

PLAINTIFF'S EXHIBIT

DOW-1002

MANUAL OF TESTING PROCEDURES
for
CHRYSTILE ASBESTOS FIBRE

ST0513801

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QUEBEC ASBESTOS MINING ASSOCIATION
ASBESTOS-CEMENT PRODUCTS ASSOCIATION

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MANUAL OF TESTING PROCEDURES FOR CHRYBOTILE ASBESTOS FIBRE

The Manual has been prepared jointly by the Asbestos Textile Institute, the Asbestos-Cement Products Association, and the Quebec Asbestos Mining Association, to provide standard methods for testing physical and chemical properties of chrysotile asbestos fibre.

The Manual is a collection of test methods for fabricators and manufacturers of asbestos who have both the facilities to make the tests with reasonable accuracy, and the personnel with the required degree of laboratory experience.

The Test Manual establishes authoritative procedures for testing milled chrysotile asbestos fibre. A number of tests used at various times in the industry, but which are now of little significance, have been omitted. It should be emphasized that the Manual is neither a guide for placing a value on asbestos fibre, nor a specification for grading it. The intention of the sponsoring organizations was to prepare a Manual that will aid consumers and potential users of asbestos products throughout the world in characterising the properties of asbestos that fit their needs. These sponsors make no recommendations or suggestions for application or use of these tests and indicate only that these tests are accepted and are proven procedures for measuring specified properties.

As in any book of this type, the tests are tentative and subject to change as more information becomes available which will increase the accuracy and adaptability of the procedures. The joint fibre associations recognize that the Manual will require revision and will issue, when necessary, revised editions to include additional tests or modifications of established procedures.

The information in the Manual represents the combined effort of the Fibre Testing Committee of the Asbestos Textile Institute, the Technical Committee of the Asbestos-Cement Products Association and the Technical and Research Committee of the Quebec Asbestos Mining Association. The sponsors acknowledge with sincere appreciation and gratitude the help of personnel who served on these Committees for the generous contribution of their knowledge and, most important, of their time and effort in proving and compiling the methods.

DEFINITIONS

SPICULES: There are two types of spicules commonly found in milled asbestos.

- 1) Rod-like pieces of unopened asbestos.
- 2) Sharp pointed pieces of non-fibrous minerals such as picrolite, brucite or other mineral matter.

PENCIL: Unopened asbestos of generally uniform diameter throughout its length, which can be fiberized.

SPELK: Synonymous with pencil.

BUNDLE: Refers to a comparatively heavy pencil which may also be partially crushed or fiberized.

HARSH: Refers to an inherent quality of a particular asbestos fibre which implies a degree of rigidity.

BRITTLE: The tendency to break readily when flexed.

SOFT: Refers to an inherent quality of a particular asbestos fibre which implies a high degree of flexibility.

CRUDY: The quality of a milled fibre containing an appreciable portion of pencils and bundles of asbestos. This is derived from the term "crude", as used in reference to unmilled asbestos.

OPEN: Open is a term used in referring to the quality of a milled fibre which does not contain an appreciable portion of pencils and bundles of asbestos.

CRUDINESS: The degree to which a fibre is crudy.

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Method of Sampling and Preparation of
Asbestos Fibres for Test Purposes

Adopted by

ACPA - 9/15/59
ATI - 9/10/59
QAMA - 10/13/59

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METHOD OF SAMPLING AND PREPARATION
OF ASBESTOS FIBRE FOR TEST PURPOSES

Scope

This method covers the procedure for sampling asbestos fibre and the preparation of the sample for testing.

1. SAMPLING

Sampling and preparation are equally as important as the testing. Every precaution should be taken to obtain and to prepare samples that will indicate the true character and condition of the asbestos fibre which they represent.

Equipment

One clean container of suitable size for each composite sample of fibre.

Procedure

- (a) Prepare a five pound composite sample from each lot consisting of 200 bags or part thereof by selecting 20 handfuls (approximately four oz.) each from a different 1/20 of the lot.
- (b) When shipments exceed a lot of 200 bags, but not in excess of 2000 bags, composite samples may be combined into one master composite sample at the discretion of the tester, if agreed between vendor and buyer.
- (c) Samples shall be kept in closed containers placed in covered storage for subsequent testing.

2. SAMPLE PREPARATION

Equipment

Smooth, clean area.

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Procedure

- (a) Each individual composite sample, or group of composites, shall first be allowed to approximate average workroom temperatures and humidity, (moisture content of sample should not exceed three per cent) and shall then be subdivided for testing in accordance with the following method.
- (b) Spread each composite as obtained in Part 1 on the smooth, clean area.
- (c) Mix the sample thoroughly with the hands by means of a gentle action. This will involve passing the fibre through the hands with gentle rubbing in order to break up and to separate all lumps and clots.
- (d) When the sample is intimately mixed, spread it out evenly over the smooth, clean area.
- (e) For master composites, quarter the sample, place aside the full thickness of two diametrically opposite quarters, and reblend the remainder. Continue this quartering until a five pound sample is obtained.
- (f) This five pound composite sample will be used as the source of fibre for each individual test.
- (g) For extraction of samples for test spread the five pound sample, or quartered fraction thereof, on a smooth, clean surface and take a sample of the desired size from each quarter using the full thickness of the pile.
- (h) When this sampling procedure is employed in a referee test procedure, acceptability shall be based on the average of the composites.

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2. (i) For best results it is recommended that the fibre be conditioned prior to testing, by means of the Johnson's Sample Conditioner, using the procedure listed in Section 3.

3. Johnson's Fibre Conditioner

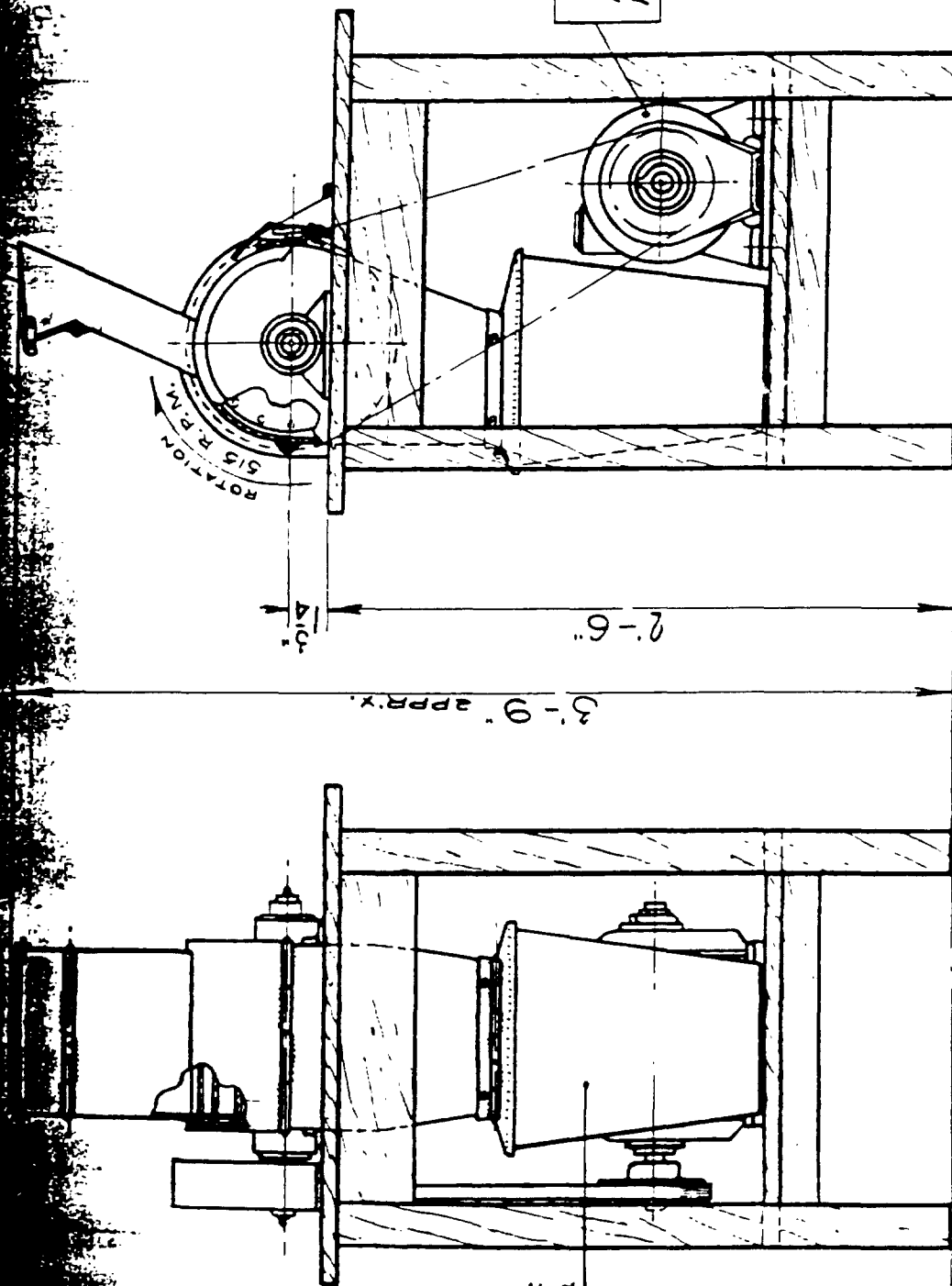
Equipment

Laboratory Fibre Conditioner	Dwg. A
Fibre Press	Dwg. B
Fibre Mold	Dwg. C

Procedure

- (a) Put into the fibre mold $1 \frac{1}{4}$ - $1 \frac{1}{2}$ lbs. of fibre. Place the mold in the fibre press and apply compressed air, pressure 85 ± 10 lbs. per sq. in. for one minute. Release pressure and remove the pressed fibre cake whole by pushing it out with the piston of the mold. Feed the pressed fibre cake into the laboratory fibre conditioner. Then re-feed this conditioned fibre into the laboratory fibre conditioner so as to give the sample a second pass. The conditioner should be operated at 525 ± 25 RPM.

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11 REMOVABLE
CONTAINER

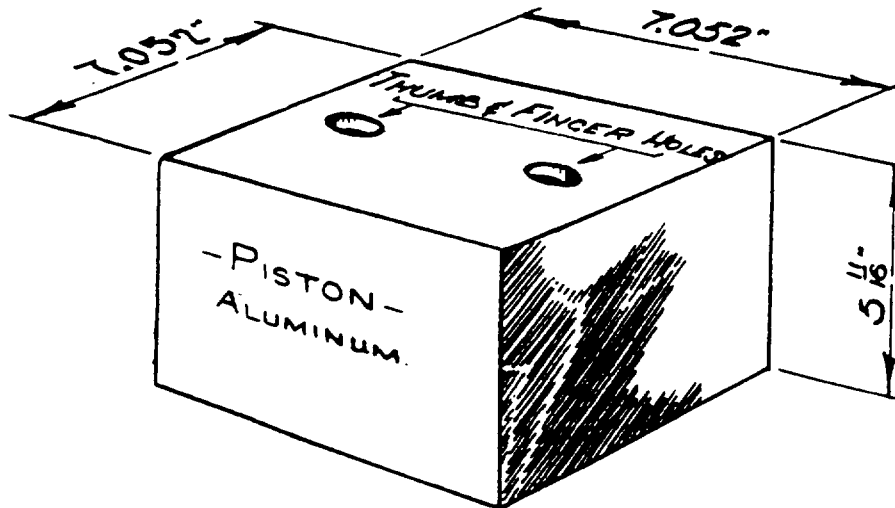
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ASBESTOS FIBER
CONDITIONER ASSEMBLY.

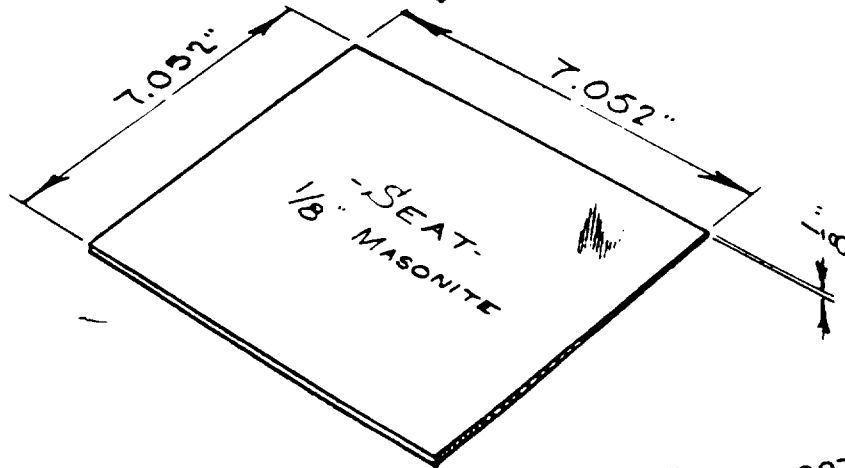
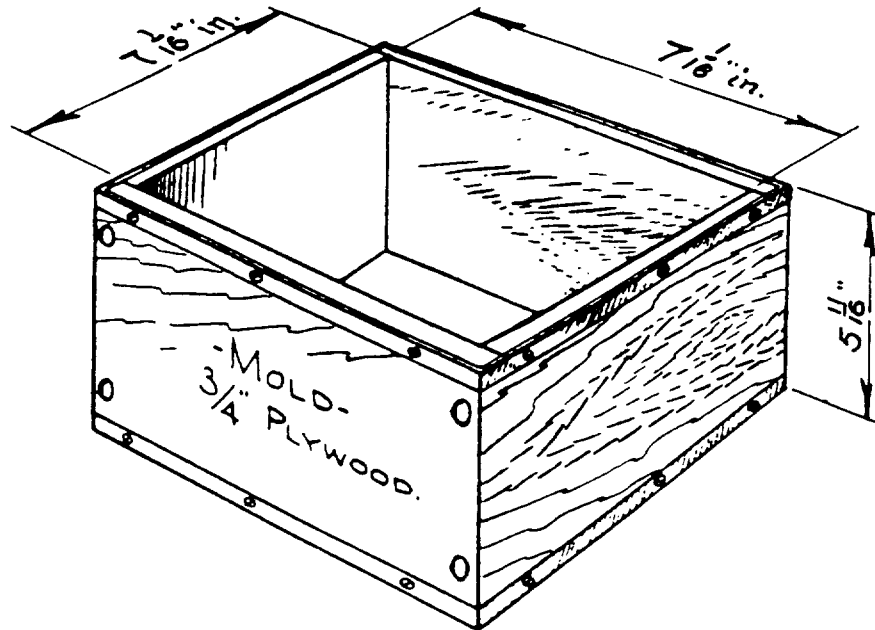
FIG. "A" JAN. 8/58

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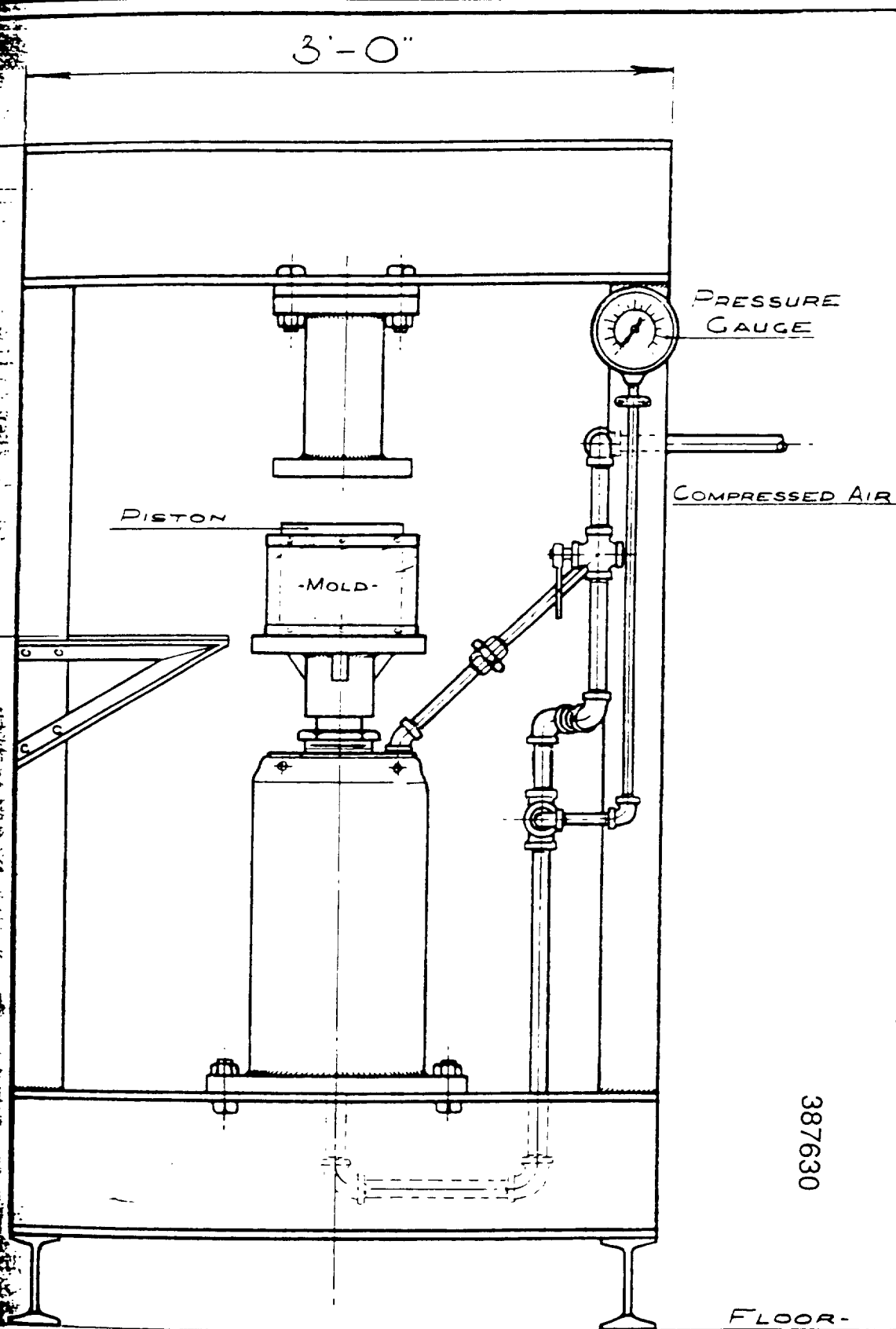


PISTON
TOP & BOTTOM
MADE OF 1/2"
OUT. WALLS
3-REINFORCE
1/4" R INSIDE



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FIBRE MOLD



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FIBER PRESS

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THE QUEBEC STANDARD ASBESTOS TEST

Adopted by

ACPA - 9/15/59
ATI - 9/10/59
QAMA - 3/ 3/59

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THE QUEBEC STANDARD ASBESTOS TEST

This method of test covers a procedure for the classification of Chrysotile Asbestos Fibre into Commercial Grades with respect to fibre length.

EQUIPMENT:

The Quebec Standard Asbestos Testing Machine, Model No. 2.

Note: The machine shall be constructed and operated according to the specification issued by the National Research Council of Canada. (Abstract attached).

A 16 oz. scoop scale with graduations of 0.2 oz. and a sensitivity of 0.1 oz.

A smooth-surfaced mixing table.

SAMPLING & PREPARATION:

According to method of sampling and preparation of asbestos fibre for test purposes.

PROCEDURE:

Sample to be tested shall be placed on the table and conditioned to normal room temperature and humidity. Examine the sample by passing through the hands to insure that clots or lumps are broken up. The moisture content of the sample should not exceed 3% (significant variations not normally encountered except when moisture content exceeds 3%). Erratic results can be expected beyond 3%. Mix thoroughly, weigh accurately into the scoop of the scale, a 16 oz. $\pm$ 0.1 oz. representative portion. Place the specimen in Box No. 1 of the testing machine, by letting it fall loosely from the scoop. The scoop shall be held from 6 to 10 inches above the screen. Take care that when the cover of the testing machine is closed, the fibre shall not be compressed.

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When setting the boxes on the machine, be sure that they are lined up true to the platform and that the ends of the boxes always face the same way each time they are used.

Start the machine and allow it to run until it automatically stops. Then, remove each tray and dump the fibre resting on each tray onto the mixing table, in individual piles, picking up and adding the loose fibre adhering to the cloth.

Weigh separately the fibre recovered from each tray; the weights in ounces of the fibre shall be recorded as the Quebec Standard Test of the fibre sample.

REPORT:

To the nearest 0.1 oz. the fibre retained in each box. An example of test results on a long (Group 3) fibre and short (Group 7) fibre would be:

	Box 1 <u>1/2"</u>	Box 2 <u>4 mesh</u>	Box 3 <u>10 mesh</u>	Box 4 <u>pan</u>	
Long Fibre	3.2	9.4	2.8	0.6	- 16.0 oz.
Short Fibre	0	0	6.2	9.8	- 16.0 oz.

QUEBEC STANDARD ASBESTOS TESTING MACHINE, MODEL 2

General Specifications

1. The Main Frame or Base shall be in one piece of grey cast iron conforming to Drawing No. 2, item No. 1*. (See original publication for Drawings.)
2. All castings shall be free of blow holes, blisters and all other faults or defects and shall be properly machined at surfaces contacting other parts of the machine moving or stationary. Other cast iron surfaces, where not machined, shall be smoothed off and painted with one coat of suitable metal paint. All castings whether aluminum or cast iron shall be finished to remove sharp edges.
3. All machine parts such as bolts, nuts, cap screws, studs, pulleys, bearings, steel springs, or any other parts, shall be used in standard engineering practice.
4. Foundations shall be of concrete, or concrete mat, allow-

ing one inch for grouting, the surface shall be level in every direction. This foundation shall be isolated from disturbing vibration caused by extraneous forces.

Main Frame or Base shall be bolted by four foundation bolts, one at each corner.

The main shaft of the machine shall rotate at a rate of 327 r.p.m. with a tolerance of plus or minus 1 r.p.m. Direction of rotation shall be counterclockwise as observed while facing machine at timing box side.

The automatic cut-off shall release the belt shifter after 600 revolutions. The machine shall then be stopped within two and one-half revolutions by the automatic application of the brake at the same time that the cut-off shall have released the belt shifter.

Each machine shall be driven by squirrel cage electric motor of not less than 2 h.p., connected to it by belt, either in one stage or by medium of one countershaft. This motor shall not be used as prime mover for any machine other than a testing machine.

Bolts and fixtures shall be kept well tightened.

The transmission belting shall be kept in good condition and shall be periodically scraped and dressed, so as to prevent slippage. Endless belting with properly glued joints shall be used.

The moving parts shall be kept greased and oiled to ensure free movement.

Bearings shall be adjusted or renewed when clearance permits a perceptible knock or play.

The machine shall be kept in adjustment conforming to the dimensions on Assembly Drawing No. 1*. (See original publication for Drawings.) When the eccentric is in high position, the common vertical through the centres of the lower and upper rocker arm bearing shall intersect at 90 degrees, the horizontal through the centre of the main shaft.

A tolerance of 1/16 inch shall be allowed on the dimensions in this Drawing No. 1 except in the case of the centre distance between upper and lower rocker arm bearings, where the tolerance shall be limited to plus or minus .005 inch. Tolerances here refer only to adjustment di-

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mensions. Table in any position shall be horizontal in the direction parallel to the main shaft and the two rocker arm shafts.

15. The Main Frame or Base, main shaft bearing housings and lower rocker arm bearing supports shall be cast in one piece of grey cast iron conforming to Drawing No. 2. Casting shall be machined where contacts are made with parts attached to it. Holes shall be drilled and tapped for various connecting parts, all as shown on Drawing No. 2 above-mentioned.
16. Timing Box Bracket shall be of grey cast iron as shown in Drawing No. 3, machined where contacting Main Frame and Timing Box. This bracket shall be attached to Main Frame by two 1/2 inch x 1 inch standard cap screws with hexagon head.
17. Shaking table and eccentric housings shall be of cast aluminum conforming to Drawing No. 4 all to be machined where contacting other parts. Item No. 4, eccentric housings shall be cast in two separate aluminum castings machined where contacting other part. Item No. 4 shall be bolted to item No. 3 by two standard hexagon head 5/8 inch by 7 inch long bolts. Shaking table assembly shall be connected to main shaft through eccentric adapters by two SKF bearings number 6222.
18. Clamping device shall be as per Assembly Drawing No. 21 and details per Drawings Nos. 22 and 23.

Note: * Send request to the National Research Council of Canada, Ottawa, Canada, for "Standard Specifications for Quebec Standard Asbestos Testing Machine".

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Testing Boxes

Testing Boxes shall consist of three boxes, a pan and all of cast aluminum, as per Drawing No. 19.

Diameter of all boxes and pans shall be 14-3/4 x 24-

over-all height of the four nested boxes without cover shall be less than 14-9/16 inches nor more than 14-5/8

with 1/2 inch opening screen cloth properly attached over-all height 3-3/4 inches.

with a 4 mesh screen properly attached - over-all height 3-21/32 inches.

with 10 mesh screen properly attached - over-all height 3-5/8 inches.

with 20 gauge g.s. sheet attached to bottom - over-all height 3-17/32 inches.

cloth to be attached to boxes with 1/8 inch flat head bolts and nuts, as per Drawing No. 19, using 20 gauge galvanized strips between screen cloth and bolt heads.

Specifications for Testing Screens

Screens for the Quebec Standard Testing Machine, Model 2 conform to the following specifications:-

Dimensions

Length: 25-3/4 inches

Width: 16-1/16 inches

Opening: 1/2 inch screen = 0.500 in.; 4 mesh = 0.187 in.;
10 mesh = 0.053 in.

Diameter: 1/2 inch screen = 0.105 in.; 4 mesh = 0.063 in.;
10 mesh = 0.047 in.

Note: The 1/2 in. screen shall have 42 holes formed by the 43 wires on length; 26 full holes formed by 27 wires on width.

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Tolerances

On wire diameter: 0.001 in.
On mesh opening : An average tolerance of +2% and -3% shall be allowed, provided that a maximum tolerance of plus or minus 5% shall not exceed 5% of the total number of openings.

Material

The screens shall be woven from a hard drawn brass wire of the composition 80-85% Cu and 20-15% Zn.

Weave

The 1/2 inch screen and the 4 mesh screen shall be woven in such a manner that the warp and shute wires are securely interlocked at intersections.

The 10 mesh screen shall be woven by the "Double Crimped" process.

General

Screen cloth shall lie flat and rigid and shall be free from wrinkles.

Cloth shall be cut in such a manner that the shute wire forms the longer dimension.

The 1/2 inch screen shall be attached to the screen box in such a manner that 24 full openings are formed with 25 wires; there will then remain 1/16 of an inch on each side between the inside of the box and the last wires.

Method for Preparation of
Crudy Fibres for the
Suter-Webb Comb Test

Adopted by

ACPA -
ATI - 9/10/59
QAMA - 4/27/61

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METHOD FOR PREPARATION OF
CRUDY FIBRES FOR THE SUTER-WEBB COMB TEST

SCOPE

1. This method covers a procedure for preparing asbestos samples containing crudy bundles of fibre to permit the taking of the representative 200-mg specimen samples required for determining the mean length and length distribution by the Suter-Webb Comb method.

OUTLINE OF METHOD

2. A 25-gm sample of the crudy fibre is passed through rolls to flatten and part the hard and tight crudy bundles of fibre, then placed in an air opening apparatus to fiberize and disperse the flattened bundles to produce a sample which is homogeneous in character. The surface area of the resulting fibre is to be 4000 ± 300 sq cm per gm, reasonably free of hard, crudy bundles of fibre, and suitable for taking the representative 200-mg sample for the Suter-Webb comb test. The surface area objective of 4000 ± 300 sq cm per gm is obtained by "trial and error", but Section 6, covering the procedure, will indicate a method for obtaining the objective for fibres of different character.

DESCRIPTION OF TERMS

3. (a) Fiberize - Opening crudy bundles of fibre into thinner cross-sections.
(b) Surface Area - As measured by the air permeability method based on D'Arcy's Law.

APPARATUS

4. (a) Rolls and tray as illustrated in Fig. 1.
(b) Air opening apparatus as illustrated in Fig. 2.
(c) Balance sensitive to .1-gm, and having a range of at least 0 to 25 gm.

(d) Stopwatch.

(e) Paint brush, 2-in. or equivalent.

(f) Forceps.

(g) Screen, Tyler 10-mesh or equivalent.

(h) Surface area apparatus.

SAMPLING

(a) Sample - The sample shall be the laboratory test sample as prescribed by the "Method of Sampling and Preparation of Asbestos Fibre for Test Purposes", or other asbestos fibre sample.

(b) Specimen for Preparation - The 25-gm specimen sample shall be derived from the laboratory test sample. Quarter the laboratory test sample down to approximately 25-gm as prescribed by the procedure "Sampling and Preparation of Fibre", Part 2, (a) through (g).

For average unmilled or semi-milled crude grades of asbestos containing a large percentage of crude fibre bundles:

Note 1 - Applicable only to those crude grades containing fibre bundles which are easily opened, otherwise follow procedure described in paragraph 6 (c).

(i) Spread the 25-gm specimen sample evenly on the galvanized sheet tray so that the fibre is one inch from all edges. Pass the fibre-covered tray slowly through the rolls. The pressure exerted by the rolls to be such that the fibre-free end of the tray is firmly gripped by the rolls. Any large pieces of crude which may prevent the tray from being drawn through the rolls should be fiberized by hand and replaced on the tray. Remove any fibre adhering to the tray, top roll and rubber scraper. Take the flattened fibre and by passing it through the fingers locate and open any compressed or crudy bundles of fibre. Special care should be taken to fiberize the large diameter pieces of crude by flexing or brooming the ends, thus making them amenable to air opening. At least 15 minutes should be allotted to this operation.

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- (ii) Shake and fluff the flattened fibre, then put loosely into the can of the air opening apparatus. Distribute the fibre evenly over the bottom of the can. Fasten the lid securely. Attach the 1/8-in. nozzle to the bottom of the can and place the hood with the perforated plate in position on top of the 100-mesh screen. Open the air valve quickly to a pressure gauge reading of 20 psi. After two minutes, close the air valve.
 - (iii) Remove the fibre from the can and again place on the tray and pass it through the rolls in the same manner as before. Again pass the fibre through the fingers, pulling apart the flattened fibre. A few large diameter pieces of crude may still be present and these are to be fiberized by hand. Five minutes is to be allotted to this operation.
 - (iv) Shake and fluff the flattened fibre, then put loosely into the opener can and air open for two minutes at 20 psi.
 - (v) Remove the fibre from the can and carefully pull apart any entangled fibre. With a brush clean the air filter twill on the lid by cleaning only a small area at a time to prevent the fibre from balling. Distribute the fibre from the twill evenly over the air-opened fibre and mix thoroughly.
 - (vi) Test for surface area. If the surface area should be higher than 4300 sq cm per gm, reduce the air blowing schedule. Should it be lower than 3700 sq cm per gm, increase the air blowing schedule. Visually inspect the fibre for crudy bundles. An acceptable prepared fibre should be reasonably free of hard, crudy bundles of fibre.
- (b) For crudy or splintery milled fibres:
- (i) Proceed as in (i) of Part 6 (a).
 - (ii) Follow step (ii) of Part 6 (a) with the exception that the air flow is maintained at 15 psi for 1-1/2 minutes.

Note 2 - When required, fibres in this class may

air-opened to a surface area of 8000 ± 500 cm^2/gm to condition any tight crudy fibre bundles present by blowing with air at 20 psi for two minutes. Any hard bundles remaining, after a surface area of 8000 ± 500 cm^2/gm has been developed, shall be sorted out and fiberized by hand or treated separately in the air opening device. Use procedure described in Part 6 (c) for fibres containing a profusion of tight, hard bundles.

(iii) Remove the fibre from the opener can and proceed as per steps (v) and (vi) of Part 6 (a).

(c) For unmilled or semi-milled crude grades of asbestos containing a large percentage of very hard and tight crudy fibre bundles and considerable magnetite:

(i) Proceed as in (i) of Part 6 (a). Fiberize only the large crudy fibre bundles which can be hand opened without effort.

(ii) Shake and fluff the compressed specimen sample by hand, then form into a cone. Starting from the top of the cone, remove the fibre in small portions, using forceps. The remaining crudy bundles will end up at the bottom of the cone. By hand, fiberize all the crudy fibre bundles and spread over the sample.

(iii) Place the sample on a 10-mesh Tyler sieve, or equivalent, and shake for 15 seconds. Set aside the minus 10-mesh material, which will contain, besides fibre, most of the magnetite.

(iv) Place the plus 10-mesh fibre in the air opener can and air open in the same manner as step (ii) Part 6 (a) for three minutes at a pressure of 20 psi.

Note 3 - Use air flow for four minutes at 20 psi for grades containing a profusion of hard crudy bundles, which are difficult to open.

(v) Proceed as in (v) Part 6 (a). A small amount of magnetite and a few crudy fibre bundles may report on the bottom of the air opener can. If so, combine with the minus 10-mesh material.

(vi) Spread the minus 10-mesh material on the tray and pass through the rolls. Pull apart and fluff any flattened or hard crudy pieces of fibre and add to sample. Mix sample thoroughly.

(vii) As most of the magnetite is loosely held in the sample, it is considered necessary to remove the major part of this granular mineral as it is likely to cause erratics in the combing results. Most of the magnetite can be eliminated by the following procedure: Thoroughly shake and fluff the prepared fibre by hand and form into a cone. With forceps starting at the top of the cone take small portions of fibre and set aside. Most of the magnetite, with a small amount of fibre, will end up on the table at the bottom of the cone. With the brush pick out the fibre and mix with the prepared sample. Discard the magnetite. Repeat the coning and fibre removal operation. A small amount of magnetite will still remain in the sample, but not sufficient to affect the reproducibility of the Suter-Webb sorter test.

(viii) Take surface area test, then proceed as in step (vi) Part 6 (a).

