GEORGIA-PACIFIC CORPORATION
Gypsum Division

June 25, 1974

PLAINTIFF'S EXHIBIT
GP-1613

SAFETY PROCEDURE

SUBJECT: Asbestos

I. OSHA Regulation

No employee shall be exposed at any time to airborne concentrations of asbestos fibers in excess of 10 fibers, longer than 5 micrometers per cubic centimeter of air.

II. Testing Method

Only the following method will be used in measuring the amount of airborne asbestos fibers: All determination of airborne fibers shall be made by the membrane filter method at 400 - 450 x (magnification) (4 millimeter objective) with phase contrast illumination.

III. Safe Exposure Limit

1. At the present time, the maximum safe exposure limit is an 8-hour average airborne concentration of 5 asbestos fibers per cubic centimeter of air. Only fibers longer than 5 microns are counted. (It takes 5,080 fibers 5 microns long to make one inch). Below this concentration, no personal protective equipment is required.

2. Air containing asbestos concentration greater than 5 fibers per cubic centimeter may be worked in for shorter periods of time without personal protective equipment provided the 8-hour average is not greater than 5 fibers. The maximum exposure at any one time, without personal protective equipment, is 10 fibers.

3. Beginning July 1, 1976, the maximum safe exposure limit will be reduced from 5 fibers to 2 fibers.

IV. Monitoring for Asbestos Dust

1. Current jobs using asbestos, or products containing asbestos, are to be tested for airborne contamination. Whenever tests are scheduled, the prescribed procedure for sample collecting will be carried out by trained technicians who will then arrange to have the samples tested.

2. The air test samples are to be taken within the breathing zones of employees, and in the general work areas.

IV. Monitoring for Asbestos Dust (Cont.)

3. After the initial determination of exposure to concentrations of asbestos fibers is made, samples shall be of such frequency and pattern as to represent with reasonable accuracy the levels of exposure of employees. In no case shall the sampling be done at intervals greater than six months for employees whose exposure to asbestos may reasonably be foreseen to exceed the limits as outlined in No. III of the Procedure.

4. Involved employees, or their representatives, are to be informed of the monitoring tests and of the results.

5. Any employee found to have been exposed to concentrations greater than the safe exposure limit is to be notified in writing within five (5) days of the findings. He is also to be notified of the corrective action to be taken.

V. Exposure

1. Employees may be exposed to asbestos dust and material when they are:

   a. Unloading, loading or handling asbestos or asbestos-contained materials.

   b. Mixing asbestos with other materials in the process of manufacturing joint systems products.

   c. Bag filling operation used in manufacture of dry compounds.

VI. Unsafe Exposure

In the event tests prove that the asbestos airborne concentration is above the acceptable threshold limit as stated in the OSHA regulations, the following precautions will be taken and strictly enforced until suitable engineering controls can be instituted to render the situation harmless.

1. Personal Protective Equipment - All employees engaged in the handling of asbestos or working in the immediate area where the asbestos airborne concentration exceeds the permissible exposure limit will wear the following personal protective equipment.

   a. NIOSH approved Respirator
   b. Head Covering
   c. Coveralls
   d. Gloves
   e. Foot Covering

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NOTE: The NIOSH approved respirator shall only be used when the airborne asbestos concentration does not exceed 10 times the permissible exposure limit in an eight-hour period.

2. Handling Asbestos

a. In all stages of handling - packaging, transporting, unloading, storing and in-plant moving - asbestos fiber bags should be handled carefully and in such a manner as to prevent the generation of dust.

b. Care should be taken to avoid puncturing the asbestos fiber bags during unloading from railroad cars, trucks or other modes of transportation. No hooks or other sharp instruments should be used. Loose fiber accumulated during transit should be picked up by vacuum cleaner before unloading.

c. Damaged bags should be immediately repaired, resealed or put into suitably closed receptacles. Any spillage of asbestos fiber should be picked up either by vacuum equipment or after thoroughly wetting, by sweeping, and should be deposited in suitable closed receptacles.

d. For storage, fiber bags should be stacked and moved with care to avoid breakage, protected from puncture by moving vehicles and, if possible, should be isolated from traffic in the warehouse and covered to prevent dust generation. To prevent deterioration of stored bags, the principle of first-in, first-out should be followed.

e. Maximum possible enclosure and fully effective dust control hoods should be provided at all stages of in-plant handling where dust is unavoidably created, such as at bag opening, fiber dumping and bag disposal operations. Loose fiber should be cleaned from work areas as described in 4, above, to prevent dust regeneration.

f. All dust from exhaust systems should be collected and safely disposed of. It should not be released into the outside air. Particular attention should be paid to the protection of the employees whose job it is to dispose of the collected dust.

g. Storage piles of bags of asbestos shall be covered with a non-clinging cover or tarp. Plastic is recommended. All spillage shall be vacuum cleaned to prevent so-called fugitive fiber release.

3. Disposal of Asbestos

a. During clean-up, dry sweeping shall not be performed.

b. If in an area where practical, the material will be wet with water, then cleaned up with shovel and brooms into the proper container.

c. If not practical to wet down the area, the clean-up of asbestos dust shall be performed with a vacuum cleaner.

3. Disposal of Asbestos (cont.)

d. All waste material including bags, boxes, cartons, etc., shall be sealed in an impermeable bag or other closed impermeable container and labelled.

e. Asbestos waste material shall not be burned as a form of disposal, it shall be buried in the ground, covered with dirt and the area posted.

