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PROCEDURES FOR DETERMINING PARTICLE SIZE, PARTICLE SIZE DISTRIBUTION, AND PACKED DENSITY OF POWDERED MATERIALS

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Procedure for Determining Particle Size, Particle Size Distribution, and Packed Density of Powdered Materials

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1. This standard has been approved by the Departments of Defense and is mandatory for use by the Departments of the Army, the Navy, and the Air Force, effective.

2. Recommended corrections, additions, or deletions should be addressed to the Standardization Division, Armed Forces Support Center, Washington 25, D. C.

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1. INTRODUCTION

1. SCOPE

1.1 This standard describes the general methods for determining particle size, particle size distribution, and packed density of powdered materials for conformance with the material requirements of applicable specifications. In the event of conflict between these methods and those in the applicable specification, the latter shall have precedence.

2. REFERENCED DOCUMENTS

2.1 The following specification and drawings of the latest issue in effect shall form a part of this standard:

SPECIFICATION

FEDERAL

RR-S-366 — Sieves, Standard for Testing Purposes.

DRAWINGS

ORDNANCE CORPS

P-72362 — Apparatus, Particle Size.

P-72534 — Density Packing Machine for Pyrotechnic. Ingredients, Assembly.

(Copies of specifications and drawings required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2. SAMPLING AND INSPECTION

1. SCOPE

1.1 This section specifies the procedures for sampling powdered materials.

2. SAMPLING

2.1 Select the required test samples from each lot of the powdered material after the powdered material has been packed and sealed for shipment. Sampling procedures shall be in acordance with the applicable specification.

(*Caution:* Exercise extreme cleanliness in handling samples. Avoid touching the powdered material with damp or soiled hands.)

2.2 Select only samples that are representative of the lot of the powdered material. Special care shall be taken to see that containers being sampled have not been subjected to segregation.

3. PACKING AND MARKING

3.1 **Packing.** Transfer samples to approved airtight rubber-stoppered glass bottles and seal the containers immediately. Keep the containers sealed and stored in a safe location at room temperature until ready for testing.

3.2 **Marking.** Label each powdered material container with the following information:

(a) Powdered material designation.
(b) Lot number.
(c) Pounds in the lot.
(d) Manufacturer's name and plant designation.
(e) Contract number.
(f) Date sample was taken.

4. INSPECTION

4.1 Before testing the powdered material, inspect the sample container to see that it is not broken, unstoppered, or otherwise damaged. Also check that it has been labeled correctly. Discard the contents of damaged or improperly labeled containers and report the condition to the Government inspector (or other proper official) at the plant.

3. TESTING

1. SCOPE

1.1 This section contains the methods of physical tests for powdered material.

1.2 Each test is considered as a separate method, and is assigned an individual method number.

2. NUMBERING SYSTEM

2.1 Test method groups. Methods are arranged in four groups according to category test. These groups are identified numerically by hundreds.

Method 100

DETERMINATION OF AVERAGE PARTICLE SIZE, 2 to 100 MICRONS (FISHER SUB SIEVE SIZER)

1. SCOPE

1.1 This method establishes a procedure for the determination of the average particle size of powdered materials.

1.2 Classification. Determination shall be made by use of the Fisher sub-sieve sizer:

Average particle diameter (2 to 35 Microns) (see 4.4.1).

Average particle diameter (35 to 100 Microns) (see 4.4.2).

2. DEFINITION

The average particle size of powdered ingredients, as determined by the air permeability method, is an average surface weighted diameter and, is a measure of the surface area per unit weight of powder. Any deviation of the particles from sphericity would increase the surface per unit weight of material. Thus, the addition of irregular particles to a sample of perfectly spherical particles would increase the average surface of the sample and consequently decrease the average particle diameter.

3. GENERAL STATEMENTS OR REQUIREMENTS

3.1 Principles of the test. The measurement of particle size by means of the Fisher sub-sieve sizer is based on the fact that gas flowing through a bed of fine-grained powder will be impeded by the external surface of the particles of the powder. The rate of flow of gas through the bed of particles is dependent upon the quantity of powder per unit area of bed, the degree of compaction of powder, and the surface area of the powdered material. The resistance offered by a sample of powdered material to a flow of air defines the average surface weighted diameter. The Gooden and Smith equation is a rapid method for calculating the average particle size of powdered material (see 4.4.2.3). The paraticle size obtained is not an absolute value. It is useful, however, for comparisons of average particle sizes based on surface area measurements. The validity of the particle size comparisons based on the Gooden and Smith equation partly on the theoretical assumptions of the equation.

3.2 Criteria for passing test.

The sample shall comply with the criteria for acceptability as specified in the specification(s) for the item.

4. DETAILED STATEMENTS OR REQUIREMENTS

4.1 Equipment.

4.1.1 *The Fisher sub-sieve sizer* (see fig. 2).

4.1.2 *Supplementary chart* (see fig. 3). A supplementary chart shall be added to the Fisher Sub Sieve Sizer in order to extend its use. This chart, based on the Gooden and Smith equation shall be used for materials having a porosity less than 0.40. It is a graphical representation of the equation when P, K, N, and A (see 4.4.2.3) are considered constant. The chart also serves as a centimeter scale for pressure readings in the calibration of the apparatus by the Fisher sub-sieve calibrator method, and in the determination of average particle diameters between 35 to 100 microns. The value of "P" is taken to be equal to the height of water in the standpipe. The loss of air through the double range flowmeter is

greater than through the single range flowmeter. Consequently, the value of "P" for the double range scale is less than that of "P" for the single range scale.

4.1.3 *Nomogram* (see fig. 4). A nomgram based on the Gooden and Smith equation, shall be added to the Fisher Sub Sieve Sizer in order to extend its use to 35 to 100 microns.

4.1.4 *Equipment for calibration and constant:*

(a) Wet test meter (see 5.1).

(b) Automatic timer.

(c) Supplementary chart.

(d) Calibrator (Fisher sub-sieve calibrator or equivalent).

4.2 **Maintenance of the instrument and general precautions.** Before the Fisher Sub Sieve Sizer shall be used for making any measurements, the following settings shall be checked:

(a) With the sample tube removed from the instrument, the level of the water in the pressure regulator standpipe shall be checked daily. The water level shall correspond exactly with the etched calibration mark on the standpipe. If necessary, water shall be removed or added to the standpipe through the glass intake arm extending through the top of the instrument cabinet.

(b) The indicating type drying agent shall be checked once every week or month, depending upon how often the instrument shall be used. If the agent is blue in color, it shall be satisfactory. When the color turns pink, the agent shall be replaced with fresh stock.

(c) The sample packing assembly shall be checked about once every month or whenever there is reason to suspect that the brass post beneath the rack and pinion has shifted in position. This adjustment shall be made by inserting both porous plugs and paper disks into the sample tube. The sample tube shall then be placed on the brass post with the lower plug in contact with the upper end of the post. With the rack on the front panel turned down, the two plugs shall be in contact when the crossbar attached to the upper end of the rack shall coincide exactly with the baseline on the calculator chart. If the plugs are not in contact or the pointer does not coincide with the baseline, adjustment shall be necessary. Adjustment of the packing assembly shall be made by loosening the setscrew holding the lower brass post in its mount. The height of the post shall then be such that the crossbar pointer coincides exactly with the baseline of the calculator chart when the porous plugs are held firmly between the brass post and flat lower end of the rack.

