



Fisheries and
Environment
Canada

Pêches
et Environnement
Canada



Environment
Canada
0015825C

Environnement
Canada

CANADA. AIR POLLUTION CONTROL
DIRECTORATE. REGULATIONS, CODES AND
PROTOCOLS REPORT EPS 1-AP

ELIAS

Standard Reference Methods for Source Testing: Measurement of Emissions of Asbestos from Asbestos Mining and Milling Operations

TD
883
C28
75-1

Regulations; Codes and Protocols
Report EPS 1-AP 75-1

Air Pollution
Control Directorate
December, 1976

ENVIRONMENTAL PROTECTION SERVICE REPORT SERIES

Reports pertaining to Regulations, Codes, and Protocols describe current legislation and administrative approaches favoured by the Environmental Protection Service

Other categories in the EPS series include such groups as Policy and Planning, Economic and Technical Review, Technology Development, Surveillance, Briefs and Submissions to Public Inquiries, and Environmental Impact and Assessment

Inquiries pertaining to Environmental Protection Service Reports should be directed to the Environmental Protection Service, Department of the Environment, Ottawa K1A 0H3, Ontario, Canada

**STANDARD REFERENCE METHODS FOR SOURCE TESTING:
MEASUREMENT OF EMISSIONS OF ASBESTOS FROM ASBESTOS
MINING AND MILLING OPERATIONS**

Air Pollution Control Directorate
Environmental Protection Service

Report EPS 1-AP-75-1

December 1976



DREI

TD

883

C 28

NO. 75-1

FOREWORD

The methods for sampling and analysis presented in this report are used in combination with those presented in Report EPS 1-AP-74-1 to determine asbestos emission levels from asbestos mining and milling operations

TABLE OF CONTENTS

	PAGE
FOREWORD	i
LIST OF FIGURES	iv
PART I SAMPLING	1
METHOD S-1 SAMPLING OF STACKS AND DUCTS IN ASBESTOS MILLING OPERATIONS	 3
S-1 1 Scope	3
S-1 2 Procedures	3
METHOD S-2 SAMPLING OF BAGHOUSE SYSTEMS IN ASBESTOS MILLING OPERATIONS	 13
S-2 1 Scope	13
S-2 2 Apparatus	13
S-2 3 Procedures	13
PART II ANALYSIS	19
METHOD A-1 ANALYSIS OF ASBESTOS SAMPLES FROM MINING AND MILLING OPERATIONS BY OPTICAL PHASE-CONTRAST MICROSCOPY	 21
A-1 1 Scope	21
A-1 2 Apparatus	21
A-1 3 Reagents	22
A-1 4 Procedure	22
A-1 5 Calculations	27
BIBLIOGRAPHY	31

LIST OF FIGURES

FIGURE		PAGE
S-1-1	ASBESTOS STACK-SAMPLING TRAIN (IN-STACK FILTER)	4
S-1-2	SECTIONED FILTER	5
S-1-3	MOISTURE ANALYSIS DATA SHEET	7
S-1-4	DATA SHEET - STACK SAMPLING	9
S-2-1	FILTER UNIT - EXPLODED VIEW	14
S-2-2	PUMP CALIBRATION ASSEMBLY	16
S-2-3	DATA SHEET - BAGHOUSE SAMPLING	17
A-1-1	FILTER-MOUNTING TEMPLATE	23
A-1-2	PORTON RETICLE	25
A-1-3	ASBESTOS FIBRE COUNTING REPORT FORM	28

PART I – SAMPLING

METHOD S-1 SAMPLING OF STACKS AND DUCTS IN ASBESTOS MILLING OPERATIONS

S-1.1 Scope

This method is applicable to the sampling of stacks or ducts in asbestos milling operations which are a source of particulate emissions to the atmosphere.

S-1.2 Procedures

Sampling shall be carried out according to the standard sampling procedures indicated in Report EPS 1-AP-74-1, *Standard Reference Methods for Source Testing. Measurement of Emissions of Particulates from Stationary Sources*. The following procedures, S-1.2.1 to S-1.2.5, refer to methods described in this report. Procedure S-1.2.6 incorporates certain modifications to the standard method and shall be used in place of Method E in the above report.

S-1.2.1 Determination of Sampling Site and Traverse Points

Method A, Report EPS 1-AP-74-1.

S-1.2.2 Determination of Stack Gas Velocity and Volumetric Flow Rate

Method B, Report EPS 1-AP-74-1

S-1.2.3 Determination of Molecular Weight by Gas Analysis

Method C, Report EPS 1-AP-74-1

S-1.2.4 Determination of Moisture Content

Method D, Report EPS 1-AP-74-1

S-1.2.5 Calibration Procedure for S-Type Pitot Tube, Dry Gas Meter, Orifice Meter, and Rotameter

Method F, Report EPS 1-AP-74-1

S-1.2.6 Sampling for Particulate Asbestos Emissions

S-1.2.6.1 Principle. Particulate matter, including asbestos particles, is withdrawn isokinetically from a number of sampling points in the stack. Sampling isokinetically means that the linear velocity of the gas entering the sampling nozzle is equal to that of the undisturbed gas stream at the sample point.

S-1.2.6.2 Apparatus

Sampling Train (Figure S-1-1).

Nozzle. A stainless steel nozzle with a sharp, tapered leading edge is required.

Probe. A Pyrex probe encased in stainless steel is required. This probe must have a heating system capable of maintaining the temperature of gas at the exit end in excess of 250°F during sampling to prevent condensation.