REPORT

7. Record the procedure finally adopted to prepare a given sample, and the surface area of the fibre tested, on the Suter-Webb report form for future reference.

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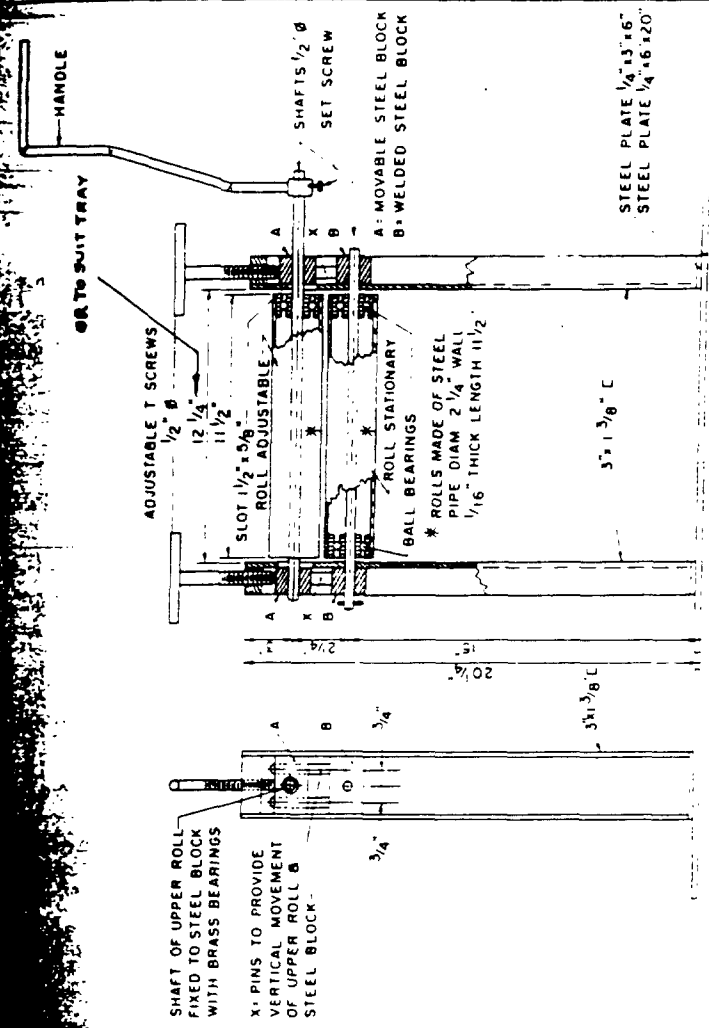


FIGURE 1

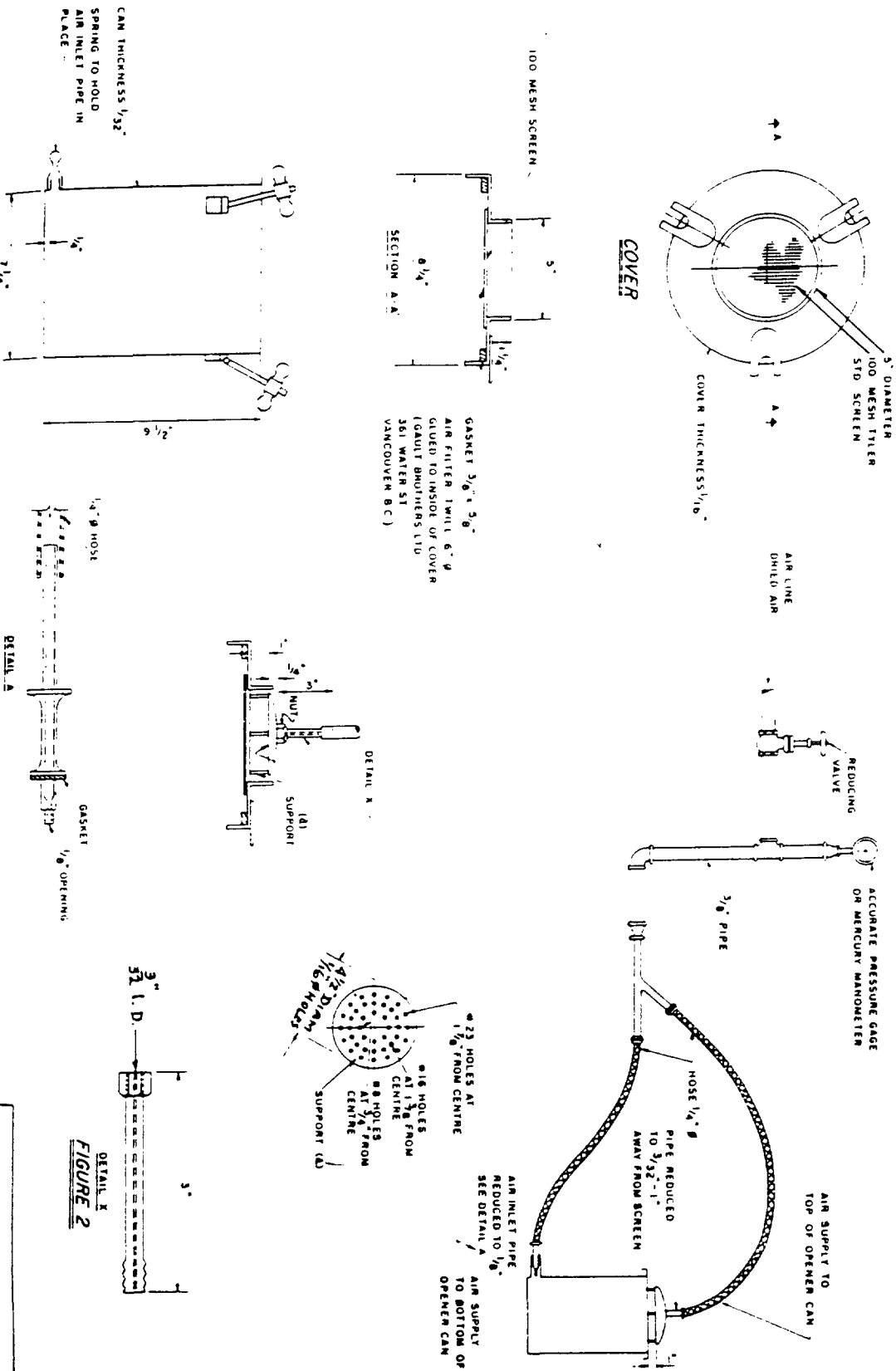
TINSMITH ROLLS TO PREPARE CRUDY OR
SPLINTERY FIBRES PREVIOUS TO AIR OPENING

SCALE 0 1/2" 1 FT

ST0513830

ST0513831

387645



Method of Test for
Measuring the Length and Length Distribution
of Asbestos Fibre by the Modified Suter-Webb
Cotton Sorter

Adopted by

ACPA -
ATI - 9/10/59
QAMA - 4/27/61

ST0513832

METHOD OF TEST FOR
MEASURING THE LENGTH AND LENGTH DISTRIBUTION
OF ASBESTOS FIBRE BY THE MODIFIED SUTER-WEBB
COTTON SORTER

SCOPE

1. This method of test is intended primarily for use in determining the mean length of a sample of asbestos containing few or no crudy bundles of fibre.* In addition, the data obtained in the test can be used to calculate the percentage of fibres by weight in each 1/8-in. length group. These in turn can be used for a detailed study of length distribution. This test is commonly limited to asbestos of Grade 3 or longer.

DESCRIPTION OF TERMS

2. (a) Mean Length - The average length of all the fibres in the sample, based on weight-length data.
(b) Crudy Bundles - Many fibre filaments still grouped together as hard, unopened bundles.

OUTLINE OF METHOD

3. A sorting apparatus consisting of a double bank of parallel combs spaced at 1/8-in. intervals, is used to align the fibre in a 200-mg specimen. The fibres are parallel in the series of combs in such a manner that all fibre lengths are grouped from a base comb. The different lengths are separated by dropping combs consecutively and arraying the fibres according to length group on a velvet covered board. The fibres in each length group are collected and weighed. From these data the mean length and length distribution can be calculated.

* The treatment for asbestos samples containing a high percentage of crudy bundles or splints is explained in the test method titled "Preparation of Crudy Fibres for the Suter-Webb Comb Test".

Modified Suter Webb Cotton Sorter - This sorter consists of two banks, each with 16 combs spaced 1/8-in. apart. The combs have two inches of needles .014-in. in diameter and 11/32-in. long, spaced 48 to the inch.

Special forceps, tips padded with hard leather, for combing and transferring fibres from one set of combs to the other. Two required.

Depressor for placing fibre in combs.

Aluminum plate covered with velvet cloth.

Fork for scooping up fibre groups off velvet cloth.

Dissecting needle.

Smooth plate for placing fibres onto velvet surface.

Special rule for measuring length of fibre groups.

Smooth pointed tweezers.

Lift for raising combs in place.

(1) Analytical balance sensitive to .1-mg and having a range of at least 0 to 200 mg.

(2) Tared watch glasses for weighing.

Sample - The sample shall be the laboratory test sample as prescribed in the "Sampling and Preparation of Asbestos Fibre for Test Purposes", or other asbestos fibre sample.

Test Specimen - The 200-mg test specimen shall be derived from the laboratory test sample. Quarter the sample down to approximately 25-gm prescribed in the Sampling and Preparation of Fibre, Part 2, (a) to (e). Divide the 25-gm sample into ten approximately equal portions and sub-divide these portions into halves. Combine a half of each of the ten portions and mix thoroughly. Repeat this procedure until the sample weight is reduced to approximately 3-gm. On a smooth clean area spread the 3-gm sample into a thin layer over an area of approximately 8" x 8" in such a manner

that pinches may be taken from all parts. Extract with tweezers five pinches of fibre from different parts, each weighing about 200 mg. Carefully separate any entangled fibre in the 1000 mg sample with dissecting needle and forceps, spread into a thin layer and extract at least five small pinches from different parts to make up the 200 ± 0.5 mg test specimen. All sampling and measuring shall be conducted in average workroom temperature and humidity.

PROCEDURE

6. (a) Combing - With the fibre sorter in front of the operator, with one bank of combs convenient to the left hand and the other to the right hand, raise all the combs in the left hand bank of combs as you face the sorter, and drop all but one comb in the right hand bank. The dropped combs will be away from the operator in the right hand bank. The 200 mg sample is divided into several tufts, which are distributed on a sheet of paper. Take one of the tufts of fibre in the fingers and align the fibre as much as possible. Now, grasp one half the long axis of the fibres with the special forceps and comb the protruding end through the single right hand comb until the fibres are thoroughly separated. Transfer the fibres to the other forceps. Now, grasping the straightened end, comb until the fibres are again completely separated. Place the combed fibres in the left hand bank of combs with the fibres as nearly as possible perpendicular to the combs, and press in place with the depressor. Collect the fibres remaining on the single comb, and repeatedly comb until all possible fibres are in the left hand comb. Fibres too short for insertion into the combs are set aside. Take each of the tufts of fibre in turn and repeat steps as for the first tuft. The fibres should be spread uniformly over the length of the left hand combs.
- (b) Transfer - Raise the dropped combs in the right hand bank. Drop the left hand combs successively. As the combs are successively dropped, the protruding fibres are extracted with the special forceps and transferred to the right hand bank of combs and pressed in place with the depressor. As they are being so laid, the protruding ends are to be brought to an even $1/8$ in. beyond the first comb by pulling out any fibres protruding more than $1/8$ in. for re-

ST0513835

insertion into the combs. The special ruler with a line drawn $1/8$ in. from one edge is used to obtain the $1/8$ in. protrusion. Any fibres too short for inserting in the right hand combs are combined with those previously set aside.

(c) Array - Revolve the apparatus 180° so that the ends of fibres that were farthest from the operator become the nearest ends. Drop combs until the first fibres appear in front of a comb. With the special forceps withdraw the fibres in small "bites" and place them in a group upon the velvet-covered plate. Record the number of combs remaining in the up position. Drop another comb and again remove all the protruding fibres and place these in another group upon the velvet-covered plate. Successively drop combs until the fibres have been withdrawn from all the combs, and the groups of fibres so withdrawn, placed, with short spacings between, upon the velvet-covered plate. Clean all combs of short fibres, and combine with those previously set aside. Collect any short fibres on and around the apparatus, and add to the shorts previously set aside. Collect and weigh the groups of fibres. The sum of the weight of the fibres must not differ from 200 mg. by more than 10 mg.

Note 1 - A proficient technician may eliminate the second combing and array after the first combing, providing he has proven that he can obtain reproducible results against the above procedure. If the second combing is eliminated, it should be noted on the Suter-Webb report.

ATIONS AND REPORT

To calculate the average length of the fibres in the test specimen we assume that the fibres protrude $1/8$ in. beyond the first comb, the spacing between the combs, and that the average length of the fibres removed after a comb is dropped is midway between the two combs. Thus the average length of the short fibres which could not be placed on the combs is taken as $1/16$ in.; the length of the fibres removed with one comb raised is $1/8 + 1/16$, or $3/16$ in.; with two combs raised is $1/8 + 1/8 + 1/16$, or $5/16$ in. Thus, starting with the short fibres set aside, the fibre length will be $1/16$ in., and passing from the first and through each succeeding comb, the fibre lengths

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will be $3/16$, $5/16$, $7/16$, etc. The weight of each group of fibres is multiplied by its average length, and the products totalled. This sum divided by the total weight of fibre gives the average length of the fibres. Record the length to one thousandths of an inch. For each laboratory sample, make two determinations for average length. If the average length of either deviates from the mean by more than ten percent, the determination is repeated. The length distribution of the fibres is obtained by calculating as a percent of the total sample, the weight of fibre for each length increment. Record to one hundredths of an inch.

ST0513837

RO TAP SCREEN ANALYSIS
FOR ASBESTOS FIBRES

Adopted by

ACPA - 9/10/59
ATI -
QAMA - 4/28/59

ST0513838

RO TAP SCREEN ANALYSIS FOR ASBESTOS FIBRES

SCOPE

This procedure covers the dry sieve analysis of asbestos fibres. It is intended to measure the apparent length and distribution of fibres in all grades with the exception of Groups 1, 2 and 3.

APPARATUS

1. Sieve Shaker - See notes.

W. S. Tyler Ro Tap Testing Sieve Shaker for 8" diameter sieves. The machine shall be equipped with Sieve Supporting Plate and Cast Iron Cover fitted with a rubber plug (Specification: No. 9 Neoprene).

Number of taps per minute shall be 154 ± 4 when operating at a speed of 285 ± 5 rpm. The shaker should be mounted on a suitable firm foundation, preferably concrete.

2. Sieves - The following Tyler Standard Mesh Sieves or U. S. equivalents shall be used for the various groups as follows:

<u>GROUP 4</u>				<u>GROUP 5</u>			
*Full height		3 mesh		Full height		4 mesh	
"	"	4 "		"	"	6 "	
"	"	6 "		"	"	10 "	
"	"	10 "		"	"	20 "	
"	"	20 "		"	"	35 "	
"	"	35 "		"	"	65 "	
"	"	Pan		"	"	Pan	

*This 3 mesh is used as a breaker and since there is no U. S. equivalent for this screen size, a 3 1/2 mesh may be used.

ST0513839

GROUP 6 & 7D

height	6 mesh
"	10 "
"	14 "
"	20 "
"	35 "
"	65 "
"	Pan

GROUPS 7 & 8 except 7D

Full height	6 mesh
" "	14 "
" "	20 "
" "	28 "
" "	35 "
" "	65 "
" "	Pan

Automatic Timer, Model 8063-B or equivalent with accuracy of ± 5 seconds.

sensitive to 0.1 gram.

compliance with "Method of Sampling and Preparation of Fibre for Test Purposes".

SIZE

4	50 grams
5	50 "
6 including 7D	100 "
7 except 7D	100 "
8	100 "

TIME

4	10 minutes
5	10 "
6 including 7D	10 "
7 except 7D	30 "
8	30 "

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PROCEDURE

The required weight of the representative sample is placed on the top sieve of the nesting. The cast iron cover is set in place and the sieve assembly is then placed in the shaker and locked in position. By means of the automatic timer the shaker is started and allowed to run for the required test period. The fibre on each screen is weighed to the nearest 0.1 gram.

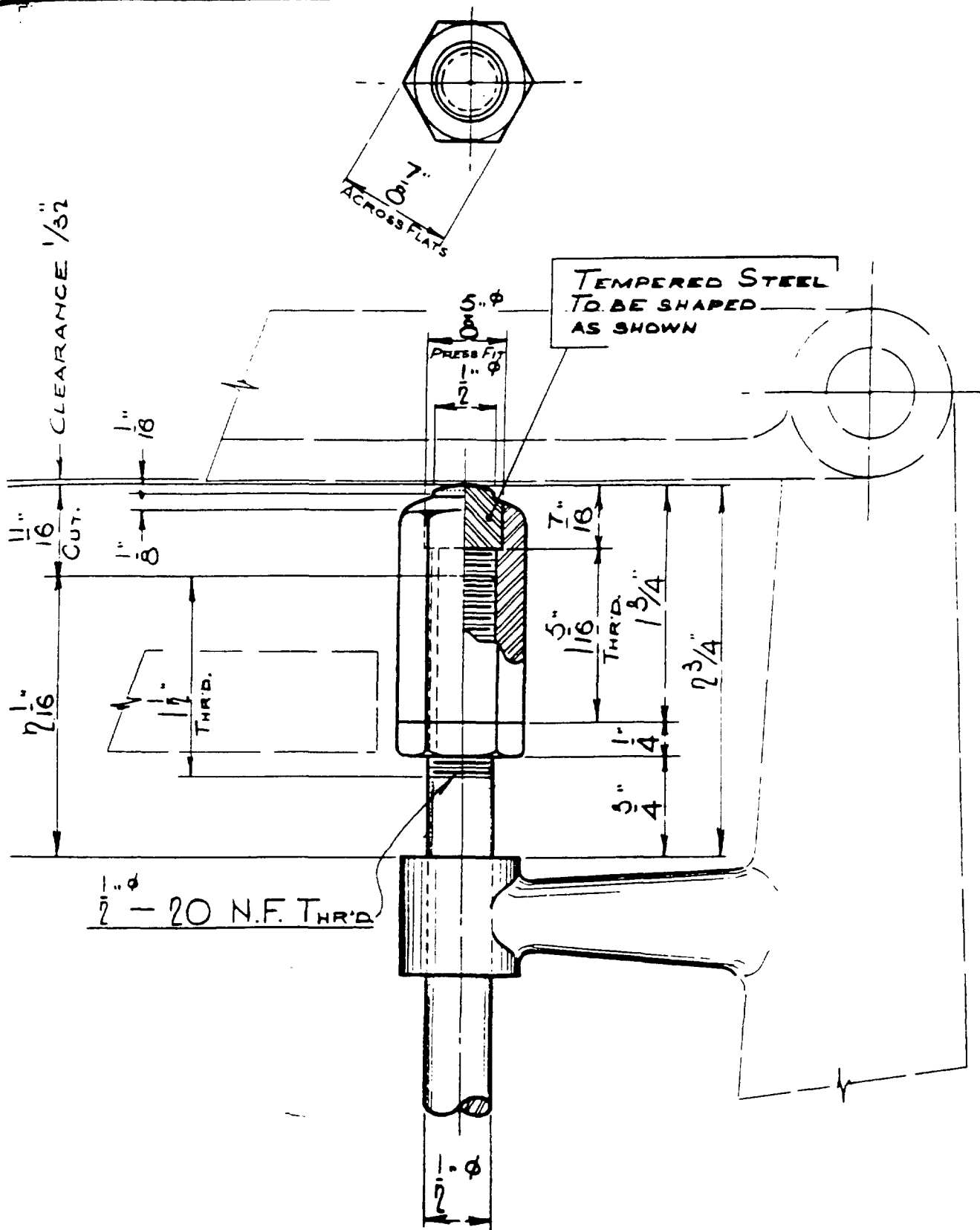
REPORT

The screen analysis is recorded as the percentage of material retained on each sieve and in the pan. Allowable error in the total should not exceed $\pm 1.0\%$.

NOTES

1. Plate Support should be set to give a clearance of $3/32" \pm 1/32"$ between top of cast iron cover and bottom of stops on Carrying Plate. (See A on Figure B). This clearance is sufficient to allow the screen assembly to be easily set in place and to rotate freely when shaker is in operation. It is important that this gap be checked periodically in order to maintain proper sieving action.
2. The height of the stopper above the top of the cast iron cover plate should be $1" \pm 1/8"$ (A plus B on Figure B) measured with hammer resting on stopper. With a new stopper this measurement should be taken only after machine has been in operation for a few hours and should be set as closely as possible to the maximum setting of $1 \frac{1}{8}"$.
3. Clearance between push rod and hammer in lower position shown at E on Figure B should be $1/16" - 5/32"$. It is necessary to make periodic adjustments to compensate for wear. This may be done by using an adjustable push rod. (See Figure A).

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TEMPERED STEEL
TO BE SHAPED
AS SHOWN

$\frac{1}{2}$ " ϕ - 20 N.F. THRD.

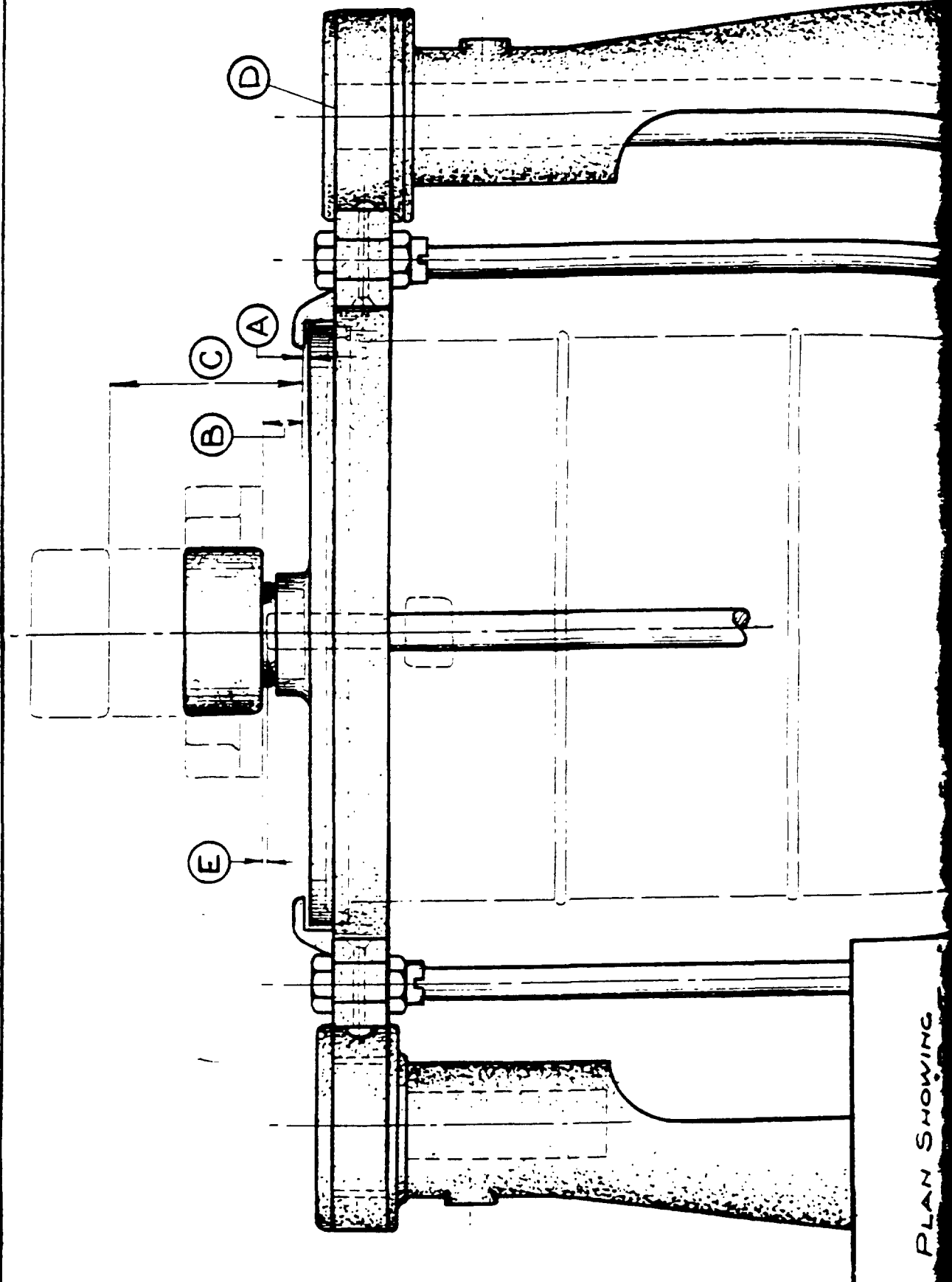
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ROTAP ADJUSTABLE
PUSH ROD

G. FIG. "A" FEB. 12/59

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Bauer-McNett Wet Classification
Test for Asbestos Fibre

Adopted by

ACPA - 9/15/59
ATI - 9/10/59
QAMA - 6/23/59

ST0513844

BAUER-McNETT WET CLASSIFICATION TEST FOR ASBESTOS FIBRE

SCOPE

1. To determine the length distribution and dust content of milled asbestos fibre by wet analysis employing the Bauer-McNett Fibre Classifier (See "Apparatus" 2(a)).

APPARATUS

- 2 (a) Bauer-McNett Fibre Classifier No. 203A (For Details see Instructions for the Installation, Operation and Maintenance of the No. 203A Bauer-McNett Fibre Classifier - The Bauer Bros. Co., Springfield, Ohio) with Improved Vacuum Cups on Drainage Tanks, similar to Appendix I attached.

Note 1. - Drainage through filter cloths or 200 M Tyler Sieve is acceptable.

2. (b) Additional apparatus required for performance of test:
 - (1) Balance (Sensitivity 0.01 Gms).
 - (2) 1000 Ml. Beaker & Stirring Rod.
 - (3) Filter Papers, Reeve Angel No. 230*, or equivalent, to fit cups on machine.
 - (4) Long-handled Bottle Brush.
 - (5) Rubber Hose 1/4-inch dia. or Copper Spray Tube (See Appendix II attached).
 - (6) Rubber Stoppers.
 - (7) Electric Drying Oven (Forced Air or Convection), or Infrared Lamp Dryer.
 - (8) Bell Timer, or Stop Watch.
 - (9) Automatic Overflow Alarm for Fourth Tank of McNett Classifier (See Appendix III attached).

2. (c) Water Supply - Sand, or Cartridge Type Filter, similar to *Cuno 1B2-2278 to be provided on water supply feeding headbox of machine to ensure a constant flow of clean water.

Footnote:- * Has been found satisfactory for this purpose.

SAMPLE WEIGHTS, SCREEN SIZES & TIME

GROUP	SCREEN SIZES (Tyler Mesh)	SAMPLE WGT (GM)	DURATION OF TEST (Mins)
(a) 3	4,14, 35,200	10	20
(b) 4,5,6 & 7D	4,14, 35,200	10	20
(c) Other 7-Grp Fibres	14,35,100,200	20	20
(d) "Floats"	80,325	20	30
(e) For reference Tyler and U. S. Equivalent sieves are listed below:-			

Tyler Series

U. S. Series

Mesh	Opening	Mesh	Opening
4	0.185 "	4	0.187 "
14	0.046 "	16	0.0469"
35	0.0164"	40	0.0165"
80	0.0069"	80	0.0070"
100	0.0058"	100	0.0059"
200	0.0029"	200	0.0029"
325	0.0017"	325	0.0017"

SAMPLE PREPARATION

- (a) In accordance with the "Standard Method of Sampling and Preparing Asbestos Fibre for Testing."
- (b) Perform McNett Tests on duplicate samples obtained by the standard sampling procedure and average the results (See "ACCURACY" 7. (a) for permissible discrepancy in test results.)

PROCEDURE

- (1) Select screens specified for the fibre grade being tested and set in place making certain that the baff-
le plates are in their slots behind the screens.

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- (b) With the rubber stoppers inserted, clean screens installed, and the tanks thoroughly washed after the preceding test, fill tanks with water.
 - (c) When the tanks are filled with water, start the motor to operate all agitators at 540 ± 40 r.p.m.; and adjust the line valve to give a flow slightly in excess of 3.0 U.S. gallons per minute to run the test. This rate of flow is obtained when a pencil-size overflow from the constant head tank occurs.
 - (d) Add test sample as specified under "SAMPLE WEIGHTS, SCREEN SIZES & TIME", obtained by the standard sampling procedure, to 800 ml of water in a 1000 ml beaker and stir until the asbestos is thoroughly dispersed.
 - (e) Pour slurry into tank No. 1 (tank with the largest screen mesh) and wash out any fibre residue in the beaker with clean water. Set timer for 20 or 30 minutes, depending upon the sample under test.
 - (f) During the operation, centre the dried filter papers, previously weighed to the nearest 0.01 gms, in vacuum cups on supporting screen, wet filter paper and clamp into position. Mark weight of filter paper, sample designation, McNett screen mesh and other pertinent data around outside edge of each filter with a soft black pencil before inserting in vacuum cups with writing against the screen.
 - (g) Open each vacuum cup valve and close the main vacuum valve.
- Note 2 - It is advantageous to perform the above two steps during the five minutes in which the McNett tanks are initially being filled.
- (h) Watch screens carefully during the test and if any tank appears to overflow, brush with a long handle brush to remove the fibres, or wash with spray pipe (See Appendix II,) applying the spray against the screen contrary to the flow of the water.
 - (i) After the running time for the sample being tested has elapsed, open the main vacuum valve until the gauge indicates a minimum vacuum of 4 inches water gauge, remove the drain plugs from each tank; and progressively increase the vacuum to the maximum desired. Failure to perform this operation slowly or

to remove all the drain plugs before any one tank is completely drained will result in ruptured filter papers.

j) During draining, remove the screens and place them in their respective tanks. Immediately after a tank has been completely drained carefully wash screen and tank using the bottle brush and hose spray to insure that all remaining fibre, including entrapped fibre and particles deposited behind the screen, are washed into the filter cup.

(k) When all the fibre in a tank is deposited into the cup and the cup itself has drained close the cup drain. Failure to close the drain after washing down and emptying the tank causes atmospheric air to enter the vacuum system and substantially lowers the vacuum of the system.

(l) After closing the cup valve, open the cup and remove the filter paper and fibre residue and dry to constant weight at $220^{\circ} \pm 5^{\circ}\text{F}$. under the infrared lamp drying unit, or in a suitable electric drying oven.

After all the samples are dried and allowed to return to room conditions, weigh the fibre residue and its filter paper together and obtain the net fibre weight of the sample by subtracting the initial weight of the filter paper and report as percent. The amount of -200M in the original sample is calculated by subtracting the cumulative weights of the material retained on the four filter papers from the original weight of the sample.

(See Note 3. concerning special crudy fibres.)

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RESULTS & SAMPLE CALCULATIONS

6. Below is an example of a typical 5-Group fibre McNett test with calculations:-

Sample: 5-Group 10 Grams, 20 Minutes

Screens	4	14	35	200 Mesh
<u>Wgt. Retained (Gms)</u>	0.5	1.3	1.9	1.4
Minus 200M-	10.0	$-(0.5+1.3+1.9+1.4) = 4.9$ Gms.		

Results:-

Screens	4	14	35	200	-200 Mesh
% Retained:	5.0	13.0	19.0	14.0	49.0%

ACCURACY

7. (a) The accuracy of this test is considered to be five per cent; i.e., the difference between two results on any one screen shall not exceed five per cent of the total sample. The results of two tests are to be averaged and reported for each fibre sample tested.
- (b) To obtain desired accuracy the following precautions should be observed:-
- (1) It is important that all material be washed from the tanks into the filter cups; and that the filter papers are handled with care.
 - (2) Fractions should be completely dried when weighed.
 - (3) Always have a slight (pencil size) overflow from the constant level tank.
 - (4) Screens should be checked regularly (at least once every operating shift) for damage, holes, warped frames, etc. Likewise the rubber gaskets should be examined and replaced if defective as this is frequently the cause of abnormal minus 200M results. The holding screws are to hold the screen plates in place against the tank body to prevent leakage around the sponge rubber gaskets. The screws should be turned tight enough to hold the screens firmly against the plate; but if turned too tight,

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or not uniformly, the screen plates may be permanently warped out of shape causing leakage past the gaskets.

- (5) The normal agitator speed (540 ± 40 r.p.m.) should be checked daily and also the belts for slippage.
- (6) Motor bearings and drive shaft should be lubricated with a good grade of ball bearing grease every three months. No lubricant is required for agitator bearings as they are of the sealed for life type.
- (7) When classifier is not in use, tanks should be kept full of water to prevent deposits forming on screens.
- (8) To obtain prescribed accuracy, screens should not be cleaned during a determination, unless tanks tend to overflow. Test results are not considered reproducible when screens are cleaned frequently and at irregular intervals during the test, due to fibres being forced through the mesh openings and the personal factors introduced by the laboratory technique involved.

Note 3. - In the case of crudy fibres, the McNett fractions may be retained for examination; and the percentage of crudy bundles and rock particles determined by an approved method.

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$\frac{3}{4}$ " ϕ Brass Nipple x 3" Long.

7" ϕ In. Dia. "Sample Cup"
MAT'L. ALUM.