(d) The level of the water in the manometer shall be checked every time a new series of particle size determinations is made. The initial level of the water meniscus in the manometer tube shall coincide exactly with the upper edge of the curved portion of the metal crossbar when the tip of the pointer coincides exactly with the baseline of the calculator chart. If necessary, adjustment of the manometer level shall be made by turning the manometer control knob. It is important to note, that the sample tube shall be removed from the instrument when either checking or adjusting the level of the water in the manometer tube.

(e) Any measurement of water level in manometer which is made with the air pump operating shall be made with airflow at a constant head. The flow of air shall be controlled by adjusting the control knob so that the bubbles rise in the standpipe at the rate of two or three per second.

(f) All measurements taken of the liquid level in the manometer on the front of the instrument shall be made with the upper edge of the curved portion of the crossbar set at the meniscus of the liquid. Readings on the manufacturer's chart or supplementary chart shall then be made with reference to the extreme tip of the pointer on the crossbar. Parallax shall be avoided when making these settings and readings.

4.3 Standardization.

4.3.1 *Fisher sub-sieve calibrator* (see 5.2) (see fig. 17). The calibrator is a synthetic ruby jewel with a precise orifice, mounted in a tube similar to a sample tube. It is a secondary standard which has been checked on a standard sub-sieve sizer (one that has previously been set to the National Bureau of Standards cement sample as the primary standard). The readings obtained are then engraved on the calibrator for a given porosity.

Procedure. The calibrator shall be used in the Fisher sub-sieve sizer in exactly the same manner as the normal sample tube with the sample all ready in place and packed. The calculator chart shall be set to indicate a porosity of 0.75 and the level of the liquid meniscus in the manometer shall be checked as described in section 4.2. The reading marked high shall be obtained with the range control set to the 0.2 to 20.0 micron range. The reading marked low shall be obtained with the range control set to the 20 to 50 micron range. In the event the readings obtained with the calibrator do not agree within 0.05 micron of the values stamped on the calibrator, the apparatus shall be standardized. The equivalent manometric deflections shall be obtained by adjusting the fine resistance wires in the capillary flowmeters of the Fisher sub-sieve sizer (see sec. 4.3.2).

4.3.2 *Procedure for adjustment of wires.* To adjust the wires, the small screw in the cap of the flowmeter shall be loosened and the wires, held by tweezers, shall be lengthened or shortened by exerting a firm but gentle pressure up or down. If the manometric deflection is to be raised, the wire shall be pressed upward into the capillary tube; if the deflection is to be lowered, the wire shall be pulled down. The screws shall be tightened and new readings shall be taken after the water has reached its maximum height. This procedure shall be repeated until the values obtained for P on the instrument correspond to the values on the calibrator.

4.4 Test procedures. Determination of particle size by means of the Fisher sub-sieve sizer shall be divided into two procedures according to the size of the average particle diameter (see 1.2). Before the Fisher sub-sieve sizer shall be used for making any determinations, the instrument settings and precautions noted in 4.2 shall be checked and the instrument shall have been recently checked with the calibrator.

4.4.1 Average particle diameter between 2 and 35 microns. The sample tube (see fig. 1) shall be placed in a vertical position in a rubber support stand. A paper disk shall be placed over the end of the sample tube. The porous plug shall then be placed on the filter paper with the perforated surface of the plug against the surface of the paper disk. The plug shall be pushed into the tube for about ½ inch, forcing the paper to crimp around the edges and precede the plug into the sample tube. The sample tube shall be inverted in the rubber support stand so that the

paper side of the plug is up. A sample of dry powder shall be weighed to + 0.01 gm. The weight of the sample shall be always equal in grams to the true density of the sample when either of the calculator charts is used. For example, 1.74 ± 0.01 gm of magnesium (density equal to 1.74) would be the weight of a powdered magnesium sample. With the aid of a funnel, the sample shall be transferred completely into the tube. A second paper disk and porous plug shall be placed into the sample tube as previously described. The sample, which is now contained between the filter papers and plugs, shall be pressed manually as tightly as possible with the aid of the plug manipulator or a metal or plastic rod. The sample, pressed as tightly as possible in this way is said to be at "minimum porosity." The sample tube shall be placed on the brass post with the lower plug in contact with the brass post. The pinion knob shall then be turned as far as it will go. The calculator chart shall be shifted laterally until the tip of the pointer coincides with the sample height curve on the chart. If the sample being tested has a porosity less than 0.40, the supplementary chart shall be used. This chart shall be attached to the back of the Fisher calculator chart. The supplementary chart shall be adjusted by aligning the baseline of the chart with the tip of the pointer. This adjustment shall be made while both plugs and both paper disks are in place in the empty sample tube. When using the supplementary chart, the height of the sample shall be read directly in cm on the ordinate scale. The chart shall be moved laterally until the tip of the pointer corresponds to the sample height line on the chart. The chart is constructed so that at this point the abscissa reading shall be equal to the ordinate reading. Once the height of the sample is determined, the calculator chart shall not be moved throughout the remainder of the determination. The sample tube shall then be mounted between the rubber-cushioned supports to the right of the brass post. The upper cap shall be screwed onto the sample tube until an airtight seal is obtained at both ends. Air at constant head shall then be passed through the sample compacted to a minimum porosity. The flow of air shall be started by throwing the electrical switch which starts the air pump. The flow of air shall be adjusted by turning the pressure control knob until bubbles rise in the standpipe at the rate of two or three per second. The liquid level in the manometer shall be allowed to rise to its maximum height. For fine powders the rise of liquid is slow; for coarse powders the rise is rapid. With the liquid level at a maximum height, the rack shall be turned until the upper edge of the crossbar coincides with the liquid meniscus in the manometer. The particle size shall be indicated directly by the location of the tip of the pointer with relation to the calculator charts. The charts shall be interpolated if the precision of the particular operation warrants it. If the average particle diameter falls in the range of 0.2 to 17.0 microns, the charts shall be read directly with the range control indicator turned to the right ("LO" position). If the particle size is in the range of 17.0 to 35 microns, the range control indicator shall be turned to the left ("HI" position), and the chart reading multiplied by two.

4.4.2 Average particle diameter between 35 and 100 microns.

4.4.2.1 *Permeability constant.* Determination of the permeability constant of the single range flowmeter. The Fisher sub-sieve sizer shall be assembled as specified in 4.4.1. The air-permeability constants of the flowmeters shall then be determined by measuring the volume of air passing through the flowmeter per unit time and pressure. The wet test meter shall be connected to the bottom end of the capillary flowmeter tubes. Care shall be taken not to damage the fine wires extending from the capillaries or to block the holes on the side of the flowmeter (see fig. 15). The sample tube containing both plugs and filter paper disks shall be placed in the Fisher sub-sieve sizer. The range control indicator, located in the upper

right corner of the Fisher sub-sieve sizer, shall be turned to the right or "read directly" position to permit calibration of the single range flowmeter tube. Air shall be passed through the flowmeter tube for approximately 15 minutes to determine whether the wet-test meter is functioning. The volume reading indicated on the dial of the wet test meter and the time of the reading as indicated on the timer, V_1, and T_1, respectively, shall be recorded. The height of the water in the manometer on the front panel of the instrument shall be read on the centimeter scale of the supplementary chart to + 0.05 cm. To obtain this value, the meniscus of the water in the manometer shall be level with the crossbar as the height of the tip of the pointer is read. This reading shall be approximately 24.5 cm for the single range flowmeter. The manometer reading shall be multiplied by 2 to obtain the air pressure, P. After approximately 1 hour the column reading and the time of the second reading (in minutes), V_2 and T_2, respectively, shall be recorded. The permeability constant, K, of the single range flowmeter shall be calculated by means of the following equation:

$$K = 1.263 \times 10^3 \cdot \sqrt{\frac{V_2 - V_1}{(T_2 - T_1)\,P}}$$

where:

K = permeability constant of the single range flowmeter.

