should be checked for

morphology (long, thin, straight-sided crystals) and electron diffraction, neither of these criteria is sufficient for positive identification of amphiboles. Ideally, double-tilting should be done to obtain two or three zone-axis diffraction patterns (Ref. 10), but the skill and/or training necessary for such an analysis can be prohibitive, even when a double tilt stage is available. Instead, the present method accepts the use of energy dispersive x-ray spectroscopy (EDS) for amphibole identification as well as for identification of chrysotile for which no distinct diffraction pattern can be discerned.

The microscope manufacturer's instructions for obtaining spectra should be followed, and the spectra should be compared to library spectra obtained from standard preparations (See Figures 5, 6, 7, 8 and 9 and Ref. 4.6). Care should be taken to obtain spectra from portions of the fibers well separated from other particulate matter if possible, to avoid the superposition of interfering spectra.

7.2.2.7 Fiber Nomenclature. Fibers are first classified as positive chrysotile (chrysotile morphology, chrysotile diffraction pattern, or EDS), tentative chrysotile (chrysotile morphology, ambiguous diffraction, ambiguous EDS, usually due to overlapping particles), positive amphibole with type (amphibole morphology, amphibole EDS), tentative amphibole (amphibole morphology, ambiguous EDS due to overlapping particles), or non-asbestos. Asbestos fibers should then be classified according to the state aggregation, either as single fibers, bundles (two or more fibers in a parallel arrangement, with fibers closer together than one fiber thickness), and mats (two or more fibers in a random arrangement, but which are separated from at least one other fiber in the mat by less than a fiber diameter somewhere along their length). See Ref. 4.6, Figure A-7 for specific fiber-counting guidelines. Note: The term "mat" should be substituted for "cluster/clump". The term "matrix/debris" in the figure is used to denote fiber, bundle, or mat attached to a non-asbestos fiber.

7.2.2.8 Additional Fiber Information. All fibers, bundles, and mats attached to non-asbestos particles are to be

counted and sized separately, and the particle itself is also to be sized (See Fig. 10 for a suggested sample count sheet). For fibers on particle surfaces, a scanning attachment with secondary electron imaging capabilities is useful. However, for fibers of which part of the length is obscured, lengths should be increased from that actually observed according to the following rules:

- 1) If the obscuring particle or grid bar is large enough for the actual length of the fiber to be twice that observed, record a value equal to twice the observed length.
- 2) If the obscuring particle or grid bar is too small for the actual length to be twice that observed, record a value equal to the length of the fiber plus the size of the particle or grid bar.
- 3) Lengths estimated in this way should be flagged on the count sheet.
- 4) Fibers that extend over more than one grid opening should be counted only once.

7.2.2.9 Filter Area to be Analyzed. Enough grid windows should be analyzed for detection of at least 100 asbestos "structures", i.e., free fibers, free bundles, free mats, or particles with asbestos attached. However, no greater than 20 300 mesh or 40 400 mesh grid windows should be analyzed. The analysis should be distributed evenly among the four grids.

7.2.2.10 Low Magnification Analysis. At a total magnification of 400-4000x, approximately 50 (300 mesh) or 100 (400 mesh) intact grid openings in each of the four grids should be examined. (This analysis is to be used for detection of "NIOSH equivalent" fibers). Methods for asbestos identification, fiber classification, and fiber sizing are identical to those used for the high magnification analysis. The differences between this analysis and the high magnification analysis are as follows:

- 1) The total filter area analyzed is at least 10 times that analyzed at high magnification.

- 2) Small fibers (as free fibers), in bundles or mats, or attached to particles, may not be detected. However, the diameter of these missed fibers will be far below the resolving power of the phase contrast light microscope. The exact diameter below which detection is impossible will depend on the magnification.
- 3) The fibers detected at low magnification should be selected by the data processing to conform closely to phase contrast detection criteria. Only fibers with diameter  $\geq 0.2$  or  $0.3 \mu\text{m}$ , with length  $\geq 5 \mu\text{m}$ , and with aspect ratio  $\geq 3:1$  should be retained as "NIOSH equivalent" fibers.
- 4) It is unlikely that, in 200 (300 mesh) grid windows, greater than 100 fibers satisfying the NIOSH size criteria will be detected. However, if this is the case, the count may be terminated at this point.