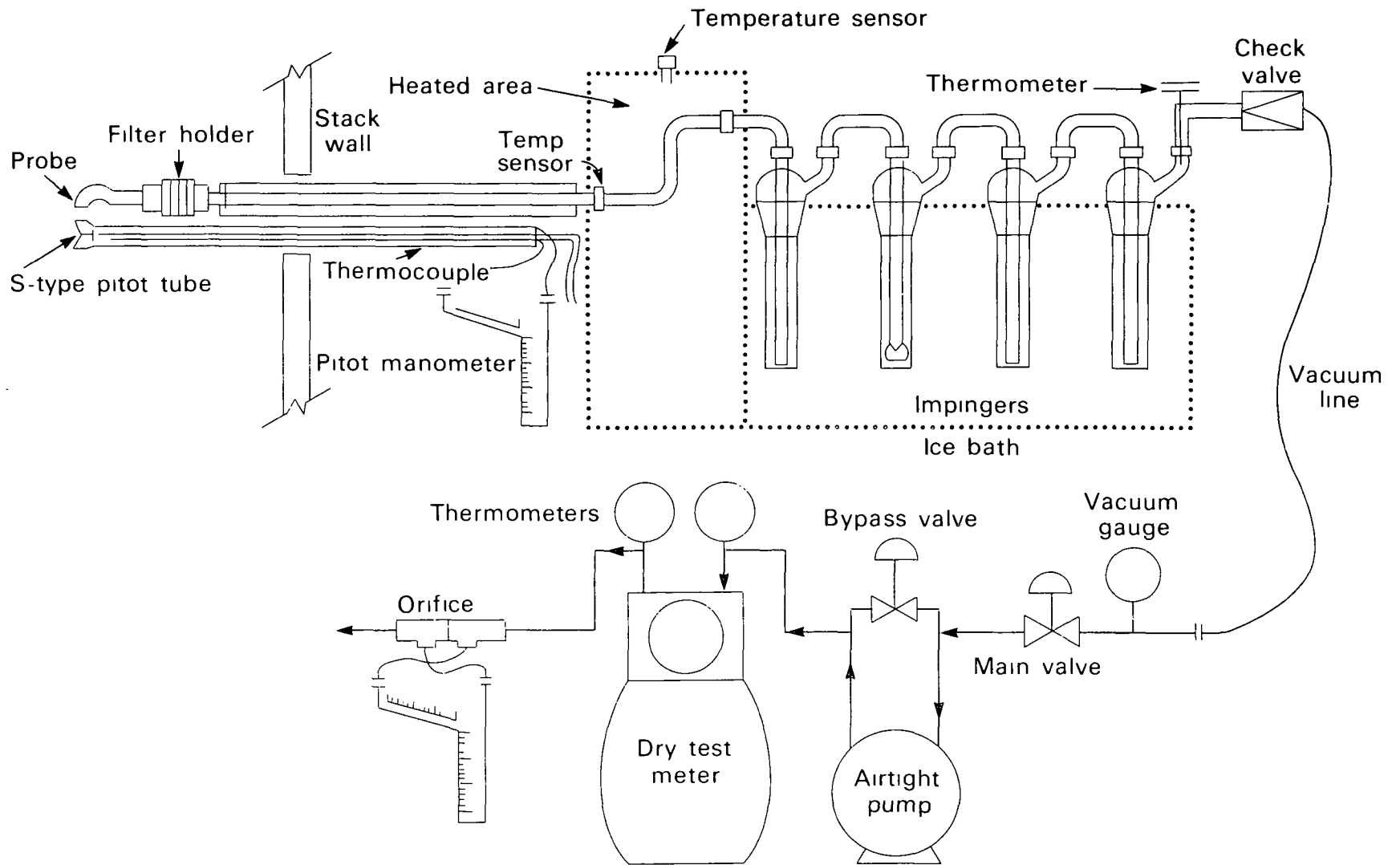


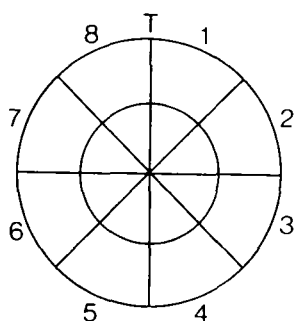
FIGURE S-1-1 ASBESTOS STACK SAMPLING TRAIN (IN-STACK FILTER)

S-Type Pitot Tube An S-type pitot tube or equivalent is attached to the probe to monitor stack-gas velocity

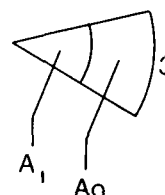
Temperature Gauge A thermocouple or equivalent, capable of measuring stack temperatures to within 1.5% of the minimum absolute stack temperature, is attached to the pitot tube

Filter Holder An in-line filter holder designed for filters 47 mm in diameter is required. It must be able to withstand temperatures up to 200°F and must include a stainless steel filter support pad and a suitable gasket

Filter. A cellulose membrane filter 47 mm in diameter, with 0.8 μm nominal pore size, is required. When supported in the filter holder there is an effective filtering area of about 960 mm². The effective filtration area is marked with a grid of eight angular 45° sectors which are each divided into an inner (A_i) and outer (A_o) radial area. The ratio A_i : A_o is 1 : 3. Each angular sector is identified by a printed number on its outer edge. A 'T' is printed to identify the top of the filter. Diagrams of the sectioned filter and a typical filter sector are given in Figure S-1-2



Complete Filter



Typical Filter Sector

FIGURE S-1-2 SECTIONED FILTER

Impingers Four Greenburg-Smith impingers are connected in series. The first, third, and fourth impingers are modified by replacing the tips and impaction plate of the standard design with a 1/2-in. I.D. glass tube extending to within 1/2 in. of the bottom of the flask. The second impinger has the standard tip and impaction plate.

Leakless Vacuum Pump. A vacuum pump capable of maintaining an isokinetic sampling rate and continuously withdrawing a portion of the stack gases through the sampling train is used. The pump is connected to the outlet of the last impinger by a vacuum line containing a gauge to measure the pump intake vacuum to within 0.5 in. Hg and a coarse adjust valve to regulate the sample flow. A bypass valve is connected across the vacuum pump to allow fine control of the sample flow.

Dry Gas Meter. A dry gas meter calibrated to within $\pm 1\%$ and equipped with inlet and outlet temperature indicators is used to determine sample volume. The dry gas meter follows the vacuum pump in the sampling train.

Orifice Meter. A calibrated orifice meter is used to measure the sampling flow rate. The orifice meter is connected to the outlet of the dry gas meter.

Differential Pressure Gauges. Inclined manometers, or equivalent, capable of measuring pitot tube velocity pressure and the pressure drop across the orifice meter to within 0.025 in. H₂O are required.

Barometer. A barometer capable of measuring atmospheric pressure to within ± 0.1 in. Hg should be used.

Sample Recovery and Analysis

Trip Balance. A balance with a 300-g capacity, capable of measuring to within ± 0.05 g is required.

Miscellaneous. A wash bottle, sample-storage containers, and a 250-ml graduated cylinder are also required.

S.1.2.6.3 Reagents

Sampling Train. Indicating-type, 6-16 mesh silica gel, dried at 350°F for 2 h, is required together with deionized or distilled water, and crushed ice.

Sample Recovery and Analysis. Deionized or distilled water is required.

S.1.2.6.4 Procedure

Sampling Train

Preliminary. Select the sampling site and minimum number of sampling points according to the procedures described in Method S-1.2.1. Determine the stack pressure, temperature, moisture, and range of velocity pressures, and use this information to calculate the required isokinetic sampling flow rate during subsequent stack-sampling tests. Determine the nozzle size required for isokinetic sampling using this data. Recommended minimum nozzle size is 1/4 in. I.D.

Preparation of Collection Train. The filter holder is loaded in the laboratory. A separate preloaded filter holder must be prepared for each sample. Place the filter in the holder so that the 'T' on the filter corresponds with an external marking on the holder. Seal the ends of the loaded filter holder with plastic plugs and place in a suitable container with the filter face in the upright position. The preloaded filter holder should be carefully conveyed to the sampling site.

Place 100 ml of deionized or distilled water in each of the first two impingers and weigh. Weigh the third impinger and leave empty. Place approximately 200 g of silica gel in the fourth impinger and weigh. Record these initial weights on the moisture analysis data sheet, Figure S-1-3.