4. Caution Signs and Labels

a. Signs

1) Caution signs shall be displayed at each location where airborne asbestos concentration exceeds the permissible level.

2) Caution signs shall be posted at such a distance from the contaminated area that employees may read the sign and take necessary protective precautions before entering the area.

3) Caution signs shall be posted at all approaches to areas where concentration exceeds the permissible level.

4) Size of caution sign 20" x 14", vertical format. The signs shall display the following legend in the lower panel, with letter sizes and styles of a visibility at least equal to that specified in this subdivision.

Legend	Notation
Asbestos	1" Sans Serif, Gothic or Block
Dust Hazard	3/4" Sans Serif, Gothic or Block
Avoid Breathing Dust	1/4" Gothic
Wear Assigned Protective Equipment	1/4" Gothic
Do Not Remain in Area Unless Your Work Requires It	1/4" Gothic
Breathing Asbestos Dust May Be Hazardous To Your Health	14 Point Gothic

4. Caution Signs and Labels (Cont.)

a. Signs (Cont.)

4) Spacing between lines shall be at least equal to the height of the upper of any two lines.

b. Labels

Caution labels shall be affixed to all raw materials, mixtures, scrap, waste, debris and other products containing asbestos fibers where the possibility exist that airborne asbestos fibers may be released in the air.

c. Label Specification

1) The caution label shall be printed in letters of sufficient size and contrast as to be readily visible and legible.

2) The caution label shall read as follows:

CAUTION

CONTAINS ASBESTOS FIBERS

AVOID CREATING DUST

BREATHING ASBESTOS DUST MAY CAUSE

SERIOUS BODILY HARM

USE APPROVED RESPIRATOR WHEN SANDING

5. Change Rooms

Employees who are exposed to asbestos fibers in excess of the permissible level shall be provided with:

a. Change Room

b. Two separate lockers, one for work clothes and one for street clothes; they shall be so separated or isolated as to prevent contamination to the employees' street clothes from his work clothes.

6. Laundering

a. Any employee who gives asbestos contaminated clothing to another person for laundering shall inform that person of the requirements in effectively preventing the release of airborne asbestos fibers and the precautions that should be taken.

6. Laundering (Cont.)

b. Contaminated clothing shall be transported in sealed impermeable bags or other impermeable containers and labeled accordingly.

VII. Medical Examinations

1. The Company shall provide medical examination relative to the exposure of asbestos fibers in the following manner:

a. Within one month after being assigned to work in an area containing asbestos dust.

b. Annual Examination.

c. Examination on termination.

NOTE: No medical examination is required of any employee, if adequate records show that the employee has been examined in accordance with this paragraph within the past one year period.

NOTE: All examinations will conform to OSHA requirements, and include as a minimum:

1. Chest X-Ray
2. Pulmonary Function Tests
   a. Forced Vital Capacity
   b. Forced Expiratory Volume at 1 second
3. Medical History to determine whether the employee has ever had any respiratory diseases.

VIII. Medical Records

Any employee who has been examined because of exposure to asbestos airborne fibers will have his medical records complete and accurate, containing all information pertaining to his medical examinations. These records shall be retained for twenty (20) years.

IX. Recordkeeping

Exposure records shall contain all information pertaining to personal or environmental monitoring and shall be retained for three (3) years. Every employee and former employee shall have reasonable access to any exposure records which indicate the employee's own exposure to asbestos fibers.

X. Housekeeping

All external surfaces in any place of employment shall be maintained free of accumulations of asbestos fibers if, with their dispersion, there would be an excessive concentration.

GEORGIA-PACIFIC CORPORATION
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June 25, 1974

SAFETY PROCEDURE

SUBJECT: Respiratory Protection Program

I. Purpose

The purpose of this program is to protect the health of all employees by preventing their exposure to harmful levels of air contaminants. Exposure to air contaminants of harmful levels will be eliminated by the application of engineering controls. In situations where engineering controls require capital expenditures and/or time for development and procurement, then interim protection will be accomplished by the use of personal respiratory protective equipment.

II. Responsibilities

A. Management will determine which areas require the mandatory use of respiratory equipment. Management will then be responsible for providing respiratory equipment which is compatible with the specific needs of each area.

B. The employees are responsible for maintaining an awareness of the respiratory protection requirements for their work area. In addition, the employees are responsible for wearing the appropriate respiratory equipment as required and for maintaining the equipment in a clean and operable condition.

III. Administration

A. The overall program administration is the responsibility of the Plant Superintendent.

B. The Supervisor of each area is responsible for ensuring that all personnel under his control are completely knowledgeable of the respiratory protection requirements for the areas in which they work. Also, each Supervisor is responsible for ensuring that his subordinates comply with all applicable facets of the respiratory program.

C. Technical support, including air sampling and laboratory analysis, is the responsibility of the Plant Superintendent.

1. A copy of technical reports and supporting data resulting from air sampling and laboratory analysis will be sent to the division Personnel Manager, accompanied by a schedule for future testing.

III. Administration (Cont.)

D. Monitoring the health of company employees via a comprehensive medical and health program is the responsibility of the Plant Superintendent.

E. The Chief Division Engineer is responsible for directing and coordinating engineering projects which are related to respiratory protection in conjunction with the Operations Manager.