V_1 = volume at first reading (cu. ft.).

V_2 = volume at second reading (cu. ft.).

T_1 = time of first reading (minutes).

T_2 = time of second reading (minutes).

P = air pressure (cm).

4.4.2.2 Determination of the permeability constant for the double range flowmeter. To determine the permeability constant for the double range flowmeter, the range control indicator shall be turned to the left or "read double" scale. The procedure outline in 4.4.2.1 shall be repeated. The permeability constants obtained for the single and double-range flowmeter tubes should correspond to the values obtained for a standard instrument. The standard permeability constants are 3.86 cms 3/2 and 7.60 3/2 for the single and double-range flowmeter tubes, respectively.

4.4.2.3 *Procedure.* The average particle size of materials having a diameter between 35 and 100 microns, when determined with the Fisher sub-sieve sizer, shall be calculated by means of the Gooden and Smith equation described below. The equation variables shall be determined with the aid of the supplementary chart. To determine the equation variables, the standard procedure described in 4.4.1 shall include the following additions and modifications:

(a) A sample of dry powder equal in weight to three times the true density, shall be weighed to ± 0.01 gm.

(b) The sample shall be placed in the sample tube as described in 4.4.1. The height of the sample in centimeters, shall be read directly on the ordinate scale of the supplementary chart. The chart shall not be moved.

The height of the water in the manometer in centimeters, shall also be read directly from the ordinate scale of the supplementary chart, following the procedure outlined in 4.4.1. The average particle diameter of the sample shall then be calculated by substituting in the Gooden and Smith Equation.

$$\mu = \frac{KHN}{(AH - N)\,3/2} \cdot \sqrt{\frac{F}{P - F}}$$

where:

μ = Average particle size, microns.

A = Cross sectional area of the sample tube, square centimeters. (1.27)

H = Height of sample, centimeters.

P = Height of water column in mano-

meter tube, centimeters, when sample tube contains two porous plugs and two filter disks only.

F = Height of water column in manometer tube, centimeters, when the sample tube contains plugs, filter disks, and sample.

K = Experimentally determined permeability constant for flowmeter, centimeters 3/2.

N = A factor which is equivalent to the weight of the sample divided by its true density.

A nomogram (see fig. 4), based on the Gooden and Smith equation, has been constructed to simplify the calculation of the average particle size of materials having a diameter greater than 30 microns. This nomogram can be used when the weight of the sample, in grams, is equal to three times the true density of the sample material. The directions for using the nomogram are as follows:

A straight edge shall be placed on the sample pressure value, F, and the sample height value, H. The point at which the straight edge crosses the "μ" scale is the average particle diameter corresponding to the experimental manometer deflection, F, and to the sample height H. When the double range flowmeter is used the value read on the "μ" scale shall be doubled to obtain the average particle diameter on the sample.

5. GENERAL INFORMATION

5.1 A precision Sargent wet-test meter has been found to be satisfactory for test.

5.2 Calibrator, Catalogue No. 14-313-7 of the Fisher Scientific Company, New York, N. Y., has been found satisfactory. (see 4.3)

Method 200

DETERMINATION OF AVERAGE PARTICLE SIZE 100 TO 1000 MICRONS (PICATINNY ARSENAL PARTICLE SIZER)

1. SCOPE

1.1 This method establishes a procedure for the determination of the average particle size of powdered materials which are in the 100 to 1000 microns range.

2. SPECIMEN

2.1 The operator should select a sample size and porosity tube to give a manometer deflection between 15 and 35 centimeters (cms). The sample size is a multiple of the density, usually 10 times, weighted to plus or minus 0.10 gram (gm).

3. DEFINITION (see method 100)

4. GENERAL STATEMENTS OR REQUIREMENTS

4.1 Principle of the test (see Drawing P-72362). The Picatinny Arsenal particle sizer shall be used for determination of powdered material in the sieve particle size range of 100 to 1000 microns. It uses the Gooden and Smith air-permeability method of measuring the specific surfaces of fine powders. A sensitive variable flowmeter on the Picatinny Arsenal particle sizer extends its range beyond that of the Fisher sub-sieve sizer. Air shall be passed into the apparatus through a reducing valve at approximately 2 pounds pressure. The air inlet shall lead to a drying bottle which shall be connected to both a standpipe and a sample tube. The standpipe shall be filled with water and shall fix the pressure of the air at a point of introduction to the sample. The excess pressure shall be released as bubbles escaping through the water. The air at this constant pressure flows downward through the sample tube in which the powder to be tested is contained. A porous plug shall support the powder in the tube. After leaving the sample tube, the air shall be divided along two paths: through a porosity tube (fritted-glass diffusion tube), and into a water filled manometer. When a sample of powder shall be added to the sample tube, the difference in pressure as read on the manometer, with and without the sample in the apparatus, shall indicate the resistance of the sample to a constant airflow.

4.2 Criteria for passing test. The sample shall comply with the criteria for acceptability as specified in the specification(s) for the item.

5. EQUIPMENT

5.1 Picatinny Arsenal particle sizer equipment (see Drawing P-72362). The Picatinny Arsenal particle sizer (see figs. 5 and 6) shall be composed of an air pressure regulator, a sample tube (see fig. 7), porous wire mesh plugs (100, 200, 235 mesh), a sample height gauge (see fig. 8), fritted-glass diffusion tubes (coarse, medium, fine), a manometer (see fig. 9), a flowmeter (consisting of the manometer and the resistance formed by the porosity tube), and other accessory equipment necessary to correlate these parts into a unit.

5.2 Nomagrams (see fig. 10, to 14). Nomograms shall be used with the Picatinny Arsenal Particle Sizer.

5.3 The following accessory equipment shall be necessary for determining the permeability constant of fritted-glass diffusion tubes.

(a) Wet-test meter.

(b) Automatic timer.

(c) Fritted-glass diffusion tubes (see 8.2).

6. MAINTENANCE OF THE PICATINNY PARTICLE SIZER.

6.1 Adjustment. Prior to determination of particle size with the Picatinny Arsenal particle sizer, the following adjustments shall be necessary:

(a) The air pressure regulator shall be set at approximately 2 pounds gauge pressure.

(b) The drying bottle shall be filled with an indicating desiccant, such as blue silica gel. The desiccant shall be checked once every week. If it is blue in color, it shall be considered satisfactory. If the color turns to pink, it shall be replaced with fresh stock.

(c) The standpipe shall be filled with water. The water level shall be approximately 5 inches from the top of the standpipe when air shall be passed through the apparatus.

(d) The manometer shall be filled to the "zero" level with distilled water to which a few drops of a color indicator, such as fluorescein green dye, shall be added.

(e) The sample height gauge shall be placed into the sample tube containing only a porous plug. The edge of the sample height gauge crossbar shall read "zero" when the tube contains only the plug.