"LOCK" — 2-REQ'D.
MAT'L. BRASS

"Lock Ring" $\frac{5}{8}$ x $\frac{1}{4}$ "

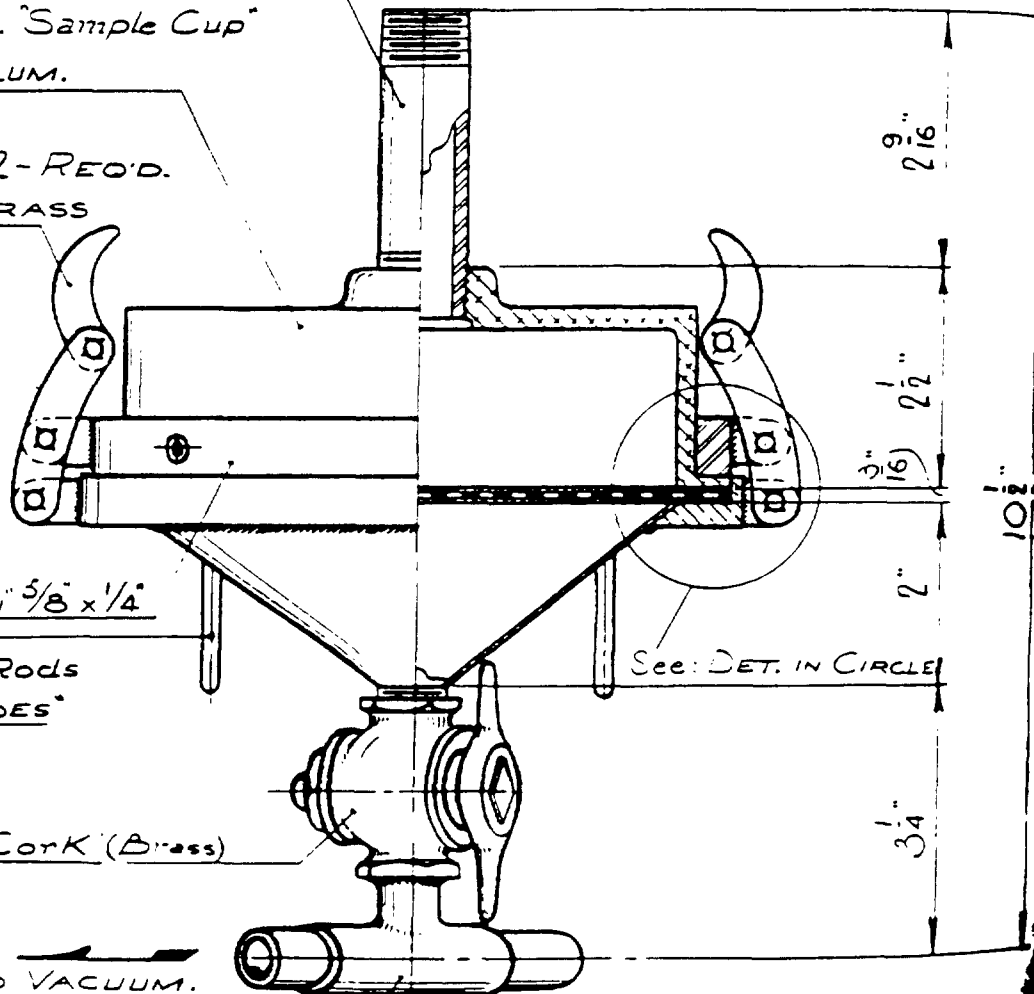
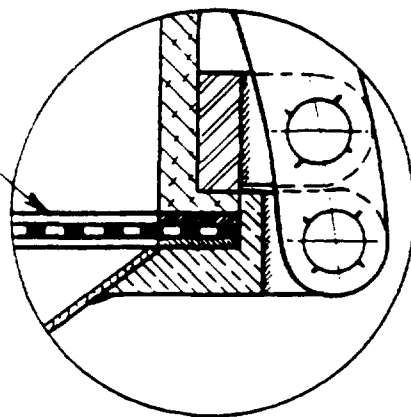
$\frac{1}{4}$ " ϕ Brass Rods
"CONE GUIDES"

$\frac{1}{2}$ " Mine Cork (Brass)

TO VACUUM.

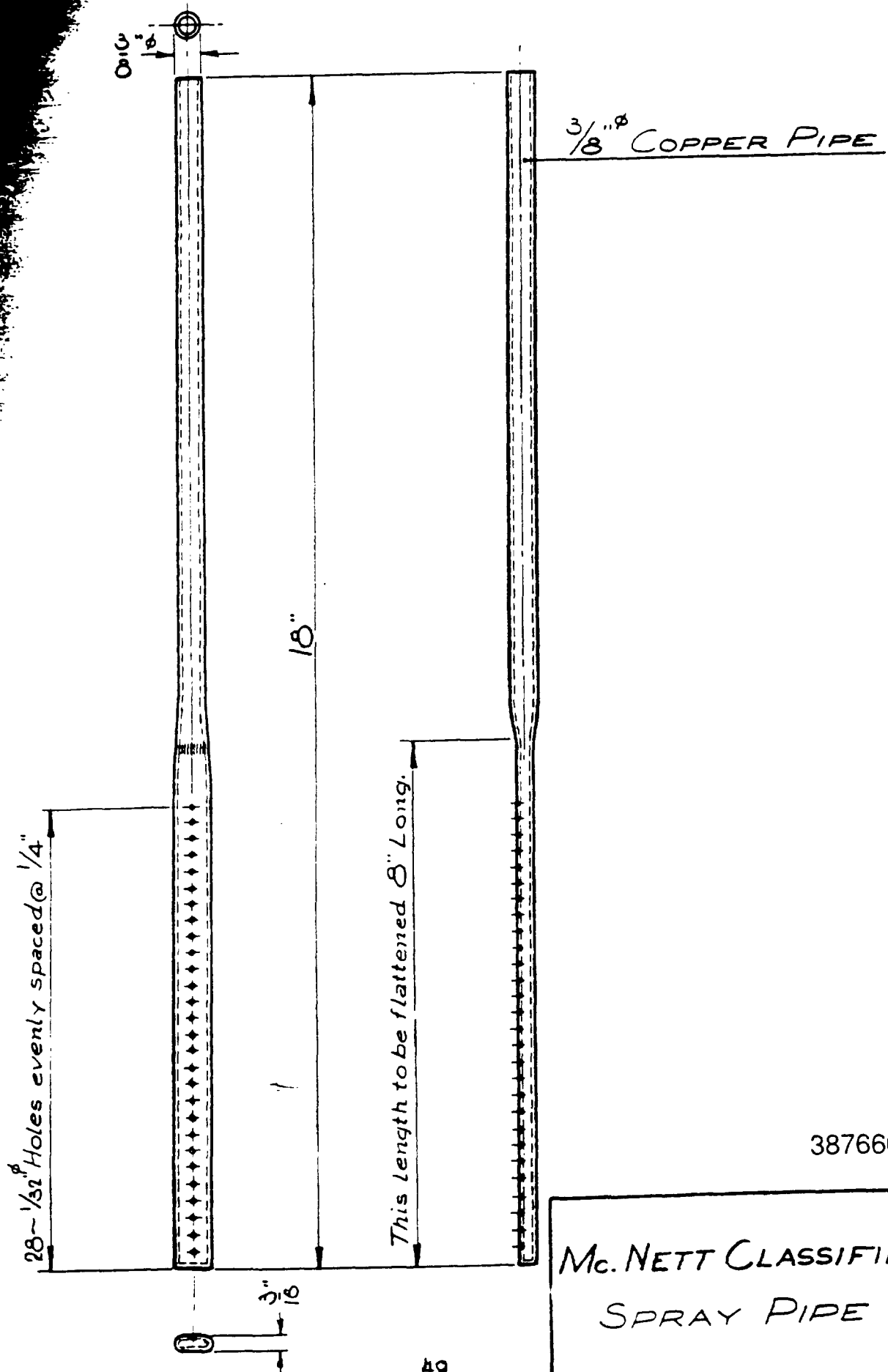
$\frac{1}{2}$ " Seamless Pipe "T"

14" x 14" Mesh
(Wire Screen)
Between Rubber
Seals



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Mc. NETT CLASSIFIER
VACUUM CUP
FOR 18.5cm. ϕ DIA. FILTER P

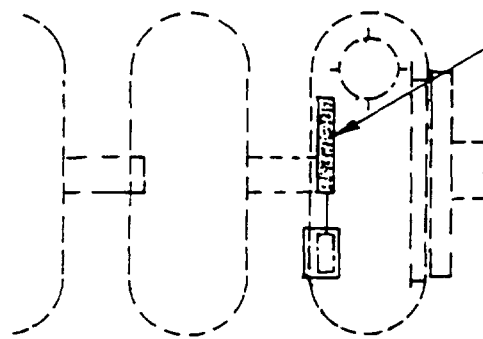


Mc. NETT CLASSIFIER
 SPRAY PIPE

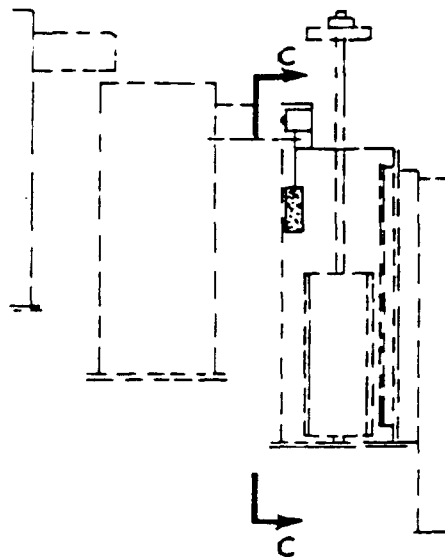
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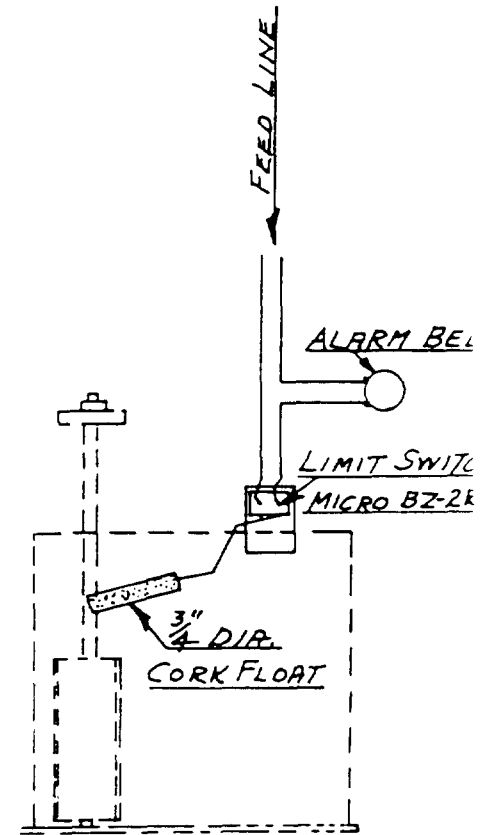


PLAN VIEW



ELEVATION

FLOAT TO BE INSTALLED IN TAN
ON OPPOSITE SIDE TO SCREEN
AND TO CLEAR IMPELLER



VIEW C-C

OVERFLOW ALARM
BAUER MCNETT CLASSIFIER
SCALE NONE

Method of Test for Length Distribution
and Dust Content of Asbestos Fibres
of 4A Grade and Longer
by Wet Screening Using the Clark Classifier

Adopted by:

ACPA -
ATI - 9/10/59
QAMA - 2/23/60

ST0513854

METHOD OF TEST
FOR LENGTH DISTRIBUTION AND DUST CONTENT
OF ASBESTOS FIBRES OF 4A GRADE AND LONGER
BY WET SCREENING USING THE CLARK CLASSIFIER

SCOPE

1. This method of test covers the Clark Classifier and procedure for determining the length distribution and the dust content of asbestos fibres of 4A grade and longer.

APPARATUS

2. (a) Nature of Apparatus - The Clark Classifier consists essentially of a semicircular tub divided into four main compartments in each of which is a circular screen or perforated disc mounted on and driven by a common shaft rotated at about 48 revolutions per minute by a geared motor. The rim of each disc and screen forms a seal by rotating between the longitudinally slit edges of a piece of soft rubber tubing mounted in a small metal channel on the inside of the tub. By means of a constant headbox, water is passed through the classifier at a chosen rate of 12.5 liters per minute. In each compartment the incoming water is directed to the bottom of the tub by means of a weir and baffle. The consequent motion of the water serves to keep the asbestos fibres from settling and to present them regularly to the rotating discs and screens. Depending on the size and the design of the disc and screen openings, a definite fibre length separation is obtained in each of the four compartments. The apparatus, illustrated in Figures 1, 2(a), 2(b) and 2(c), shall consist specifically of the parts described in paragraphs (b) to (i).
- (b) Classifier - Clark Model 46, four screen pulp classifier as shown in Figure 1.
- (c) Perforated Discs - Three perforated discs fabricated of Plexiglas and conforming to the design shown in Figures 2(a), 2(b) and 2(c). The disc for the first compartment shall contain 1/2" perforations; the disc for the second compartment shall contain 1/4" perfora-

tions; the disc for the third compartment shall contain 1/8 inch perforations.

- (d) Screen - 30 mesh screen as supplied with the Clark classifier. The 30 mesh screen shall be used for the fourth compartment.
- (e) Balance - Balance capable of weighing five grams to the closest 0.001 gram.
- (f) Drying Oven - Standard gravity convection type drying oven capable of maintaining 220°F plus or minus 5°.
- (g) Beaker - Beaker of 1500 milliliter capacity.
- (h) Filter Paper - Qualitative filter paper discs, 90 millimeters in diameter.
- (i) Timer - Timer capable of being read to the nearest 0.5 second. The timer shall be accurate to one per cent or less.

SAMPLING OF ASBESTOS FIBRE

- 3. The sampling shall be conducted in accordance with the "Standard Sampling and Preparation Procedures for Asbestos Fibre".

PROCEDURE

- 4. (a) Size of Sample - The weight of sample used for this test shall be 5.000 plus or minus 0.001 grams.
- (b) Preparation of Equipment for Classification - A drain cup, fitted with a filter paper disc which has been weighed to the closest 0.001 gram, shall be placed on each of the four drain pipes. A No. 3 rubber stopper shall be inserted into each of the drain pipes and the drain plug shall be inserted into the outlet line of the flow box leading to the first compartment. The water-rate to the compartments shall then be adjusted to 12.5 plus or minus 0.1 liters per minute and rotation of the discs and screen shall be started.

Note 1 - The No. 3 rubber stoppers are used in the compartments instead of the supplied drain plugs to eliminate tangling of the fibres on the drain plug stems.

ST0513856

Note 2 - The water rate to the compartments is adjusted by means of the metering valve located in the line from the constant headbox to the tub. Once this valve has been set, no further adjustments are necessary. Care shall be exercised in setting the water rate to the constant headbox that a continual overflow is obtained.

- (c) Wet Screening Test - When overflow from the last compartment of the classifier is obtained, the fibre sample shall be placed in a 1500 milliliter beaker to which approximately one liter of water has been added and shall be stirred to give a uniform slurry. The timer shall then be started and the slurry shall be poured into the weir box of the first compartment over approximately a one minute period. Care shall be exercised that the beaker is rinsed thoroughly.

During classification, the discs and screen shall be kept free of excess fibre accumulation by periodically flushing with water and any fibre which tends to settle in the compartments shall be stirred into suspension by squirting with a small quantity of water. At 20 minutes in the time schedule, the water flow to the compartment shall be turned off and the disc and screen rotation shall be stopped.

Note 3 - The water flow to the compartments can be stopped most conveniently by removing the drain plug in the outlet line of the flow box leading to the first compartment.

- (d) Collection and Drying of Fractions - The rubber stoppers shall be removed from the drain pipes and the compartments allowed to empty. The discs, screen, and compartments shall be rinsed thoroughly and the excess water shall be removed from the fibre pads on the drain cups by blowing air down each drainpipe. The drain cups shall then be removed and the four filter paper discs containing the fibre pads shall be dried in a gravity convection oven for approximately two hours at 220°F. plus or minus 5°. After drying, the filter paper discs containing the fibre pads shall each be weighed to the closest 0.001 gram.

Note 4 - When rinsing the compartments, special care shall be exercised to remove any fibre caught in the rubber seals and to flush the drain pipes thoroughly.

Note 5 - Since the fibre and the filter paper are not dried initially, the filter paper discs containing the fibre pads shall be allowed to absorb atmospheric moisture for approximately one hour after removal from the oven and prior to weighing.

CALCULATION AND REPORT

5. (a) The weight of fibre in each of the four fractions shall be determined to the closest 0.001 gram. The per cent fibre in each of the four fractions shall then be calculated to the closest 0.1 per cent and the per cent dust (minus 30 mesh material) shall be determined by difference.
- (b) Report the weight per cent of fibre in each of the four fractions and the weight per cent dust (Minus 30 mesh material) to the closest 0.1 per cent.

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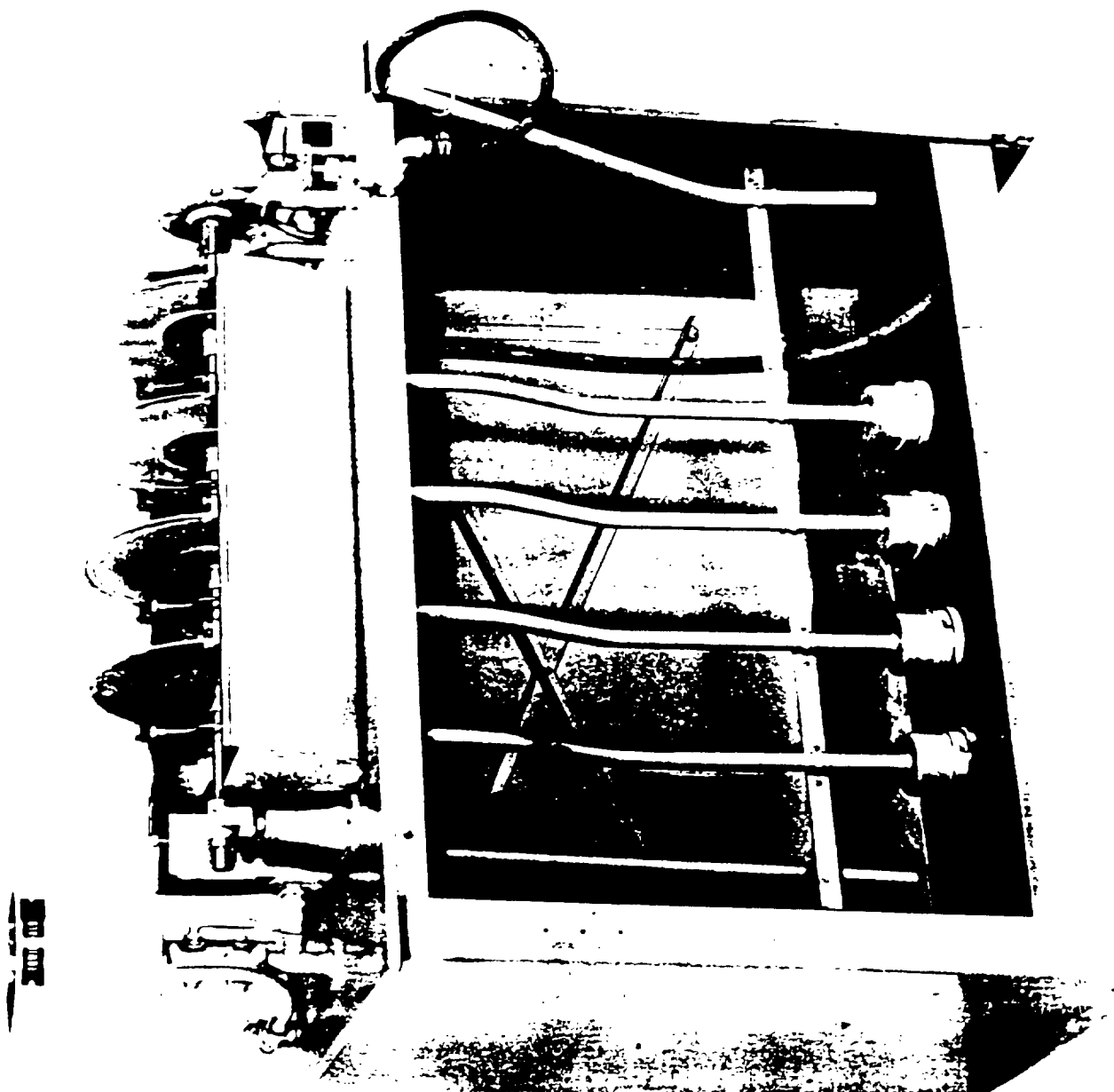
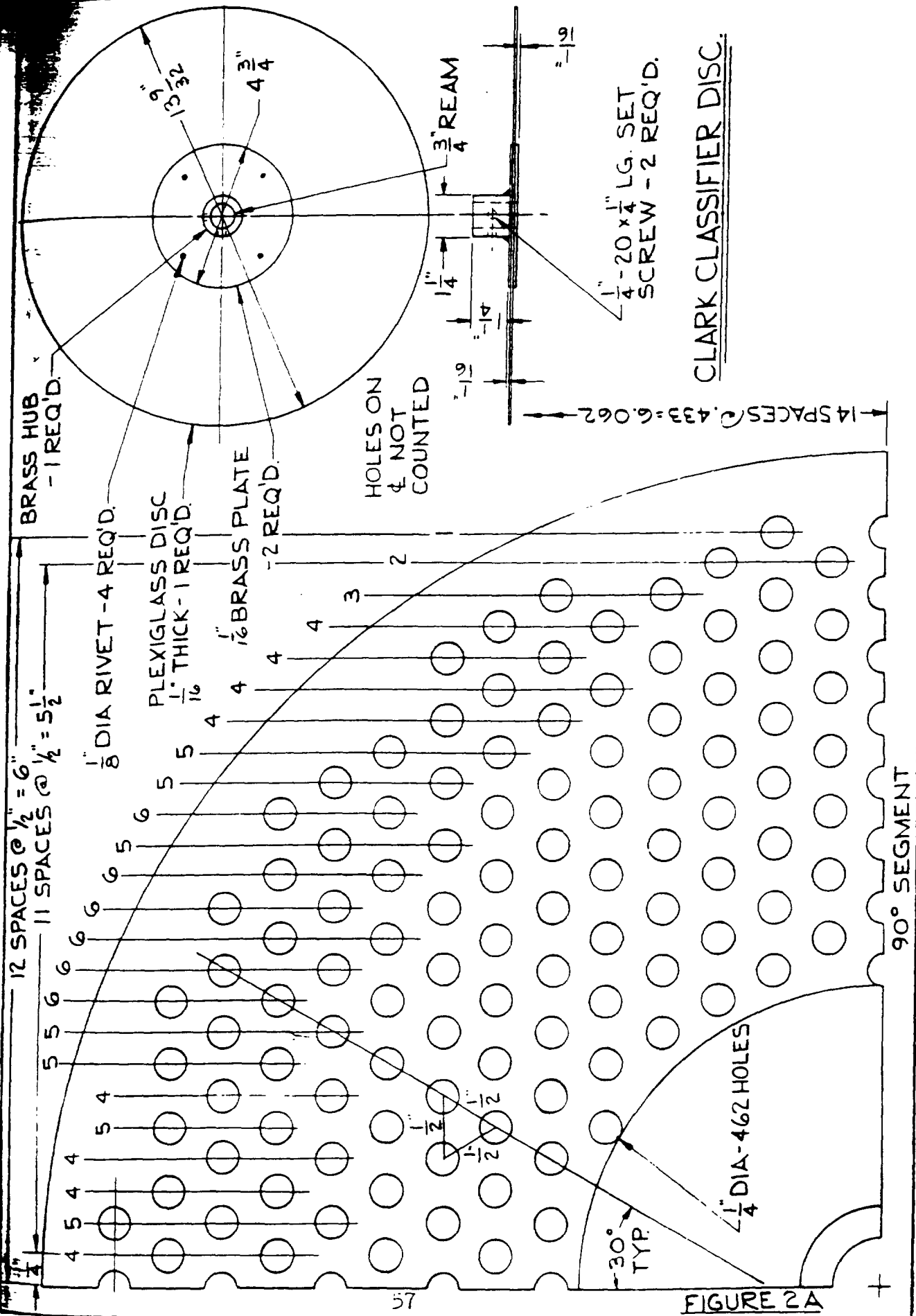


FIG.1.-Clark Classifier

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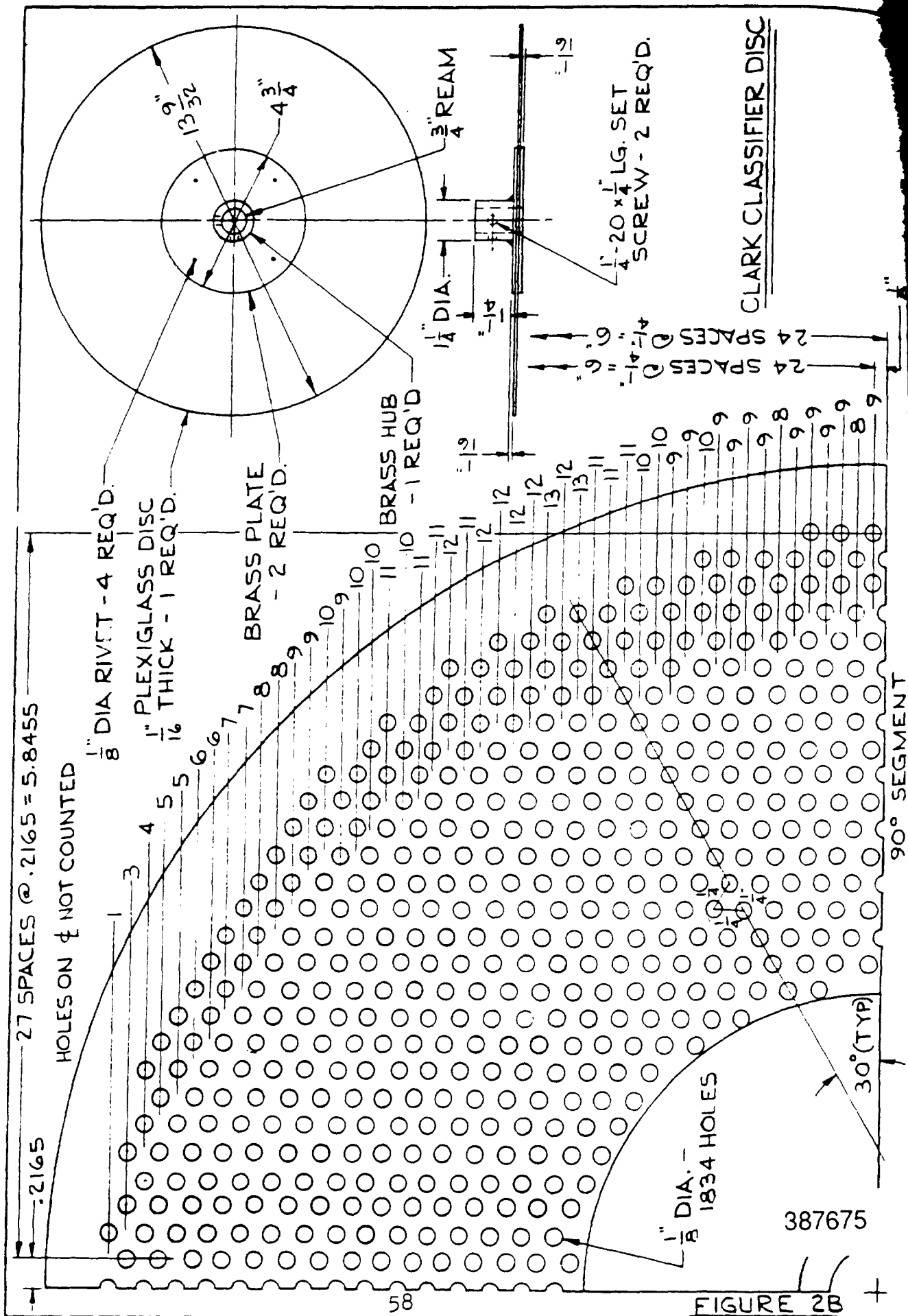


FIGURE 2B

90° SEGMENT

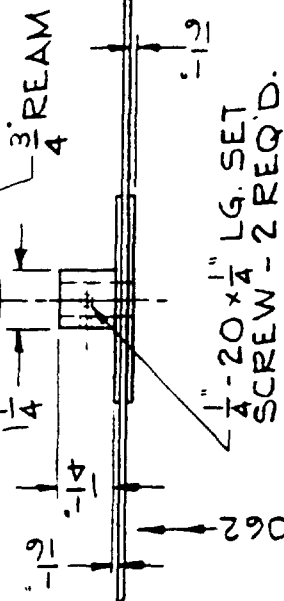
6 SPACES @ 1" = 6"
5 SPACES @ 1" = 5"

BRASS HOPE
1 REQ'D

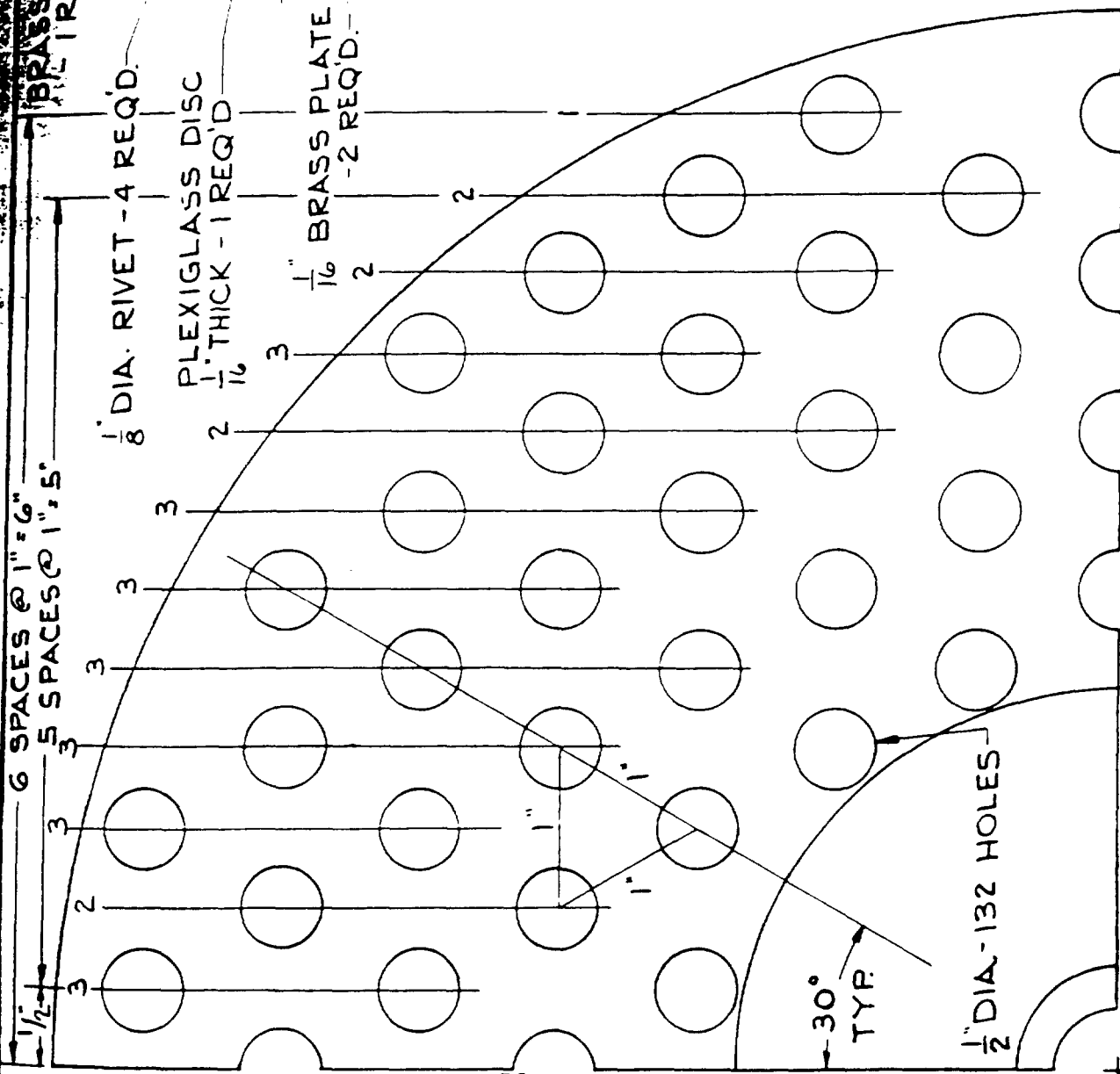
1" DIA. RIVET - 4 REQ'D

PLEXIGLASS DISC
1" THICK - 1 REQ'D

1" BRASS PLATE
- 2 REQ'D



CLARK CLASSIFIER DISC



90° SEGMENT

ST0513863

Method of Test for Length Distribution and Dust
Content of Asbestos Fibres Shorter Than 4A Grade by Wet
Screening Using the Clark Classifier

Adopted by:

ACPA - 9/15/59

ATI -

QAMA - 2/23/60

METHOD OF TEST FOR LENGTH DISTRIBUTION AND DUST
CONTENT OF ASBESTOS FIBRES SHORTER THAN 4A GRADE
BY WET SCREENING USING THE CLARK CLASSIFIER

SCOPE

1. This method of test covers a procedure for determining the length and the dust content of asbestos fibre by the Clark Classifier Wet Screen Method and is applicable to the fibre grades used in asbestos-cement products.

EQUIPMENT

2. (a) The equipment, illustrated in Figures 1, 2(a), 2(b), 3(a) and 3(b), shall consist specifically of the parts described in paragraphs (b) to (c).
- (b) The equipment for classification shall consist of the following:
 - (1) Classifier - Clark Model 46, four screen pulp classifier as shown in Figure 1.

The Clark Classifier consists essentially of a semi-cylindrical tub divided into four main compartments in each of which is a circular screen or perforated disc mounted on and driven by a common shaft rotated at about 48 revolutions per minute by a geared motor. The rim of each disc and screen forms a seal by rotating between the longitudinally slit edges of a piece of soft rubber tubing mounted in a small metal channel on the inside of the tub. By means of a constant headbox, water is passed through the classifier at a chosen rate of 12.5 liters per minute. In each compartment, the incoming water is directed to the bottom of the tub by means of a weir and baffle. The consequent motion of the water serves to keep the asbestos fibres from settling and to present them regularly to the rotating discs and screens. Depending on the size and the design of the disc and screen openings, a definite fibre length separation is obtained in each of the four compartments.