### 7.3 Data Reduction

7.3.1 Data reduction is greatly facilitated by access to a computer. The program should use as input the following quantities: The total air volume filtered ( $V_a$ ), the effective filter area ( $A_f$ ), the area of a grid opening ( $A_g$ ), and the magnification at which fibers were sized  $M$  (or the horizontal and vertical magnification [ $M_H$  and  $M_V$ ] if fibers were sized with the screen tilted). The fiber counts read by the program should include all information entered on the count sheet, including grid opening number, fiber type (positive or tentative chrysotile, positive or tentative amphibole), fiber classification (fiber, bundle, or mat), structure classification (free or attached to particle), length flag (length augmented due to obscuring of an end), and size information (length  $L_i$  and width  $W_i$ , or width with horizontal and vertical components  $L^H_i$  and  $L^V_i$  of length if the screen is tilted, and estimated thickness of mat). Size of particles which have asbestos attached should also be entered, as well as some code to designate which fibers, bundles, or mats are attached to each particle.

7.3.2 Calculated quantities should include the concentration of total asbestos fibers ( $C_T$ ) with 95% confidence limits in fibers/ $\text{m}^3$ , the

concentration of asbestos structures ( $C_S$ ) with 95% confidence limits in structures/ $m^3$ , the concentration of free fibers ( $C_F$ ) with 95% confidence limits in free fibers/ $m^3$ , asbestos mass concentration ( $C_M$ ) in  $ng/m^3$ , and NIOSH equivalent fiber concentration ( $C_{NE}$ ) with 95% confidence limits in fibers/ $m^3$  0.3  $\mu m$  diameter, 5  $\mu m$  length. For example,  $C_T$  is calculated as,

$$(4) \quad C_T(\text{fibers} / m^3) = \frac{N_T A_f}{n A_g V_a (m^3)}$$

where  $N_T$  is the total number of fibers detected in  $n$  grid openings. A similar calculation is carried out for  $C_S$ ,  $C_F$ , and  $C_{NE}$ . Confidence limits should only be calculated if the fiber (or structure) counts are randomly distributed among grid windows (See Ref. 4.12 for statistical tests for random distributions and confidence interval calculation). Calculations should be done on each of the four fiber types, as well as for total asbestos (positive and tentative chrysotile, and positive and tentative amphiboles).

Mass concentration in  $ng/m^3$  should be calculated from the total estimated volume  $V_i$  (in units of  $10^{-9} \text{ cm}^3$  of each asbestos fiber, bundle, or mat, assuming specific gravities (SG) of 2.5 for chrysotile and 3.25 for amphiboles), by