F. The Safety Supervisor will, in addition to the line organization, maintain surveillance via spot checks of employees who are working in areas where respiratory protective equipment is required.

IV. Basic Program

A. Respirator Selection and Use - Respirator selection shall be on the basis of need according to the air contaminants to which the employee is exposed.

B. Respirator Availability - The supervision of each department will make a respirator available immediately to each employee who is placed as a new hire or as a transferee in any job which requires respiratory protection. Replacement respirator will be made available as needed. Each man receiving a respirator will sign the Respirator Responsibility Form. This form states that the employee receiving the respirator is responsible for its care and condition. Should it be lost, or should the employee fail to turn it in when assigned to another department or when he or she leaves the employ of the Company for any reason, he or she shall be charged for the cost of the respirator.

C. Employee Training - Each employee, upon assignment to a respirator area, will be briefed by his supervisor relative to the respirator program. He will review the Respirator Information Sheet for his job. Also, he will be fully instructed in the use and care of his respirator.

D. Respirator Inspection and Maintenance

1. The wearer of a respirator will inspect it daily whenever it is in use.

2. Supervision in each area and the Safety Supervisor will periodically spot check respirators for fit, usage and condition.

IV. Basic Program (Cont.)

D. Respirator Inspection and Maintenance (Cont.)

3. Respirators which are in daily use will be cleaned on a daily basis by the employees to whom they are assigned.

4. Respirators which are individually assigned shall be either marked or stored in such a manner so as to assure that they are worn only by the individual to whom they are assigned.

E. Monitoring

In order to assure the adequacy of the respiratory program and to provide for a continuing healthful environment for the employees, monitoring operations will be conducted on a periodic basis.

1. Environmental - Personal samplers and ambient air samples will be used in accordance with accepted industrial hygiene standards in order to periodically sample plant work areas. The results of this sampling will point out the areas where respiratory protection is required. It will also document the type of equipment which should be worn.

2. Medical - Two types of monitoring comprise the medical aspects of the program.

a. Pre-employment physical examinations are conducted in order to assure that they are in a healthy condition.

b. Periodic physical examinations will be given to regular employees in order to assist them in maintaining their health.

RESPIRATOR PROCEDURE

SUBJECT: Respirator Maintenance, Care and Cleaning

I. Purpose

The purpose of this procedure is to train employees required to wear respirators in proper respirator maintenance, care and cleaning.

II. Respirator Issue

A. The respirators currently being issued at our Chicago joint systems operation is the Style 560 - Wilcon respirator, and

B. 3M Brand Disposable Respirator No. 8710.

III. Maintenance and Inspection

A. The wearer of the respirator will inspect it daily to insure proper function and worn parts replacement.

B. The department supervisor and the safety supervisor will periodically spot check respirators for fit, usage and condition.

IV. Care and Cleaning

A. The Wilson 560 respirator is designed with a changeable filter feature. It requires two (2) R-60 disposable filters which are to be replaced daily.

B. A thorough cleaning and sterilizing of this respirator is required daily. Cleaning is accomplished with warm water and a disinfectant. (For exact cleaning procedure, see your supervisor.)

V. Respirator Replacement

When your respirator becomes worn or does not function properly, contact your supervisor immediately. He will make the necessary repairs and/or replacement of worn parts or replace your old respirator with a new one if replacement is so indicated.

VI. Respirator Storage

Respirators which are individually assigned shall be either marked or stored in such a manner so as to assure that they are worn only by the individual to whom they are assigned.

INFORMATION SHEET

RESPIRATOR RESPONSIBILITY FORM

I. PURPOSE

To eliminate and control the excessive cost of replacing respirators that have not been properly maintained or turned in when employees transfer or leave the employ of the company.

II. REQUIREMENT

Each employee receiving a respirator shall sign the Respirator Responsibility Form.

III. TYPE OF PROTECTIVE EQUIPMENT

The foremen issuing the protective equipment will indicate on the form what has been issued by completing the appropriate portion of the Respirator Responsibility Form.

IV. ROUTING

Send the form to the Personnel Office to be placed in the employee's file.

RESPIRATOR RESPONSIBILITY

1. I _____ have received on this date _____ a respirator and have been instructed on its use. It is my responsibility to keep clean and in good operating condition this respirator.

   I agree to pay the cost of the respirator should I fail to turn it in to my foreman on being assigned to another department or leaving the employ of the company for any reason.

2. I _____ have received on this date _____ a disposable respirator and have been instructed on its use and replacement.

_____
Employee Signature

_____
Date

_____
Foreman Signature

_____
Department

HOUSEKEEPING INSPECTION PROCEDURE

The purpose of the housekeeping inspection procedure is to provide our joint systems operations a systematic method of control of housekeeping and to establish a record of this control to satisfy the Environmental Protection Agency and OSHA of our efforts to provide our employees with a work atmosphere which is in compliance with the Occupational Safety and Health Standards.

I. Plant Housekeeping Report

The form is simple and self-explanatory. The inspection areas may need some modification from one location to another. The second column is for indicating if a given area was inspected or not, and we have also provided some room for comments.

II. Rating Evaluation Sheet

The rating evaluation sheet is provided to assist inspectors in achieving uniform ratings for conditions observed during inspections. The evaluation sheet does not include all of the conditions to be inspected, but it is designed to give you a feel for classifying specific conditions into a rating category.