6.2 Permeability constant.

6.2.1 Determination of the permeability constant for the porosity tubes (the fritted-glass diffusion tube). Since the accuracy and reproducibility of particle size measurements made with the Picatinny Arsenal particle sizer shall depend on calibration of the porosity tubes, it shall be necessary to determine the permeability constants of each tube. Each tube shall be cleaned by washing with 1:1 hydrochloric acid and rinsing with distilled water, and dried for 4 hours by heating in an oven at 110° C. The tubes shall be stored in a desiccator until ready for use. The dried tube shall be clamped in an upright position into the apparatus (see fig. 5) and the open end shall be connected to the wet-test meter. The empty sample tube shall be inserted into the instrument. The air pressure shall be adjusted so that air bubbles escape through the water at the rate of 2 or 3 per second. Air shall be passed through the system for approximately 15 minutes to determine whether the pointer on the wet-test meter is moving at a constant rate. The volume and the time reading, T_1, in minutes, shall be recorded. The top and bottom levels of the water column in the manometer shall be read. The sum of the volume reading on the wet-test meter, V_2, and the time of the second reading, T_2, shall be recorded. The permeability constant of the porosity tube shall be calculated by means of the following equation:

$$K = 1.263 \times 10^3 \cdot \sqrt{\frac{V2 - V1}{(T_2 - T_1)\ P}}$$

where:

K = permeability constant of the porosity tube.

V_1 = volume at first reading (cu. ft.).

V_2 = volume at second reading (cu. ft.).

T_1 = time of first reading (minutes).

T_2 = time of second reading (minutes).

P = air pressure (cm).

Calibration of these tubes shall be repeated once every month. When not in use, the porosity tubes shall be stored in a desiccator.

7. TEST PROCEDURES

7.1 General test procedure. A porous plug shall be placed in the sample tube by gently sliding it into position until it rests at the bottom of the tube. The tube shall be placed in a sample tube holder. A representative sample of powder shall be weighed to plus or minus 0.01 gm. The amount of powder required for analysis depends on the nature of the particles. A multiple of the density of the material, usually 10 times shall be used. With the aid of a funnel, the sample shall be transferred into the sample tube containing a porous plug. The sample tube containing the sample shall be lightly tapped to permit the particles to settle in a uniform bed. Tapping for 1 minute is usually adequate. The sample tube shall then be inserted into the apparatus. Air shall be passed through the system so that 2 or 3 air bubbles per second escape through the water in the standpipe. A porosity tube shall be selected to give a manometric deflection, F, between 15 and 35 cm. The manometric deflection shall be recorded when it has attained a constant value. The permeability constant of the porosity tube, K, shall be noted. The sample tube shall be removed from the apparatus and the height of the sample, H, shall be measured with the sample height gauge. The sample tube shall be emptied, cleaned, and containing only the porous plug used in the above particle size determination, shall again be placed in the apparatus. The air pressure shall be regulated as specified above, and the top and bottom levels in the manometer shall be read. This manometric reading is equivalent to the pressure, P, of the air entering the sample.

7.2 Calculation of particle size. There are two techniques available for determining the average particle size of powdered material.

7.3 Calculation by substituting the proper values in the Gooden and Smith equation:

$$\mu = \text{KHN divided by } (AH\text{–}N)^{\frac{3}{2}} \text{ times } \sqrt{F \text{ divided by } (P - F)}$$

where:

μ = average particle size, microns.

A = cross sectional area of the sample tube, square centimeters.

H = height of sample, cm.

P = initial air pressure, cm; i.e., the pressure of air entering the sample tube.

F = the pressure of air after passing through the sample, cm.

K = experimentally determined permeability constant of the porosity tube, cm 3/2.

N = a factor which is equivalent to the weight of the sample divided by its true density.

7.4 Determination of average particle size by nomograms. Determination of average particle size by means of 3 nomograms where the 2 parts of the equation, the porosity part, KHN divided by (AH–N) 3/2 and the pressure part $\sqrt{F \text{ divided by } (P - F)}$, are equated to α and β, respectively, is as follows:

(a) In Nomogram I, P is aligned with F, and β shall be read on the third scale (see fig. 10).

(b) In Nomogram II, the porosity, K, aligned with the height of the sample, H, where A and N are constant values (A=2.00; N=10, 5, or 1), gives a value for α which shall be read on the third scale (see figs. 11, 12, or 13 as applicable).

(c) In Nomogram III, α is aligned with β and the average particle size or the sample, in microns, shall be read directly on the third scale (see fig. 14).

8. GENERAL INFORMATION

8.1 A precision Sargent Wet-Test Meter has been found to be satisfactory for test.

8.2 Fritted-glass diffusion tubes manufactured by the Corning Glass Works of Corning, N. Y., have been found satisfactory.

Method 300

DETERMINATION OF PARTICLE SIZE DISTRIBUTION BY SIEVE ANALYSIS

1. SCOPE

1.1 This method establishes a procedure for determining the particle size distribution of powdered materials by sieve analysis for materials with the average particle size greater than 35 microns.

2. SPECIMEN

2.1 The sample size shall be 100 gm weighed to within 0.1 gm selected after the material has been passed through the riffle-splitter.

3. GENERAL STATEMENTS OR REQUIREMENTS

3.1 **Principle of the test.** Sieve analysis determines the particle size distribution of materials in the particle size range of 35 to 1000 microns. The test consists of shaking samples of powdered material through a sieve arrangement and weighing the powder collected on each sieve and in the pan.

3.2 **Criteria for passing test.** The sample shall comply with the criterion for acceptability as specified in the specification(s) for the item.

4. EQUIPMENT FOR THE TEST

4.1 **Riffle-splitter** (see 5.1). The riffle-splitter, an apparatus for dividing a sample into statistically equivalent fractions shall be used in the sample preparation.

4.2 **Mechanical shakers.** Either one of the following mechanical shakers shall be used for test:

4.2.1 *"Ro-Tap" apparatus.* The "Ro-Tap" apparatus is a mechanical shaker geared to produce 300 plus or minus 15 gyrations and 150 plus or minus 10 taps of the striker per minute.

4.2.2 *"End-shake" apparatus.* This is a mechanical shaker with a sieve arrangement for half-height and full- height sieves. In this apparatus, the nest of sieves reciprocates in a direction lengthwise of the gear box.

4.3 **Sieves.** U. S. standard sieves conforming to Specification RR-S-366 shall be used for test.

4.4 **Balance.** A balance accurate to plus or minus 0.1 gram (gm.) shall be used.

5. PREPARATION FOR TEST

5.1 **Sample preparation.** The accuracy and precision of the sieve analysis method depend largely on the sampling of the material to be analyzed. Each sample container shall, therefore, be tumbled repeatedly for at least 10 minutes to insure homogeneity of the powdered material. Samples of the material shall be taken from several parts of the container, blended, and then divided with a riffle-splitter (see 4.1) until a homogeneous sample is obtained. One hundred gm of this sample shall be weighed on a balance to within 0.1 gm and set aside for the sieve analysis test.

5.2 **Sieve preparation.** The sieve shall be cleaned and dried thoroughly before being used for sieve analysis. They shall be cleaned by prolonged soaking in water aided by brushing with a camel's-hair brush or, where absolutely necessary, with a fairly limber paint brush. The use of any brush should be

kept to an absolute minimum for screens finer than 270 mesh, since too sturdy a brush may result in irreparable damage to the screen. Suitable solvents may be used except in the case of resinous or plastic materials (unless positively necessary) where evaporation of the contaminated solvent will leave a film of the plastic material and cause serious "blending" in the finer screens. A sharp object shall never be used to remove particles that are lodged in the openings. The sieves used for particle size determination shall be kept exclusively for that purpose.