Plant _____

Location _____

Test _____

Date _____

Test conducted by _____

Impinger number	Contents	Weight (gm)
1	Water	final _____ initial _____
2	Water	final _____ initial _____
3		final _____ initial _____
4	Silica gel	final _____ initial _____

Total moisture (V_w)* _____ ml

$$* \text{ Volume of water, ml} = \frac{\text{weight of water}}{1\text{g/ml}}$$

FIGURE S-1-3

MOISTURE ANALYSIS DATA SHEET

At the sampling site, attach a loaded filter to the probe and then attach the nozzle to the holder. Align the nozzle so that the "T" on the filter will be at the top of the filter holder with respect to the nozzle tip. Attach the probe to the sampling train so that the final configuration is as indicated in Figure S-1-1. Check the sampling unit for leaks by plugging the nozzle inlet with a rubber stopper and pulling a 15 in. Hg vacuum. If the needle on the dry gas meter moves, a leakage rate not in excess of 0.02 ft³/min at a vacuum of 15 in. Hg is acceptable. After leak checking, adjust the heater to provide a minimum gas temperature of 250°F at the probe outlet. Adjust the temperature in the heated area ahead of the impingers to 225°F. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger as low as possible, preferably at 70°F or less. Temperatures above 70°F may result in damage to the dry gas meter from either condensation or excessive heat.

Particulate Train Operation. To begin sampling, position the sampling nozzle at the first sampling point (traverse point). Point the nozzle directly into the approaching gas stream and secure the entire apparatus to the support system. Immediately start the vacuum pump and adjust the sampling flow rate to isokinetic conditions. Sample for at least 2 min at each sampling point, sampling time must be the same for each point. Maintain isokinetic sampling throughout the sampling period by making the necessary adjustments in the sampling flow rate as stack conditions change, or as the buildup of particulate matter on the filter affects the flow. Nomographs are available, or can be constructed, which aid in the rapid adjustment of the sampling rate without other computations. For each run, record the data required on the data sheet in Figure S-1-4. Record instrument readings at intervals consistent with the test duration established for each point. For example, if testing for a minimum of 2 min per point, record readings every minute and whenever flow adjustments are necessary. The time between readings should not exceed 2 min. When the traverse is completed, turn off the vacuum pump and record the final instrument readings. Transfer the sampling apparatus to the other sampling port and repeat the sampling procedure. When the second traverse is completed, remove the sampling apparatus from the stack and handle in accordance with the sample recovery procedure described below.

Sample Recovery

At the sampling site, disconnect the probe from the sampling train. Carefully disconnect the filter holder from the probe and avoid jarring or knocking the unit. Once removed, hold the filter holder in a horizontal position so that the exposed filter surface is facing upward at all times and carefully disconnect the nozzle. Seal the filter holder with plastic plugs and place upright in a container for transfer to the analytical laboratory.

Sample Analysis

Filter. The filter is analysed for asbestos content according to the analytical procedure (A-1) in Part II. For each filter sample obtained, a minimum of four *alternate* sectors must be analysed (1, 3, 5, and 7, or 2, 4, 6, and 8). The result for the filter sample is the average of the results for the four or more sectors analysed.

Impingers. Determine the final weights of all four impingers and their contents to the nearest 0.5 g and record on the moisture analysis data sheet (Figure S-1-3).

Plant _____ Nozzle diameter _____ in
 Location _____ Probe length _____ ft
 Test no _____ Probe temperature _____ °F
 Date _____ Dry gas meter calibration factor _____ in H₂O
 Ambient temperature _____ °F Pitot calibration factor _____
 Barometric pressure _____ in Hg P_s = _____ in H₂O
 Moisture content (assumed) _____ % Test by _____
 Stack diameter _____ ft

Point	Time	Stack gas temp (°F)	Velocity pressure (in H ₂ O)	Orifice pressure (in H ₂ O)	Gas meter vol (ft ³)	Gas meter temp (°F)		Last impinger temp (°F)	Pump vacuum (in Hg)
						Inlet	Outlet		

FIGURE S-1.4 DATA SHEET - STACK SAMPLING

Total moisture (V_{lc}) is the total volume of liquid collected in all impingers

S.1.2.6.5 Calculations

Average Dry Gas Meter Temperature and Average Orifice Pressure Drop. Refer to data sheet (Figure S-1-4).

Dry Gas Volume. Correct the sample volume measured to reference conditions (77°F and 29.92 in. Hg), using Equation S-1-1.

$$\begin{aligned} \text{(Equation S-1-1)} \quad (V_{m})_{ref} &= V_m \left(\frac{T_{ref}}{T_m} \right) \left(\frac{P_{bar} + \frac{\Delta H}{13.6}}{P_{ref}} \right) \\ (V_{m})_{ref} &= 17.95 \left(\frac{V_m}{T_m} \right) \left(P_{bar} + \frac{\Delta H}{13.6} \right) \end{aligned}$$

where

- $(V_{m})_{ref}$ – sample volume corrected to reference conditions, ft³
- V_m – volume of gas measured by dry gas meter at meter conditions, ft³
- T_{ref} – absolute temperature at reference conditions, 537 °R
- T_m – average dry gas meter temperature, °R
- P_{bar} – barometric pressure, in. Hg
- ΔH – average pressure drop across orifice (see Figure S-1-4), in. H₂O
- 13.6 – conversion factor, in. H₂O/in. Hg
- P_{ref} – absolute pressure at reference conditions, 29.92 in. Hg

Volume of Water Vapour. Convert the volume of water condensed during the stack test to a volume of water vapour at reference conditions using Equation S-1-2

$$\begin{aligned} \text{(Equation S-1-2)} \quad (V_w)_{ref} &= V_{lc} \left(\frac{\rho_{H_2O}}{M_{H_2O}} \right) \left(\frac{RT_{ref}}{P_{ref}} \right) \left(\frac{1}{453.6} \right) \\ &= 0.0479 V_{lc} \end{aligned}$$

where

- $(V_w)_{ref}$ – volume of water vapour in gas sample at reference conditions, ft³
- V_{lc} – total volume of liquid collected in impingers (see Figure S-1-3), ml
- ρ_{H_2O} – density of water, 0.9982 g/ml

M_{H_2O}	-	molecular weight of water, 18 lb/lb-mole
R	-	ideal gas constant, 21.83 in Hg ft ³ /lb-mole °R
T_{ref}	-	absolute temperature at reference conditions, 537°R
P_{ref}	-	absolute pressure at reference conditions, 29.92 in Hg
453.6	-	conversion factor, g/lb

Moisture Content Calculate the moisture content of the stack gas using Equation S-1-3.

$$\text{(Equation S-1-3)} \quad B_{wo} = \frac{(V_w)_{ref}}{(V_m)_{ref} + (V_w)_{ref}}$$

where

B_{wo}	-	proportion by volume of water vapour in the gas stream
$(V_w)_{ref}$	-	volume of water vapour in the gas sample at reference conditions (see Equation S-1-2), ft ³
$(V_m)_{ref}$	-	volume of gas through dry gas meter at reference conditions, ft ³