III. Inspection Frequency

The frequency of inspection is left to the discussion of the local manager dependent on need.

IV. Recordkeeping

The completed Plant Housekeeping Report forms should be filed and kept for a minimum of three years.

RATING EVALUATION

Poor - Conditions which would constitute a poor rating would include: Excessive accumulations of dust on floors, machines, building supports, stored materials, etc. Clothing, refuse, bottles, rags, etc., not properly stored or disposed of. Any unsanitary or unclean conditions existing in toilets, washrooms, showers or drinking fountains. Stored material upset, damaged stored materials not repaired, unnecessary accumulations of scrap, paper, bags, etc. Water, oil or grease spills or leaks, severe disorderly arrangement of materials, broken equipment such as pallets, bags, etc., and other dirty, unsightly or disorderly conditions not otherwise specified.

Fair - Conditions which would constitute a fair rating would include: Moderate accumulations of dust on floors, machines, building supports, stored materials, etc. Conditions existing in washrooms, showers, toilets or drinking fountains which would indicate that cleaning had been done; however, better cleaning is necessary. Stored materials improperly piled or arranged. Materials protruding into aisleways. Scrap, paper, bags, etc., left lying on floor, on loads or found on the ground, and other conditions which need improvement not otherwise specified.

Good - Conditions which would constitute a good rating would include: Light accumulations of dust on floors, machines, building supports, stored materials, etc. Conditions of the washrooms, showers, toilets and drinking fountains indicate that adequate cleaning is achieved; however, there is still room for improvement. Stored materials properly piled and arranged; however, straighter stacking and a more orderly arrangement of materials can be achieved. An occasional piece of scrap, paper or bag lying on the floor, on loads or found on the ground. These and other conditions not otherwise specified are satisfactory; however, improvement can still be achieved.

Excellent - Conditions which would constitute an excellent rating would include: Virtually no accumulations of dust on floors, machines, building supports, stored materials, etc. The washrooms, showers, toilets and drinking fountains indicate a superior job of cleaning. Stored materials properly piled and arranged in a complete orderly fashion. No scrap, paper or bags on floors, loads or ground. These and other conditions not otherwise specified are outstanding.

PLANT HOUSEKEEPING REPORT

JOINT SYSTEMS PLANT

PLANT LOCATION _____ DATE OF INSPECTION _____

INSPECTION AREA	INSPECTED - YES/NO COMMENTS	RATING			
		POOR	FAIR	GOOD	EXCEL-LENT
OUTSIDE PLANT					
Front of Plant					
Limestone Unloading					
Dust Collection					
INSIDE PLANT					
Dock					
Blender					
Hammer Mill					
Storage Bin					
Packer					
Ready-Mix Mixer					
Railroad Unloading					
Main Aisle of Plant					
Finished Goods Warehouse					
Raw Material Storage					
Lunch Room					
Rest Room					
Laboratory & Maintenance					
Office					
GENERAL PLANT EVALUATION					

EXHIBIT F

(Responsive to Interrogatory # 102)

IX. APPENDIX I

AIR SAMPLING METHOD - MEMBRANE FILTER

General Requirements

The following sampling and anlytical methods for fiber counting are adapted from the NIOSH membrane filter method for evaluating airborne asbestos fibers [90].

(a) Air samples representative of the breathing zones of workers must be collected to characterize the exposure from each job or specific operation in each work area.

(b) Samples collected shall be representative of the exposure of individual workers.

(c) Suggested records:

(1) The date and time of sample collection.

(2) Sampling duration.

(3) Total sample volume.

(4) Location of sampling.

(6) Other pertinent information.

Sampling

(a) Samples shall be collected so as to be representative of the breathing zones of workers without interfering with their freedom of movement.

(b) Samples shall be collected to permit determination of TWA exposures for every job involving exposure to fibrous glass in sufficient

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numbers to determine the variability of exposures in the work situation.

(c) Equipment

The sampling train consists of a membrane filter and a vacuum pump.

(1) Membrane filter: Samples of fibrous glass are collected in the breathing zones of the workers using a personal sampler with cellulose ester membrane filter. The filter is a 0.8-μm pore size mixed cellulose ester membrane mounted in a open-face sampling cassette which can be attached to the worker near his or her breathing zone.

(2) Pump: A battery-operated pump, complete with clip for attachment to the worker's belt, capable of operation at 2.5 liters/minute or less.

(d) Calibration

The personal sampling pump should be recharged prior to calibration and then calibrated against a bubble meter, wet test meter, spirometer, or similar device at a flowrate of 1.0 to 2.5 liters/minute. The sampling train used in the calibration (pump, hose, filter) shall be equivalent to the one used in the field. The calibration should be performed to an accuracy of $\pm$ 5%.

(e) Sampling Procedure

(1) Sampling is performed using an open-face membrane filter cassette.

(2) The sampler shall be operated at a flowrate between 1.5 and 2 liters/minute.

(3) The temperature and pressure of the atmosphere being sampled are measured and recorded.

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(4) One membrane filter is treated in the same manner as the sample filters with the exception that no air is drawn through it. This filter serves as a blank.

(5) Immediately after sampling, personal filter samples should be sealed in individual plastic filter holders for shipment. The filters shall not be loaded to the point where portions of the sample might be dislodged from the collecting filter during handling.