$$(5) \quad C_m = \frac{SG A_f V_i}{n A_g V_a (m^3)}$$

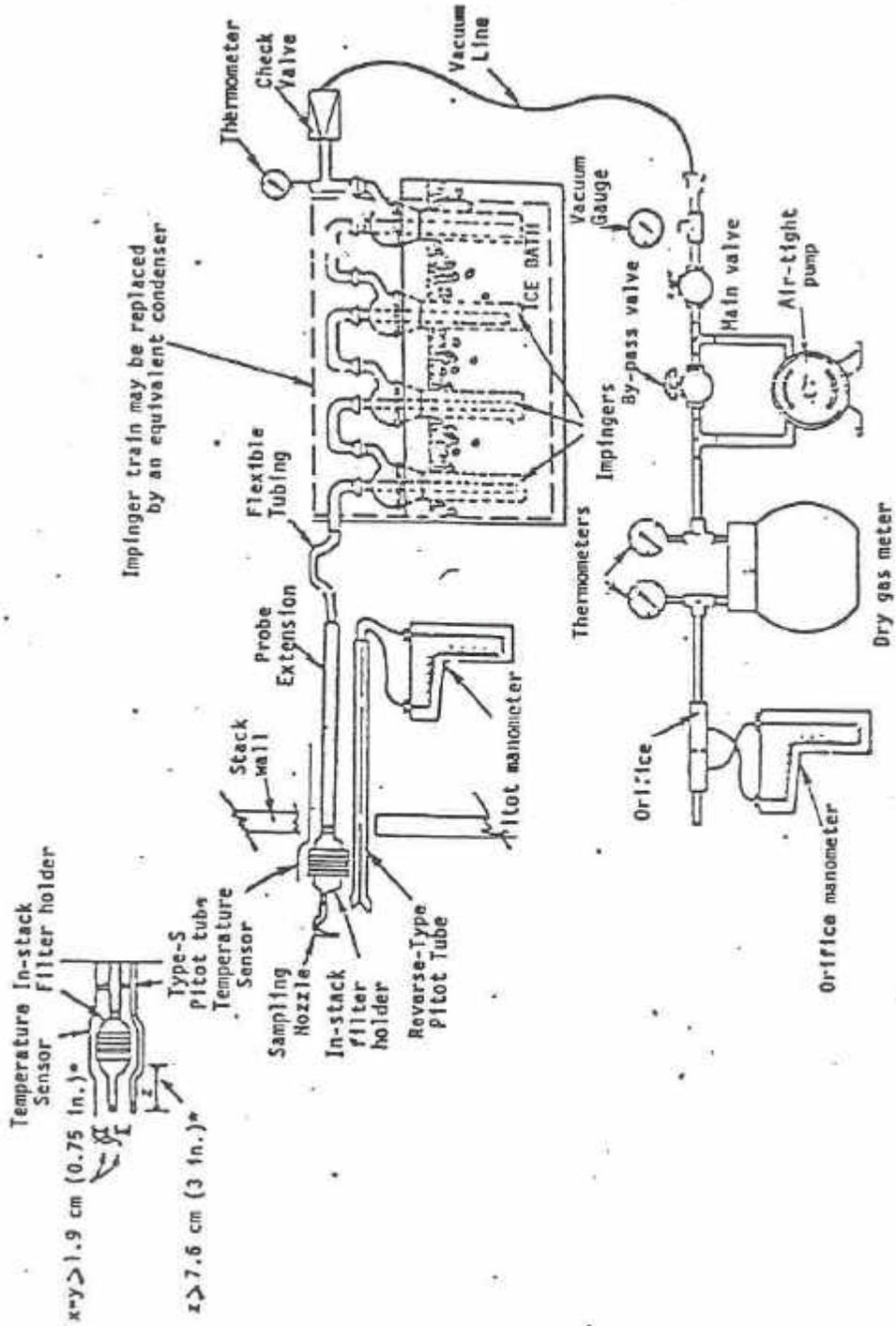
Means, standard deviations, geometric means, and minus one and plus one geometric standard deviation values should be calculated for lengths and widths. Also, it is desirable to have some graphical or tabular representation of length and width distributions. (See Figure 11 for a sample report sheet).

## 8. REFERENCE

Yamate, G. Methodology for the Measurement of Airborne Asbestos by Electron Microscopy, Final Report, Contract #68-02-3266, Environmental Monitoring and Systems Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, NC. (1982).