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Note 1 - The Clark apparatus was designed for the classification of wood pulp. In adapting the apparatus for the classification of asbestos fibre, it was necessary to provide additional agitation in the various compartments to minimize settling of the crudier bundles.

- (2) Perforated Discs - Two perforated discs fabricated of Plexiglas and conforming to the design shown in Figures 2 (a) and 2 (b). The disc for the first compartment shall contain 1/4 inch perforations; the disc for the second compartment shall contain 1/8 inch perforations.

- (3) Screens - 30 and 100 mesh screens as supplied with the Clark Classifier. The 30 mesh screen shall be used for the third compartment; the 100 mesh screen shall be used for the fourth compartment.

Note 2 - The 30 mesh screen has openings 0.0203 inch in width; the 100 mesh screen has openings 0.0060 inch in width.

- (4) Nozzle Assembly - Nozzle assembly conforming to the basic design shown in Figures 3 (a) and 3 (b).
- (5) Balance - Capable of weighing five grams to the closest 0.001 gram.
- (6) Drying Oven - Standard gravity convection type drying oven capable of maintaining $220^{\circ}\text{F} \pm 5^{\circ}$.
- (7) Graduate Cylinder - Stoppered graduate cylinder of 500 ml capacity.
- (8) Filter Paper - Qualitative filter paper discs, 90 mm in diameter.
- (9) Timer - Capable of being read to the nearest 0.5 second and accurate to one per cent or less.

- (c) The equipment for projection shall consist of the following:

- (1) Projector - Vu-Graph overhead projector, Catalog No. 2025, or equivalent.
- (2) Screen - 70 x 70 inch Da-Lite Challenger or equivalent.

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- (3) Bottles - Four ounce cork stoppered glass bottles.
- (4) Dishes - Three inch Petrie dishes.
- (5) Compasses - Six inch compass and beam compass with two 12-inch extensions.

SAMPLING OF ASBESTOS FIBRE

- 3. The sampling shall be conducted in accordance with the Standard Sampling and Preparation Procedures for Asbestos Fibre.

PROCEDURE

- 4. (a) Size of Sample - The weight of sample used for this test shall be 5.000 ± 0.001 grams.
- (b) Preparation of Equipment for Classification - The nozzle assembly shall be positioned on the front of the classifier such that a nozzle extends into each compartment. Each nozzle shall be so adjusted that it is directed at a point approximately $1 \frac{1}{2}$ inches to the left of the disc or screen and approximately $7 \frac{1}{2}$ inches (measured along the circumference of the tub) from the top front edge of the compartment. A drain cup, fitted with a filter paper disc which has been weighed to the closest 0.001 gram, shall be placed on each of the four drainpipes. A drain plug shall then be inserted into each of the drain pipes and into the outlet line of the flow box leading to the first compartment.

With the nozzles in the first three compartments closed, the water rate to the fourth nozzle shall be adjusted to 800 ± 20 mls per minute. The water rate to the compartments shall then be adjusted to 12.5 ± 0.1 liters per minute and rotation of the discs and screens shall be started.

Note 1 --To eliminate tangling of the longer fibres on the drain plug stems, No. 3 rubber stoppers shall be used in the first two compartments instead of the supplied drain plugs.

Note 2 - The water to the classifier and the nozzle assembly shall be filtered to remove any foreign material present in the piping.

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Note 3 - The water rate to the compartments is adjusted by means of the metering valve located in the line from the constant headbox to the tub. Once this valve has been set, no further adjustments are necessary. Care shall be exercised in setting the water rate to the constant headbox that a continual overflow is obtained.

- (c) Wet Screening Test - When overflow from the last compartment of the classifier is obtained, the fibre sample shall be mixed with approximately 400 mls of water in a 500 ml stopper graduate by oscillating 40 complete times in one minute. The timer shall then be started and the fibre slurry shall be poured into the weir box of the first compartment over approximately a 30 second period. Care shall be exercised that the graduate cylinder is rinsed thoroughly.

After addition of the slurry, water from the nozzle assembly shall be fed alternately at approximately 30 second intervals into compartments 1, 2, 3 and 4 with this order of agitation being observed during the entire classification cycle. During classification, the discs and screens shall be kept free of excess fibre accumulation by periodically flushing with water. At 6 1/2 minutes in the time schedule the water flow to the compartments and the nozzle assembly shall be turned off, and the disc and screen rotation shall be stopped.

Note 4 - The water flow to the compartments can be stopped most conveniently by removing the drain plug in the outlet line of the flow box leading to the first compartment.

- (d) Collection and Drying of Fractions - The drain plugs shall be removed from the drain pipes and the compartments allowed to empty. The discs, screens and compartments shall be rinsed thoroughly and the excess water shall be removed from the fibre pads on the drain cups by blowing air down each drain pipe. The drain cups shall then be removed and the four filter paper discs containing the fibre pads shall be dried in a gravity convection oven for approximately two hours at $220^{\circ}\text{F} \pm 5^{\circ}$. After drying, the filter paper discs containing the fibre pads shall each be weighed to the closest 0.001 gram.

Note 5 - When rinsing the compartments, special care shall be exercised to remove any fibre caught in the rubber seals and to flush the drain pipes thoroughly.

Note 6 - Since the fibre and the filter paper are not dried initially, the filter paper discs containing the fibre pads shall be allowed to absorb atmospheric moisture for approximately one hour after removal from the oven and prior to weighing.

- (e) Length Measurements - The fibre pads shall be removed from the filter paper discs and placed in individual four ounce bottles. The bottles shall then be filled approximately half way with filtered water and the fibre mats dispersed by shaking. Small portions of each of the fractions shall be transferred to individual Petrie dishes using a pair of tweezers. Enough water shall be added to just cover the dish bottom and the fibre shall be distributed uniformly throughout the dish.

Each of the four fractions shall then be projected onto a screen at a magnification of 20 diameters and 50 representative fibres from each fraction shall be measured for length.

Note 7 - In the measurement of long and highly opened fibres, it is essential, for representative results, that a reasonable separation be obtained in the Petrie dish and that the individual fibres measured be made as straight as possible. In difficult cases the use of a 0.15 per cent solution of Aerosol OT instead of water will aid in the dispersion.

Note 8 - For length measurements, it is both convenient and more accurate to span the fibres with a compass and to add the individual lengths graphically on a sheet of paper.

Note 9 - Although a skilled operator can accurately select by eye typical fibres for measurement, it is preferable to use the procedure of measuring all fibres in order which touch two perpendicularly intersecting lines, centered in the projected field, until the required number of fibres has been measured.

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CALCULATION AND REPORT

5. (a) The weight of fibre in each of the four fractions shall be determined to the closest 0.001 gram and the arithmetical average length of each of the four fractions shall be determined to the closest 0.001 inch.

The per cent fibre in each of the four fractions shall be calculated to the closest 0.1 per cent and the per cent dust (minus 100 mesh material) shall be determined by difference. The average "dust free" length of the fibre shall then be calculated to the closest 0.001 inch using the following equation:

$$\text{Average Length} = \frac{(\%1) (L1) + (\%2) (L2) + (\%3) (L3) + (\%4) (L4)}{(\%1 + \%2 + \%3 + \%4)}$$

where:

% = weight per cent of fibre held in compartment,
L = arithmetical average length of fibre held in compartment in inches.

Note - For 3 and 4-grade chrysotile fibres and for blue asbestos, the following length constants may be used for the second, third and fourth compartments in the calculation of average "dust free" length:

	<u>Chrysotile</u>	<u>Blue</u>
Compartment 2	0.183	0.194
Compartment 3	0.103	0.123
Compartment 4	0.041	0.055

For 5, 6 and 7-grade chrysotile fibre the length constant for the second compartment shall not be used.

- (b) Report the weight per cent of fibre in each of the four fractions and the weight per cent dust (minus 100 mesh material) to the closest 0.1 per cent. Report the average length of each of the four fractions and the average "dust free" length of the fibre to the closest 0.001 inch.

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L4)

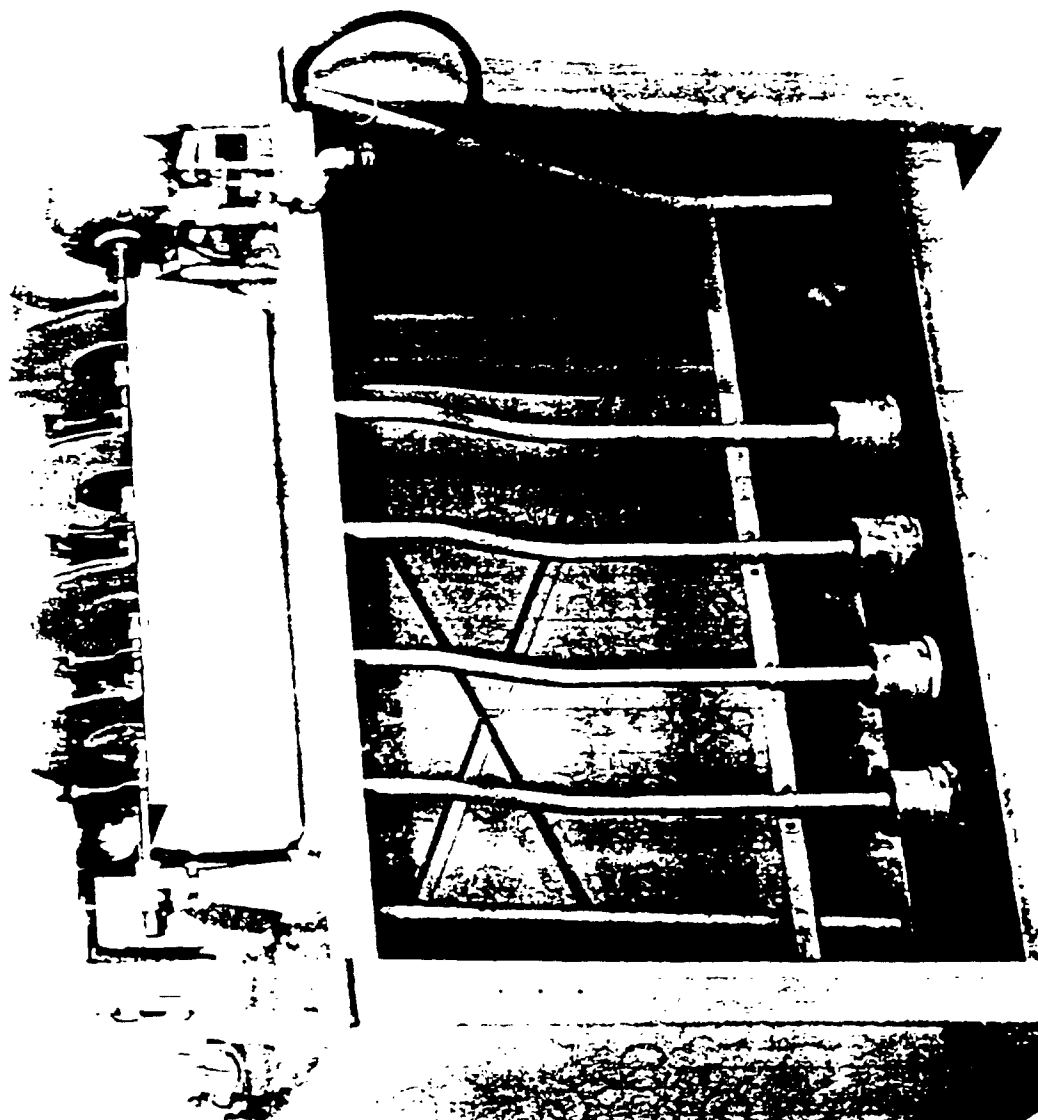


FIG. 1 - Clark Classifies
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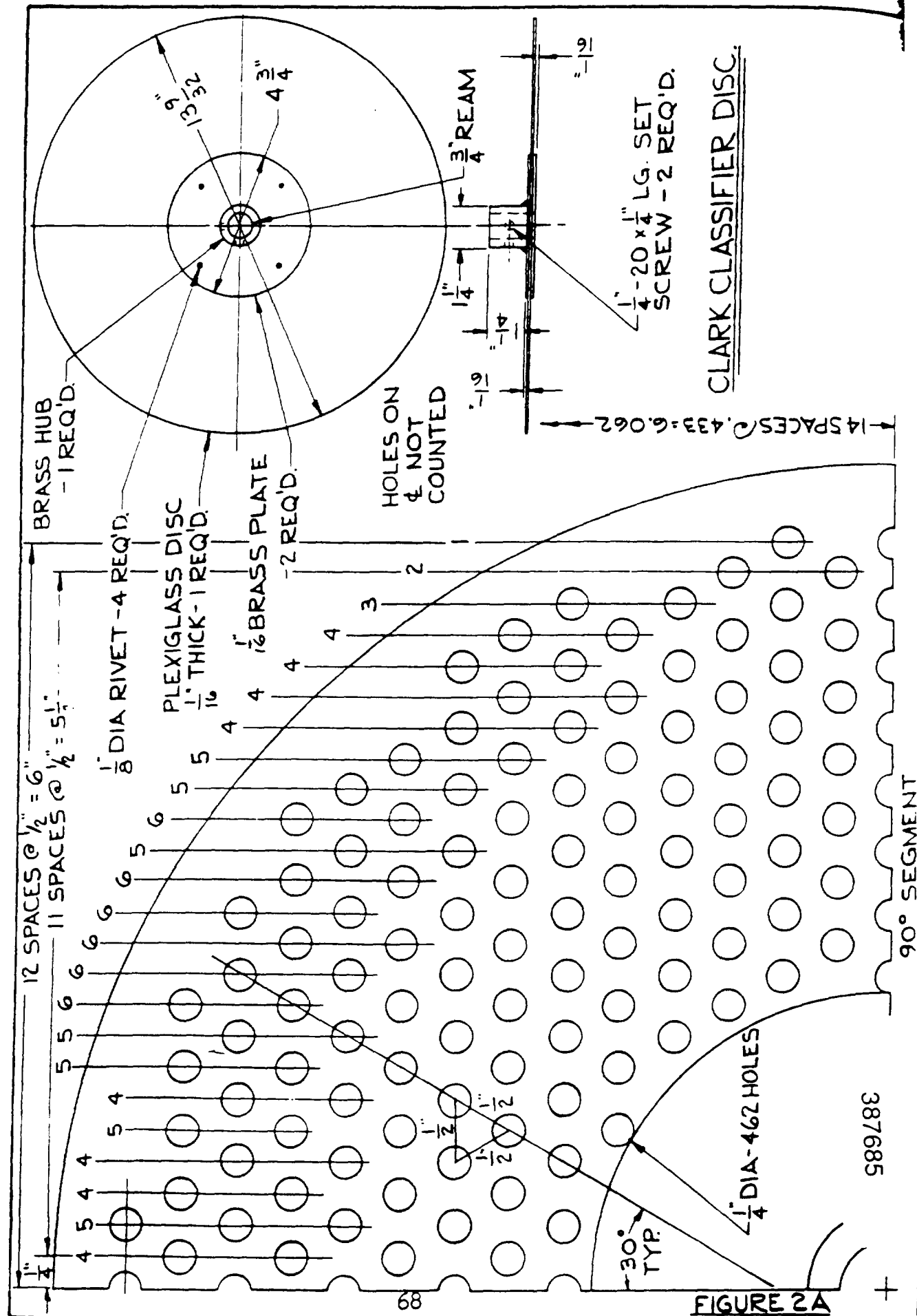
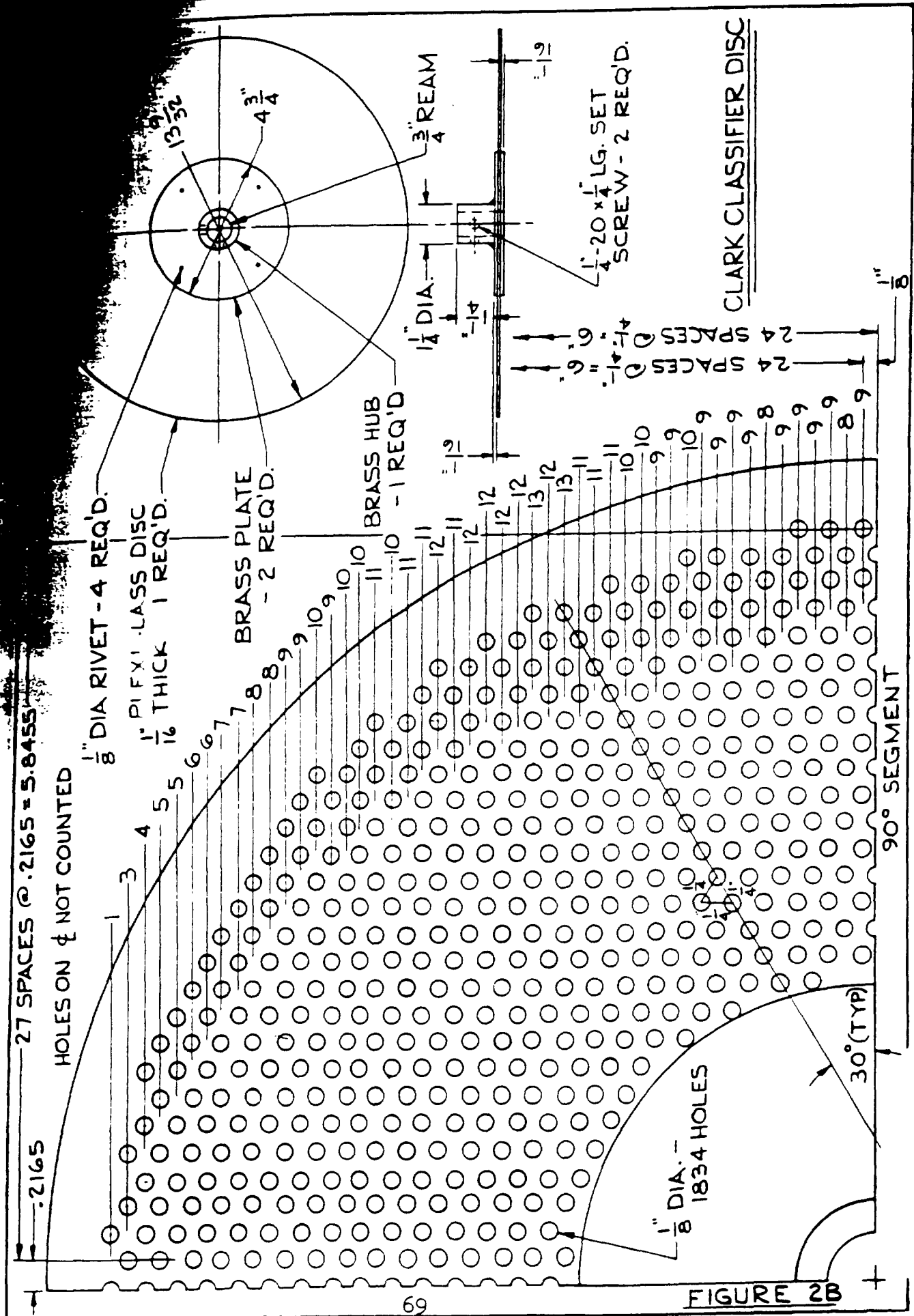


FIGURE 2A



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Wet Volume Test

Adopted by:

ACPA - 2/14/61
ATI -
QAMA - 9/13/60

WET VOLUME TEST

SCOPE

This test is designed to appraise all grades of asbestos fibre in terms of its buoyancy in water.

EQUIPMENT

1. Balance - capacity greater than 100 grams.
- accuracy ± 0.05 grams.

2. Glass Graduated Cylinders

Capacity	-	2000 ml
Subdivisions	-	20 ml
inside diameter	-	80 mm
wall thickness	-	3 mm
Graduate - total length	-	482.6 $\pm$ 1.6 mm
- from bottom of base to 2000 ml mark	-	398.0 $\pm$ 6 mm
Stopper	-	Type suitable for Mechanical Inversion

(Graduates - supplied by Scientific Glass Co.
Bloomfield, New Jersey)

<u>Fibre Grade</u>	<u>Sample Weight</u>	<u>Final Reading Time</u>
3-4-5 and 6	30 gms.	2 hrs.
7	50 gms.	4 hrs.

Type of Machine - Recommend mechanically driven inverter* capable of rotating 2000 ml cylinder 30 rpm's with the axis of rotation passing through the centre of the bottle.

SAMPLING

According to "Method of Sampling and Preparation of Asbestos Fibres for Test Purposes".

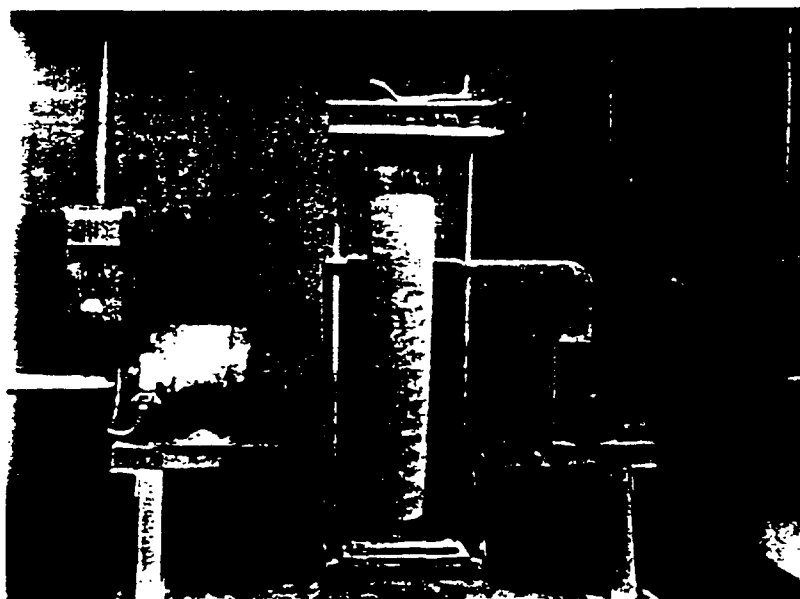
* Illustration of one type of mechanically driven inverter which has been found satisfactory, attached hereto.

PROCEDURE

1. Sample as per above and carefully weigh up the required fibre.
2. Add distilled or clean water at $77^{\circ} \pm 3^{\circ}\text{F}$ to the cylinder, filling to the 1000 ml mark. Place the required sample of asbestos in the cylinder and add distilled or clean water to the 2000 ml mark.
3. Place cylinder carefully in mechanically driven inverter and rotate 30 times in one minute. After the initial shaking cycle let stand for ten minutes and then repeat the shaking cycle making 30 complete inversions in one minute.
4. Place the cylinder on a vibration free table.
5. Record the wet volume of the fibre suspended in water in ml after two hours for groups 3, 4, 5 and 6 and four hours for group 7.

(A complete inversion consists of a rotation through 360° to original vertical position.)

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ST0513878

Mechanically Driven Inverter
for Wet Volume Test

ST0513879

DYCKERHOFF
AIR PERMEABILITY TEST
FOR ASBESTOS FIBRES

Adopted by

ACPA - 2/14/61
ATI - 3/ 2/61
QAMA - 9/13/60

DYCKERHOFF AIR PERMEABILITY TEST FOR ASBESTOS FIBRES

SCOPE

1. To measure the comparative degree of openness or fiberization of milled asbestos fibre by the air permeability method.

APPARATUS

2. (a) Dyckerhoff Automatic Air Permeability Tester, Blaine Design, Type LLD complete, consisting of built in electronically controlled stop watch, mechanical filling manometer, observation window in rear, inside lighting system, enclosed in a dust-proof, sheet metal case with improved cell design and special brass cell spacer. (See Appendix and Bulletin No. 7202 Chemisches Laboratorium fur Tonindustrie, Berlin, Germany for further details.)
- (b) Additional apparatus and materials required for performance of test.
 - (1) Balance (Sensitivity ± 0.1 gms).
 - (2) Filter paper supplied by manufacturer, or equivalent.
 - (3) Funnel, wide mouthed.
 - (4) Tamping tool (supplied with instrument).
 - (5) Manometer fluid.
- (c) Source of electrical power.

Note 1 - As equipment is German made, state power requirements when ordering; and consider replacement parts on account of delivery time involved.
- (d) Calibrating & Reference Standards
 - (1) Low: Silex Silica
 - (2) High: Kaolin

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Note 2 - Obtainable from the Industrial Minerals Sub-Division, Mineral Processing Division, Department of Mines and Technical Surveys, Ottawa, Ontario, Canada with instructions.

SAMPLE WEIGHT

3. Fifty (50) grams of fibre for all grades.

SAMPLE PREPARATION

4. (a) In accordance with the "Method of Sampling and Preparation of Asbestos Fibres for Test Purposes".
- (b) Derive testing specimen from laboratory sample, prepared in accordance with 4(a) above, in the following manner:
 - (1) Spread sample on a smooth working face by layers to form a flat pile of uniform thickness (approx. 1/2 inch) and quarter.
 - (2) Spread reduced sample again, as described in (b) (1) above, and select a 50-gram test specimen by taking pinches from different sections of the pile until a sample is obtained which will require minimum adjustment to the desired weight.

Note 3 - When pinches are taken be careful that each fraction contains the total cross-section of the pile from top to bottom at the point it is taken, including any grit or fines which may have segregated at the bottom.

- (3) Perform air permeability on two (2) distinct samples thus obtained from the original laboratory sample supplied by taking at least two (2) readings on each cell loading and average results (See "ACCURACY" for expected accuracy of test results).

PROCEDURE

5. (a) Check for Air Leaks: Before using or calibrating instrument check system for air leaks as follows:
 - (1) Seal off the air intake part where sample cylinder fits by coating the edges of the air

port with petroleum jelly and sliding a piece of glass over the intake port.

- (2) Apply vacuum to manometer through use of hand wheel.
- (3) Wait five minutes until the oil drains from the sides of the manometer, then observe levels of fluid. If it remains stationary there are no leaks. If it moves, examine the tubing and check-valve and correct any defects before proceeding further with test.

Note 4 - Minute leaks might exist in a system, however, without having a significant effect upon the air permeability value. Changes in the manometer level of less than 0.1" in ten minutes may be neglected. Erratic readings can be caused by fines collecting in the rubber check-valve. This valve must be cleaned regularly.

- (b) Calibration of Instrument: Verify calibration daily or as required with both low and high standard reference samples in the following manner.

- (1) Insert standard reference sample (low or high as desired).
- (2) Perform air permeability test by taking two separate readings and average results.
- (3) If average reading departs from standard reference by more than $\pm 2.0\%$ check system for leaks or other defects.
- (4) The liquid level in the "U" tube at zero pressure must be at the indicated etch mark on the tube. (See attached sketch).
- (5) The standard cell or sample holder supplied by the manufacturer with the Dyckerhoff apparatus shall be used.

- (c) Weigh out 50 ± 0.1 gram sample.

- (d) Place perforated plate in bottom of cell and filter paper on top of perforated plate.

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- (e) Divide 50-gram sample into four approximately equal parts. Pack fibre into cell one part at a time, keeping the bed level uniform, and compress after each addition with the tamping tool provided.

Note 5 - Any lumps or knots of matted fibre still remaining in sample should be disentangled before cell loading is commenced.

Note 6 - Care should be taken not to compress fibre beyond final plug length or required porosity of 70.0% assuming an average specific gravity of 2.55 for chrysotile asbestos.

- (f) When all fibre has been added, compress and place filter paper on top of the fibre plug.
- (g) Place brass spacer screen side down upon filter paper; insert plunger into sample cell and compress until plunger is seated flush with top of cell.
- (h) Fasten clips on plunger and cell to prevent fibre springback. Test samples under compression.
- (i) Turn the suction handwheel to the filling position to displace the liquid in the "U" tube leg. Then turn it to the measuring position and reset the clock to zero. This procedure starts the automatic process which will indicate on the clock the time required and then stop the test.
- (j) Record time to the nearest second after test is completed.

RESULTS & ACCURACY

- 6. (a) Take four air permeability readings as described under "PROCEDURE" on each fibre sample under test, and report average value.
- (b) The expected maximum difference between any individual reading and the average is $\pm 3.0\%$. When maximum difference is exceeded, repeat test by taking readings on a new cell loading.
- (c) Another means of measuring the degree of openness of fiberization of milled asbestos fibre is to use the various apparatus commonly employed for surface

area determinations by the air permeability method. The "effective" surface area thus obtained may be correlated with Dyckerhoff time readings.

Note 7 - A constant temperature and humidity atmosphere of 75°F and 50% RH is recommended for more uniform results.

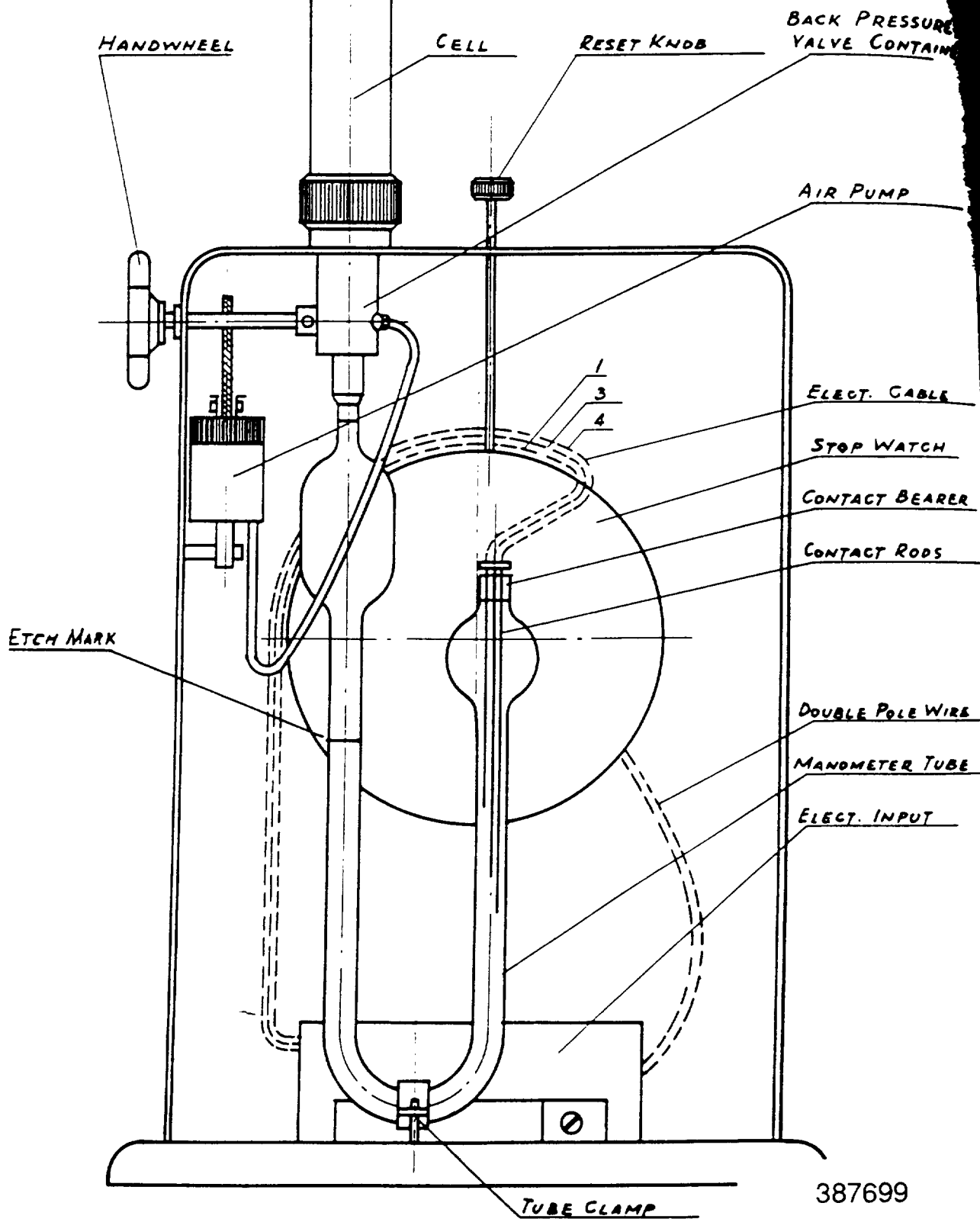
REFERENCES

1. Kozeny: Ger. Wien. Akad, 1927, Vol. 136a, p. 271.
2. Carman: Tran. Inst. Chem. Eng., 1937, Vol. 15, p. 150 and J.S.C.I., Vol. 56.
3. Lea and Nurse: J.S.C.I., 1939, Vol. 58, p. 277.
4. Rigden: J.S.C.I., 1943, Vol. 62, p. 1.
5. J. M. Dalla Valle, C. Orr, Jr. and R. R. Cornwall, "Limitations of the Arealometer Method for the Measurement of Fibre Diameters", Textile Research Journal, 1950, Vol. 20, pp. 676-82.