(f) Optimum Sampling Times

A requirement for a minimum count of 100 fibers or 20 fields has been determined to be the optimum choice to achieve low variability of the fiber count (as approximated by a Poisson distribution) and reduced counting times. In other words, the optimum fiber density on the filter should be 1 to 5 fibers/microscope counting field. To estimate optimum sampling times, the approximate field area of the counting scope and the pump flowrate must be known in advance.

The following equation is used to calculate the range of optimum sampling times which can then be plotted on log-log paper:

$$\text{Minutes} = \frac{(FB/FL)(ECA/MFA)}{(FR)(AC)}$$

where: FB/FL = 1 to 5 fibers/field

ECA = Effective collecting area of filter in square millimeters (855 square mm for 37-mm filter)

MFA = Microscope field area in mm (generally 0.003 to 0.006 square mm)

FR = Pump flowrate in cc/minute

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AC = Air concentration of fibers in fibers/cc

(NOTE: If air concentrations are expressed in fibers/cu m they must be changed to fibers/cc for this equation.)

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X. APPENDIX II

ANALYTICAL METHOD - FIBER COUNT

Principle of the Method

(a) Environmental dust samples are collected by drawing air through a membrane filter by means of a battery-powered personal sampling pump.

(b) The filter is transformed from an opaque solid membrane to a transparent, optically homogeneous gel.

(c) The fibers are sized and counted by phase-contrast microscopy at 400-450X magnification.

Range and Sensitivity

(a) This method has been successfully applied at concentrations of 10,000 to 20,000,000 fibers/cu m (0.01 to 20 fibers/cc) for fibers longer than 5 μm. Large deviations from the specified conditions of the method may result in filters with either too few or too many fibers. Too few fibers will yield air concentration estimates of low statistical precision.

(b) A sensitivity of 10,000 fibers/cu m (0.01 fiber/cc) has been reported [JM Dement, written communication, 1975] based on a 4-hour sample at 2 liters/minute air flow.

Interferences

All particulates, such as asbestos or mineral wool, with a length-to-width ratio of 3 to 1 or greater, and length greater than 10 μm should, in

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the absence of other information, be considered as glass fibers and counted as such. Asbestos interference can be eliminated using phase contrast, polarized light microscopy.

Advantages of the Method

(a) The fiber count method allows for repeated counts, and storage for counting at a later time. The method consumes only part of the filter, thereby allowing for at least one repl cate sample analysis at a later time.

(b) Fiber counts are assumed to be more toxicologically significant than fiber weight for fibers less than 3.5 μm in diameter.

(c) Fiber size determinations may be performed.

Disadvantages of the Method

(a) The fiber count method is slow and tedious.

(b) Variation in counts may be significant between different observers.

(c) The sensitivity of the method is dependent on the sampling time and flowrate. The sensitivity and useful range of this method has not been determined specifically for fibrous glass but is based on the method recommended for asbestos.

Apparatus

(a) Optical Equipment

(1) Microscope body with binocular head, 10X Huygenian

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eyepieces, and Koehler illumination.

(2) Porton reticle.

(3) Mechanical stage, and stage micrometer with 0.01-mm subdivisions.

(4) Abbe or Zernike condenser fitted with phase ring with a numerical aperture equal to or greater than the numerical aperture of the objective.

(5) A phase-ring centering telescope or Bertrand lens and a green filter if recommended by the microscope manufacturer.

(6) Fiber mounting equipment

(A) Microscope slides, and cover slips, usually 0.17 mm thick.

(B) Scalpel, tweezers, lens tissues, and glass rod or spatula for mounting procedures.

(b) Wheaton Balsam Bottle.

Reagents

(a) Dimethyl phthalate.

(b) Diethyl oxalate.

Analysis of Samples

(a) Calibration and Standardization

(1) Porton Reticle and the Counting Field

The fiber count procedure consists of comparing fiber length with calibrated circles, and counting all fibers > 10 μm in length within a

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given counting field. A Porton reticle is used for this purpose. The Porton reticle is a glass plate inscribed with a series of circles and rectangles. The square on the left, divided into six rectangles, is defined as the counting field.

(2) Placement in Eyepiece

Place the Porton reticle inside one Huygenian eyepiece, resting it on the field-limiting diaphragm. Keep the reticle clean, since dirt on the reticle will be in focus and will complicate the counting and sizing process.

(3) Stage Micrometer

The Porton reticle cannot be used for counting until it has been properly calibrated with a stage micrometer. Most stage micrometer scales are approximately 2 mm long, divided into units of 10 μm.

(4) Microscope Adjustment

When adjusting the microscope, follow the manufacturer's instructions while observing the following guidelines.

(A) The light source image 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.

(5) Porton Reticle Calibration Procedure

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Each eyepiece-objective-reticle combination on the microscope must be calibrated. Should any of the three be changed (disassembly, replacement, zoom adjustment, etc) the combination must be recalibrated. Calibration may change if the interpupillary distance is changed. For proper calibration, the following procedure should be followed closely.