Figure 427-1

PARTICULATE SAMPLING TRAIN EQUIPPED WITH IN-STACK FILTER



\* Suggested (interference-free) spacings

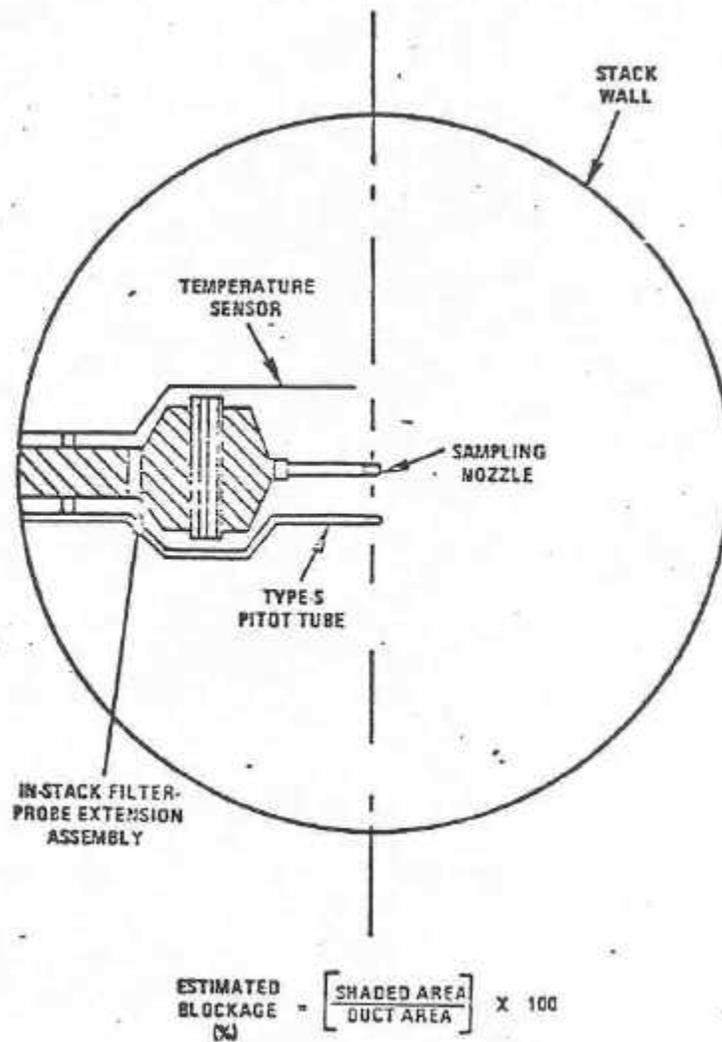


FIGURE 427-2. Projected area model of cross-section blockage (approximate average for a sample traverse) caused by an in-stack filter holder-probe extension assembly.



FIGURES

## FIGURE CAPTIONS

1. Plot of coefficient of variation (CV) versus structure count.
2. Plot of CV versus total figure count.
3. Electron diffraction (microdiffraction) pattern of right cylindrical orthochrysotile fiber. Silicate spacing is indicated.
4. Microdiffraction pattern of helical clinochrysotile fiber.
5. Energy dispersive x-ray spectrum (EDS) of chrysotile fiber. Copper peaks are from copper grid.
6. EDS of amosite fiber.
7. EDS of crocidolite fiber. Titanium peaks are from titanium grid.
8. EDS of tremolite fiber.
9. EDS of actinolite fiber.
10. Suggested count sheet format.
11. Sample report sheet.