6. TEST PROCEDURE

6.1 For normal powders. A nest of five sieves shall usually be employed for a sieve analysis. However, the number of sieves and their size shall depend on the distribution and nature of the material being used. If a nest of five sieves is used, the following procedure shall apply:

The sieves shall be selected so that two are coarser than the mean size of the sample (as determined by an air-permeability method), and the remaining sieves shall be finer than the mean size. For example, if a sample has an air permeability mean diameter of 130 microns, the sieves used would be 80, 100, 120, 140, and 170 mesh, respectively. The sieves shall be assembled with the coarsest sieves on top, and the finest sieve fitted to the bottom pan. The rims of the sieves shall then be taped to prevent dusting. The entire nest of sieves shall be placed into a mechanical sieve apparatus such as the "Ro-Tap" (see 4.2.1) or the "End-Shake" (see 4.2.2) and shaken for 30 minutes. If there is evidence of a film of powder adhering to the bottom of a sieve, it shall be brushed with a soft camel's-hair brush or, in the case of more sensitive compositions, a brush with antistatic attachment, and added to the next finer sieve. After the shaking has been completed, the stack of sieves shall be disassembled and the sieve fraction shall be removed from each sieve and placed in a labeled container. A funnel large enough to completely contain the sieve shall be used to minimize the sample loss. The sieve shall be inverted into the top of the funnel and all of the powder particles shall be removed by brushing the screen surface with a suitable brush. Where a film of powder adheres to the bottom of the screen it should be added to the next smaller size screen using a camel's-hair brush (with suitable antistatic attachment for sensitive powders). Each of the sieve fractions shall be weighed to within 0.1 gram.

6.2 For sensitive powders. For powders which may be classified dangerous such as finely divided titanium and zirconium and mixtures containing these powders, the machine should be grounded and wherever possible the operation carried out behind a suitable barricade by remote control. Operators shall observe all safety precautions, including the use of proper clothing. Wet sieving may be used to separate the different sizes where agglomeration due to static charges may be expected. (The type of solvent and directions for drying shall be stated in the specification.) A liquid antistatic agent shall be used to coat the screens, sieves, and walls.

6.3 Calculation. Each of the sieve fractions shall be weighed to within 0.1 gm. The weight percent of the sample retained on each sieve shall then be calculated by dividing the weight of powder on each sieve by the total weight of powder recovered. The cumulative percent of material passing through each sieve shall be determined by adding together the percentage of powder on the finer sieves and pan. The percent of powder finer than the corresponding size of the opening on each sieve, in microns, shall be plotted on log-probability paper against the size of the opening of the respective sieve. A straight line of best fit shall be drawn through the plotted points. The geometric mean, which represents the average size of the particles shall be obtained by reading the 50 percent size. The standard deviation, which represents the distribution of the par-

ticles shall be determined by first reading the 84.1 percent size and dividing this value by the 50 percent size. These values, which characterize the distribution, shall be reported. A sample tabulation of the data is as follows:

U.S. sieve number	Opening of sieve (microns)	Weight on sieve (grams)	Percent on sieve	Percent through sieve
80	177	6.9	7.0	93.0
100	149	22.9	23.0	70.0
120	125	35.8	36.0	34.0
140	105	23.8	23.9	10.1
170	88	9.2	9.2	0.9
(Thru 170 on pan)			0.9	
Total		99.5	100.0	

The geometric mean and standard deviation of this sample are equal to 135 microns and 1.2, respectively.

7. GENERAL INFORMATION

7.1 General information.

7.1.1 The Fisher riffle-splitter has been found to be satisfactory.

7.1.2 The end-shake testing sieve shaker, a product of the Newark Wire Cloth Company of Newark, N. J. has been found to be satisfactory.

Method 400

DETERMINATION OF THE PACKED DENSITY OF POWDERED MATERIALS

1. SCOPE

1.1 This method establishes a procedure for the determination of the packed density of powdered materials.

2. SPECIMEN

2.1 The sample size shall be the weight of the material filled to 100 ml mark weighed to within 0.10 gm.

3. DEFINITION

3.1 Packed density. The packed density of powdered materials is the weight per unit volume of material which has been packed until it has attained its most compact form. Packed density indicates the loading density of loose pyrotechnic powders.

4. GENERAL REQUIREMENTS OR STATEMENTS

4.1 Description of test.

4.1.1 The test consists of an instrumental procedure to determine the packed density of powdered material.

4.1.2 Results obtained using the packed density apparatus specified on Drawing P-72534 will be equivalent to those obtained using "hand-tapped" techniques. However, the instrumental method is safer, more rapid, and has greater precision.

4.2 Criteria for passing test.

4.2.1 The sample shall comply with the criterion for acceptability as specified in the specification(s) for the item.

4.3 Equipment for test.

4.3.1 Packed density apparatus (see fig. 18). The apparatus used for determining the packed density of powdered materials shall be composed of the following parts:

(a) Motor-driven revolving cam assembly.

(b) An automatic timer.

(c) Sample assembly consisting of 100 cubic centimeter (cc.) graduate cylinder and cylinder holder.

4.3.2 *Balance.* A balance accurate to plus or minus 0.1 gm shall be used.

4.4 Test procedure.

4.4.1 *General.* A clean, dry graduate cylinder shall be weighed to plus or minus 0.1 gm on a balance. The graduate cylinder shall be filled to the upper etched mark with the test sample (e.g., the 100-cc mark for a 100-cc graduate cylinder). The cylinder containing the poured sample shall be weighed to plus or minus 0.1 gm. The weight of the sample is the difference between the weight of the graduate with the sample and the empty graduate cylinder. The graduate cylinder containing the sample shall be stoppered and taped to prevent powder from escaping. It shall be inserted in the apparatus by placing it first into the upper circular holder from below and then into the lower holder so that the base of the cylinder rests on the rubber covered block. The apparatus shall be turned on for 10 minutes by means of an automatic timer. This causes the motor to revolve the cam at a constant rate of about 60 revolutions per minute, raising and simultaneously

turning the graduate cylinder part way around before it drops to the rubber covered block. This repeated lifting and dropping jars the material and causes closer packing of the particles. At the end of 10 minutes the timer shall automatically break the circuit. The volume of the sample in the graduate cylinder shall be read and recorded.

4.4.2 *Calculation.* The packed density of the sample shall be calculated by means of the following equation:

Packed density = Weight of sample in the cylinder divided by volume of sample in the cylinder.

Sample calculation:

Weight of graduate cylinder with sample	= 216.6 gm.
Weight of graduate cylinder, empty	= 121.2 gm.
Weight of sample	= 95.4 gm.
Volume of sample in the cylinder after packing	= 82.1 cc.
Packed density	= 95.4 divided by 82.0
Packed density	= 1.16 gm/cc

Copies of specifications, standards, drawings, and publications required by contractors in connection with specific procurement functions may be obtained from the procuring agency or as directed by the contracting officer.

Copies of this standard for military use may be obtained as indicated in the foreward to the Department of Defense Index of Specifications and Standards.

Copies of this standard may be obtained for other than official use by individuals, firms, and contractors from the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C.

Both the title and the identifying symbol number should be stipulated when requesting copies of military standards.