Using a 10X objective, place the stage micrometer on the mechanical stage and focus and center the image. Change to the 40-45X objective and adjust the first scale division to coincide with the left boundary of the Porton rectangle. Count the number of divisions between the left and right boundaries of the long horizontal dimension of the largest rectangle, estimating any portion of the final division. This measurement represents 200 L units and the measurement is then divided by 200 to find "L." The large rectangle is 100 L units long on the short vertical dimension. The calculated "L" is inserted into the formula $D = L(2N)1/2$ where "N" is the circle number (indicated on the reticle) and "D" is the circle diameter. Since the circle diameters vary logarithmically, every other circle doubles in diameter. For example, number three is twice the diameter of number one; number four is twice the counting field area consisting of the left six smaller rectangles can be calculated from the relation 10,000 L. The reticle calibration is now completed for this specific objective-eyepiece-recticle combination.

(b) Preparation of Mounting Solution

An important part of the sample evaluation is the mounting process which involves a special mounting medium of prescribed viscosity. The proper viscosity is important to expedite filter clearing and to minimize particle migration. Once the sample has been mounted, an elapsed time of

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approximately 15 minutes is needed before the sample is ready for evaluation.

Combine the dimethyl phthalate and diethyl oxalate in a 1 to 1 ratio by volume and pour the solution into a Wheaton balsam bottle. Add 0.05 gram of new membrane filter/ml of solution to reach the necessary viscosity. The mixture must be stirred periodically until the filter material is dissolved and a homogeneous mixture is formed. The normal shelf life of the mounting solution is about 6 months. Approximately 300 samples can be prepared from 20 ml of mounting solution.

(c) Sample Mounting

Cleanliness is important. The working area must be kept clean to prevent sample contamination and erroneous counts. The following steps should be followed when mounting a sample.

(1) Clean the slides and cover slips with lens tissue. Lay the slide down on a clean surface with the frosted end up. It is good practice to rest one edge of the cover slip on the slide and the other edge on the working surface. By doing this, you keep from becoming contaminated.

(2) Wipe all the mounting tools clean with lens tissue and place them on a clean surface (such as lens tissue). When mounting a series of filters, wipe the scalpel clean before cutting a sector of each sample [see (5) below].

(3) Apply a small drop of mounting solution onto the center of the slide with a glass rod. It may be necessary to adjust the quantity of solution used or the size of the wedge. The correct amount will result in the solution extending only slightly beyond the filter boundary. If the

quantity is greater than this, adverse particle migration may occur.

(4) With a spatula or a supplemental glass rod, spread the mounting media into a triangular shape. The size of this triangle should coincide with the dimension of the filter wedge.

(5) Separate the middle and bottom sections of the field monitor case to expose the fragile filter. Cut a triangular wedge from the center to the edge of the filter using a scalpel. The size of the wedge should approximate one-eighth of the filter surface. The filter should be handled gently so that no material will be lost.

Grasp the filter wedge with tweezers on the outer area of the filter which was clamped between the monitor case sections. Do not touch the filter with fingers. Place the wedge, fiber-bearing side up, upon the mounting medium.

(7) Lift the cover slip with the tweezers and carefully place it on the filter wedge. Once this contact has been made, <u>do not reposition the cover slip</u>.

(8) Label the slide with the sample number and current date before proceeding to the next filter.

(9) The sample should become transparent after about 15 minutes. If the filter appears cloudy, it may be necessary to press <u>very lightly</u> on the cover slip. This is rarely necessary, however.

(10) Examine the slide within 3 days. The sample mount should be discarded after 3 days if it has not been counted because crystals which appear similar to glass fibers may begin to grow at the mounting media/air interfaces; they seldom present any problems if the slide is examined within 3 days. In any case, do not perform counting or

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sizing around the edges of the filter.

(d) Counting and Sizing--Finding and Inspecting Counting Fields

Place the slide on the mechanical stage and position the center of the wedge under the objective lens and focus upon the sample. Nearly all of the particulates (particles and fibers) will be found in the upper 10-15 μm of the filter surface. When counting and sizing, continued use of the fine focus control is required to insure that nothing is missed. Start counting from one end of the wedge and progress along a straight line to the other end (count in either direction from circumference to wedge tip). Haphazard fields are selected without looking into the eyepieces by slightly advancing the slide in one direction with the mechanical stage control.

(e) Achieving Comparable Results

(1) Size only those fibers with a length-to-width ratio equal to or greater than 3:1.

(2) Count only fibers greater than 10 μm in length. (Be as accurate as possible in accepting or rejecting fibers near this length).

(3) Count up to 100 fields if necessary to yield a total count of at least 100 fibers. Count at least 20 fields even if more than 100 fibers are counted.

(4) Select the field of view without looking through the microscope's eyepieces to minimize unconsciously selecting "heavy" or "light" areas.

(5) The fields are selected along the entire length of a radial line running between the outside perimeter and the tip of the wedge.

(6) When an agglomerate (mass of material) covers a significant portion of the field of view (approximately 1/6 or greater), reject the field and select another. (Do not include this field in the number of fields counted.) Record the agglomerated field even though it is not included in the count.

(7) Bundles of fibers are counted as one fiber unless both ends of a fiber crossing another can be clearly resolved.

(8) For fibers that cross either one or two sides of the counting field, the following procedure is used to obtain a representative count. First, arbitrarily select: a) the left and bottom sides, and b) the upper and lower left corners and vertical direction as "decision aids."

Then count any fiber greater than 10 micrometers in length, but only if the fiber:

a. lies entirely within the counting area, or

b. crosses the left or bottom sides, or

c. crosses the upper or lower left corners, or

d. crosses both the top and bottom sides.