Acceptable Results The percent isokinetic variation (%I), calculated for each sample point using Equation S-1-4, should fall within the range 90% < %I < 110%

[Equation S-1-4]

$$\text{(Equation S-1-4)} \quad \%I = \frac{\frac{V_m}{t} \left(\frac{1}{1 - B_{wo}} \right) \left(P_{bar} + \frac{\Delta H}{13.6} \right) T_s}{0.3272 (N_d)^2 \left(\frac{T_{mi} + T_{mo}}{2} \right) P_s U_s} \times 100$$

where

I	-	ratio of the sampling velocity through the nozzle to stack gas velocity
V_m	-	volume of gas sampled through gas meter at meter conditions for each point sampled, ft ³
t	-	sampling time for each point sampled, min
B_{wo}	-	proportion by volume of water vapour in the gas stream (see Equation S-1-3)
P_s	-	absolute stack-gas pressure, in Hg
T_s	-	absolute stack-gas temperature for each point sampled, °R
T_{mi}	-	dry gas meter, absolute inlet temperature for each point sampled, °R

T_{mo}	-	dry gas meter, absolute outlet temperature for each point sampled, °R
P_{bar}	-	barometric pressure at the sampling site, in Hg
U_s	-	stack gas velocity at each point sampled, ft/s
N_d	-	inside diameter of sampling nozzle, in
ΔH	-	pressure drop across orifice meter for each point sampled, in H ₂ O
0.3272	-	conversion factor, (s/min) (ft ² /in ²) (π)
13.6	-	conversion factor, in H ₂ O/in Hg

METHOD S-2 SAMPLING OF BAGHOUSE SYSTEMS IN ASBESTOS MILLING OPERATIONS

S-2.1 Scope

This method is employed where method S-1 cannot be applied and where access to the clean-air side of the baghouse is possible. The equipment and procedures required for sampling in baghouses by an open-face-filter method employing membrane filters are described. Handling of the samples after recovery is also described.

S-2.2 Apparatus

Filter Holder. A standard, 37-mm acrylic plastic filter holder with a filter support pad is required. Figure S-2-1 is an exploded schematic of this filter holder.

Filters. A cellulose acetate membrane filter, 37 mm in diameter with 0.8 μm pore size, is used. When supported on the filter holder there is an effective filtering area of about 855 mm^2 . The effective filtration area is printed with a grid which divides it into eight, 45° angular sectors. Each sector is further divided into an inner (A_i) and outer (A_o) radial area. The ratio A_i/A_o is 1/3, (Figure S-1-2).

Pump. A portable vacuum pump capable of maintaining a sampling rate of 2 lpm for 4h or more is required. A bypass valve is connected across the vacuum pump to allow control of the sample flow rate.

Rotameter. A calibrated rotameter, accurate to within $\pm 2\%$, is required for adjustment of the flow rate through the filter unit.

Temperature Gauge. A thermometer is required which will measure ambient baghouse temperatures to within $\pm 1^\circ\text{C}$.

Barometer. A barometer capable of measuring absolute atmospheric pressure to within 2 mm Hg is required.

Cellulose Collars. These collars are used to seal the filter holders so that air cannot bypass the filter.

S-2.3 Procedures

S-2.3.1 Sampling Sites. For any particular baghouse system a minimum number of filter samples must be taken through the area of the baghouse to obtain a representative test result for that system. Thus, a valid test result for any baghouse system will be the arithmetic average of all the filter samples taken to yield a representative sampling of the air in that system.

The minimum number of filter samples to be taken is a function of the exhaust capacity of the system and the practicalities involved in sampling large capacity units. To ensure a representative sampling of the system with a reasonable number of filter samples, Equation S-2-1 is used to determine the minimum number of filter samples which must be taken to yield a valid test run.

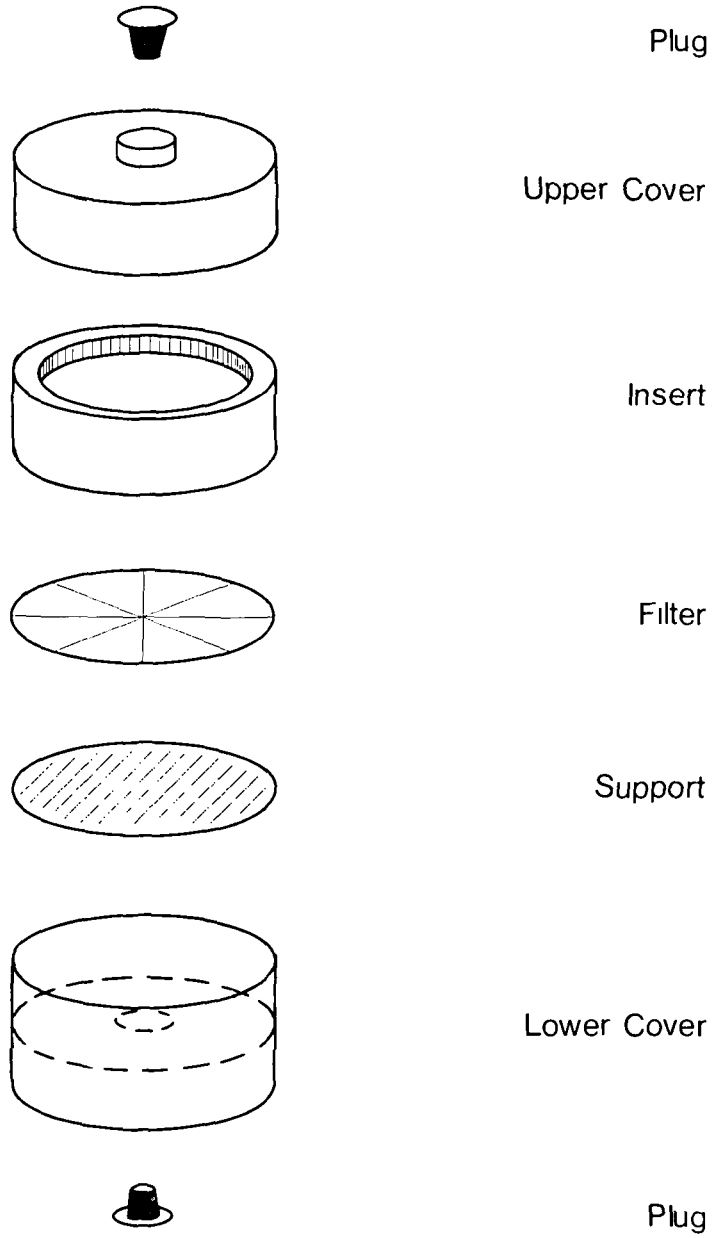


FIGURE S-2-1 FILTER UNIT - EXPLODED VIEW

$$\text{(Equation S-2-1)} \quad N = 0.22 \times (\text{CFM})^{0.25}$$

where

- N - minimum number of filter samples
 CFM - rated exhaust capacity of baghouse, cfm

When applying this equation, any fractional number must be rounded off to the next highest integer. When small-capacity baghouse units are encountered at least three filter samples must be taken to yield a valid test run, regardless of the result obtained using Equation S-2-1.