Custodian:
Army—Ord
Navy—WEP
Air Force—WADD

Preparing activity:
Army—Ord

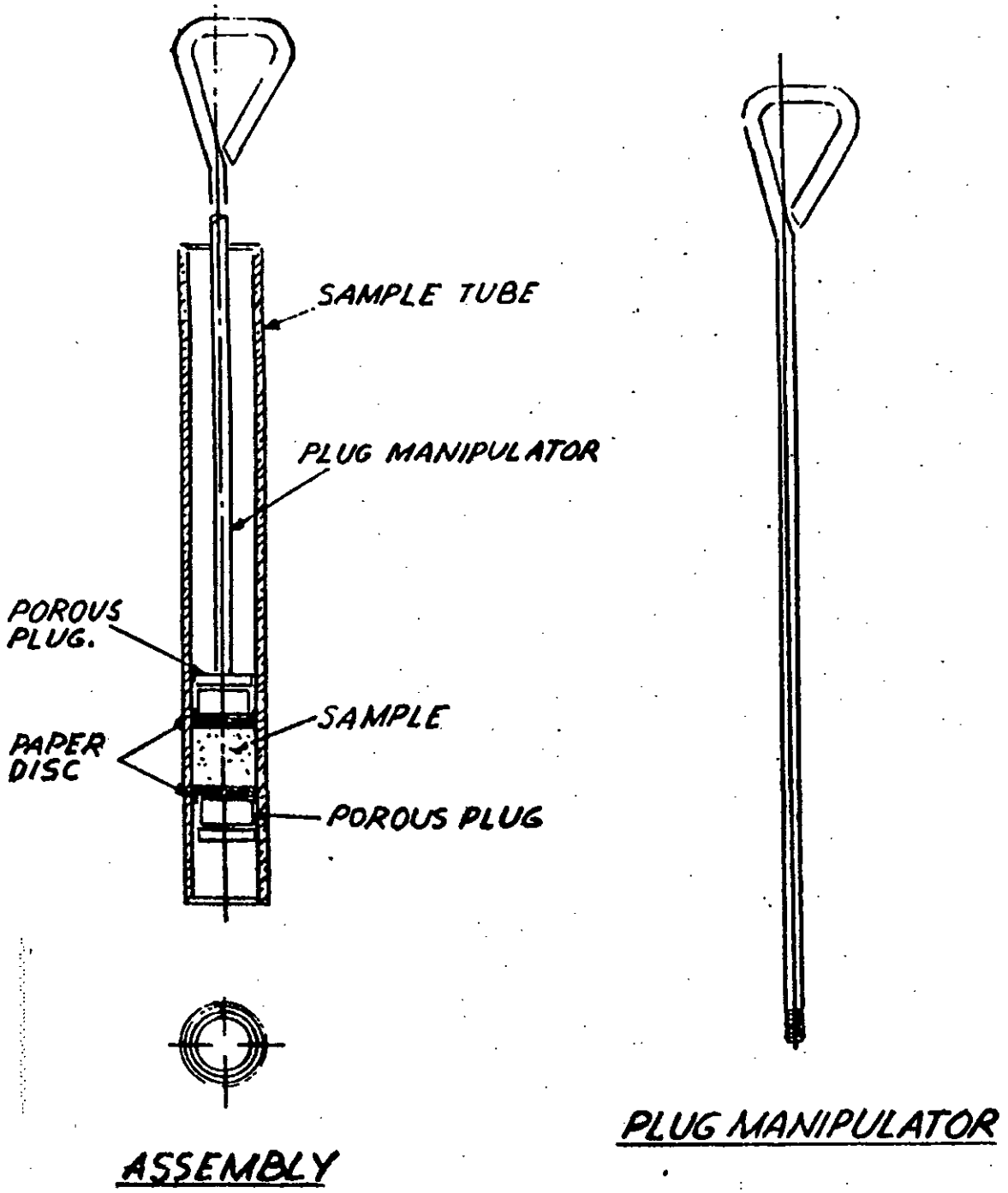


FIGURE 1.