Reject and do not count all other fibers.

Calculations of Airborne Concentrations

Glass fiber airborne concentration may be calculated from th following formula:

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

where:

C = Airborne fiber concentrations in fibers >10 μm/cu m.

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F = Average fiber count in fibers >10 μm/field.

B = Average fiber count of blank(s) or control filter(s) in fibers >10 μm/field. (It is subtracted to eliminate the error or background contamination.)

W = 855 square mm for 37-mm diameter filters (the portion of the membrane filter which is exposed when mounted in the field monitor case, ie, the effective filter area).

A = The area of the counting field of a calibrated reticle expressed in square mm/field

V = Total air volume collected through filter expressed in milliliters.

XI. APPENDIX III

AIR SAMPLING METHOD - TARED FILTER

Sampling

Breathing zone samples of the total airborne material are collected on a tared 37-mm filter of 0.8-μm pore size, low ash, polyvinyl chloride, mounted in a filter holder with a 4-mm opening. The sample is collected for a 30-minute period at a sampling rate of 2 liters/minute. A personal sampling technique is employed, with the sampler head fastened to the worker's clothing in the breathing zone. Battery-powered personal sampler pumps, such as those used in the sampling train of the Coal Mine Dust Personal Sampling Units, approved under the provisions of 30 CFR 74 or their equivalent are used to draw air through the filters.

Calibration of Personal Sampler

The accuracy of environmental monitoring can be no greater than the accuracy of the volume of air which is measured. Therefore, the accurate calibration of a sampling device is essential to the correct interpretation of an instrument's indication. The frequency of calibration is dependent on the use, care, and handling of the pump. Pumps should also be recalibrated if they have been misused or if they have just been repaired or received from a manufacturer. If the pump receives hard usage, more frequent calibration may be necessary. Regardless of use, maintenance and calibration should be performed on a regular schedule and records of these should be kept.

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Ordinarily, pumps should be calibrated in the laboratory. The accuracy of calibration is dependent on the type of instrument used as a reference. The choice of calibration instrument will depend largely upon where the calibration is to be performed. For laboratory testing, primary standards, such as a spirometer or a soapbubble meter, are recommended, although other standard calibration instruments, such as a wet-test meter or dry gas meter, can be used. The actual setups will be similar for all in truments.

Instructions for calibration with the soapbubble meter follow. If another calibration device is selected, equivalent procedures should be used. Since the flowrate given by a pump is dependent on the pressure drop of the sampling device, in this case a filter, the pump must be calibrated while operating with a representative filter in line. The calibration system should be assembled in this order: soapbubble meter, water manometer, filter, and pump.

(a) Check the voltage of the pump battery with a voltmeter to ensure adequate voltage for calibration and charge the battery if necessary.

(b) Turn on the pump and moisten the inside of the soapbubble meter by immersing the buret in the soap solution and drawing bubbles up the inside until they travel the entire buret length without bursting.

(c) Adjust the pump rotameter to provide the desired flowrate.

(d) Check the water manometer to ensure that the pressure drop across the sampling train does not exceed 13 inches of water at 1 liter/minute.

(e) Start a soapbubble up the buret and measure with a stopwatch the time required for it to move between calibration marks.

(f) Repeat the procedure in (e) above at least twice, average the results, and calculate the flowrate from the volume between the preselected marks divided by the time required for the soapbubble to traverse the distance.

(g) Record the volume measured, elapse time, pressure drop, air temperature, atmospheric pressure, serial number of the pump, the date, time, and name of the person performing the calibration.

(h) The rotameter reading should be corrected for temperature and pressure, if necessary.

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XII. APPENDIX IV

ANALYTICAL METHOD - GRAVIMETRIC ANALYSIS

Principle of the Method

A known volume of air is drawn through a tared polyvinyl chloride filter to collect fibrous glass.

The sample-containing filter is removed from the cassette and dried over a desiccant to constant weight and weighed using a suitable microbalance. If tha desiccated sample and filter exceeds the weight of the filter by more than 5 mg then the sample and filter is ashed in a platinum crucible. The crucible is heated to a constant weight and weighed using a microbalance.

Range and Sensitivity

Although this method has not been validated for fibrous glass, it has been validated for other substances, such as carbon black, that have a recommended environmental limit similar to fibrous glass. This method for fibrous glass has been validated for carbon black over the range of 1.86-7.7 mg/cu m at an atmospheric temperature and pressure range of 18-25 C and 749-761 mm Hg, using a 200-liter sample. Under the conditions of sample size (200 liters), the working range of the method is estimated to be 1.5-10 mg/cu m or a 0.3-2 mg total weight of material collected on the filter. It was also validated for a 100-liter sample over the range of 7.8-27.7 mg/cu m at atmospheric temperature and pressure conditons as above.

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The method may be extended to higher sample concentrations by collecting a smaller sample volume; however, no more than 1.5 to 2 mg of material should be collected on any filter because greater amounts will be lost due to flaking.

Interferences

The presence of any other particulate material in the air being sampled will be a positive interference since this is a gravimetric method. Those materials that volatilize or combust at 600 C or less will not be interferences.

Information on any other particulate materials present should be solicited. If the concentration of other particles is known, then the fibrous glass concentration can be determined by the difference. If other particulate matter is known to be present and its concentration cannot be determined, then this method will not provide a limited measure of the fibrous glass concentration.