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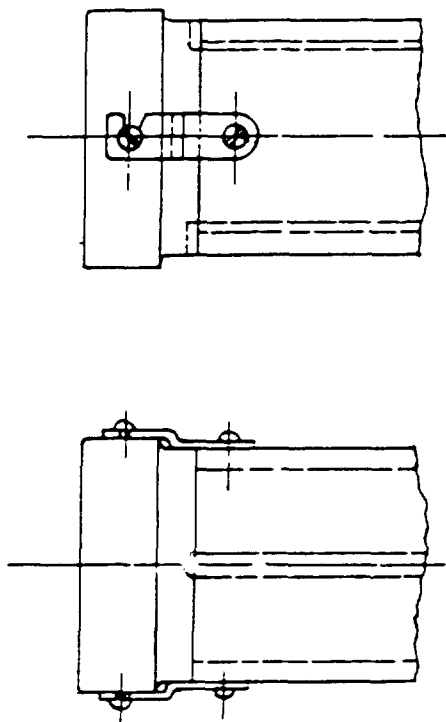
FIGURE 3



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DYCKERHOFF
SCHEMATIC DRAWING
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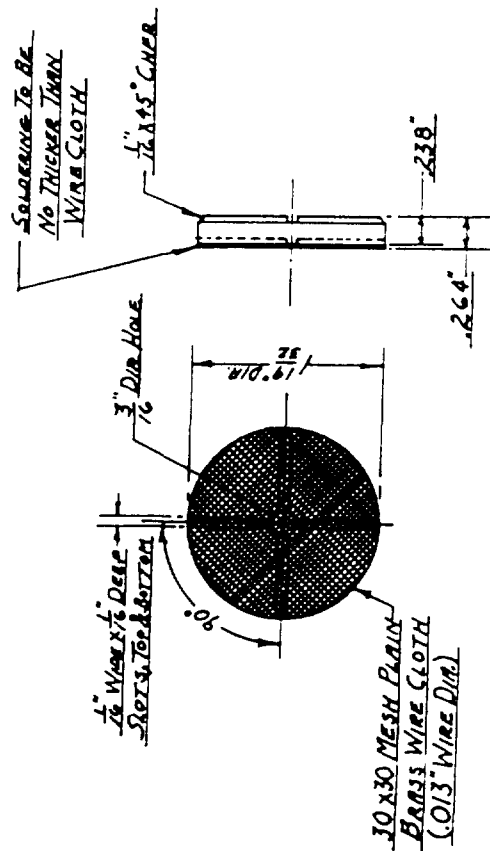
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DYCKERHOFF CELL CATCHES

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MATERIAL - BRASS

DYCKERTIOFF SPACER

Asbestos Fibre Strength Unit Test

Adopted by

ACPA - 2/14/61
ATI -
QAMA - 2/23/60

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ASBESTOS FIBRE STRENGTH UNIT TEST

Photographs illustrating the test equipment are given in Figures 1 and 2.

The following is a detailed description of the test procedure:

1. Fibre Preparation - Fibre being evaluated shall be tested in an open condition.

(a) Opening Procedure

- (1) Ball Milling Operation - Three hundred (300) grams of fibre are placed in a porcelain ball mill.

Successive use of several sets of balls is desirable in order to enable the operator to use the ball mill continuously. The balls are removed from the ball mill with the fibre and brushed or wiped clean of fibre while a duplicate set of balls is being used in the mill.

Fibre charge	300 grams
Ball charge	6 kg
Number of balls	80 (approx.)
Speed of rotation	65 rpm
Milling time	Samples shall be ball milled for that time which ultimately gives the highest strength to the asbestos-cement cakes. *

Balls are to be discarded and replaced when their diameter is 35 mm or less.

- (2) Disintegrator Operation - After ball milling the fibre is passed twice in succession through the small laboratory disintegrator, as specified.

* For chrysotile fibre from the Jeffrey and Thetford areas, 60 min. ball milling time has been found by testing to give the highest strength to the asbestos-cement cake.

The 300 grams of ball milled fibre are fed by hand through the disintegrator. The fibre sample should be fed into the disintegrator in about 1 1/2 to 2 min. at a reasonably steady rate so as to maintain a relatively constant disintegrator speed.

The following perforated steel plates should be used with the various fibre grades:

<u>Fibre Grade</u>	<u>Diameter of Holes in Perforated Plates</u>
3	10 mm
4	7 mm
5	5 mm
6	3 mm

After each disintegration, the apparatus should be cleaned. After the first disintegration, any bundles of fibre which have flocked together shall be separated by hand before the second disintegration. After the second disintegration the fibres which have flocked together and remain within the steel housing shall be removed and discarded.

- (3) Fibre Mixing - After the second disintegration the fibre shall be mixed in a tin mixing can. The mixing can is rotated on a roll table for 10 min. at a speed of 56 rpm.

2. Asbestos-Cement Sample Procedure

- (a) Equipment - The major items of equipment are given below:

- (1) Weighing - A scale or balance capable of weighing to the nearest gram is used for dry mix preparation. A scale capable of weighing to the nearest 0.1 gram is used to obtain volume and dry weights of individual asbestos-cement cakes after curing. The latter scale must be of a design to permit weight measurement of an individual cake when completely immersed in water.
- (2) Dry Mixing - Dry mixing of the asbestos and cement binder constituents is done in a special mixing can. For dry mixing, a roll table or equivalent

equipment for rotating the mixing cans at 78 rpm is required.

- (3) Wet Mixing - Wet mixing shall be with the mixer and cone conforming to specifications. The agitator shall rotate at 600 rpm in the axis of the cone. The agitator shall be placed as closely as possible to the bottom of the cone without touching it during rotation.
- (4) Pressing - The press must be capable of applying a pressure sufficient to obtain with 145 grams of dry stock (and water of hydration), a dry (cured cake) density of 1.60 gm/cm^3 . Testing experience has shown that a total gauge pressure of 34,000 lbs. between platens is required.

The press assembly is designed for pressing a cake to a constant thickness of 6 mm which produces a density of approximately 1.6 gm/cm^3 . This is done by using stop gauge bars to maintain a constant clearance between the bottom of the top die and the base of the confining mold during forming of the cake (dwell time). The press assembly is designed for clearance of less than 6 mm to allow for the effect of "springback" and to produce a final cured cake thickness of 6 mm (of the desired constant density).

The recommended press assembly consists basically of the following parts in the order of their vertical position from the top:

- (i) Device for Holding and Lowering the Confining Mold - This consists of a cylinder and piston (mounted on the upper cross-head of the press) with a yoke attached to the top of the piston. The side rods of the yoke project through the press cross-head connection to the confining mold. In addition, an L-shaped frame, pivoted centrally from the lower part of the cylinder, supports the piston and yoke during removal of the cake from the confining mold.
- (ii) Top Platen - This includes the top die mounted on springs against a supporting plate (above) and gauge bars surrounding the top die, rigidly fixed to the supporting plate.

- (iii) Confining Mold - The top die shall fit into the confining mold (during formation of each cake) with a clearance on four sides with a minimum of 0.051 mm to a maximum of 0.152 mm in order to allow seepage of surplus water during initial compression of the slurry.

The top inside edges of the confining mold are rounded to facilitate initial contact with the top die.

- (iv) 40-mesh copper wire screen.
- (v) 15-mesh brass wire screen.
- (vi) Perforated steel plate.
- (vii) Grooved bottom platen.
- (viii) Platen base.
- (5) Suction System - A suction system is used to provide a way to remove the water collected on the top of the mold during pressing. A thin tubular metal nozzle connected by rubber tubing to a receiving vessel (vacuum flask) is used to remove the water.
- (6) Moist Air Cabinet - A humidity cabinet for moist curing of cakes after forming.
- (7) Steam Cure Tank - An autoclave and controls required for curing cakes after moist cure.
- (8) Saturating Tank - A suitable tank for saturating one day's production of cakes is required.
- (9) Drying Oven.
- (10) A Testing Machine for transverse center loading of the samples to destruction is required.
- (11) Micrometer.
- (b) Raw Materials - It is important that a large quantity of Portland cement and finely ground silica be obtained in order to provide a constant source of these materials for fibre evaluation for a minimum period

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of one year. It is essential that the properties of these materials remain constant in order to avoid differential effect of these materials on the ultimate strength of the asbestos-cement samples, independent of the effect of fibre on strength.

- (1) Portland Cement.
- (2) Silica - (Finely ground quartz).
- (3) "Prepared" Water - The water used in the preparation of the samples shall be distilled water saturated with lime and gypsum (chemically pure). This water will be prepared by adding 2 grams of hydrated lime and 3 grams of gypsum ($\text{Ca SO}_4 \cdot 2 \text{ H}_2\text{O}$) per liter. It shall be allowed to stand for 24 hrs (agitating water from time to time), after which it is siphoned off into a clean bottle.

(c) Preliminary Steps

- (1) Preparation of the Perforated Plate and Screens - The screens and plate should be cleaned before the forming of each cake, using running water and a stiff bristle brush. The screens are then brush-coated with a very light machine oil (Socony-Vacuum 133B Process Oil or the equivalent; if this is not available, a mixture of 10 parts kerosene to 1 part raw linseed oil is satisfactory). The screens and plate are then placed under the confining mold on the bottom platen ready to receive the slurry.
- (2) Preparation of the Mold and Die - The mold and die should be brushed with the oil or linseed oil-kerosene mixture before the forming of each cake.

(d) Cake Formation - Evaluation of one fibre sample shall consist of forming and testing of ten 206 x 76.2 x 6 mm asbestos-cement cakes.

- (1) Batch Formulation - Quantity of dry mix (1700 grams) sufficient to prepare at least 11 test cakes* (145 grams per cake) is mixed according to a predetermined proportion based on empirical results and the chosen strength standard.

* This allows sufficient dry mix for forming one more cake than the required 10 in case of accidental damage to one cake during forming.

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To calculate the quantity of each material used in the mix for each cake (145 grams of dry mix), the following formulas are used:

$$F \text{ (fibre)} = \frac{145 \times K_A}{1000 + K_A} = \text{fibre weight in grams}$$

where K_A = quantity of fibre used in making sample, expressed in Kg of fibre/1000 Kg of binder

$$C \text{ (cement)} = 0.6 (145 - F) = \text{cement weight in grams}$$

$$S \text{ (silica)} = 0.4 (145 - F) = \text{silica weight in grams}$$

$$F + C + S = 145 \text{ grams}$$

- (2) Dry Mixing - The dry stock should be mixed for 5 min. in the specified mixing cans at 78 rpm. After mixing the stock is transferred from the mixing can to a jar or bag with as little vibration or agitation as possible so as to prevent any separation of constituents within the batch.

Prior to weighing the 145 grams of dry stock, the entire stock of 1700 grams is placed on a 3' x 3' polyethylene or similar sheet and rolled by taking opposite ends of the sheet using two rolls per end for a total of 4 rolls. Repeat this rolling technique before taking each 145 grams of stock.

- (3) Slurry Preparation - After rinsing the mixing cone a rubber stopper is inserted at the bottom and 410 cc of prepared water at 74F are poured into the cone. An additional 40 cc of the prepared water will be added to wash down the cone and stirrer after the stock has been added. 145 grams of dry stock are added and the mixer is started. The timing is continuous from this point, the time at which the mixer is started being zero.
- (4) Mixing - (Time 0 to 2 min.) The stirrer shall rotate at 600 rpm during the mixing.
- (5) Transfer of Slurry - (Time 2 to 2 3/4 min.) Initially the confining mold is resting on the

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screens on top of the bottom platen which is pulled forward on the platen base to facilitate transfer and distribution of the slurry. The yoke (and piston) for holding the confining mold is in the bottom position. At the end of the second min. (after mixing) the slurry is poured from the bottom of the mixing cone into the mold, using the chute. A rubber spatula and hoe are used to scrape the slurry remaining in the chute into the mold and to distribute the slurry evenly in the mold.

- (6) Dewatering of the Cake - (Time $2 \frac{3}{4}$ to $4 \frac{1}{2}$ min.) After removal of the chute and mixing assembly, the confining mold and the bottom platen together are pushed back beneath the top die so that the confining mold hooks contact the yoke rods between the set screw collars.

The cycle is started by opening the safety switch and closing the foot switch. The low pressure timer is activated. Timing is controlled by turning the low pressure relief valve handle counterclockwise to obtain minimum pressure. (Zero pressure reading on the gauge.) The valve handle should be given one additional turn after the zero reading is obtained. At this setting the top die will contact the slurry in from 9 to 10 seconds. In other words, the rate of rise is approximately 0.2 inches/sec (5mm/sec). When the top die is in contact with the slurry, water is forced out through the screens and between the mold and the die. Water pressed from the cake is drained from the platen base into a settling tank to prevent clogging of the laboratory drain. The water between the mold and the die is drawn off through the automatic vacuum system. As a result, the operation is now automatic until the end of the dwell time cycle and the operator can now make preparations for the next sample to be pressed.

- (7) Applying Forming Pressure - (Time $4 \frac{1}{2}$ to 5 min.) After the dewatering interval, Timer No. 1 automatically shuts off and Timer No. 2 starts building up to the required high pressure. This rate of pressure rise is controlled by adjusting the micrometer valve.

- (8) Dwell Time - (Time 5 to 6 min.) A total pressure of 34,000 lbs. (gauge) is automatically maintained by Timer No. 2 for one minute. This pressure is maintained by the value set on the high pressure relief valve.
- (9) Removing the Cake from the Confining Mold - At the end of the dwell time interval the motor automatically stops and the Waterman No. 2 valve opens permitting the ram to drop to the desired distance to remove the cake. This distance is controlled by closing the toggle switch. The top die is forced away from the supporting plate of the top platen by springs. At the same time since the confining mold is supported rigidly by the holding device, the top die passes through the confining mold, exposing the cake for removal. A flat asbestos-cement or Lucite plastic sheet approximately 9 x 22 cm is put on top of the screens on the bottom platen to receive the sample. Asbestos-cement sheets, if used, should be coated or partially impregnated with an alkali resistant moisture-proofing compound such as an epoxy resin. The maximum recommended clearance between the bottom of the cake and the top surface of the asbestos-cement sheet should be 1/4 in. in order to minimize the distance required for the cake to drop.

If the cake does not fall of its own accord, it is carefully stripped from the die by inserting a thin spatula between the cake and the die. The asbestos-cement or plastic sheet with the cake on top is removed from the press and the rough edges smoothed by the spatula. The cake is numbered for identification. Clean the screens and the perforated plates and place on the grooved platen for the next test. The confining mold is lowered onto the screens by releasing the holding device.

After formation of each cake, remnants of the slurry should be wiped from the confining mold and the die with emphasis on the grooves on the sides of the die in order to prevent clogging of the automatic vacuum system. The water removal system must be kept clean if reproducible results are to be obtained.

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The above procedure is repeated for each cake. At the end of each day's operation the equipment should be thoroughly cleaned and oiled.

(e) Cake Curing

(1) Moist Cure - Immediately after forming one set of ten the cakes on the flat asbestos-cement supports are stacked one on top of the other in groups of ten in the moist cure cabinet. The minimum period of moist cure is 16 hrs. This will increase somewhat, depending upon when the sample was made.

(2) Steam Cure - After removing the asbestos-cement sheets from between the cakes, the cakes are transferred from the moist cure cabinet to a flat rack on which they are stacked side by side between pegs in groups of five. It is important to lean each cake against the cake next to it, allowing a slight space between cakes at the bottom edge. The rack is placed in the autoclave with a large asbestos-cement sheet completely covering the rack to prevent condensed water from dripping on the cakes during cure. The autoclave is sealed and the steam pressure raised immediately to curing pressure.

Cakes made on the same day should be steam cured at the same time. The actual period of steam cure is 20 hrs. at 7.03 kg/cm^2 (100 psi).

(3) Saturation - After steam curing the samples shall be immersed in water for 24 hrs. at a temperature of approximately 75F. Cakes should be stacked on edge with the top edge approximately 25 mm or 1 in. below the water surface.

(f) Cake Testing - The order of sample testing shall be as follows:

- a. Determine volume by displacement of saturated sample.
- b. Determine saturated weight (in air).
- c. Determine saturated transverse strength.
- d. Determine specimen thickness.
- e. Determine dry specimen weight.

(1) Volume - The sample volume shall be determined

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by weighing each saturated cake completely immersed in 75F service water drawn fresh each day. The difference in weight between the saturated weight in air and the immersed weight equals the volume in cubic centimeters.

- (2) Saturated Weight - Cakes shall be removed from the water one at a time and immediately wiped free of excess surface water by means of a slightly wet cloth, the excess water of which has been driven off by wringing, and then weighed to 0.1 gram. The cakes are now covered with a damp cloth until the transverse testing is begun. Remove one cake at a time for test, keeping the others covered by the damp cloth.
- (3) Transverse Testing - Each saturated cake is then broken in flexure as a simply supported beam with the center load being applied across the entire width to the smooth (top) side. A 15.2 cm span shall be used and the loading rate shall average 36 kg/min.
- (4) Sample Dimensions
 - (i) Thickness shall be measured at three points across the width of the cake directly at the break. Individual thickness values shall be recorded to the nearest .01 mm.
 - (ii) Cake width shall be considered a constant value.
- (5) Drying - After the samples are broken they shall be dried for 24 hrs. at 220 to 230F.
- (6) Dry Weight - The dry sample weights shall be recorded to 0.1 gram.
- (g) Calculation - The following theoretical calculations shall be made for each individual cake tested.

(1) Dry Density

$$\frac{\text{Dry Cake weight in grams}}{\text{Cake volume in cc}} = \text{Density in gm/cm}^3$$

$$\text{Volume} = \text{Saturated Weight} - \text{Immersed Weight in Grams}$$

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(2) Transverse Strength

Modulus of Rupture

$$MR = \frac{3}{2} \frac{Pl}{b t^2}$$

P = breaking load in kg
 b = width in centimeters = 7.62 cm
 t = thickness at break in cm
 l = span = 15.2 cm

$$MR_T = \frac{(3) (P) 15.2}{(2) (7.62) (t^2)} = \frac{(3) P}{t^2} \text{ kg/cm}^2 \text{ (Equation \#1)}$$

Example:

For a cake with a 40 kg breaking load and a thickness of 0.600 cm

$$MR_T = (3) \frac{40}{0.36} = 333 \text{ kg/cm}^2$$

- (3) Correcting Modulus of Rupture (Unit Strength) to Common Density - To adjust all MR values to a common density and thus eliminate small differences in unit strength values due to differences in density, the following calculations should be performed. The common (standard) density of 1.60 gm/cm³ has been chosen as standard:

$$MR_A = \frac{(1.60)^2 (MR_T)}{(\text{Dry density of test cake})^2} \text{ (Equation \#2)}$$

where:

$$MR_A = \text{MR at } 1.60 \text{ gm/cm}^3$$

$$MR_T = \text{MR from test results}$$

Example:

For a dry cake density of 1.58 gm/cm³ and MR_T of 333 kg/cm²

$$MR_A = \frac{(1.60)^2}{(1.58)^2} (333) = 341 \text{ kg/cm}^2^*$$

* Since this strength exceeds the allowable range described in Section 5, it should be rerun at a lower fibre content.

Any individual value MR_A varying from the average by more than 7.5% of the average (including ten results) shall be eliminated and a new average strength calculated.**

- (4) Reference Strength Level - A standard reference strength for the asbestos-cement samples has been taken as 275 kg/cm^2 at a dry cake density of 1.60 kg/cm^3 .
- (5) Calculating Fibre Content to Give Standard Strength - The fibre content of the mix should be chosen so that the average MR_A lies somewhere between $261\text{--}288 \text{ kg/cm}^2$. If the MR_A exceeds this range, a new series of test specimens should be prepared at a higher or lower fibre content as required.

The MR_A value of the test sample will usually vary somewhat from the standard reference strength, and the fibre content of the mix must be altered slightly to attain the proper strength level. Experience has shown that this correction is most easily and accurately made by assuming a linear relationship between MR_A and the ratio of fibre weight: dry mix weight - fibre weight. Mathematically this may be expressed as follows:

$$\frac{(F_T)}{(145 - F_T)} \cdot \frac{(275)}{(MR_A)} = \frac{(F_A)}{(145 - F_A)} \quad (\text{Equation \#3})$$

where the total mix weight per cake is 145 grams and

F_T = weight of fibre in grams per cake as tested

F_A = adjusted fibre weight in mix so as to give standard strength

hence:

% fibre required in mix to give standard strength

$$= \frac{F_A}{145} \times 100$$

** If more than three such samples vary from the average of 7.5% the whole test will be repeated.

or from Equation #3

$$\% \text{ fibre required in mix} = \frac{(275 F_T) (100)}{MR_A (145 - F_T) + 275 F_T}$$

(Equation #4)

This may of course be expressed in terms of the % fibre present in the original mix as tested. For example, in these terms Equation #4 becomes

$$\% \text{ fibre required in mix} = \frac{(275 Q_f) (100)}{MR_A (1.00 - Q_f) + 275 Q_f}$$

(Equation #5)

where: Q_f = ratio of fibre weight to total dry mix weight

Either Equation #4 or Equation #5 may be used to calculate the fibre content required to give standard strength.

- (6) Calculation of Strength Units - Asbestos fibre possesses the ability to impart strength to an asbestos-cement product. That is to say, every fibre grade contains a certain quantity of strength giving units. The quantity of fibre required in an asbestos-cement furnish varies inversely with the number of strength units it possesses. For example, if X amount of a fibre possessing 100 strength produces a product of a given strength, 2X would be required to produce the same product from a fibre possessing only 50 strength units.

The following definition is the basis for the strength unit test:

A fibre that gives the standard strength at the standard density when used as 10% of the furnish is defined as having 100 strength units.

Therefore, by knowing the % fibre required in the mix to give standard strength, it is possible to calculate the strength units as follows:

$$\text{strength units} = \frac{1000}{\% \text{ fibre required in mix}} \quad (\text{Equation \#6})$$

(7) Typical Analysis of Data

Mix Composition

$$Q_f = .125$$

$$F_T = \text{fibre weight in grams/cake} = (145) (.125) = 18.1 \text{ g}$$

$$C_T = \text{cement weight in grams/cake} = (0.6) (145-18.1) = 76.1 \text{ g}$$

$$S_T = \text{silica weight in grams/cake} = (0.4) (145-18.1) = 50.8 \text{ g}$$

$$\text{Total weight/cake in grams} = 145.0$$

Average Strength Data

$$P = \text{breaking load} = 31 \text{ kg}$$

$$T = \text{thickness at break} = 0.600 \text{ cm}$$

$$\text{Density} = 1.58 \text{ gm/cm}^3$$

Calculations

$$MR_T = \frac{3P}{T^2} = \frac{(3)(31)}{(.6)^2} = 258 \text{ kg/cm}^2 \quad (\text{Equation \#1})$$

$$MR_A = \frac{(1.60)^2 (MR_T)}{\text{Density}} = \frac{(1.60)^2 (258)}{(1.58)^2} = 265 \text{ kg/cm}^2 \quad (\text{Equation \#2})$$

$$\begin{aligned} \% \text{ fibre required} &= \frac{(275)(F_T)(100)}{MR_A (145-F_T) + 275 F_T} \quad (\text{Equation \#4}) \\ &= \frac{(275) (18.1) (100)}{(265) (145-18.1) + (275)(18.1)} = \\ &= \frac{497,750}{33,600 + 4,980} \\ &= \frac{497,750}{38,580} = 12.9\% \end{aligned}$$

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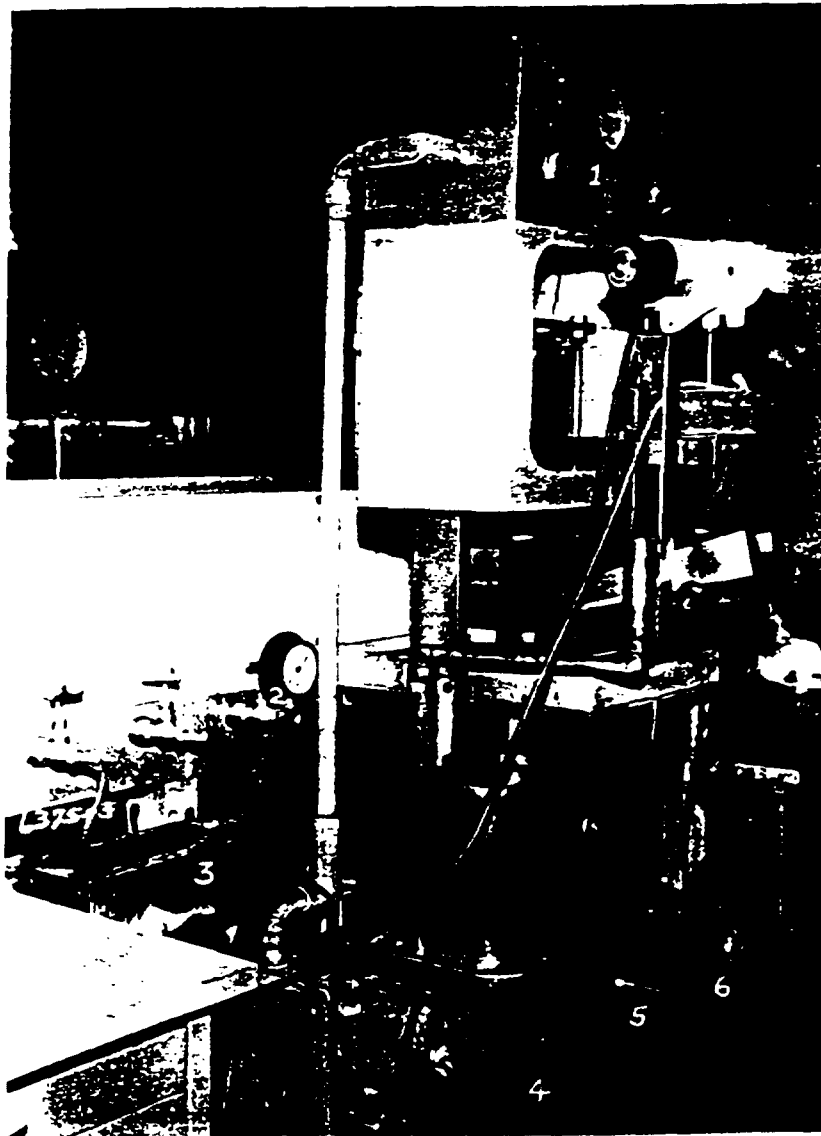
or using (Equation #5)

$$\begin{aligned}\% \text{ fibre required} &= \frac{(275) (.125) (100)}{(265) (1-.125) + (275) (.125)} \\ &= \frac{3437.5}{232.0 + 34.4} = \frac{3437.5}{266.4} = 12.9\%\end{aligned}$$

$$\text{Strength Units} = \frac{1000}{12.9} = 78 \quad (\text{Equation \#6})$$

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FIGURE 1
THE SEMI-AUTOMATIC PRESS

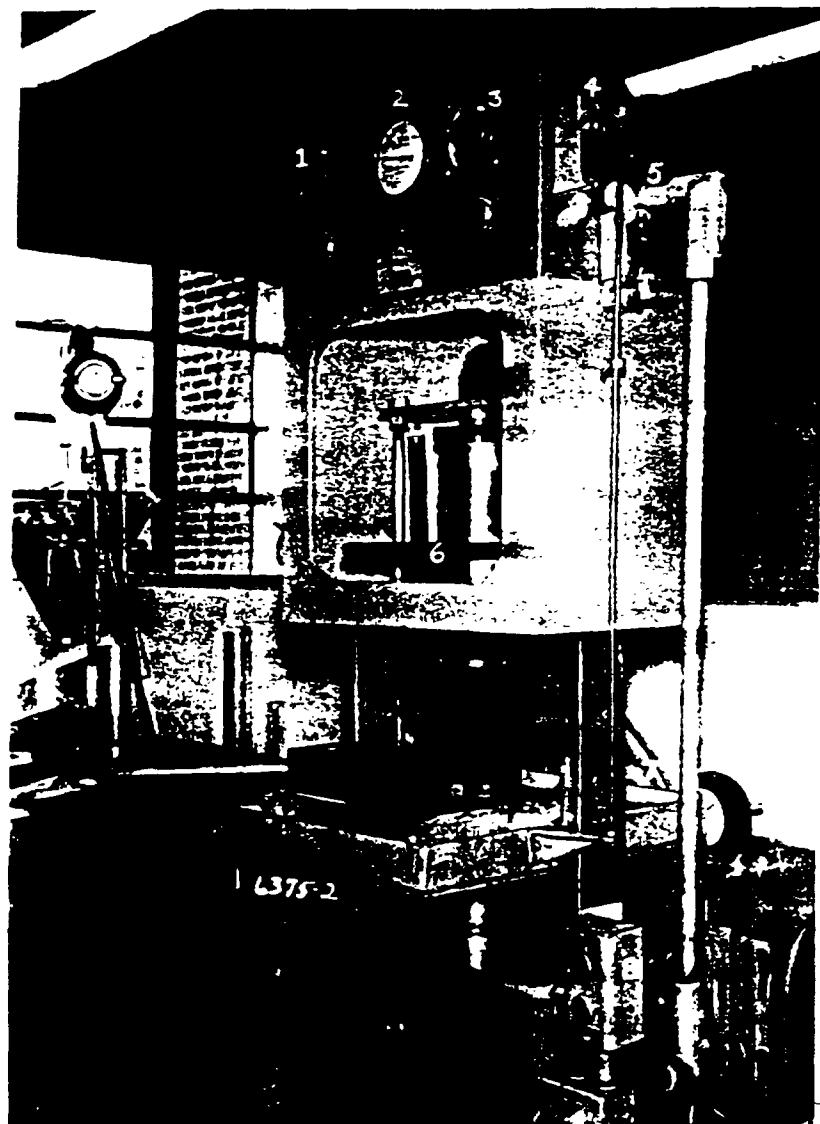


1. Toggle switch
2. Vacuum gauge
3. Settling tank
4. Low pressure control valve
5. Dump valve
6. High pressure control valve

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FIGURE 2

THE SEMI-AUTOMATIC PRESS



1. Low pressure timer (No. 1)
2. High pressure gauge
3. High pressure timer (No. 2)
4. Lower limit switch
5. Top limit switch
6. Confining mold drop lever
7. Vacuum line
8. Safety switch

(The foot switch is not shown)

Method of Test for
Tensile Strength of Asbestos Fibre

Adopted by

ACPA -
ATI - 9/10/59
QAMA -

ST0513906

METHOD OF TEST FOR
TENSILE STRENGTH OF ASBESTOS FIBRE

SCOPE

1. This method of test covers a procedure for determining the tensile strength of asbestos fibre, expressed in grams per denier, where a denier is defined as the weight in grams per 9000 meters of length.

APPARATUS

2. (a) The apparatus shall consist specifically of the parts described in paragraphs (b) to (i).
- (b) Tensile Tester - The tensile tester shall be a vertical pendulum type with capacities of zero to ten pounds and zero to 20 pounds and shall have a constant rate of traverse of 12 inches per minute.
- (c) Balance - The balance shall be capable of weighing ten milligrams to the closest 0.01 milligram.
- (d) Mounting Cards - The mounting cards shall be constructed of cardboard approximately 0.01 inch thick and shall conform to the design shown in Figure 1.
- (e) Cutter - The cutter shall consist of two razor blades mounted 1.00 plus or minus 0.02 centimeter apart.
- (f) Scale - The scale shall be at least ten centimeters in length and shall be graduated to 0.02 centimeters or less.
- (g) Cement - Epoxy resin. Mix two parts by weight of Araldite 502 resin with three parts by weight of ASP 400 clay. Blend 25 parts of this mix with one part by weight of HN 951 hardener and sufficient ASP 400 clay to give a high consistency.

NOTE: - The Araldite 502 resin and the HN 951 hardener are supplied by CIBA Products Company, Kimberton, Pa.; the ASP 400 clay is supplied by Minerals and Chemicals Company, Menlo Park, New Jersey.

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(h) Tweezers.

(i) Dissecting Needle.

SAMPLING OF ASBESTOS FIBRES

3. The sampling shall be conducted in accordance with the Standard Sampling and Preparation Procedure for Asbestos Fibre.

PROCEDURE

4. (a) Size of Sample - The weight of sample used for this test shall be approximately 100 grams.
- (b) Selection of Fiber Bundles for Test - The 100 gram sample shall be divided into eight equal portions and five fibre bundles shall be selected from each portion.

NOTE 1: - In the evaluation of crudes preferably ten to 20 pieces of crude shall be selected and an equal number of fibre bundles shall be removed from each piece so that a total of 40 bundles are obtained.

NOTE 2: - The fibre bundles shall be continuous and free of faults, as determined by visual inspection, and shall have a length of at least 1.5 centimeters and a weight of at least one milligram.

- (c) Preparation of Fibre Bundles for Test - Each of the 40 fibre bundles shall be cut to a length of 1.00 plus or minus 0.02 centimeter and shall be dissected until the weight is 0.50 plus or minus 0.05 milligram. The length of each specimen shall be checked with a scale and the weight of each specimen shall be recorded to the closest 0.01 milligram.

Each specimen shall be placed on a mounting card so that it lies across the center of the slit perpendicular to the long dimension and shall be attached at the ends with cement. The cement shall be allowed to cure for a minimum of 16 hours at room conditions not in excess of 80 degrees Fahrenheit.

NOTE 3: - The resin cure can be accelerated by heating, but the resultant wicking action causes the fibre specimens to become brittle.

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- (d) Tensile Strength Test - Each mounted specimen shall be placed between the grips of the tensile tester and the mounting card cut through to the slit on both sides. The tensile tester shall then be set in operation and the breaking load recorded to the closest 0.1 pound.

CALCULATION AND REPORT

5. (a) The tensile strength of each of the 40 specimens shall be calculated to the closest 0.1 gram per denier using the following equation.

$$\text{Tensile Strength} = (\text{load}) / (1.98) (\text{weight})$$

where

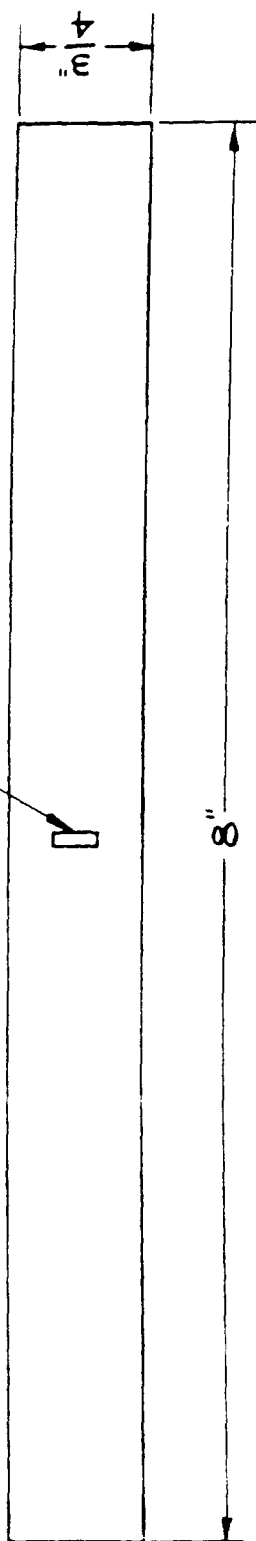
load = breaking load of specimen in pounds
weight = weight per unit length of specimen
in milligrams per centimeter.