Locate the minimum number of filter samples as evenly as possible throughout the bagroom. Exact locations are to be agreed upon by the inspector and the person responsible for sampling. Prepare a schematic of the bagroom layout indicating locations of samples and fans with respect to the bags. For a valid test run, all samples should be taken simultaneously whenever possible. If restrictions on time or supply of equipment preclude simultaneous sampling, all samples must be taken within two days.

S-2.3.2 Preparation of Filter Unit. Using a pair of clean tweezers place a filter, with the printed side up, on the support pad of the holder. Replace the insert and upper cover of the holder, insert the plugs and then place a wet cellulose collar around the unit so that it seals the joint between the insert and lower cover of the holder. Allow the collars to dry and store the prepared units upright, in a suitable container, for transport to the sampling site.

S-2.3.3 Sampling. Before sampling, ensure that the pump batteries are fully charged and that the pump is operational. Using a suitable piece of tubing, attach the filter unit to the inlet of the pump. Remove the upper cover, exposing the filter surface. Adjust the pump withdrawal rate to 2 lpm using a previously calibrated rotameter, as shown in Figure S-2-2. Print the sample number on the filter unit and record all pertinent sampling data on the data sheet, shown in Figure S-2-3. Remove the calibration assembly and allow the pump to run for up to 4h depending on the concentration expected. The minimum sampling time is that required to achieve the optimum filter loading for analysis, 5 asbestos fibres per field. When sampling, the printed filter face is generally pointed downward. If the general flow of the air stream is known, the filter face should be placed parallel to the air flow. At the end of the sampling period check the flow rate as before and record this value. If the difference between initial and final rates is less than 0.3 lpm the sample is acceptable and the arithmetic average of the two values is used as the sampling rate. If the difference in values is greater than 0.3 lpm the sample is discarded and another taken in the same location within two days.

On completion of sampling, carefully remove the filter unit from the pump, replace the top cover and sealing plugs and place the sample in a container with the printed surface of the filter facing upward. When conveying the samples to the analytical site avoid jarring the units and ensure that they are maintained upright at all times.

S-2.3.4 Analysis. The filter samples are analysed according to Method A-1 in Part II. At least one of the eight sectors of the filter must be analysed, usually sector 1. Replace the rest of the filter in the holder, seal and store upright for future evaluation if necessary.

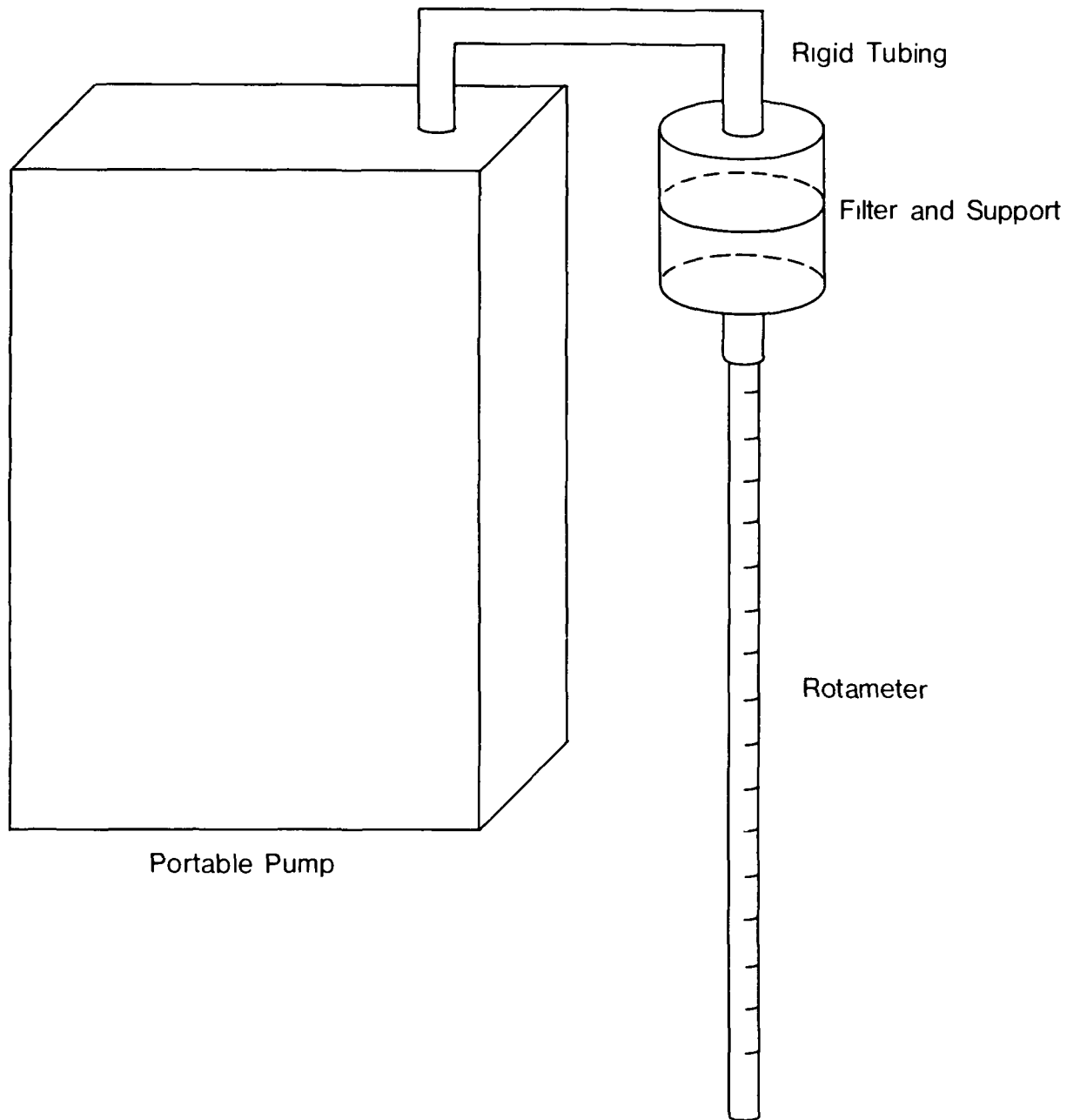


FIGURE S-2-2 PUMP CALIBRATION ASSEMBLY

Plant _____ Baghouse Temperature _____
 Baghouse _____ Absolute Pressure _____
 Sampled by _____ Date _____

Filter no	Sampling rate (lpm)			Time of sampling		Remarks
	Initial	Final	Difference	Start	Stop	