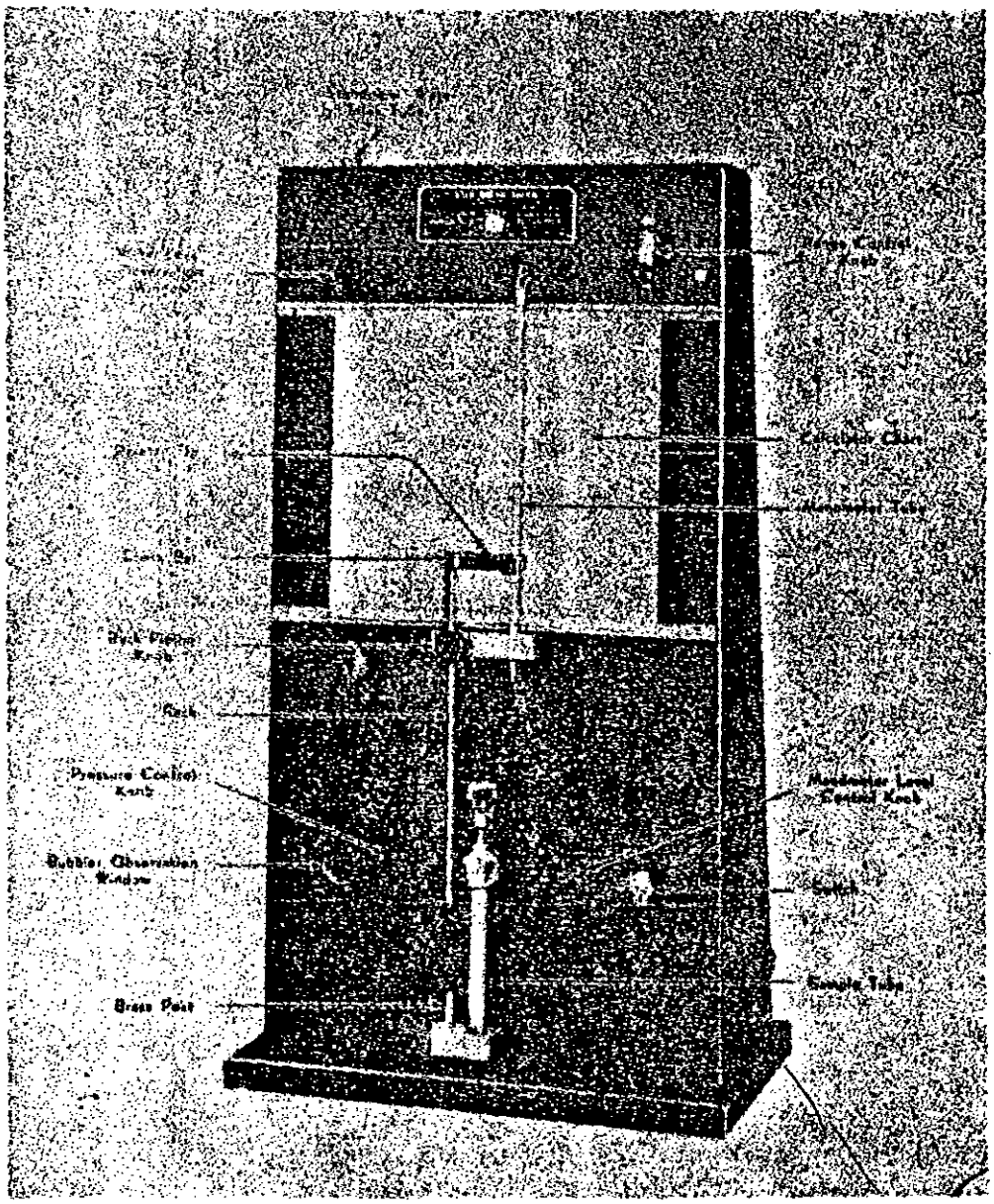


FIGURE 2. *The Fisher sub-sieve sizer.*

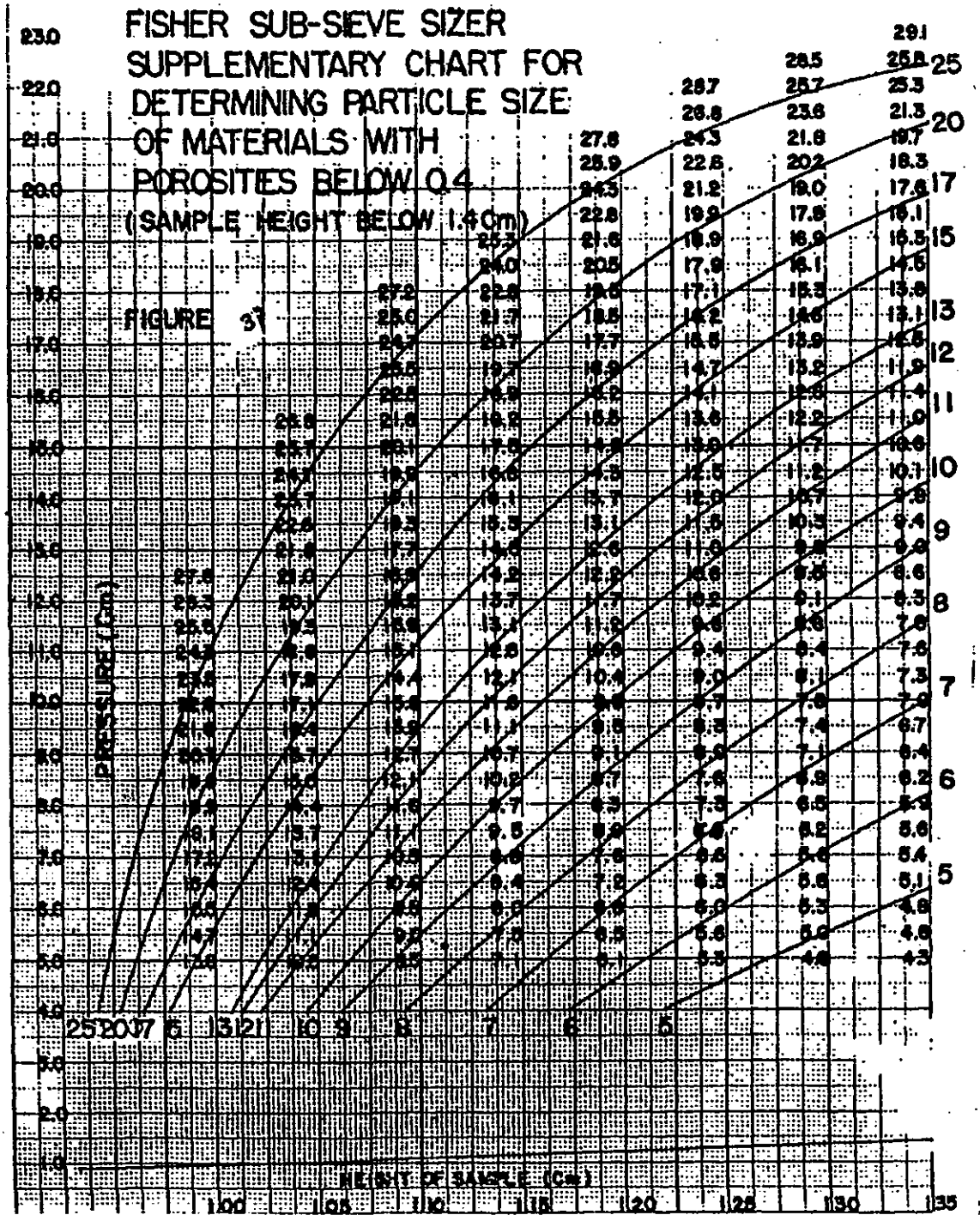


FIGURE 3.

NOMOGRAM FOR AIR PERMEABILITY EQUATION

$$A = \frac{KHN}{(AN-N)} \% \sqrt{\frac{2F}{P-2F}}$$

WHERE N=3, A=1.27, K=3.64 AND P=97.

(FOR USE WITH FISHER SUB SIEVE SIZER)

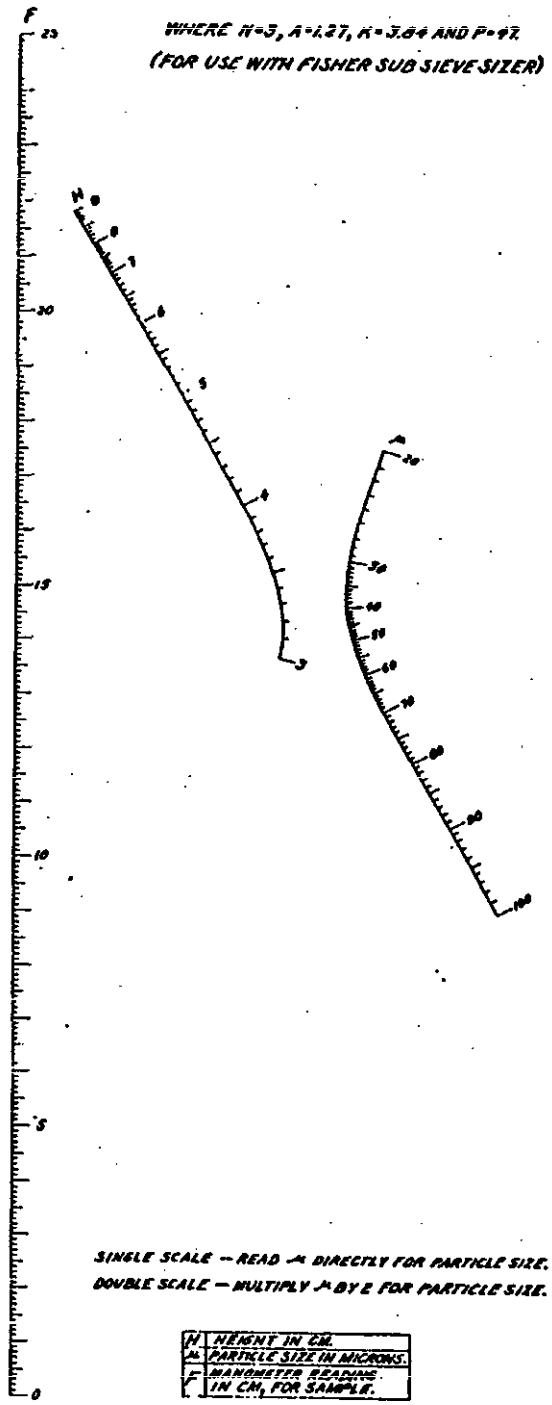


FIGURE 4.