- (b) Report the average tensile strength of the 40 specimens to the closest 0.1 gram per denier.

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SLIT (0.25" x 0.05")



CARDBOARD MOUNT FOR TENSILE STRENGTH

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Asbestos Fibre Evaluation
Relative Strength in A/C Products

Adopted by:

ACPA - 9/14/60

ATI -

QAMA -

ASBESTOS FIBRE EVALUATION
RELATIVE STRENGTH IN A/C PRODUCTS

SCOPE

To determine the reinforcing value of asbestos fibres in asbestos cement relative to a fibre chosen as "standard".

APPARATUS

1. Mixing Equipment: Patterson-Kelley Model LB-558 laboratory blender complete with two one-quart and one two-quart plastic shells.
2. Vacuum System: As detailed in Figure No. 1.
3. Vacuum Pump: "Cenco Pressovac 4" or equivalent. An air aspirator (as detailed in Figure No. 2), connected to a compressed air line through a pressure reducing valve, may be used in place of the vacuum pump.
4. Mold Assembly: As detailed in Figure No. 3.
5. Hydraulic Press: 10-ton Carver, laboratory model.
6. Humidity Cabinet: Designed for saturated atmosphere at ambient temperature. (A Harshaw No. H-18877 Stainless Steel desiccating cabinet has been found satisfactory for this purpose.)
7. Laboratory Autoclave: Capable of maintaining a saturated steam pressure of 125 pounds per square inch gauge.
8. Drying Oven: Standard gravity convection type, capable of maintaining 220 degrees Fahrenheit plus or minus 5 degrees.
9. Flexural Tester: With five inch span and capable of close readings in the zero to 300 pound range. (A 300 pound proving ring assembly with continuous reading dial, used in conjunction with a Carver press, has been found preferable to other testing devices.)
10. Laboratory Balance: Capable of weighing 500 grams to 0.1 gram.

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11. Graduated Cylinder: 1000 ml capacity.
12. Filter Paper: 15 cm circular, Whatman No. 40 and Whatman No. 50.
13. Beaker: 5500 ml, stainless steel.
14. Plexiglas Squares: 7 inches x 1/8 inch thick.
15. Glass Jars: wide mouth, screw cap, 32 ounce capacity.
16. Spatula: Stainless Steel, with narrow blade.
17. Thermometer: Range minus 4 to plus 220°F in two degrees.
18. Stop Watch.

RAW MATERIALS

1. Obtain 1000 pounds each of Portland cement and 90-200 mesh pulverized silica. These materials, which will serve as standards, should be thoroughly mixed and stored in air-tight containers.
2. Obtain 250 pounds of asbestos fibre (preferably an asbestos-cement 3-grade) and prepare in an appropriate manner. This fibre, which will serve as the standard, should be thoroughly mixed before and after processing and should be stored in a dry place in clean cloth sacks.
3. For each fibre to be evaluated, obtain the sample by the "Method of Sampling and Preparation of Asbestos Fibres for Test Purposes".

SAMPLE PREPARATION

1. For each evaluation, premix 1290 grams of cement and 860 grams of silica in the two-quart shell by rotating in the blender for 15 minutes. This cement-silica mixture, which will be sufficient for a 15 sample evaluation, should be stored in air-tight containers until needed.
2. Prepare three cakes each of the following compositions using the standard fibre (weigh to the closest 0.1 gram):
 - 3% Fibre - 4.5 grams fibre, 145.5 grams cement-silica mix
 - 9% Fibre - 13.5 grams fibre, 136.5 grams cement-silica mix
 - 15% Fibre - 22.5 grams fibre, 127.5 grams cement-silica mix

Also prepare six cakes of the following composition using the fibre to be evaluated (weigh to the closest 0.1 gram):

15% Fibre - 22.5 grams fibre, 127.5 grams cement-silica mix

The order of making should be staggered by preparing one cake each of 3%, 0% and 15% standard fibre followed by two cakes of the unknown, with this order being repeated until a total of 15 samples has been prepared.

3. Using the one-quart shells, dry mix the materials for each cake by rotating in the blender for five minutes and then add to 600 ml of tap water (at $77^{\circ}\text{F} \pm 3^{\circ}$) and wet mix for an additional five minute period. (It is found desirable to reserve one of the shells for dry mixing only, using the other for the wet mixing operation. Further, as a matter of convenience, the materials for one sample can be wet mixed while those for the next are being dry mixed.) Be certain that both shells are thoroughly cleaned after each use.
4. During the mixing operation, fit the Buchner funnel with a sheet of whatman No. 40 filter paper, wetting the paper to position it firmly. Then level the funnel by eye.
5. With the stopcock between the filter flask and vacuum source closed, adjust the vacuum to 16 inches of mercury.
6. When mixing is complete, swirl the shell vigorously to maintain a uniform slurry concentration and pour the material into the Buchner funnel.
7. When all of the slurry has been transferred, apply the vacuum noting the time. (A stop watch is recommended for all time schedules.)
8. Rock the funnel continually during filtration to distribute the fibre.
9. As soon as the water has been drawn off (as indicated by a decrease in vacuum) again note the time. The number of seconds from vacuum application to this point is recorded as the filtering time.
10. Continue vacuum application for 30 seconds to draw off any water remaining in the bottom of the Buchner.

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11. Remove the funnel from the flask, place a sheet of Whatman No. 50 filter paper on the cake, invert the funnel and remove the cake onto a tared Plexiglas square.
12. Remove the Whatman No. 40 from the cake bottom and replace it with a sheet of No. 50.
13. Then weigh the cake (to the closest 0.1 gram) on a balance tared for the Plexiglas and the two sheets of No. 50 filter paper.
14. Carefully place the mold over the cake, then invert so that the cake falls into the mold. This places the original bottom of the sample on the bottom of the mold with a sheet of Whatman No. 50 filter paper on each side.
15. Insert the plug into the mold, invert so that the plug is on the bottom, and place the assembly in the press. Apply pressure at a uniform rate such that a total load of 14,150 pounds is obtained over a 30-second period.
16. Allow the cake to compact for 30 seconds and then release the pressure.
17. Remove the mold from the press. Then remove the plug from the mold and run the spatula around the edge of the cake to free it.
18. Invert the mold on a Plexiglas square to remove the cake, peel off the upper filter paper, place the tared Plexiglas on top, invert, and peel off the bottom filter paper.
19. With the spatula smooth outwards any extruded flash from the pressing operation.
20. Then weigh the cake (to the closest 0.1 gram) on a balance tared for the Plexiglas.

CURING CYCLE

1. When the series of cakes is finished, store the stack in the humidity cabinet with a Plexiglas square on the bottom and top and between each sample.
2. After 24 hours, remove the cakes from the humidity cabinet and autoclave for 20 hours using saturated steam at 125 lbs. per square inch gauge. (Relative to autoclav-

ing, the system should be purged of air prior to the start of the steaming cycle and the charging and blow-down operations should be performed slowly over approximately 15 minute periods.

3. Finally rack the cakes for free air circulation and dry in the gravity convection oven at $220^{\circ}\text{F} \pm 5^{\circ}$ for 24 hours. Then obtain the dry weights to the closest 0.1 gram.

TESTING PROCEDURE

Test each cake in flexure on a five inch span. After the first break, place the two halves together, rotate the sample 90 degrees, and retest. Tests should be performed as soon as possible after removal from the oven so as to eliminate the effect of excessive moisture absorption.

CALCULATIONS

1. Record the average filtering time in seconds for the fibre being evaluated.
2. Calculate the per cent water remaining after filtration and after pressing for the unknown fibre (to the closest 0.1 per cent) as follows:

$$\begin{aligned} \text{Per cent water after filtration} &= \\ &(\text{Average filtered weight}) - 150.0 (100)/(150.0) \end{aligned}$$

$$\begin{aligned} \text{Per cent water after pressing} &= \\ &(\text{Average pressed weight} - 150.0) (100)/(150.0) \end{aligned}$$

3. Obtain the average breaking load in pounds for the unknown fibre and the 3%, 9% and 15% standard cakes.
4. Using a relative strength of 20 for 3%, 60 for 9% and 100 for 15%, calculate the least squares line of breaking load versus relative strength for the standard fibre, as illustrated in the following example:

Assume that the average breaking loads for the 3%, 9% and 15% standard cakes are 90.0, 171.0 and 240.0 pounds respectively. The following table is then set up, where X is the relative strength and Y is the corresponding breaking load:

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ST0513917

<u>X</u>	<u>Y</u>	<u>(X)(Y)</u>	<u>(X)²</u>
20	90.0	1,800.0	400
60	171.0	10,260.0	3,600
<u>100</u>	<u>240.0</u>	<u>24,000.0</u>	<u>10,000</u>
Total 180	501.0	36,060.0	14,000

The average X and Y values are then calculated as:

$$M_x = 180/3 = 60$$

$$M_y = 501.0/3 = 167.0$$

The slope of the least squares line is next calculated as:

$$m = \frac{36,060.0 - (60)(501.0)}{14,000 - (60)(180)} = \frac{36,060 - 30,060}{14,000 - 10,800} = \frac{6000}{3200} = 1.875$$

The Y-intercept of the least squares line is finally calculated as:

$$b = 167.0 - (1.875)(60) = 167.0 - 112.5 = 54.5$$

The equation for the least squares line of breaking load versus relative strength for the standard fibre is then:

$$Y = 1.875 x + 54.5$$

5. From the equation for the standard, calculate the relative strength of the unknown fibre (to the closest 0.1) from its average breaking load, as illustrated in the following example:

For an unknown fibre having an average breaking load of 210.0 pounds, the relative strength would be:

$$X = (210.0 - 54.5)/(1.875) = 155.5/1.875 = 82.9$$

RESULTS

1. **Filtering Time:** This time will vary with both the type of fibre used and the degree to which the fibre has been prepared. Since the end of filtration is taken as the point at which a decrease in vacuum occurs (caused by loss of the water seal around the cake edge), and is not based on the time required to remove a specified amount of water, it is dependent not only on the rate at which water is removed from the A/C slurry, but also on the water retention properties of the fibre used. Conse-

quently, without the per cent water remaining after filtration, the filtering time has no definite meaning.

2. **Filtered Weight:** The filtered weight, in terms of the per cent water held after filtration, indicates the water retention properties of the fibre used and also varies with the fibre type and the degree of preparation. In conjunction with the filtering time, it has special application in the formulation of wet machine furnishes since, in wet machine operation, the water content of the asbestos-cement film on the felt is controlled by passing the film over a vacuum box.
3. **Pressed Weight:** The pressed weight, in terms of the per cent water held after pressing, is an indication of the resistance to water removal under pressure. The value is a guide as to the moisture content of the rolled product in wet machine operation and as to the moisture content of pressed sheets.
4. **Relative Strength:** The relative strength, which expresses the reinforcing characteristics of the fibre relative to the standard fibre as 100, is used in effectiveness of furnish calculations to control the strength of asbestos-cement products.

Use of relative strength values in calculating the effectiveness of furnish can best be illustrated by the following example:

The relative strength of a blend containing 80 pounds of Fibre A (relative strength 100), 50 pounds of Fibre B (relative strength 72), and 20 pounds of Fibre C (relative strength 32) is:

$$\frac{(80)(100) + (50)(72) + (20)(32)}{(150)} = 81.6$$

In a furnish containing 12.0 per cent of this blend, the effectiveness of furnish would be:

$$(81.6)(12.0) = 979$$

It should be emphasized that no general correlation exists between the effectiveness of furnish value and the various A/C processes. However, within any one process there is a definite relationship between the furnish calculation and product strength.

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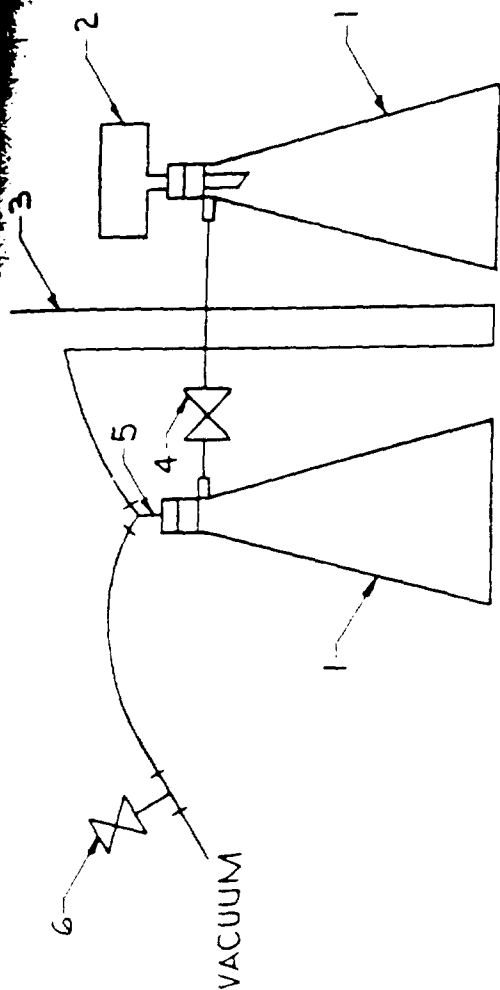
APPENDIX

SAMPLE PREPARATION

One, two or three unknown fibres can be evaluated against the same standard within any one run, the only limiting factors being time and the capacity of the two-quart shell. If more than one fibre is evaluated, it is not possible to premix the cement and silica together, but each must be premixed separately and weighed separately. For a three fibre evaluation, consisting of 27 cakes, premix 2200 grams of cement and 1500 grams of silica and weigh out as follows:

3% Fibre Cakes - 87.3 grams cement, 58.2 grams silica
9% Fibre Cakes - 81.9 grams cement, 54.6 grams silica
15% Fibre Cakes - 76.5 grams cement, 51.0 grams silica

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VACUUM SYSTEM

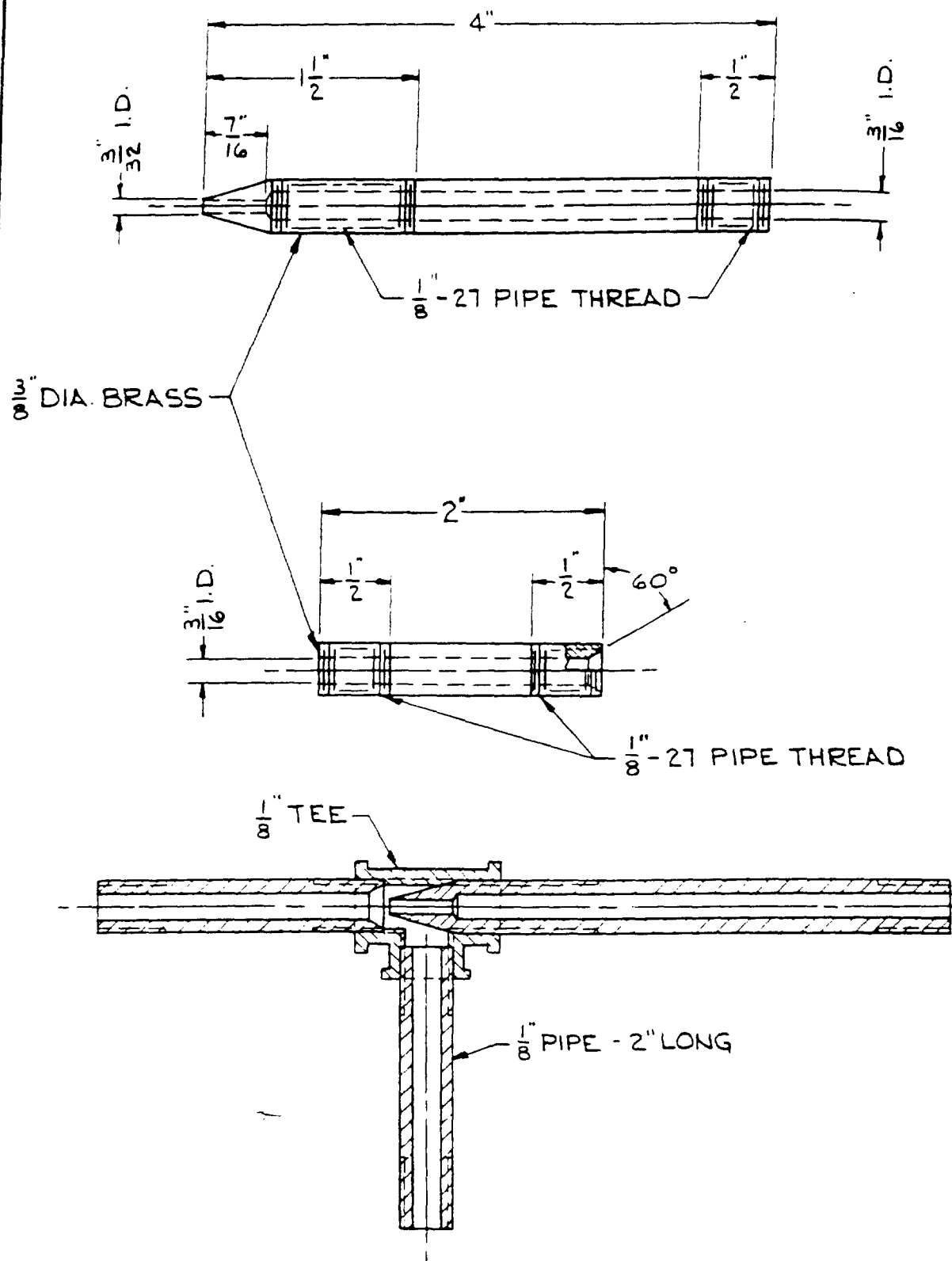
- (1) FILTER FLASK (2000 ML.)
- (2) BUCHNER FUNNEL (51 MM. I.D.)
- (3) MERCURY MANOMETER
- (4) BRASS STOPCOCK ($\frac{1}{4}$ " BORE)
- (5) BRASS Y-TUBE ($\frac{5}{16}$ " BORE)
- (6) NEEDLE VALVE ($\frac{1}{4}$ " I.P.S.)
- RUBBER TUBING ($\frac{1}{4}$ " I.D. VACUUM TYPE)

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FIGURE 1

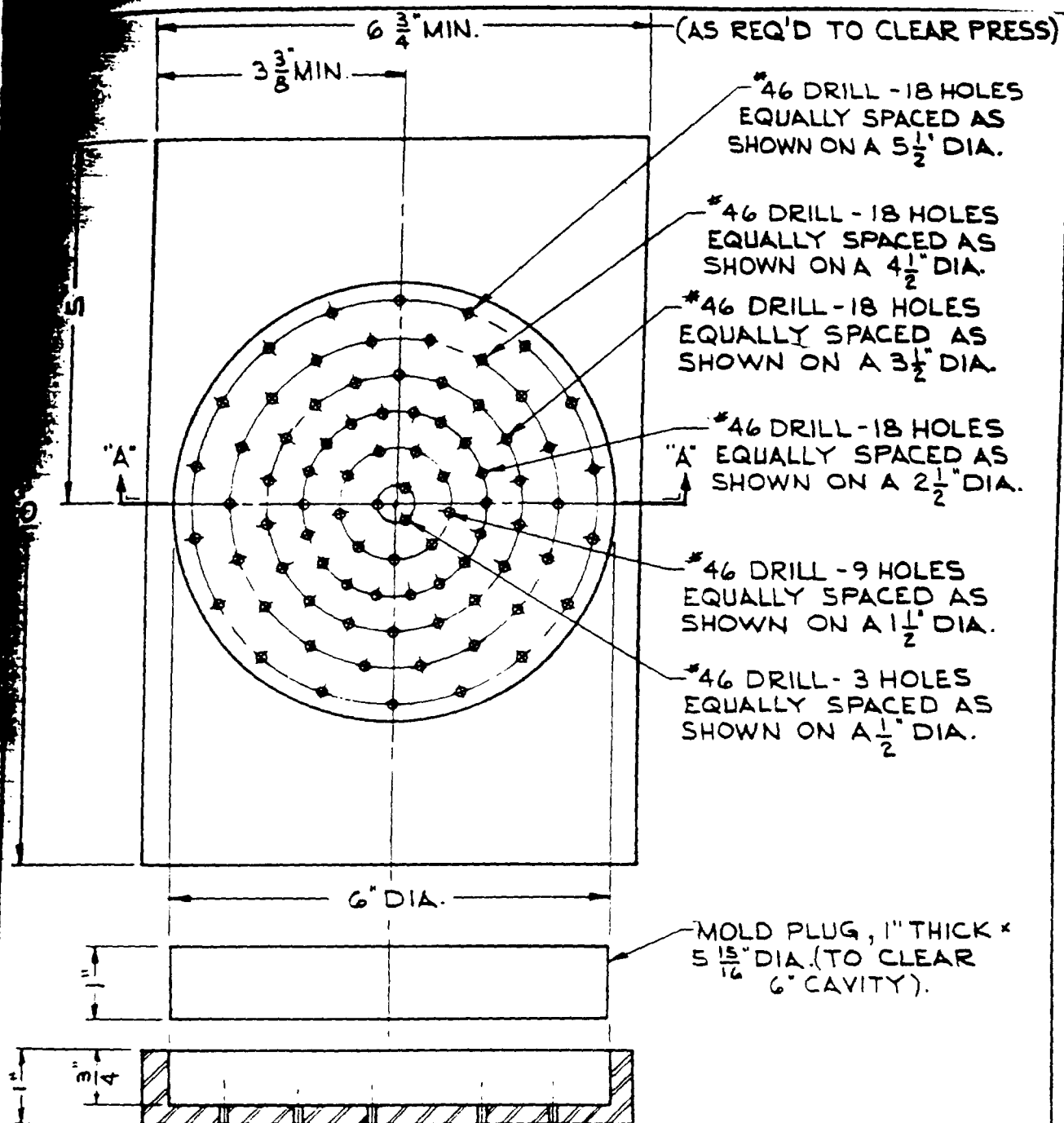
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VACUUM ASPIRATOR ASSEMBLY

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CONSTRUCTION NOTES:

1. MOLD MAY BE MADE CIRCULAR (6 $\frac{3}{4}$ " O.D) INSTEAD OF RECTANGULAR.
2. DRILL & TAP CENTER OF PLUG TO DEPTH OF $\frac{1}{2}$ " FOR $\frac{1}{2}$ " - 13 BOLT.
3. MATERIAL - COLD ROLLED STEEL.

PRESS MOLD

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FIGURE 3

ST0513923

CRUDY CONTENT DETERMINATION
FOR
GRADES 4D AND SHORTER

Adopted by:

QAMA - 6/20/61
ATI -
ACPA -

CRUDY CONTENT TEST

SCOPE

1. (a) This test is to measure by water elutriation a residue of material that indicates the crudiness and/or grit content in milled fibre.
- (b) The limitation of this test is for Q.S. grades of 4D to 7F inclusive.

APPARATUS

2. (a) The apparatus as described by the drawings No. A and B.
- (b) Stop watch.
- (c) 1000 cc beaker.
- (d) Waring blender Model No. 702 CR-W.
- (e) A balance to weigh 10 grams to an accuracy of ± 0.01 gm.
- (f) A Buchner funnel and filter or 200 mesh Tyler sieve.
- (g) Drying oven or infra-red lamp units.

PROCEDURE

3. (a) Sample Preparation - In accordance with the Method of Sampling and Preparing Asbestos Fibre for Testing a laboratory sample will be taken.

The test specimen will be selected from the above-mentioned sample as follows:

- (1) Spread the sample on a smooth working surface by layers to form a flat pile approximately $1/2$ " thick and quarter.
- (2) Spread the reduced sample again and select 10 grams by taking pinches from different sections

in the pile until the specimen is obtained which will require minimum adjustment to obtain the desired weight (10 grams $\pm$ 0.01 gms).

Note: Special attention must be made to ensure that each pinch of fibre picks up the full cross-section of the pile, including any grit or fines which may have segregated to the bottom.

- (3) Two determinations of crudy content must be made on distinct samples thus obtained from the laboratory sample. The two results should then be averaged. (See Accuracy).

- (b) Fill the tube to half its height with water.
- (c) Add the sample to the column from the top as a slurry in 200-400 cc of water.
- (d) Bubble air through the sample for one minute to give thorough mixing.

Note: Ensure that no loss of sample takes place.

- (e) Reduce the air rate to approximately 50 cc/min. and turn on the water to a 3500 cc per minute rate.
- (f) After nine minutes turn off the air bubbles completely and increase the water rate to 5400 cc per minute.
- (g) After five minutes shut off the water.
- (h) Pinch the inlet tubing, remove from the water air-line, and drain the collected crude into the beaker. The first two liters of water usually contain all the crude and the remainder of the water may be discarded.
- (i) Wash down the lower end of the tube by squirting water into the inlet tube for a moment and add this to the beaker.
- (j) Filter, dry and weigh the crude and grit.
- (k) If it is so desired, the grit content may be measured as follows:

Place the weighed material in a Waring blender with about 250 cc of water and beat for one minute until

ST0513925

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the crude is pulpy and well fiberized.

Wash the material into a large beaker of warm (but not boiled) water, stir well and decant the fibre away from the grit.

Filter, dry and weigh the grit (it may be necessary to hand-pick and discard some unfiberized crude).

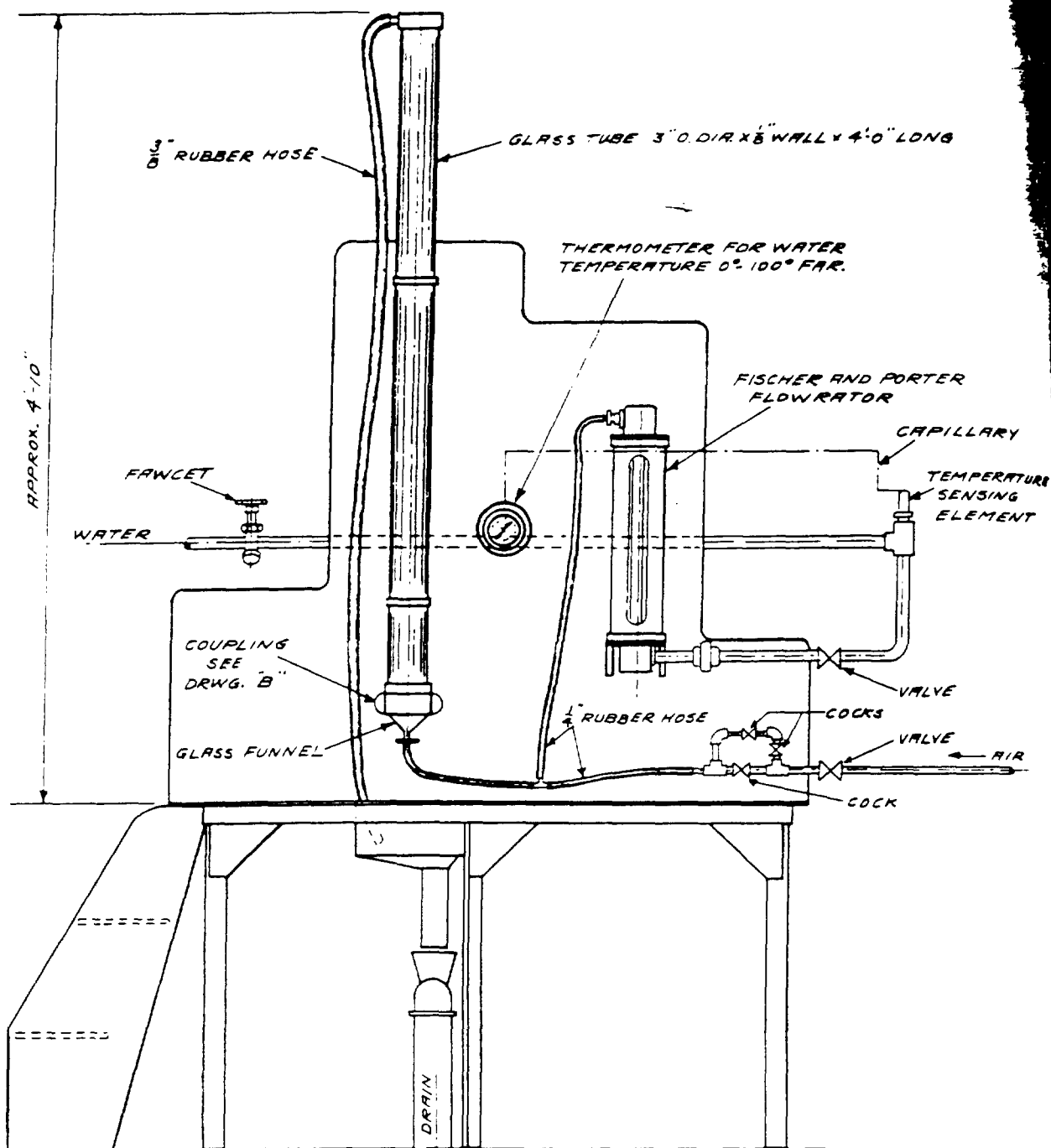
Subtract from the previous total Paragraph 3 (j) to obtain per cent crude.

RESULTS & ACCURACY

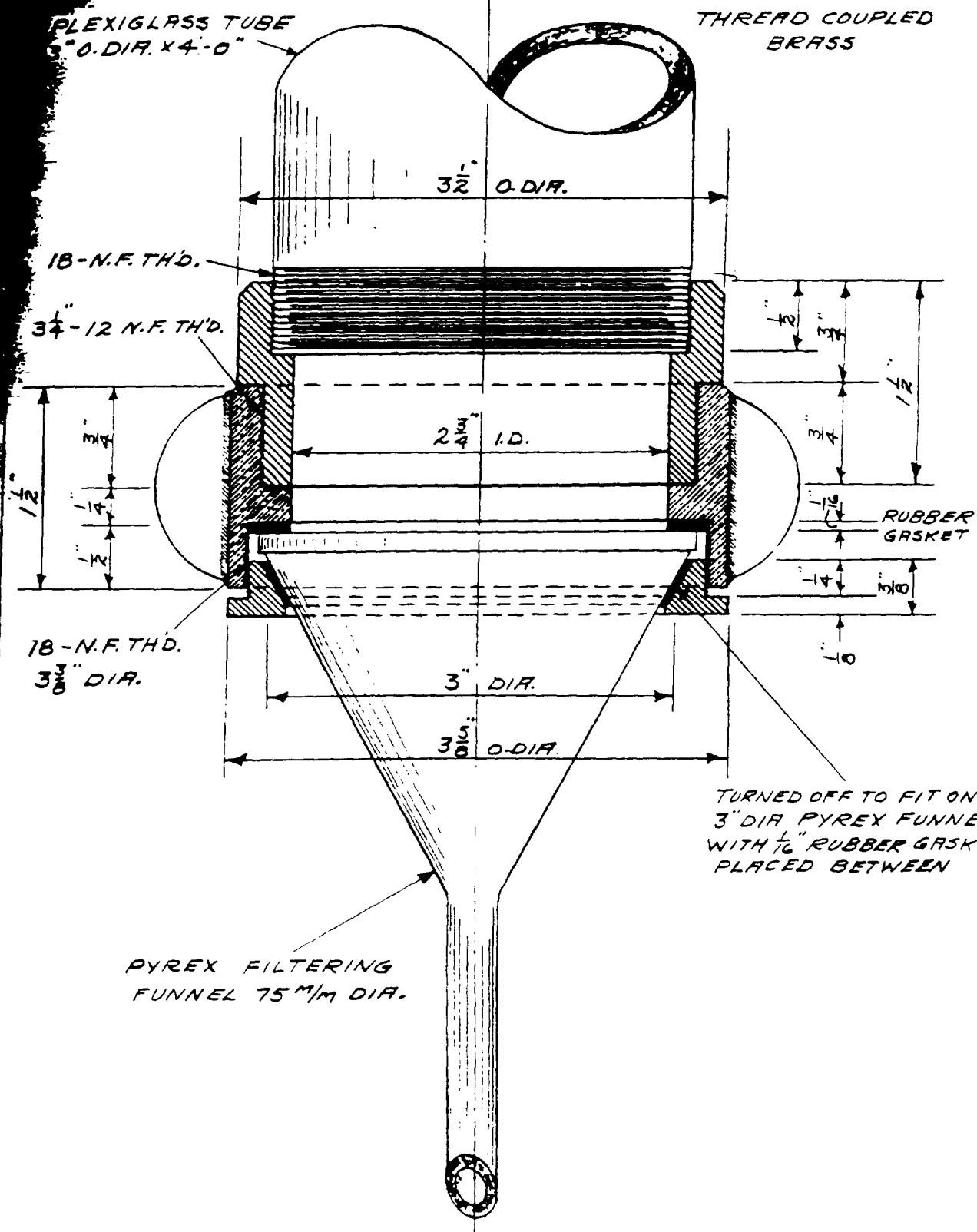
4. (a) The weighed residue described under "PROCEDURE" paragraph 3 (j) is reported as the percentage of grit and crude. The average of two results must be reported.
- (b) The weighed residue as described under "PROCEDURE" paragraph 3 (k) is reported as the percentage of crude. The average of two results must be reported.
- (c) The expected maximum difference between any individual readings and the average is $\pm 3\%$. When the maximum difference is exceeded, repeat the test.