Baghouse schematic indicating sampling locations

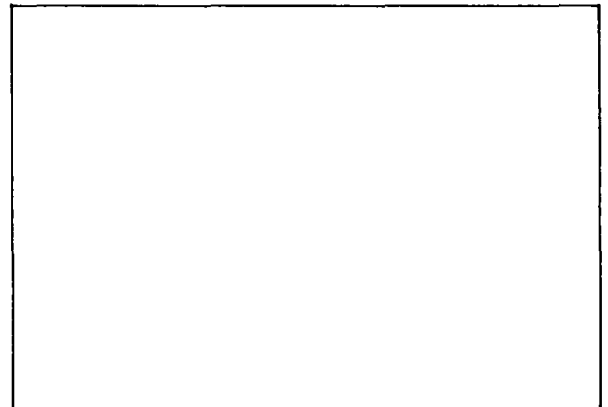


FIGURE S-2-3

DATA SHEET - BAGHOUSE SAMPLING

PART II – ANALYSIS

METHOD A-1 ANALYSIS OF ASBESTOS SAMPLES FROM MINING AND MILLING OPERATIONS BY OPTICAL PHASE-CONTRAST MICROSCOPY

A-1.1 Scope

The equipment and procedures necessary for mounting, sizing, and counting asbestos fibres obtained by standard sampling techniques are described. The method applies to samples collected on membrane filters and considers only those asbestos fibres greater than 5 μm in length with an aspect ratio of at least 3:1.

A-1.2 Apparatus

A-1.2.1 Filter-Mounting Equipment. Filter samples should be mounted in a clean environment to avoid contamination. The following items will facilitate the mounting of the sample.

Microscope Slides. Glass slides 25 x 75 mm are used. Those with a frosted end are preferable so that sample data can be easily recorded.

Cover Slips. Cover slips of a size sufficient to cover the filter wedge are required. A No. 1 1/2 (17 mm) cover slip is generally used as most objectives are optically corrected for this thickness.

Scalpel. A scalpel is required to cleanly and easily section the filter sample.

Tweezers. A pair of tweezers is required to remove the filter sample from its holder and to place the sector to be analysed on the glass slide.

Lens Tissue. Lens tissue (lint-free) is used to clean all mounting equipment and slides prior to use.

Glass Rod. A glass rod is required to spread the mounting solution on the slide.

Wheaton Balsam Bottle. This bottle is used to hold the mounting solution and has a special top which helps prevent contamination of the solution. A glass rod is included for dispensing the solution.

A-1.2.2 Optical Equipment. A light-field microscope fitted with phase-contrast accessories and capable of achieving a magnification of 400X is required. The following parts and accessories must be available with this unit:

- (a) 10X Huygenian eye-piece,
- (b) Koehler illumination,
- (c) Mechanical stage;
- (d) Abbe or Zernike condenser fitted with a phase ring with a numerical aperture (N.A.) equal to or greater than the N.A. of the objective,
- (e) 40X - 45X positive phase-contrast achromatic objective (N.A. 0.65-0.75),
- (f) Phase ring-centering telescope,
- (g) Porton reticle,
- (h) Stage micrometer (2 mm divided into units of 0.01 mm).

A-1.3 Reagents

A-1.3.1 Mounting Solution. Reagent-grade dimethyl phthalate and diethyl oxalate which are free of particles and colour are required. Filter material identical to that used in sampling is also required.

A-1.4 Procedure

A-1.4.1 Preparation of Mounting Solution. Prepare a 1:1 solution of dimethyl phthalate in diethyl oxalate by mixing together equal volumes of these reagents. For each millilitre of this solution add 0.05g of filter medium and stir until the filter dissolves. This mounting solution is stable for up to six months but only a small amount should be prepared at any time since very little is required to mount each sample. About 300 samples can be prepared from 20 ml of mounting solution.

A-1.4.2 Sample Mounting. The exposed filter is carefully sliced into the appropriate number of sectors for analysis of the sample. (Refer to sampling methods S-1 and S-2 for number of sectors per filter sample which must be analysed.) Each sector is a 45° wedge of the filter sample.

With the glass rod supplied with the Wheaton Balsam bottle apply a small drop of mounting solution to a cleaned glass slide. It is important to avoid using an excessive amount of solution in order to prevent migration of the asbestos particles. To lessen this problem, it is useful to prepare a template on which a 45° wedge in the shape of a sample sector has been etched (Figure A-1-1). The glass slide is placed over this template so that the wedge appears in the centre of the slide. A drop of mounting solution is then placed in the centre of the wedge. The size of the drop is correct when, on settling, it touches or slightly overlaps the edges of the wedge.

With a clean glass rod, spread the spot of mounting solution so that it coincides with the shape of the filter wedge.

Using tweezers, grasp the sector to be mounted by its unexposed outer edge and place the sector, exposed side up, on the mounting medium.

Carefully place a clean cover slip over the filter sector. Do not reposition the cover slip once contact has been made.

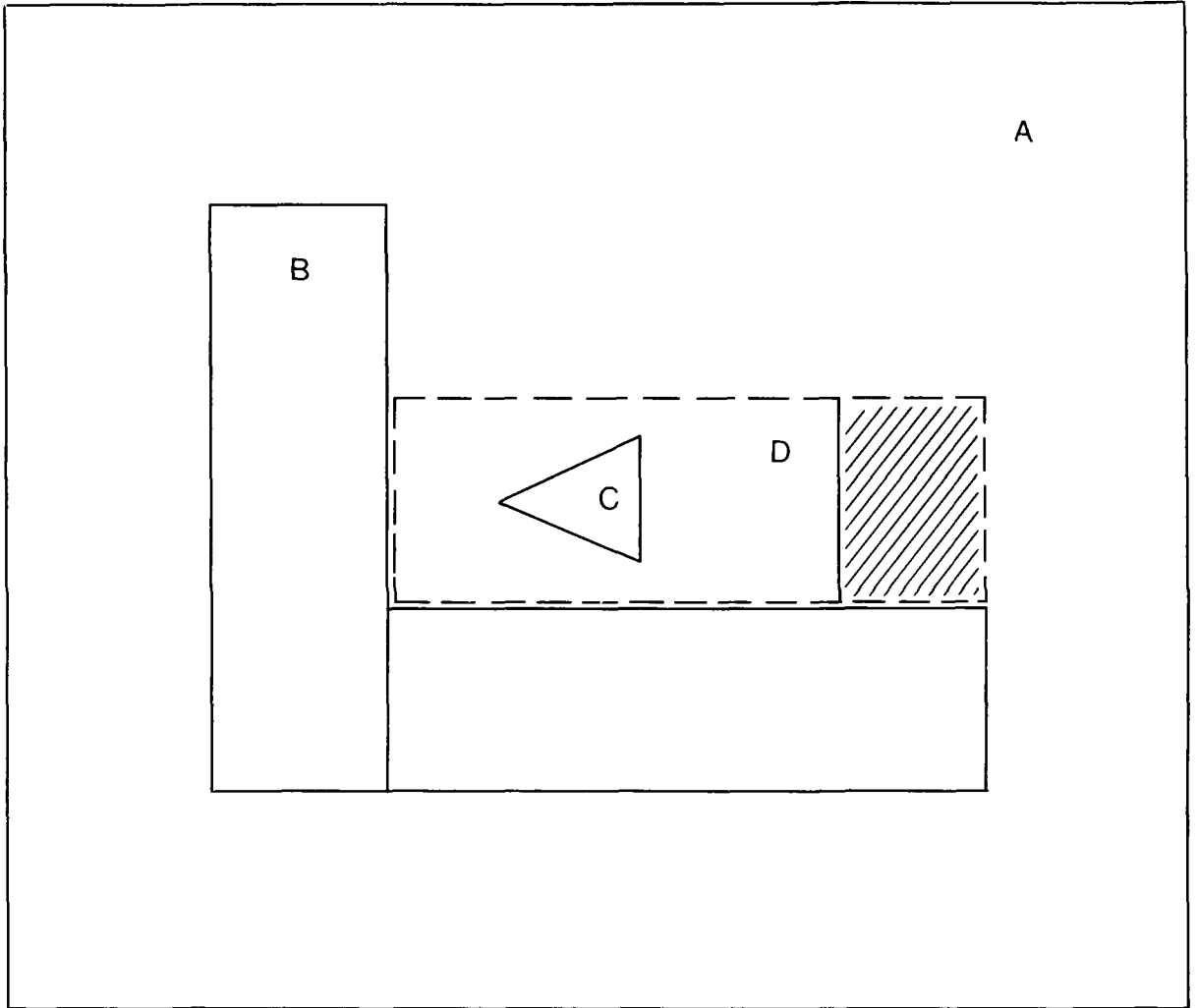
Label the slide with a sample number and mounting date.