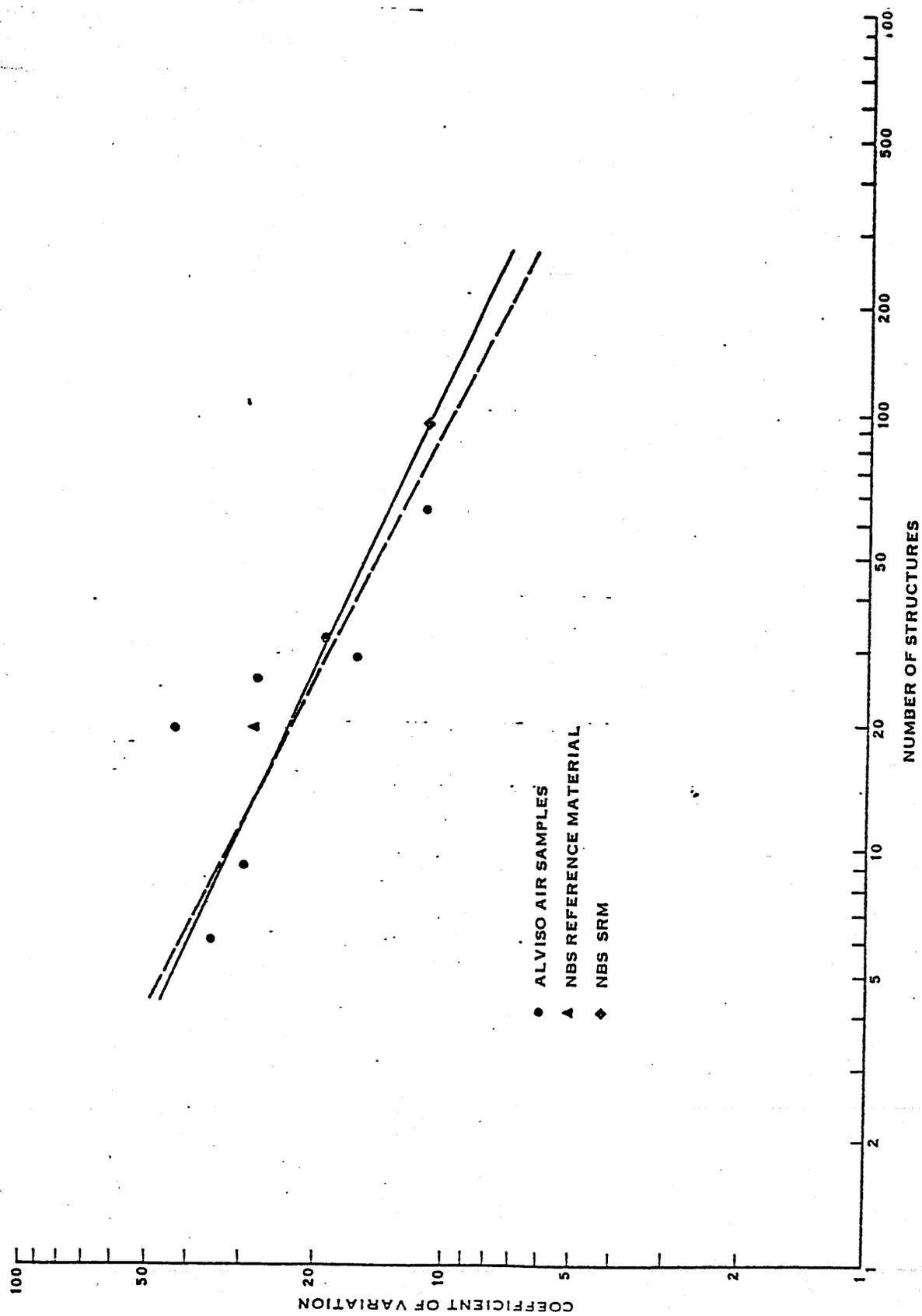


Figure 1

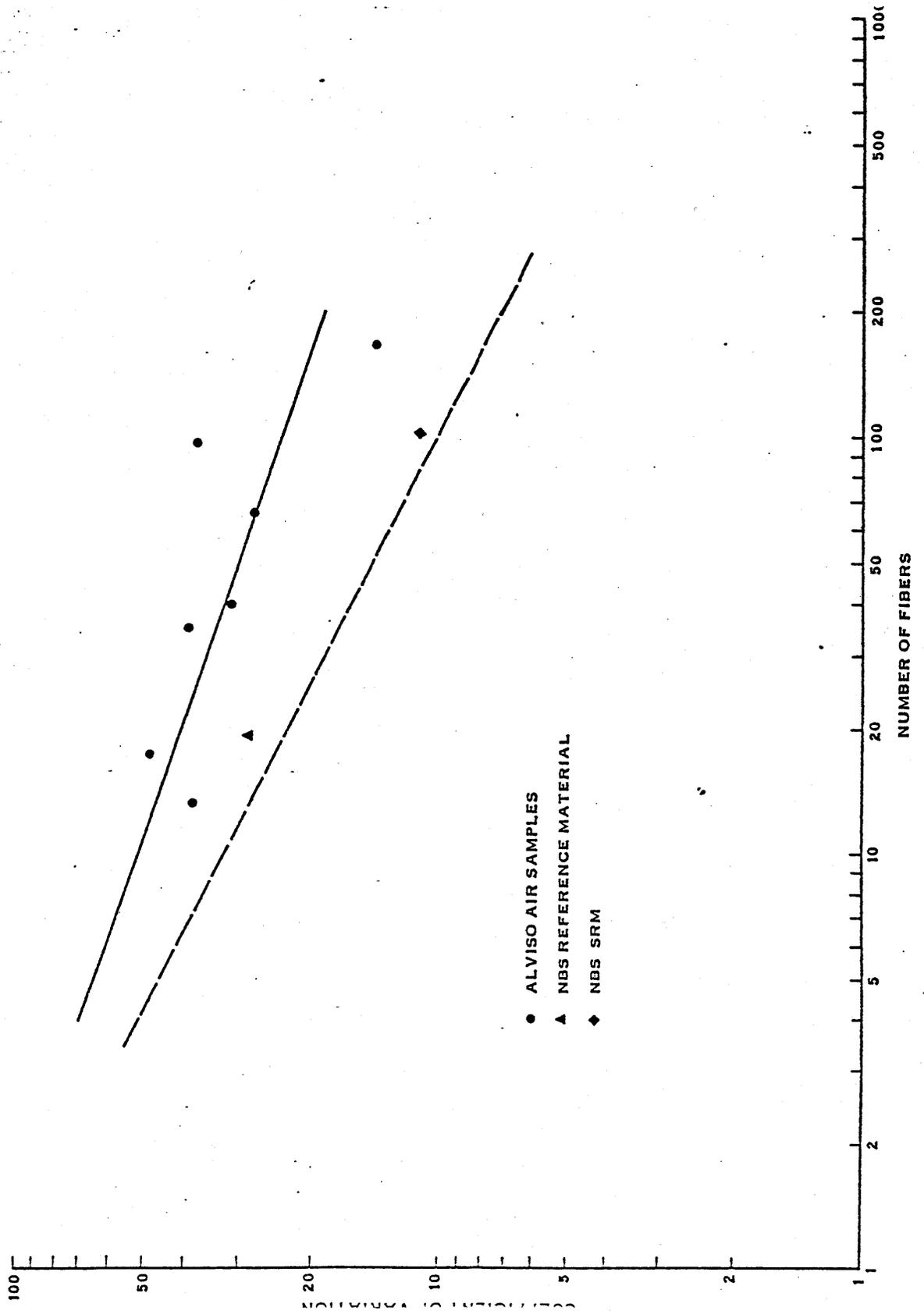


Figure 2

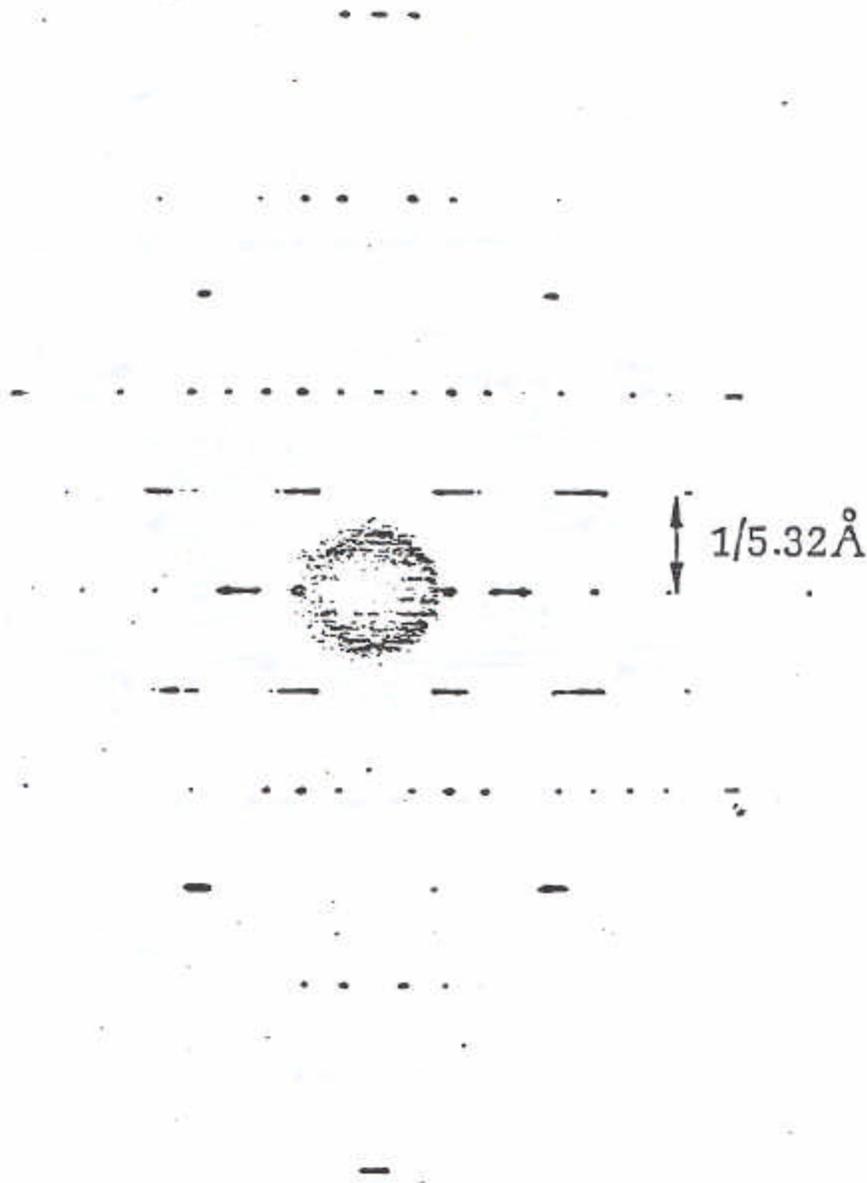


Figure 3. Electron diffraction (microdiffraction) pattern of right cylindrical orthochrysotile fiber. Silicate spacing is indicated.

[reserved for figure 4]

[reserved for figure 5]

[reserved for figure 6]

[reserved for figure 7]

[reserved for figure 8]

[reserved for figure 9]

[reserved for figure 10]

[reserved for figure 11]

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