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CRUDY CONTENT APPARATUS
DRAWING A



THREAD COUPLED
BRASS

$$3\frac{1}{2} \text{ Q.D./M.}$$

1B-N.F. TH'D.

3 1/4 - 12 N.F. TH'D.

 $2\frac{3}{4}$ | 1.0.

RUBBER
GASKET

18 - N.F. THO.
3 3/4" DIA.

3" DIA.

3030 0.01A

TURNED OFF TO FIT ON A
3" DIA PYREX FUNNEL
WITH $\frac{1}{16}$ " RUBBER GASKET
PLACED BETWEEN

PYREX FILTERING
FUNNEL 75 mm DIA.

CRUDY CONTENT APPARATUS-COUPPLING
DRAWING "B"
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Color Test
for
Asbestos Fibre

Adopted by

ACPA -
ATI - 9/ 8/60
QAMA - 9/13/60

COLOR TEST FOR ASBESTOS FIBRE

SCOPE

1. To determine the color and difference in color and shade of asbestos fibre.

APPARATUS

2. (a)* Photovolt photoelectric reflection meter model #610.
(b)* Search unit #610-T or 610-Y equipped with a green, amber and blue tristimulus filters.
(c)* Calibrated gray working standard plaque, catalogue No. 6163 of 60% reflectance.
(d)* Calibrated dark gray working standard plaque, catalogue No. 6163 of 44% reflectance.
(e)**Hydraulic press.
(f)**Compression cylinder with inside diameter of 1 1/8 in. which is approximately 1 square inch in area.

SAMPLING

3. In accordance with the "Method of Sampling and Preparation of Asbestos Fibres for Test Purposes".

PROCEDURE

4. (a) The details of the operation of this apparatus and the theory behind it are clearly described in the manufacturer's instructions.
(b) The procedure for running the test is as follows:
 - (1) Switch on the photovoltmeter and search unit and allow them to heat up for a minimum of 30 minutes.
 - (2) Balance the photovoltmeter against the working standard plaque for the filter being used.

* Photovolt Corporation, 95 Madison Avenue, New York, N. Y.

** F. S. Carver Inc., 1 Chatham Road, Summit, New Jersey

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- (3) Weigh out 10 grams of fibre and compress to 16,000 psi on the pellet using the press and compression cylinder.
- (4) Remove pellet from the compression cylinder. Be sure to test the pellet side next to the compression plunger.
- (5) Place the search unit on the pellet and take readings using the blue, green & amber tristimulus filters.
- (6) Place the working standard plaque back on the search unit after each sample reading and calibrate to insure proper readings.
- (7) Smoking will upset the search unit readings.
- (8) The 44% reflectance plaque is for very dark fibre only. The 60% reflectance plaque shall be used normally.
- (9) A voltage regulator should be used if the voltage is variable.

RESULTS AND SAMPLE DATA

5. (a) Assuming we are checking the green spectrum, we calibrate the galvanometer using the plaque and setting the galvanometer at the reading printed on the plaque for green, i.e. 73.5.
- (b) The sample is then placed on the search unit and the galvanometer reading is taken. The calibration is checked again by a second standard plaque reading.
- (c) The before and after test calibration and test reading is recorded as:

<u>FILTER</u>	<u>READING</u>	<u>CALIBRATION</u>	
		<u>BEFORE</u>	<u>AFTER</u>
Green	80.3	73.5	73.5

ACCURACY

6. (a) The accuracy shall be ± 0.50 units of reflectance of the galvanometer. Readings on separate samples of the same material must be within 1 galvanometer unit of each other. Readings on different samples that are 1.0 or more galvanometer units different are considered significant.

Air Classification Test

Adopted by:

ACPA - 9/15/59
ATI -
QAMA - 4/28/59

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AIR CLASSIFICATION TEST

SCOPE

1. (a) This test indicates the relative proportions of dust (less than 40 microns), fibre, and granular material (rock) in a sample of milled asbestos fibre.
- (b) The percentage of these components in milled fibre furnish a guide to its cleanness and general degree of effectiveness when used in asbestos cement products.

EQUIPMENT

2. (a) Balance (sensitivity 0.01 gm).
- (b) Stop Watch.
- (c) Rectangular Metal Scoop, 1 3/4 x 7 x 1 1/2 in.
- (d) Six Inch Classifier with Mechanical Agitator and Accessories. (Refer to Fig. 1, page 3). Also refer to construction details (Fig. 2, Fig. 3, Fig. 4).
- (e) Orifice #3, 0.415 in. diameter.
- (f) Brass Tube 5 mm inside diameter, 3 in. long.
- (g) Rubber Stopper to fit small end of separator.
- (h) Wire 12 in. long (bent in hook shape at one end, see Fig. 2, page 4).
- (i) Hose or Leather Strap 15 in. long.
- (j) 28 Mesh Tyler Standard Screen, 8 in. diameter.
- (k) 35 Mesh Tyler Standard Screen, 8 in. diameter.
- (l) Pan, 8 in. diameter, for above screens.
- (m) Ro-Tap Sieve Shaker.

SAMPLING

3. According to "Method of Sampling and Preparation of Asbestos Fibres for Test Purposes".

PROCEDURE

4. (a)	Sample from <u>Group</u>	Sample Weight <u>in gms</u>	Air Jet <u>Type</u>	Test Time <u>in Min.</u>	Screen <u>Mesh</u>	*Ro-Tap Time in <u>Min.</u>
	3	5	Tube	9	35	1
	4	10	Tube	9	35	1
	5	10	Tube	9	35	1
	6	10	Tube	9	35	1
	7	10	Tube	9	28	1

* Ro-Tap without hammer.

- (b) Push the 5 mm diameter brass tube through the rubber stopper so that it does not project more than 1/8 in. beyond the small end of the stopper. Insert stopper in small end of separator and clamp firmly in place, in such a manner that this jet can rotate freely. Attach the radius arm of the agitator to the jet, and connect the jet to the flange containing the #3 orifice, by means of a 3 ft. section of 1/4 in. or 3/8 in. diameter rubber tubing.
- (c) Charge the sample to the classifier by means of the rectangular scoop, then start agitator and turn on the compressed air and adjust the pressure so as to register 1.0 cm on the mercury manometer. Adjust water manometer to 8.0 cm and raise the mercury manometer to 15.5 cm. Start the stop watch. At three minute intervals shake the separator by striking it sharply with the short length of rubber hose or strap. Care must be taken to never strike the 6 in. diameter cylinder connecting the two cones.
- (d) At the end of the time allotted for the fibre under test (see table) shut off the air and place screen (35 or 28 mesh) and pan under the separator. Remove the rubber stopper and let the fibre fall onto the screen. If it chokes in the opening, loosen it with the short length of wire used expressly for that purpose.

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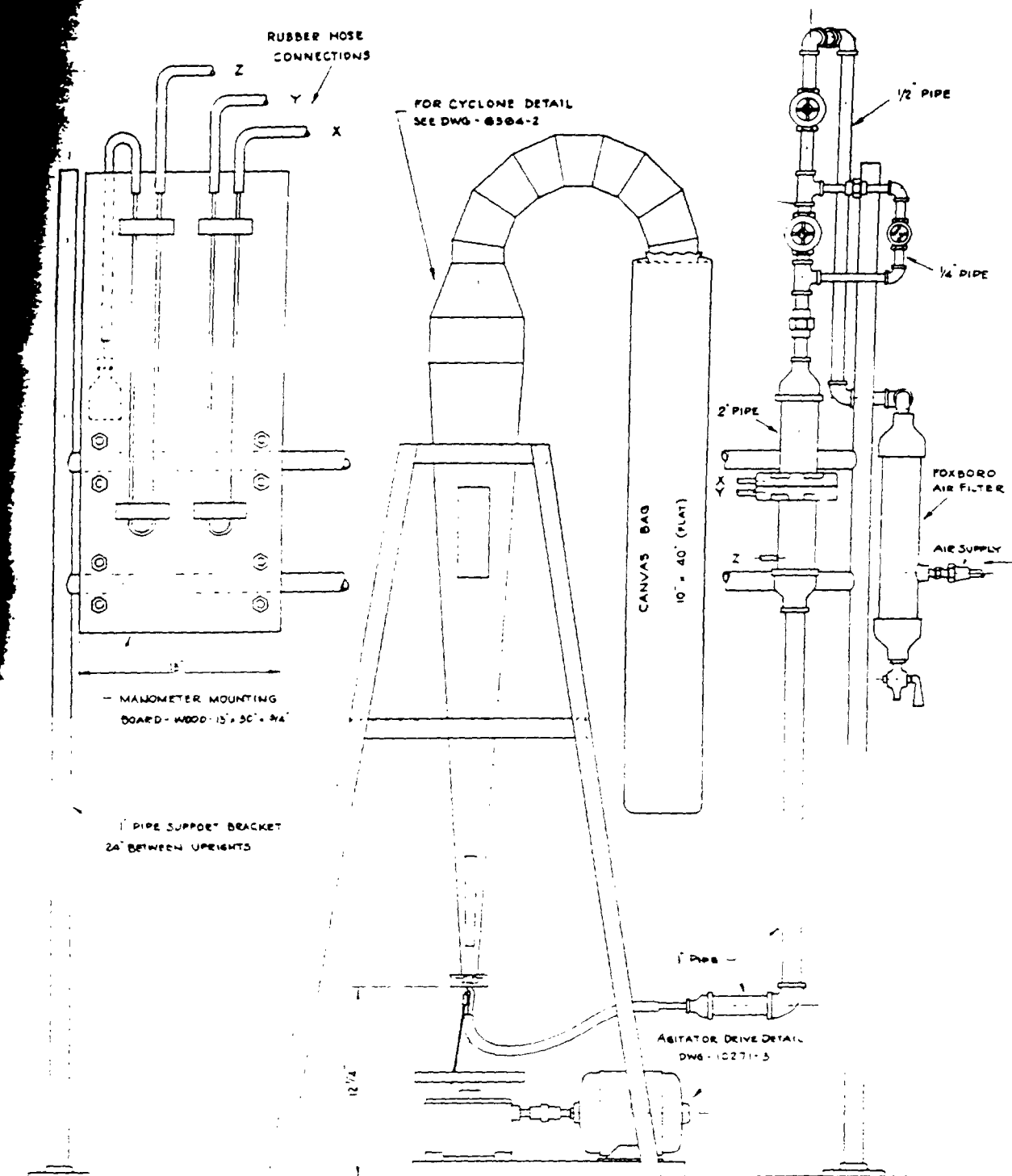
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- (e) Shake down the dust and rock on a section of the screen not covered by fibre, by rapping the two sides of the cone. Blow any remaining residue in the jet onto the rock laden section of the screen, using an air pressure of not more than 1.0 cm of mercury.
- (f) Place the screen and pan in the Ro-Tap sieve shaker and run for one minute without using hammer. Weigh, to the nearest 0.01 gm, the material on the screen, and record as fibre. Weigh the contents of the pan and record as the granular content. Obtain the dust content by difference between the original sample weight and the combined weights of the fibre and granular contents.

REPORT

5. (a) Report the results in per cent.
- (b) An example of the test results on a 6D fibre would be as follows:

Fibre	(+40 microns)	41.6%
Dust	(-40 microns)	46.4
Granular		<u>12.0</u>
		100.0



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AIR CLASSIFICATION APPARATUS

FIGURE 1.

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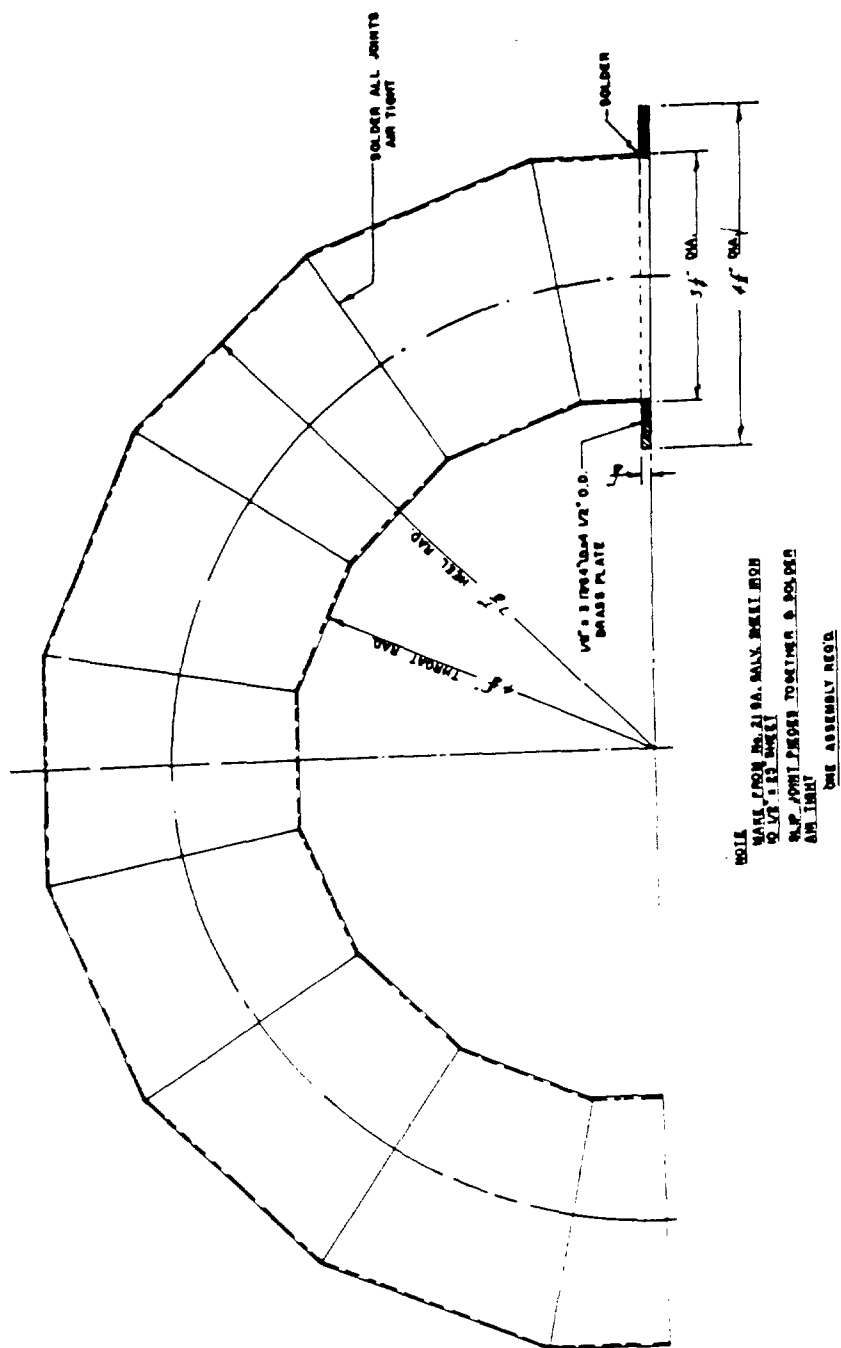


FIGURE 3

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FIGURE 4

MAGNETIC RATING OF ASBESTOS
USED FOR ELECTRICAL PURPOSES

Adopted by:

ACPA -
ATI - 9/10/59
QAMA -

Permission to reprint
received from A.S.T.M.

ST0513940

MAGNETIC RATING OF ASBESTOS USED FOR
ELECTRICAL PURPOSES¹

ASTM Designation: D1118-57

SCOPE

1. This method covers two procedures for determining the magnetic rating of asbestos used for electrical purposes. The free iron and magnetic iron oxide (Fe_3O_4) is determined by a magnetic analyzer and is expressed empirically as magnetic rating (MR). Experience has shown that when used for electrical insulation it is desirable that asbestos have a low magnetic rating. The two procedures described in this method are both based on the principle of comparing the electro-magnetic effect of an unknown sample of asbestos with the electromagnetic effect of a known standard.

CALIBRATING STANDARDS

2. (a) A magnetic analyzer covering the usual range of magnetic rating (MR) is designed for two sensitivities: one with a full scale deflection equal to an MR of one, and the other with a full scale deflection equal to an MR of six. The sensitivity is controlled by a transfer switch incorporated in the apparatus. For the initial calibration, three to five standards, differing in value to cover the test range of the scale, shall be used. A calibrating standard with an MR of one contains 0.18 g. of National Bureau of Standards standard sample No. 29a of iron ore magnetite² uniformly distributed over the space occupied by a specimen

¹ Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D13 on Textile Materials.

² Standard Sample No. 29a is no longer available from the National Bureau of Standards. A new standard magnetite sample suitable for this test is available from the Asbestos Textile Institute, Mr. M. C. Shaw, Philadelphia Textile Institute, Schoolhouse Lane, Philadelphia 44, Pa.
--Ed.

under test ($3/4$ in. in diameter by $2\ 7/8$ in. in length). For preparing calibrating standards of different values, the amount of magnetite shall be varied directly with the value of the desired standard (Note). The calibrating standards shall conform to the following requirements: Since the amount of magnetite used in a calibrating standard is not sufficient to fill the space needed for a test specimen, the magnetite shall be thoroughly mixed with an inert material such as zinc oxide so that it will pack firmly in the space provided. The containers for the calibrating standards shall be made of a non-magnetic and non-metallic material and have a chamber for the standard mixture with the same dimensions as the space in the test specimen holder. A convenient container is a wooden tube 1 in. in outside diameter with an inside diameter of $3/4$ in. and a length of $4\ 7/8$ in. with two end plugs $3/4$ in. in diameter by 1 in. in length. One plug shall be cemented in place before, and the other after, the standard material is packed. The cement used shall be non-magnetic.

Note: In setting up the calibrating standards it is desirable to have the magnetic rating equivalent to the percentage of magnetic material present in the specimen to be tested. Because of the variation in the permeability of the oxides, it may not be possible to accomplish this accurately.

- (b) Secondary standards for routine measurements can be made using smaller containers, that will fit in the test specimen holder, and having sufficient magnetite to give a deflection equal to the calibrating standard corresponding to the maximum scale value. One secondary standard is necessary for each scale range.

PROCEDURE A

PRINCIPLE OF OPERATION OF APPARATUS

3. A schematic drawing of the apparatus for Procedure A is shown in Fig. 1. The apparatus consists of a testing solenoid I having a primary winding M to produce a strong magnetic field, and secondary windings S and B to measure the change of flux caused by the specimen, a class A audio amplifier, A, to increase the signal sufficiently to operate an indicating instrument I. This apparatus operates from a 115-v., 60-cycle power supply.

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The secondary winding S is wound in two equal sections and connected so that their voltages oppose each other when the primary winding M is energized. Coil B is used in series with S to balance out any small voltage difference between the two sections, so that the voltage to the amplifier is zero when no specimen is in the coil. Under this condition, a small amount of magnetic material in one section of S will cause a small voltage at the input to the amplifier due to the increased magnetic flux in that section. This voltage is amplified sufficiently by A to be read by instrument I. It has been found that the readings of I are nearly directly proportional to the magnetic rating up to a magnetic rating of six. The primary and secondary windings of the testing solenoid, the amplifier gain, and the indicating instrument sensitivity can be varied as long as the over-all amplification is sufficient to give a full-scale deflection for the standard corresponding to the maximum scale value.

TESTING SOLENOID

4. The testing solenoid shall have a primary winding of 17 layers of 0.057-in. enameled single cotton covered copper wire (approximately 4200 turns). This coil shall be wound on a compound tube 2 1/4 in. in outside diameter with an inside diameter of 2 in., a length of 18 in., and end flanges 5 in. square by 1 in. in thickness. Inside of and concentric with the primary winding is the main secondary winding. This winding shall be in two sections on a compound tube 2 in. in outside diameter with an inside diameter of 1 in. and a length of 18 in. having two winding spaces each 3 in. in length with a diameter of 1 1/2 in. located an equal distance from the ends of the tube and 1 1/8 in. apart. Each section shall be wound with one layer of 0.010 in. enameled single silk covered copper wire (approximately 220 turns). These coils shall be electrically connected so that their voltages are opposing each other. A small sliding coil, B, Fig. 1, shall be connected in series with the secondary winding and is used for fine adjustment of the secondary voltage. This coil shall be made to slide in the end of the secondary winding coil form with provision to lock it in place at any position up to 2 in. inside the form. The winding space for the sliding coil shall be 1 in. in outside diameter with an inside diameter of 7/8 in. and a length of 1/4 in. The number of turns on this coil is determined during test as follows:

Energize the primary coil from a 115-v., 60-cycle source and measure the voltages across the secondary coils. Then connect these coils in series so that their combined voltage is near zero. Then using a more sensitive indicating instrument, remove turns from the stronger section one turn at a time until it is within one turn of being exactly equal to the other section. Then wind the sliding coil with a sufficient number of turns so that the sections are exactly balanced at some point within its limits of motion and unbalanced in opposite directions at the extreme positions the sliding coil can take. The final balancing is usually made with the complete analyzer apparatus and the proper balance is indicated when the indicating instrument will show an appreciable deflection with the balance coil at one extreme of its limits of motion and then decrease to approximately zero and then increase to an approximately equal unbalance as the coil is moved to its other extreme limit.

TEST SPECIMEN HOLDER

5. Test specimens shall be placed in a holder to facilitate setting them in and removing them from the testing solenoid. This specimen holder shall be slightly less than 1 in. in diameter so that it will easily slide inside the secondary coil. It shall be made with a handle at one end and a cork at the other end leaving a space $3/4$ in. in diameter by $2 \frac{7}{8}$ in. in length for the test specimen. The handle shall be made with a stop and its length shall be such that the specimen holder is centrally located under the nearest section of the secondary winding when the stop is against the solenoid.

AMPLIFIER

6. The amplifier shall be a two-stage resistance-coupled class A amplifier, using a pentode (type 57) for the first stage and a triode (type 56) for the second stage. The input is transformer coupled with a high secondary-to-primary-turn ratio transformer. The secondary of the input transformer is shunted by a resistance to change from the high sensitivity scale to the low sensitivity scale. This shunt circuit is controlled by a switch mounted on the amplifier panel. The amplifier gain is controlled by varying the screen grid voltage of the type 57 tube with the potentiometer G, Fig. 1. The indicating instrument I, included in the amplifier, is a d-c. milli-ampere meter with a sensitivity of 1 ma. for a full scale deflection. This instrument is used with a copper oxide

ST0513944

instrument rectifier for use on alternating current. It is coupled to a 30-henry choke in the output of the type 56 tube by means of two 2- μ f capacitors.

INITIAL CALIBRATION

7. Use the amplifier with the indicating instrument to balance the secondary windings. After this has been done adjust the over-all amplifier gain to give full-scale deflection on the high-sensitivity (MR1) scale, when an MR1 calibrating standard is in the testing solenoid and the gain control G, Fig. 1, is near its mid-position. (Before inserting the calibrating standard, balance the secondary voltage to a minimum.) Switch the amplifier to the low sensitivity (MR6) scale, insert the MR6 calibrating standard in the testing solenoid and adjust the shunt on the input transformer to give full-scale deflection with the gain control G at the same position as used in adjusting the first scale. Then recheck these adjustments to make sure the sensitivities are correct before calibrating the scales. Calibrate the scale by adjusting the equipment to give a full-scale deflection for the calibrating standard corresponding to its maximum value, then substitute other calibrating standards and record the deflection obtained with them. This shall be done on each scale. If desired, a special scale can then be marked according to these readings to eliminate correcting all subsequent readings made on the analyzer.

PROCEDURE

8. After the initial calibration is completed, to determine the magnetic rating of a test specimen of asbestos, first energize the apparatus and permit it to warm up for a few minutes to become stable. Then balance the secondary coils with the balancing coil until the reflection on the indicating instrument is a minimum at full sensitivity. Decrease the sensitivity, place the calibrating standard for the range of test desired in the testing solenoid and adjust the amplifier gain control to give a full-scale deflection equal to the magnetic rating of the calibrating standard. Then remove the calibrating standard, place a 10-g. specimen of asbestos in the specimen holder and insert in the solenoid. The indicating instrument will then give the magnetic rating of the specimen.

PROCEDURE B

PRINCIPLE OF OPERATION OF APPARATUS

9. A schematic drawing of the apparatus for Procedure B is shown in Fig. 2. The apparatus consists of a series-parallel network of two duplicate test coils T_1 and T_2 , two duplicate capacitors C_1 and C_2 , a rectifying unit R , and an indicating instrument I . With an a-c. voltage impressed on the circuit, the current flow is as indicated by the arrows in Fig. 2. Arrows of solid lines indicate the current flow during the positive half cycles, and arrows of dotted lines indicate the current flow during the negative half cycles. A study of this diagram will show that due to the action of the rectifier, capacitor C_1 alternately carries the positive current of coil T_1 and the negative current of coil T_2 while capacitor C_2 alternately carries the positive current of coil T_2 and the negative current of coil T_1 . Therefore, if the currents in both coils are equal, a stable condition exists and no current flows in the indicating circuit. If, however, the currents are unequal, a voltage difference will arise between the two capacitors causing a current to flow through the indicating instrument I .

COILS

10. The two coils shall be wound on a non-magnetic and non-conducting coil form approximately 6 in. in length with an inside diameter of 1 to 2 in. and shall have several thousand turns each (Note). They shall be as nearly identical in resistance and inductance as is practicable.

Note: In one satisfactory apparatus the coils were layer wound with 10,000 turns of 0.0179 in. enameled copper wire on a spool 6 $\frac{3}{8}$ in. in length and with an inside diameter of 1 $\frac{3}{4}$ in. and having end flanges 4 in. in diameter by $\frac{1}{16}$ in. in thickness.

CAPACITORS

11. The capacitors shall be of high quality and have equal reactances (Note).

Note: In one satisfactory apparatus the capacitors were 10- μ f. pyranol capacitors insulated for 330 v., a-c.

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RECTIFYING UNIT

12. The rectifier shall be made from two conventional full wave copper oxide rectifiers as shown in Fig. 3. Fig. 3 (a) is the conventional full wave rectifier with the d-c. terminals marked "+" and "-" and the a-c. terminals marked "~". Fig. 3 (b) shows the connections to be made between two conventional rectifiers to give the desired rectifying unit for this apparatus. Fig. 3 (c) is a simplified sketch of Fig. 3 (b) corresponding to the sketch of the rectifier unit in Fig. 2.

INDICATING INSTRUMENT

13. The details of the indicating instrument are shown in Fig. 4. This shall consist of a portable galvanometer G, a resistance network for adjusting the over-all sensitivity and for changing the scale range, and a capacitor for by-passing any alternating current that may be present. The galvanometer shall have the following characteristics:

Galvanometer resistance, ohms.....	50
Period, sec.....	3
Sensitivity, μ amp. per 1-mm. scale division.....	0.040
Extreme critical damping resistance, ohms..	700
Scale length, mm.....	100

The resistance network shall be made up of stable resistors having low-temperature coefficients. The values of these resistances are approximate as final adjustment must be made for the particular equipment being used. The capacitor shall be a 50-v. electrolytic capacitor.

TEST SPECIMEN HOLDER

14. The test specimen holder shall consist of a wooden tube 1 in. in outside diameter with an inside diameter of $3/4$ in. and a length of $4 \frac{3}{8}$ in. and with two wooden stoppers that extend $3/4$ in. into each end of the tube.

INITIAL CALIBRATION

15. (a) With this apparatus the balance is greatly affected by the proximity of any magnetic material, as such material changes the inductance of the coils. For this reason magnetic materials should not be used except those that are unavoidable and whose effect

can be balanced out, such as the capacitors and the assembly bolts through the rectifier. If these parts are magnetic, the coils shall be mounted with their axes horizontal and parallel, with the capacitors and rectifiers located midway between them. There shall be at least 3 in. between the sides of the coils and the capacitors. A simple balancing arrangement consists of a non-metallic plug, capable of fitting tightly into the end of one of the coils, with a magnetic machine screw through its center. The center of the plug should be drilled and tapped so that the machine screw can be screwed in or out of the coil. A locking nut should be used to hold this screw in place after balance has been obtained. The length and size of this screw can be determined when the procedure described in Paragraph (b) is performed.

- (b) For the initial balance, the sensitivity of the indicating instrument must be greatly reduced. This can be done by connecting a variable resistance of approximately 0 to 100 ohms across the instrument connections to the capacitors. Set the resistor at 0, shorting out the instrument, energize the circuit with a 115-v., 60-cycle supply, and then increase the resistance slightly until a few millimeters deflection is obtained on the galvanometer. It will then be found that bringing a magnetic screw near the end of one of the coils will cause the galvanometer deflection to decrease to zero and then reverse as the screw is pushed further into the coil. This indicates that the screw is bringing the coils into balance and then unbalancing them in the opposite direction. Then fit the plug into the desired coil and lock the screw in the position for zero balance. Increase the shunt across the instrument and adjust the balancing screw to maintain the balance until the coils are balanced with the resistance at its highest value. Remove the resistance and readjust the balance. The instrument resistances may now be adjusted to give the desired scale sensitivities. Place an MR1 calibrating standard in the test coil, throw the sensitivity switch to the MR1 position, and vary R4 to give 100-mm. deflection. If desired, a smaller variable resistance can be placed in series with R4 to give fine adjustment of the sensitivity. Throw the sensitivity switch to the MR6 position and place an MR6 calibrating standard in the test coil. Adjust R1 keeping the sum

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of $R1 + R2 = 700$ ohms, until a 60-mm. deflection is obtained on the galvanometer. If calibrating standards of other MR values are available, other points on the scale can be checked and a correction curve obtained, although the deflections are nearly directly proportional to the MR values over the range of values used in this apparatus.

PROCEDURE

16. Turn the sensitivity switch to the MR6 position, Fig. 4, and energize the apparatus with a 115-v., 60-cycle supply. Balance the apparatus by adjusting the balance screw to bring the galvanometer deflection to zero. Throw the sensitivity switch to the MR1 position, Fig. 4, and rebalance the apparatus, locking the balancing screw in place. Place the MR1 calibrating standard in the test coil, and adjust the sensitivity to give a galvanometer deflection equivalent to an MR of one. Insert the 10-g. test specimen of asbestos in the test coil and read the resulting deflection of the galvanometer. If the deflection is off the scale, throw the sensitivity switch to the MR6 position and read the deflection.

Note: The procedure here described is applicable only under those conditions wherein the magnetic particles being tested exhibit no directional properties or when the particle distribution is such as to give the ultimate in random orientation. The magnetic particles present in chrysotile asbestos, however, are most frequently needle-like or pseudo-fibrous and as such exhibit directional properties with regard to magnetic permeability. Therefore, there must be an understanding as to the extent of the fibrous characteristics and the grain size distribution of the magnetic particles present in the sample under test if a proper evaluation of the resulting magnetic rating is to be truly factual. In view of the fibrous characteristics of the magnetic particles and the resultant directional magnetic properties, the method of sample preparation and the placement of the sample in the sample holder must be carefully pursued in an endeavor to obtain a test specimen that will exhibit a maximum in random orientation.

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CALCULATION

17. The magnetic rating of the test specimen is proportional to the deflection, calculated as follows:

$$\text{MR for the MR1 scale} = \frac{\text{deflection}}{100} \times 1$$

$$\text{MR for the MR6 scale} = \frac{\text{deflection}}{60} \times 6$$

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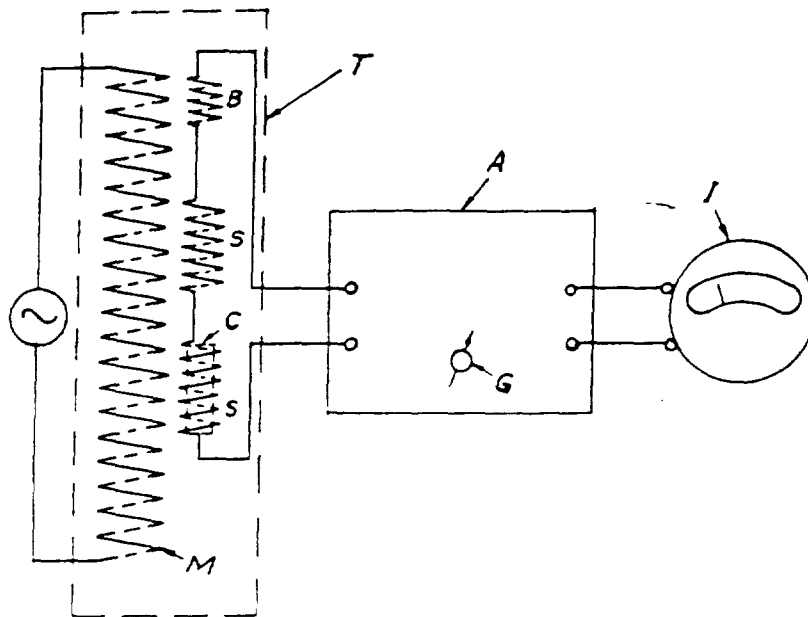


Fig. 1. - Schematic Diagram of Apparatus for Procedure A.

- T - Test solenoid
- M - Magnetizing coil
- S - Secondary coils
- B - Balancing coil
- C - Test sample
- ~ - Alternating current source
- A - Amplifier
- G - Amplifier gain control
- I - Indicating instrument

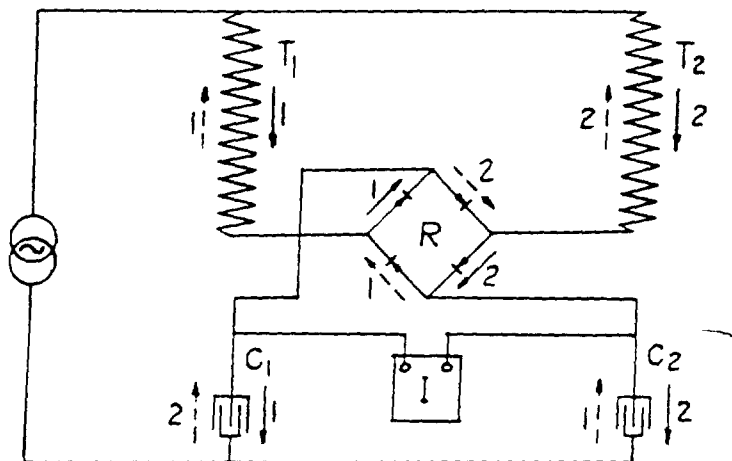


Fig. 2. - Schematic Diagram of Apparatus for Procedure B.