The sample should become transparent within 15 min but approximately 1 h should be allowed for all graininess to disappear before the slide is counted.

If the edge of the cover glass is not made air-tight to form a 'sealed' mount, the slide should be counted within three days. A sample with a sealed mount must be counted within 25 days but preferably should be counted as early as possible.

A-1.4.3 Optical System - Adjustment and Calibration

A-1.4.3.1 Microscope Adjustment. The microscope is adjusted, according to manufacturer's instructions, to produce positive phase-contrast images using Koehler illumination. The following general guidelines must be observed.



Scale = Actual Size

LEGEND

- (A) Template Body
- (B) Perpendicular Raised Edges
- (C) 45° Wedge Shape
- (D) Glass Slide

DETAILS

- ¼" white acrylic plastic sheet
- plastic rulers glued to body
- etched into body with scribing tool
- placed over wedge and held in position by raised edges

FIGURE A-1-1 FILTER-MOUNTING TEMPLATE

- (a) The light source must be in focus and centered on the condenser iris or annular diaphragm
- (b) The object for examination must be in focus
- (c) The illuminator field iris must be in focus, centered on the sample, and opened only to the point where the field of view is illuminated
- (d) The phase rings (annular diaphragm and phase-shifting elements) must be concentric

A-1.4.3.2 Calibration of Porton Reticle. The porton reticle is a glass plate inscribed with a series of circles and rectangles (Figure A-1-2). This reticle is used to determine fibre length and also to define a field area in which the asbestos fibres can be counted. The fibre lengths are determined by comparison with the circles on the reticle. The square on the left of the reticle, which is divided into six rectangles, is the counting field area (f.a.)

The porton reticle rests on the field-limiting diaphragm in the Huygenian eyepiece, and remains in sharp focus, superimposed on the microscope's field of view.

In order to evaluate the asbestos fibre concentration of the sample, the reticle must be properly calibrated. The calibration of the reticle will vary with the specific eyepiece-objective-reticle combination used. Should any of these be changed (i.e. disassembly, replacement, zoom adjust, etc) the combination must be recalibrated. The stage micrometer is used to calibrate the porton reticle as follows

- (a) Clip the stage micrometer onto the mechanical stage,
- (b) Focus on the micrometer scale at the 40X - 45X objective magnification,
- (c) Align the left boundary of the large rectangle on the reticle with the first scale division of the micrometer,
- (d) Determine the length of the large rectangle in millimetres by counting the number of scale divisions of the micrometer along this length.

When the length of the large rectangle has been determined, the field area (f a) and circle diameters (D_n) can be calculated from the following relationships

- (a) The length of the large rectangle is defined as 200L units.
- (b) The width of the large rectangle is 100L units
- (c) Thus the area of the counting field (f a) is $(100L)^2$
- (d) The diameters of the circles are given by the equation