Precision and Accuracy

The precision and accuracy of the total sampling and analytical method has not been determined specifically for fibrous glass; however, it has been determined for other substances, such as carbon black, with a similar recommended limit. For carbon black, the coefficient of variation for the total analytical and sampling method in the range of 1.86-7.7 mg/cu m was 0.056. This value corresponds to a 0.20 mg/cu m standard deviation at the Occupational Safety and Health Administration (OSHA) carbon black

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standard level. A collection efficiency of greater than 98.7% was determined for the collection medium at the 2X level; thus, no bias was introduced in the sample collection step. Likewise, no significant bias in the analytical method is expected other than normal gravimetric errors. The coefficient of variation is a satisfactory measure of both accuracy and precision of the sampling and analytical method.

Advantages and Disadvantages of the Method

The analysis is simple but the method is nonspecific and subject to interference due to presence of other nonvolatile or combustible particulates in the air being sampled.

Apparatus

(a) Sampling Equipment

The sampling unit for the collection of personal air samples for the determination of fibrous glass has the following components:

(1) The filter unit, consisting of the filter media, cellulose supported pad and 37-mm three-piece cassette filter holder.

(2) Personal sampling pump: A calibrated personal sampling pump whose flow can be determined to an accuracy of ±5% at the recommended flowrate. The pump must be calibrated with a filter holder and filter in the line.

(3) Thermometer.

(4) Manometer.

(5). Stopwatch.

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(b) Polyvinyl chloride membrane filter; 37-mm diameter, 0.8-micrometer pore size.

(c) Plastic Petri dish-filter holder or equivalent for storage and weighing.

(d) Desiccator.

(e) Platinum Crucible.

(f) Platinum-tipped or Nichrome Forceps.

(g) Platinum or Silica Triangles.

(h) Microbalance capable of weighing to 10 micrograms. Particular care must be given to proper zeroing of the balance. The same balance should be used for weighing filters before and after sample collection.

Reagents

Drierite or any other suitable desiccant.

Analysis of Samples

(a) Preparation of Filters

All filters must be dried and weighed prior to use.

(b) Sampling Requirements and Shipping of Samples

(1) To collect fibrous glass, a personal sampler pump is used to pull air through a polyvinyl chloride membrane filter. The filter holder is held together by tape or a shrinkable band. If the filter holder is not tightened snugly, the contaminant will leak around the filter. A piece of flexible tubing is used to connect the filter holder to the pump. Sample at a flowrate of 1.5 to 2 liters per minute. After sampling,

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replace small plugs to seal filter cassettes.

(2) Blank

With each batch of ten samples submit one filter from the same lot of filters which was used sample collection to exactly the same handling as the samples except that no air is drawn through it. Label this as a blank.

(3) Shipping

The filter cassettes should be shipped in a suitable container designed to prevent damage in transit.

(c) Analysis of Samples

(1) If the outer surface of the cassette filter holder is heavily coated with dust, carefully swab the outer surface with a moist paper towel before opening the cassette so as to minimize sample contamination. Discard paper towel.

(2) Open the cassette filter holder and carefully remove the polyvinyl chloride membrane filter from the holder and cellulose support pad with the aid of filter tweezers. Transfer filter to a filter holder.

(3) Dry the filter to constant weight in a desiccator containing a dessicant. This takes about 12 hours.

(4) Weigh the filter using a microbalance. If the weight of filter contents exceeds 5 mg then put filter and contents in a clean, dried, and tared crucible.

(5) Put the crucible in a muffle furnace and ash at 600 C to constant weight. When handling the platinum crucible platinum-tipped or nichrome forceps should be used. If it is necessary to hold or stabilize

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the crucible platinum or silica triangles should be used. Iron forceps should never be used for crucibles that are above 500 C because iron will alloy with platinum. Very hot crucibles should not be put into the desiccator. The crucible should be allowed to cool in the air until the temperature has fallen below 100 C. Then it may be placed in the desiccator.

(6) Weigh the crucible using a microbalance.

Calibration and Standards

The microbalance should be properly zeroed for all weighings and preferably the same microbalance should be used for weighing filters before and after sample collection. The balance should be maintained and calibrated with National Bureau of Standards (NBS) Class M weights.

Calculations

(a) Record the tare weight, in μg, of the dry filter before sampling.

(b) Record the weight, in μg, of the dried, sample-containing filter.

(c) The difference between these two weights represents the μg of sample.

(d) Corrections for the blank must be made for each sample. (If found to be necessary, corrections should also be made for other particulate matter.)

μg sample = μg found in sample filter

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μg blank = μg found in blank filter

(e) The concentration of the analyte in the air sampled can be expressed in mg per cu m (μg/liter = mg/cu m) by the following equation:

$$\text{mg/cu m} = \frac{\mu\text{g found (section (d))}}{\text{Vs}}$$

Vs = Volume of air in liters at 25 C and 760 mm Hg

(f) If the ashing procedure is to be performed record the tare weight, to the nearest μg, of the dry crucible before adding the filter.

(g) Record the weight, to the nearest μg, of the crucible and contents after the sample-containing filter has been ashed.

(h) The difference between these two weights represents the μg of sample.

(i) Corrections for the ashed blank filter must be made for each sample.

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