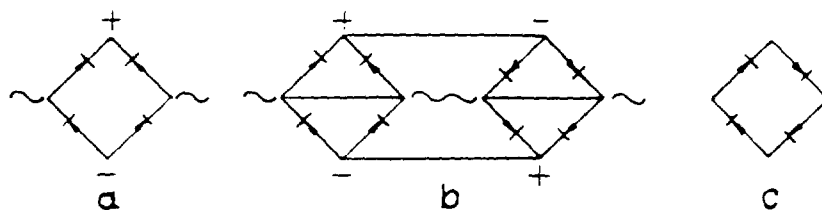


Fig. 3 - Schematic Diagram of Rectifiers and Connections for Procedure B.

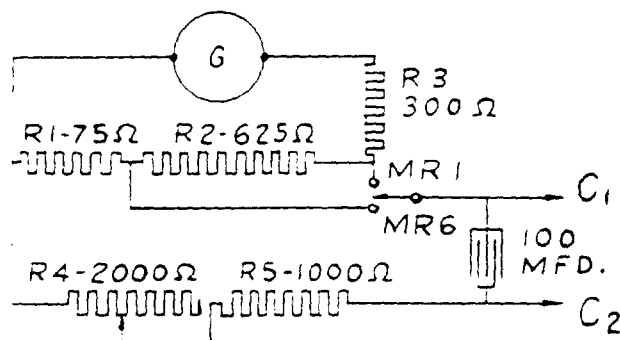


Fig. 4 - Schematic Diagram of Indicating Instrument for Procedure B.

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Methods of Quantitative Analysis
of Asbestos Fibres

Adopted by

ACPA - 2/14/61
ATI - 9/ 8/60
QAMA -

METHODS OF QUANTITATIVE ANALYSIS OF ASBESTOS FIBRES

SCOPE

1. These methods describe procedures for the quantitative analysis of chrysotile for (a) Silica (SiO_2), (b) Ferric Oxide (Fe_2O_3), (c) Alumina (Al_2O_3), (d) Calcium Oxide (CaO), (e) Magnesium Oxide (MgO), (f) Carbonates, (g) Water of Crystallization.

OUTLINE OF METHOD

2. The samples are treated as silicates not decomposed by acids and are therefore fused with Na_2CO_3 to convert to soluble sodium salts. From the hydrochloric acid solution of the melt, silica is first removed by dehydration and precipitation. The filtrate containing iron and aluminum plus calcium and magnesium is treated with bromine water to oxidize the iron to the ferric state and the latter together with the aluminum are precipitated with ammonia and ignited. To differentiate between Fe_2O_3 and Al_2O_3 , the ignited residue is fused with potassium pyrosulfate and the iron in the melt determined volumetrically. Another aliquot portion of the solution of the melt is used for determination of alumina by use of 8 - hydroxy quinoline. The filtrate resulting from the Fe_2O_3 and Al_2O_3 precipitation contains calcium (if present) and magnesium. The calcium is precipitated from this filtrate, after the latter is acidified, as calcium oxalate. The latter is ignited and weighed as CaO . The filtrate after removal of the calcium is made acid with conc. HNO_3 and evaporated to dryness to insure removal of ammonium salts, as the latter will prevent complete precipitation of the magnesium. The residue is dissolved in dilute HCl and the magnesium precipitated with $(\text{NH}_4)_2\text{HPO}_4$ in ammoniacal solution. The precipitate is carefully ignited and weighed as $\text{Mg}_2\text{P}_2\text{O}_7$. The water of crystallization is determined by ignition loss and corrected for carbonate content.

SAMPLING

3. (a) Sample - The sample shall be the laboratory test sample as prescribed in the "Method of Sampling and Preparation of Asbestos Fibres for Test Purposes" or other asbestos fibre sample.

- (b) Test Specimen - The 0.5 gram test specimen shall be derived from the laboratory test sample. Quarter the sample down to approx. 25 grams as prescribed in the Sampling and Preparation of Fibre, Part 2, (a) through (g). Divide the 25 gram sample into ten approx. equal portions and sub-divide these portions into halves. Combine a half of each of the ten portions and mix thoroughly. Repeat this procedure until the sample weight is reduced to approx. 3 grams. On a smooth, clean area spread the 3 gram sample into a thin layer over an area of approx. 8" x 8" in such manner that pinches may be taken from all parts. Extract with tweezers five bundles of long fibre free from rock, grit or dust, each weighing about 0.5 gram. Spread into a thin layer and extract at least five bundles of the fibre from different parts so that when these are combined and dried to constant weight at 110°C (230°F) the test specimen will weigh 0.5 ± 0.02 grams.
- (c) Preparation of Sample - The test specimen is placed in a tared porcelain crucible and after brought to constant weight at 110°C (230°F) is cooled in a desiccator and weighed on a sensitive gram balance to the fourth decimal place. This weight minus the tare is the sample weight (W) for (a) SiO_2 , (b) Fe_2O_3 , (c) Al_2O_3 , (d) CaO and (e) MgO . (Different samples must be used for carbonates and water of crystallization.) The crucible with specimen is then placed in a furnace at 800°C (1470°F) for 1 hour. After cooling, the specimen is reduced to as fine a mesh as possible with an agate mortar and pestle. About 3.0 grams Na_2CO_3 are added and ground intimately with the specimen for an intimate mixture. The contents of the mortar are then transferred to a 30 ml capacity platinum crucible using a camel hair brush to sweep the last of the mixture from the mortar. Successive small amounts of additional Na_2CO_3 are then ground in the mortar and transferred in like manner to the platinum crucible, to insure removing the last trace of specimen from mortar to the crucible. Then sprinkle a little additional Na_2CO_3 on top of the intimate mix of powdered specimen and the Na_2CO_3 , place a platinum lid on the crucible.
- (d) Apparatus:
- (1) Mortar and Pestle, 125 ml capacity. Agate.
 - (2) Platinum Crucible, 30 ml capacity.

- (3) Oven for drying at 110°C (230°F).
- (4) Muffle type electric furnace for ignition at 800°C (1470°F).
- (5) Sensitive chemical gram balance.
- (6) Porcelain Crucible, 20 ml capacity.
- (7) Camel Hair Brush.
- (8) Desiccator.

(e) Reagents:

- (1) Na_2CO_3 , Anhydrous, C.P.

DETERMINATION OF SILICA

4. (a) Procedure - Heat the covered platinum crucible containing the intimate mix of powdered specimen and flux at first over a small flame to drive out any moisture present. Gradually raise the temperature until the highest heat of a good Meker burner is obtained. As soon as the mass melts quietly and there is no further evolution of carbon dioxide, the decomposition is complete. Wind a piece of platinum wire into a spiral and insert it into the fused mass. Remove the flame, allow the crucible to cool in the air somewhat and then play a stream of water upon the outside of the crucible. As soon as the crucible does not hiss when the water strikes it, quickly introduce enough water into the crucible to cover the melt. After about a minute carefully pull on the wire; usually the melt can be withdrawn from the crucible. If it does not come out easily, it can often be loosened from the crucible. If it does not come out easily, it can often be loosened by carefully heating the crucible; place the melt in a 300 ml casserole, add 25 ml of water, cover the casserole and carefully add 25 ml of 6 N. hydrochloric acid. A lively evolution of carbon dioxide at once takes place, but as the silicic acid separates, the inner part of the cake gradually becomes coated with a film of silicic acid which protects it from the further action of the acid. Consequently, it is necessary to break up the cake from time to time by means of a glass rod, until finally there is no evolution of a gas and no more hard lumps remain. After the evolution of carbon dioxide has

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nearly ceased, wash off the under side of the watch glass, raise it by placing a glass triangle under it, and evaporate to dryness. Heat the residue for at least an hour at 110°C to dehydrate the silica.

Moisten the dry powder with conc. hydrochloric acid and allow the covered dish to stand 10 minutes at the ordinary temperature in order that the basic salts and oxides formed during the evaporation and drying may once more be changed to chlorides. Then warm gently, dilute with 100 ml water, heat to boiling, and after the silicic acid has settled, filter through a well-fitted ashless filter paper. Wash the residue three or four times by decantation with hot 2 N. hydrochloric acid, then transfer to the filter and wash with hot water until free from chloride. Place the precipitate in a platinum crucible and set aside for the time being. The separation of the silicic acid is not quite complete; as much as 2 mg may remain in the filtrate. To remove this, once more evaporate the solution to dryness on the water bath, and again heat at 110°C for an hour, moisten the residue with 5 ml of conc. hydrochloric acid, and allow to stand not more than 15 minutes. Warm, dilute to 100 ml, heat to gentle boiling, and filter through a new and correspondingly small, ashless filter, washing with hot 2 N. acid and with water as before. Save the filtrate and analyze it for Fe_2O_3 , Al_2O_3 , CaO and MgO as will be subsequently described.

Combine the latter wet filter with that one previously obtained and ignite the wet filters containing the silica in a platinum crucible. Keep the temperature low until all the carbon is consumed, and do not allow the filter to catch fire. Finally, cover the crucible and ignite over a Meker burner, cool and weigh as impure silica.

The silica thus obtained is not absolutely pure. To test its purity, moisten it with water, add a drop of conc. sulfuric acid and about 5 - 10 ml of pure hydrofluoric acid. (Do not measure this acid in a glass graduate. Do not inhale the fumes. If spilled on the hands, wash them immediately under the water tap.) Place the crucible in an air bath and evaporate under a hood until no more vapors are expelled. Then remove the excess sulfuric acid by heating over a free flame. Raise the temperature gradually and finally

heat the crucible over a Meker burner and again weigh. Repeat the treatment with sulfuric and hydrofluoric acids, without adding any more water, until the contents of the crucible (usually Fe_2O_3 and Al_2O_3) are at a constant weight. Deduct this amount from the weight of impure silica and add it to the precipitate obtained with the ammonia in the subsequent analysis.

(b) Calculations:

$$\frac{\text{Wt. of Silica}}{\text{Wt. of Sample (W)}} \times 100\% = \% \text{SiO}_2$$

(c) Apparatus:

- (1) Platinum Crucible
- (2) Platinum Wire
- (3) 300 ml Casserole
- (4) Glass Triangle
- (5) Vented Chamber at 110°C
- (6) Ashless Filter Paper (Whatman No. 41)
- (7) Water Bath
- (8) Meker Burner
- (9) Vented Hood

(d) Reagents:

- (1) 6 N. Hcl
- (2) 2 N. Hcl
- (3) Conc. Hcl
- (4) Conc. H_2SO_4
- (5) Pure HF

DETERMINATION OF FERRIC OXIDE

5. (a) Procedure - The filtrate obtained after precipitation of the silicic acid in the last procedure should be

treated as follows. Oxidize the iron back to the ferric state by adding bromine water and boiling until the excess of the latter is expelled. After this add 10 ml of 2 N. ammonium chloride solution, several drops of methyl red indicator, and precipitate the aluminum and iron from the boiling hot solution by adding dilute ammonia free from carbonate until the solution turns yellow. Allow the precipitate to settle, filter, and wash twice by decantation with hot water. Redissolve by running hot 2 N. hydrochloric acid through the filter into the beaker containing the greater part of the precipitate. Repeat the precipitation with ammonia as before, and after filtering and washing by decantation, transfer the precipitate to the filter and wash until free from chloride with water containing 2% ammonium nitrate. Save the filtrate for CaO and MgO determinations.

Allow the precipitate to drain as completely as possible, and ignite wet in the crucible containing the residue obtained from the treatment of the impure silica with sulfuric and hydrofluoric acids. After igniting strongly over a Meker burner, weigh the crucible; its contents represent the sum of Al_2O_3 and Fe_2O_3 .

For the determination of the Fe_2O_3 , fuse the mixed oxides with potassium pyrosulfate in a silica crucible using 15- 20 times as much $\text{K}_2\text{S}_2\text{O}_7$. Before starting the fusion, heat the $\text{K}_2\text{S}_2\text{O}_7$ in the silica crucible until fumes of sulfuric anhydride evolve. This makes it anhydrous so that steam will not come off during the fusion. Add the mixture of oxides to this and heat slowly with a small flame until complete fusion occurs. Add a small amount of additional $\text{K}_2\text{S}_2\text{O}_7$ at the last. When the fusion is complete, make a spiral from a platinum wire and insert in the melt. When solidified, remove by pulling on the wire. Dissolve the melt in HCl (1:1 by vol.) stirring until all is dissolved. Dilute this solution to a definite volume and take one half of it for the Fe_2O_3 and the other half of it for the Al_2O_3 determination.

Heat the liquid taken for the Fe_2O_3 to about 80°C and while still hot reduce the ferric iron by adding stannous chloride drop by drop from a buret until the yellow color of the FeCl_3 just disappears, using a white piece of paper under the beaker for a better background of color change. Cool the solution to 15°C ,

and while stirring add 10 ml of HgCl_2 solution. Formation of a light silky precipitate indicates conditions are correct. Stir the solution vigorously for 1 minute, add 3 drops of diphenylamine indicator solution and dilute to 150 - 200 ml with cold distilled water. Titrate with a standardized solution of $\text{K}_2\text{Cr}_2\text{O}_7$ until the blue color persists. The concentration of the $\text{K}_2\text{Cr}_2\text{O}_7$ solution should be such that 1 ml of the standard solution is equivalent to .006 grams Fe. From the titration, calculate the Fe to Fe_2O_3 .

(b) Calculations:

$$\frac{(\text{ml } \text{K}_2\text{Cr}_2\text{O}_7 \text{ titer}) \times .006 \times \frac{2}{1} \times 1.43 \times 100\%}{\text{Sample Wt. (W)}} = \% \text{Fe}_2\text{O}_3$$

(c) Apparatus:

- (1) Meker Burner
- (2) Platinum Crucible
- (3) Platinum Wire
- (4) Silica Crucible
- (5) Two 50 ml Burets
- (6) Ashless Filter Paper (Whatman No. 41)

(d) Reagents:

- (1) Bromine Water
- (2) 2 N. NH_4Cl
- (3) 2 N. NH_4OH
- (4) 2 N. HCl
- (5) 2% NH_4NO_3
- (6) $\text{K}_2\text{S}_2\text{O}_7$ C.P.
- (7) 1:1 by Vol. HCl
- (8) Stannous Chloride

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Prepare by dissolving 15 grams Tin metal C.P. in 350 ml hot HCl (Sp. Gr. 1.19) and dilute to 1 liter.

(9) Mercuric Chloride
(Saturated solution)

(10) Diphenylamine Indicator (1 gram dissolved in 100 ml H_2SO_4
(Sp. Gr. 1.84)

(11) Potassium Dichromate, approx. 0.1 N.

Prepare by dissolving 4.9 grams in water and diluting to 1 liter. Standardize against a known weight of pure iron using same method as in the procedure.

DETERMINATION OF ALUMINUM OXIDE

6. (a) Procedure - Use the other half of the solution from the last procedure made with the 1:1 HCl. Dilute to approx. 200 ml and heat to 70°C. Add an excess of the reagent 8 - Hydroxyquinoline. Slowly add 2 N. ammonium acetate until a permanent precipitate is formed, and then add 20- 25 ml more. Allow the precipitate to settle, filter through a weighed Gooch crucible, wash with cold water, dry at 120 - 140°C, and weigh as $Al(C_9H_6ON)_3$ containing 11.1% Al_2O_3 .

(b) Calculations:

$$\frac{\text{Wt. of } Al(C_9H_6ON)_3 \times .111}{\text{Sample Wt. (W)}} \times 100\% = \% Al_2O_3$$

(c) Apparatus:

- (1) Tared Gooch Crucible with asbestos mat.
- (2) Filter suction flask and aspirator.

(d) Reagents:

- (1) 8 - Hydroxyquinoline, prepared by dissolving 25 grams in 60 ml glacial acetic acid. Dilute with cold water to 2 liters.
- (2) 2 N. CH_3COONH_4

DETERMINATION OF CALCIUM OXIDE

7. (a) Procedure - Make the filtrate, obtained from the ammonia precipitation of Fe_2O_3 and Al_2O_3 in the previous procedure, acid with HCl . Bring to a volume of 400 ml, heat to $80 - 90^\circ\text{C}$, and slowly add, while stirring, 30 ml of hot 0.5 N. ammonium oxalate solution. Slowly add NH_4OH until the solution is slightly ammoniacal and allow the precipitate to stand several hours before filtering.

If considerable magnesium is present in the solution, some magnesium oxalate will come down with the calcium oxalate. Since chrysotile contains a large amount of magnesium, it is best to redissolve the calcium oxalate precipitate and repeat the precipitation, using a filter paper for filtering off the precipitate. When all the solution has passed through the filter, wash the precipitate with about 15 ml hot water. Then rinse the precipitate back into the original beaker by holding the funnel in an inverted position and directing a stream of hot water against it. Replace the funnel in the support and wash the filter with about 25 ml of hot 3 N. hydrochloric acid. Heat the acid in a test tube, pour it upon the upper edge of the paper, and catch the liquid as it runs through the filter in the beaker containing the precipitate. Finally wash the filter with a little hot water (and with dilute ammonia if it is to be used again for filtering the next precipitate). Heat the dilute acid in the beaker and add a little more acid if necessary to dissolve the precipitate completely. Dilute the solution to about 250 ml and repeat the precipitation of the oxalate at the boiling temperature, adding ammonia and 5 ml more of the ammonium oxalate reagent. Since the solution already contains oxalic acid equivalent to the calcium, only a little more reagent is necessary.

Weighing as CaO . Use an ashless paper for collecting the precipitate. Wash the precipitate with hot water until free from chloride, and save the filtrate for the magnesium determination. Transfer the precipitate to a weighed platinum crucible. Ignite carefully with the flame at the mouth of the crucible until the precipitate is dry, and then heat with a small flame at the base of the crucible until all the paper is decomposed without letting it take fire. Then gradually raise the temperature and heat over a Meker burner for

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an hour with the crucible in an upright position and covered. Cool to about 100°, place in a desiccator, and weigh after 15 minutes. Repeat the heating until after cooling a constant weight is obtained. The calcium oxide is somewhat hygroscopic but is not difficult to weigh if it has been washed free from chloride. Call the weight constant if it agrees within 0.2 mg with the previous weight.

(b) Calculations:

$$\frac{\text{Wt. CaO}}{\text{Wt. Sample (W)}} \times 100\% = \% \text{ CaO}$$

(c) Apparatus:

- (1) 600 ml Beaker
- (2) Glass Funnel
- (3) Platinum Crucible
- (4) Ashless Filter Paper (Whatman No. 40)
- (5) Meker Burner
- (6) Desiccator

(d) Reagents:

- (1) 3 N. HCl
- (2) 0.5 N. Ammonium Oxalate
- (3) 2 N. NH_4OH

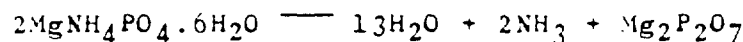
DETERMINATION OF MAGNESIUM OXIDE

8. (a) Procedure - Add 75 ml of concentrated HNO_3 to the combined filtrates and washings from the $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ precipitation, and evaporate to dryness on the steam bath or hot plate. Do not boil the solution or there will be loss by spattering. Keep the beaker covered with a watch glass supported above the upper rim of the beaker. A glass triangle, or glass supports which are made for this purpose, should be used to support the watch glass. To the small residue obtained, add 2 ml of concentrated HCl and 25 ml of water. Heat

nearly to boiling, and, after a few minutes, filter off the silica residue through a small filter. Wash the beaker, and filter thoroughly with hot water. The silica comes from the action of reagents on reagent bottles or on the beakers used in the analysis.

Dilute the solution to about 150 ml; add about 1.2 g of $(\text{NH}_4)_2\text{HPO}_4$ dissolved in a little water and a few drops of phenolphthalein indicator solution. Heat nearly to the boiling point, and then slowly add 1.5 N. Ammonia water until a faint pink color is obtained and a slight precipitation takes place. Stir well for about a minute, touching the sides of the beaker as little as possible. When the precipitate has become distinctly crystalline, add more ammonia until a deep color is obtained with the phenolphthalein. Allow the solution to cool, then add one-fifth the solution's volume of concentrated ammonium hydroxide and allow to stand over night. Continue the analysis as follows:

Weighing as $\text{Mg}_2\text{P}_2\text{O}_7$.



Use an ashless filter paper for filtering off the magnesium ammonium phosphate precipitate. After washing it with 1.5 N. NH_4OH , moisten with a saturated solution of ammonium nitrate in 1.5 N. NH_4OH , dry and ignite very slowly and carefully, and weigh. The precipitate contains 36.21% MgO .

(b) Calculations:

$$\frac{\text{Wt. } \text{Mg}_2\text{P}_2\text{O}_7 \times .3621}{\text{Sample Wt. (W)}} \times 100\% = \% \text{MgO}$$

DETERMINATION OF CO_2 FROM CARBONATES (By Knorr Alkalimeter Method as Proposed in ASTM D-13 Supplement 1958)

9. (a) Sampling - The sample shall be taken in the same manner from the laboratory test sample from which specimens were taken for the preceding analyses. A specimen shall be taken, weighing approx. 1 gram and dried to constant weight at 110°C (230°F) weighing to the nearest 0.001 gram. Call this sample weight W.

(b) Procedure - Check the gas flow train to insure freedom from leaks. Aspirate a current of air through the sy-

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stem at the rate of about two bubbles per minute for a period of 10 min. Stop the air current and remove the absorption tubes. Place the tubes within the balance case and allow it to stand for several minutes. When ready for weighing, open the stopcock momentarily and close. Weigh and repeat the above procedure. The second weight should agree with the first to within 0.005 g. If it does not, repeat this process until the two successive weighings agree. When a constant weight is reached, replace the absorption tubes in the gas train.

Transfer about 1 g of the sample, weighed to the nearest 0.001 g, to the distillation flask. Wash down any adhering particles on the inside neck of the flask with distilled water. Add enough distilled water to the flask so when the apparatus is reconnected the tip of the dropping funnel will be submerged about 5 to 10 mm. Place 50 ml of HCl (1:1) in the dropping funnel and replace the guard gas-absorption tube at the top of the funnel. Start the flow of water in the condenser and open all the stopcocks except the one on the dropping funnel. Turn on the aspirator for medium suction. Adjust the stopcock on the dropping funnel so the suction will draw the acid slowly into the flask. When all the acid is in the flask, fully open the funnel stopcock and pull air through the system at the rate of two or three bubbles per second. After the reaction in the flask has subsided, warm the contents gently to boiling. When steam starts to condense in the condenser, turn off the heat. Continue to draw air through the apparatus for 30 min. Re-weigh the absorption bulb as before. The gain in weight represents the CO₂ in the sample.

(c) Calculations:

$$\frac{\text{Wt. of CO}_2 \text{ Absorbed}}{W_1 (\text{Sample Weight})} \times 100\% = \% \text{ CO}_2 \text{ in Asbestos}$$

(d) Apparatus:

Sensitive Chemical Gram Balance
Knorr Alkalimeter, consisting of the following parts connected as shown in the appended sketch Fig. No. 1.

- (1) Gas Washing Bottle, A, containing concentrated H₂SO₄ which serves both to indicate the rate of flow and to prevent any water vapor entering the system from the atmosphere.

- (2) Drying Tube or Cylinder, B, filled two-thirds full of soda-asbestos absorbent (Ascarite) and one-third full of a drying agent to remove all CO_2 from the air.
- (3) Knorr Alkalimeter Unit, C, which consists of a dropping funnel, a distillation flask, and a condenser fitted with standard-taper joints to form a unit assembly.
- (4) Gas Washing Bottle, D, containing a solution of 5 to 10 per cent by weight of Ag_2SO_4 in concentrated H_2SO_4 . This serves to absorb any water vapor that escapes from the condenser and to remove any HCl from the evolved gases.
- (5) Drying Tube, E, containing CuSO_4 to absorb any H_2S generated by the absorption of HCl .
- (6) Drying Tube, F, filled with a suitable drying agent to effect complete drying.
- (7) Absorption Tube, G, filled two-thirds full of soda-asbestos absorbent (Ascarite) and one-third full of a drying agent, to absorb the CO_2 .
- (8) Absorption Tube, H, filled with a drying agent and soda-asbestos absorbent (Ascarite) in the reverse direction to prevent CO_2 from entering the system.
- (9) Trap, I, to prevent back flow of water from the aspirator.
- (10) Valve or Pinched Tubing, J, to control the rate of air flow pulled through the system by the aspirator.

(e) Reagents:

- (1) Conc. H_2SO_4
- (2) Soda-asbestos Absorbent (Ascarite)
- (3) 10% by wt. Ag_2SO_4
- (4) CuSO_4
- (5) 1:1 by vol. HCl

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DETERMINATION OF WATER OF CRYSTALLIZATION

10. (a) The sample shall be taken in the same manner from the laboratory test sample from which specimen were taken for the preceding analyses. A specimen shall be taken, weighing about 5 grams and dried to constant weight at 230°F, (110°C), weighing to the nearest 0.001 gram. Call this sample weight W_2 .

(b) Procedure - Ignite in a tared porcelain crucible the sample which has been dried to constant weight, by placing the crucible and contents in an electric furnace maintained at 1700 to 1750°F (927° - 954°C) and continue the ignition for 1 hour. Cool in an oven at 110°C and then in a desiccator. Then re-weigh to nearest 0.001 gram.

(c) Calculations:

$$\frac{\text{Original Wt.} - \text{Oven Dry Wt.}}{\text{Original Wt.}} \times 100\% = \% \text{ Absorbed Moisture}$$

$$\frac{\text{Oven Dry Wt.} - \text{Ignited Wt.}}{\text{Oven Dry Wt.}} \times 100\% = \% \text{ Ignition Loss}$$

The equation for calculating the % water of crystallization provides for ignition loss due to evolution of CO_2 from carbonates even though in some cases there may be no CO_2 evolved.

$$\frac{W_2 (100\% - \% \text{ CO}_2) - W_2 (100\% - \% \text{ Ign. Loss})}{W_2 (100\% - \% \text{ CO}_2)} \times 100\% = \% \text{ Water Crystallization}$$

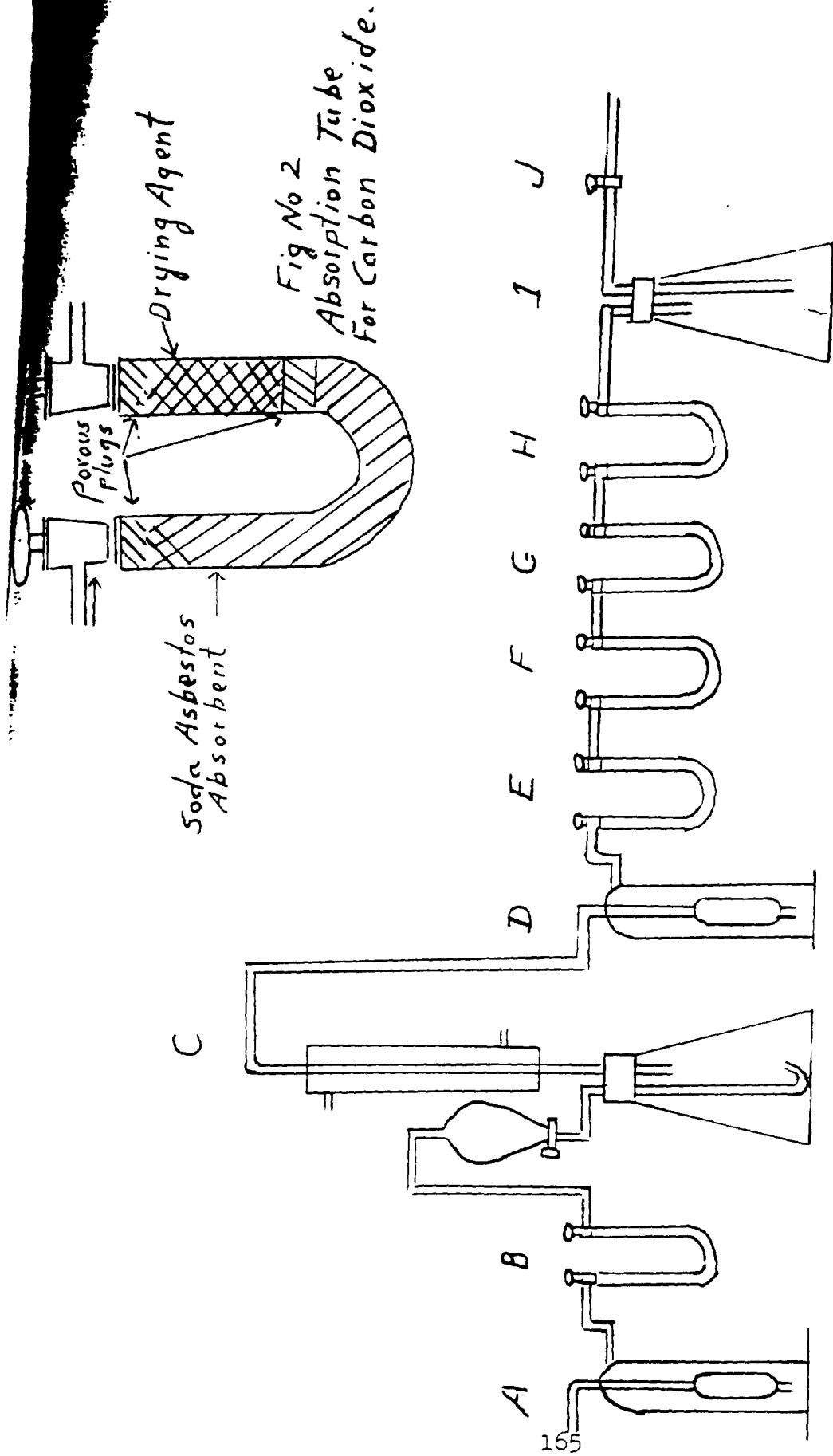


Fig. No 1. Knorr Alkalimeter for Determination of Carbon Dioxide.

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Method for Measuring Resin Pick-Up
of Asbestos Fibre

Adopted by

ACPA -
ATI - 6/ 8/61
QAMA -

METHOD FOR MEASURING RESIN PICK-UP
OF ASBESTOS FIBRE

SCOPE

1. This method of test covers a procedure for determining resin pick-up by asbestos fibre. It is intended to provide comparative values between a sample of fibre and a standard or between two different samples of fibre when the resin is of a liquid phenolic classification.

It has been established that the apparent surface area of the fibres, as measured by air permeability, and bulk density are major factors influencing the amount of resin picked up by the fibres.

OUTLINE OF METHOD

2. A weighed sample of fibre is compressed to a 6-in. x 6-in. pad by a constant applied load which is subsequently released. The pad of fibre, supported by a perforated screen, is then immersed in a low viscosity phenolic resin solution of specified solids content for a fixed time period. After removal and allowed to drain freely, the sample is dried and the resin solids content is determined by a standard ignition method for asbestos-phenolic products.

APPARATUS

3. (a) Screens - Brass wire with 16 mesh openings and measuring 6-in. x 6-in.
- (b) Mold - Metal mold having a cavity measuring 6-in. x 6-in. x 4-in. deep.
- (c) Press - Shall have a capacity of at least 3600 lb. total force to produce a pressure of 100 psi on an area of 36 sq. in. Daylight should be no less than 8 inches with an 8 inch ram movement.
- (d) Balance - Sensitivity of 0.01 grams and capacity of weighing 50 grams.
- (e) Stop watch

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- (f) Glass tray - Size to hold 6-in. x 6-in. screen with fibre pad and capacity of 2 quarts, measuring approximately 12-in. x 7-in. x 2-in. deep.
- (g) Drying oven - Standard drying oven with solvent exhaust and capable of maintaining 350°F plus or minus 5°.
- (h) Muffle furnace - Capable of ignition at 1480°F plus or minus 20°F.
- (i) Dessicator
- (j) Resin - Phenolic varnish soluble in ethyl alcohol.
- (k) Alcohol - Commercial grade ethyl.

SAMPLING OF ASBESTOS FIBRE

- 4. The sampling shall be conducted in accordance with the Standard Sampling and Preparation Procedure for Asbestos Fibre.

PROCEDURE

- 5. (a) Fifty grams of fibre shall be evenly distributed in the cavity of the mold with a screen placed at the bottom of cavity.
- (b) A load of 100 psi shall be applied for one minute and released.
- (c) The compressed pad of fibre and supporting screen shall be removed from the mold as a unit and placed into the tray containing 1500 cc of resin solution of 20 per cent solids and 80 per cent alcohol content.
- (d) Pad of fibre shall be allowed to absorb resin solution and become submersed without assistance.
- (e) After five minutes of soaking the screen and fibre shall be removed and allowed to drain freely without squeezing.
- (f) Upon drying to about 15 per cent solvent content either in the air or low temperature vented oven (120-140°F), the sample shall be tested for resin content by selecting three specimens of about 15 gm each from the impregnated fibre.

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- (g) Since resin content is based on bone dry weight, the specimens shall be dried for 30 minutes at 350°F plus or minus 5°, removed from oven and cooled to room temperature in a dessicator. Weights shall be taken to the nearest 0.01 gm and recorded.
- (h) The specimens shall then be ignited for two hours in a muffle furnace at 900°F followed by one hour at 1480°F plus or minus 20°F.
- (i) The specimen shall be removed from muffle and allowed to cool to room temperature before re weighing to nearest 0.01 gram.

CALCULATION AND REPORT

6. W_b = Bone dry weight after 1/2 hour at 350°F.

W_a = Ash weight after 1 hour at 1480°F.

$$\% \text{ resin solids} = 100 - \left[\frac{W_a}{W_b \times .86} \times 100 \right]$$

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