$$D_n = L \sqrt{2^n}$$

where

n - circle number

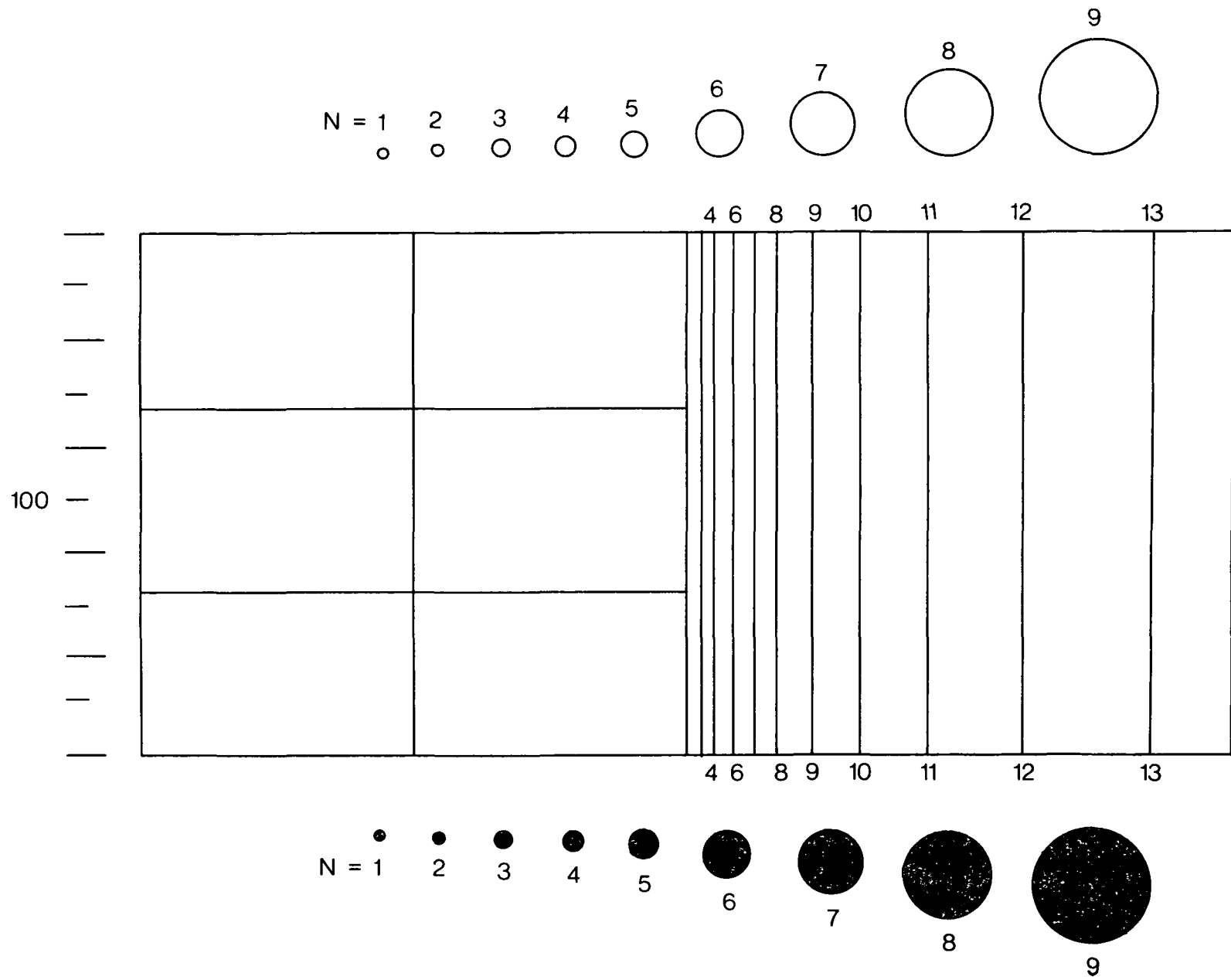


FIGURE A-1-2 PORTON RETICLE

Example

Suppose the length of the rectangle as measured by the stage micrometer was 132 mm

$$\begin{aligned} \text{Then, } 200L &= 132 \text{ mm} \\ L &= 00066 \text{ mm} = 66 \mu\text{m} \end{aligned}$$

$$(i) \quad \begin{aligned} \text{Counting field area (f a)} \\ f a &= (100L)^2 = 00436 \text{ mm}^2 \end{aligned}$$

$$(ii) \quad \begin{aligned} \text{Circle diameters (D}_n\text{)} \\ D_1 &= 66 \sqrt{2^1} = 93 \mu\text{m} \\ D_2 &= 66 \sqrt{2^2} = 132 \mu\text{m} \end{aligned}$$

Since the circle diameters vary logarithmically, every other circle doubles in diameter

Thus,

$$\begin{aligned} D_3 &= 2 \times D_1 = 186 \mu\text{m} \\ D_4 &= 2 \times D_2 = 264 \mu\text{m} \\ D_5 &= 2 \times D_3 = 372 \mu\text{m} \\ D_6 &= 2 \times D_4 = 528 \mu\text{m} \\ \text{etc} \end{aligned}$$

A-1.4.4 Counting and Sizing.

(a) For each field investigated, count only those fibres $>5 \mu\text{m}$ in length with an aspect ratio of at least 3:1

(b) Count any fibre $>5 \mu\text{m}$ if it is entirely within the field area

(c) If some portion of the fibre extends out of the field area then only count that fibre if it crosses one or both of adjacent preselected sides. It is convenient to use the left and bottom sides of the field area as reference sides.

(d) Count all fibres $>5 \mu\text{m}$ present in a total of 100 fields. Count 25 fields in the inner area (A_i) of the wedge and 75 fields in the outer area (A_o).

(e) Select the counting fields without looking into the eyepiece

(f) Select the fields in a straight line running from the tip of the wedge (centre of filter) to the centre of the arc side (circumference of the filter). If further counts are required to achieve the necessary 100 fields, select them along straight lines parallel to and slightly above or below the original counting line.

(g) Do not count a field containing more than 20 fibres unless there is very little fibre overlap and few background particulates.

(h) When an agglomerate of particulates covers $1/6$ or more of the field area, reject the field and count another.

- (i) Size and count only free fibres which are unattached and have both ends visible
- (j) When counting a field, adjust the fine focus control so that any fibres embedded in the filter matrix are not missed
- (k) Count at least one unexposed blank filter, treated in the same manner as the samples except that no air is drawn through it, in order to obtain the background fibre loading for the samples
- (l) Record the fibre counts for each sample on the appropriate form, Figure A-1-3.

A-1.5 Calculations

A-1.5.1 Standard Sample Volume.

A-1.5.1.1 Stack Samples - Equation S-1-1 in Part I is used to determine the standard sample volume at reference conditions expressed in cubic feet. Using Equation A-1-1, convert this value to the standard sample volume at reference conditions expressed in cubic centimeters

$$\text{(Equation A-1-1)} \quad V_s = 28.32 \times 10^3 \times (V_m)_{ref}$$

where

- V_s - sample volume corrected to reference conditions (760 mm Hg, 298°K), cc
- $(V_m)_{ref}$ - sample volume corrected to reference conditions (29.92 in Hg, 537°R), ft³
- 28.32×10^3 - conversion factor, cc/ft³

A-1.5.1.2 Baghouse Samples - Use Equation A-1-2 to determine the sample volume corrected to standard conditions of 760 mm Hg and 298°K

$$\text{(Equation A-1-2)} \quad V_s = V_a \frac{T_{ref}}{T_a} \frac{P_a}{P_{ref}} = 0.392 \frac{^{\circ}\text{K}}{\text{mm Hg}} \frac{(V_a) (P_a)}{T_a}$$

where

- V_s - sample volume corrected to reference conditions, cc
- V_a - sample volume measured during sampling, cc
- T_{ref} - absolute temperature at reference conditions, 298°K
- T_a - absolute temperature during sampling, °K
- P_{ref} - absolute pressure at reference conditions, 760 mm Hg
- P_a - absolute pressure during sampling, mm Hg

A-1.5.2 Concentration of Asbestos Fibres. Use Equation A-1-3 to determine the concentration of asbestos fibres in the sample

$$\text{(Equation A-1-3)} \quad C = \frac{(S-B) \times (A)}{(f a) \times (V_s)}$$

where

C	-	asbestos fibre concentration, fibres/cc
S	-	average asbestos fibre count of sample, fibres/field
B	-	average asbestos fibre count of blanks, fibres/field
A	-	effective filtering area of filter sample, mm ²
f a	-	field area of counting field, mm ²
V _s	-	sample volume corrected to reference conditions, cc

BIBLIOGRAPHY

Bumbaco, M J , Williams, B D , and Shelton, J.H , *Study of Interlaboratory Count Correlation and Fibre Distribution on Asbestos Stack Samples*, Environment Canada, Report EPS 5-AP-7 (Sept 1976).

Environment Canada, Air Pollution Control Directorate, *Standard Reference Methods for Source Testing. Measurement of Emissions of Particulates from Stationary Sources*, Report EPS 1-AP-74-1 (Feb 1974)

Liedel, N A , Bayer, S G , and Zumwalde, R D , *USPHS/NIOSH Membrane Filter Method for Evaluating Airborne Asbestos Fibres*, U S Dept of Health Education and Welfare, National Institute for Occupational Safety and Health, Cincinnati, Ohio (Nov 1973)