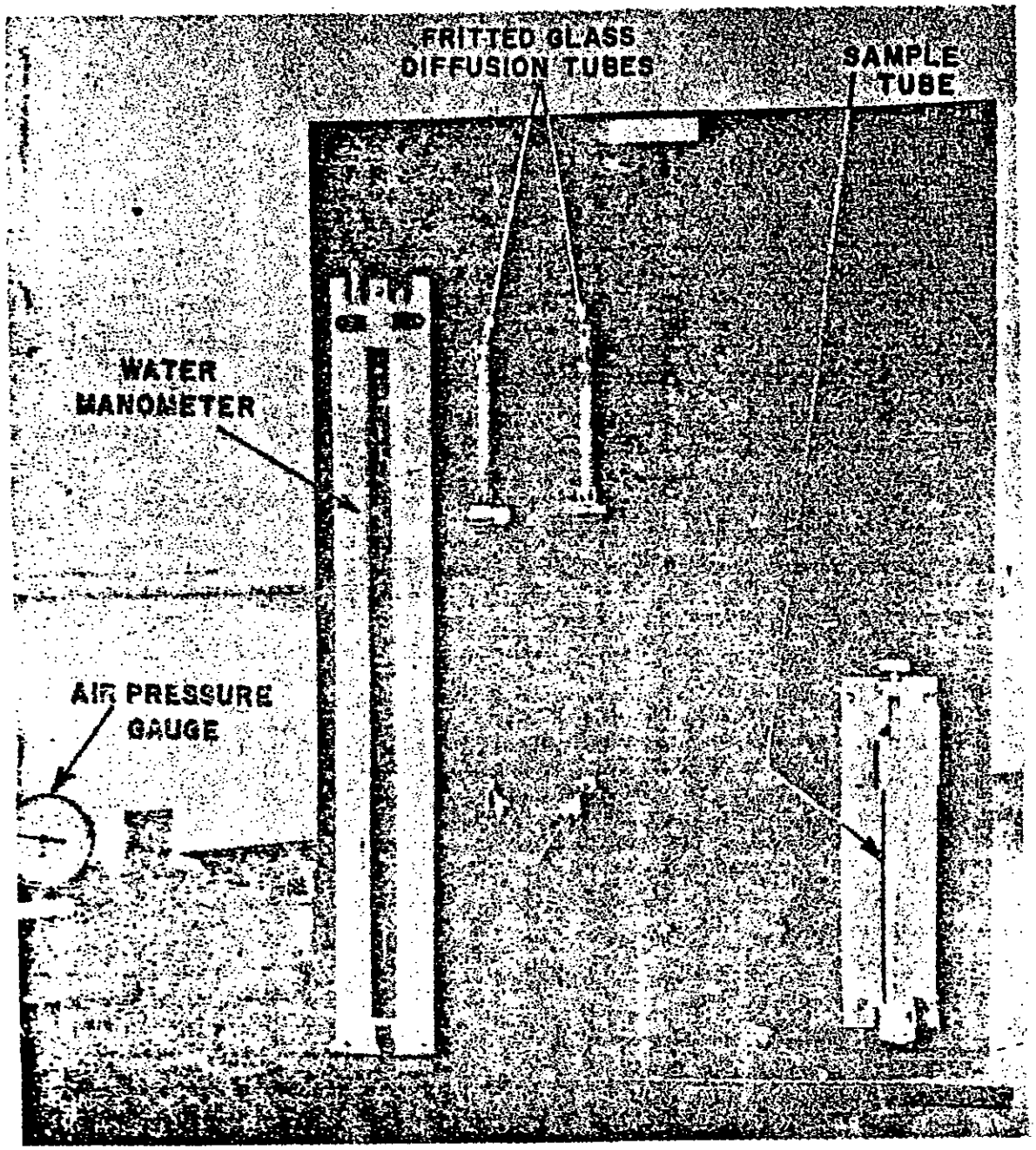


FIGURE 5. *Front view, Picatinny Arsenal particle sizer.*

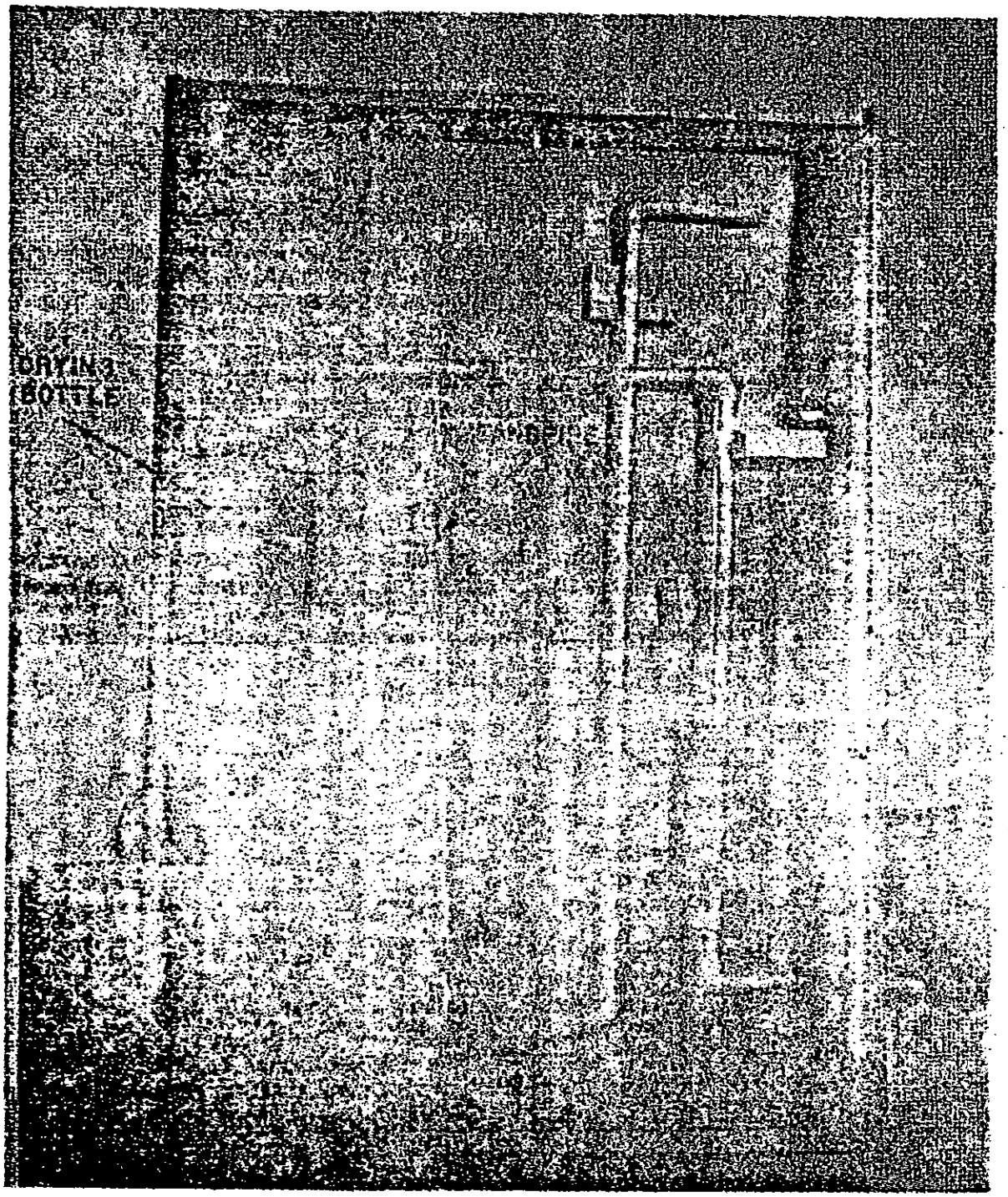


FIGURE 6. *Back view, Picatinny Arsenal particle sizer.*

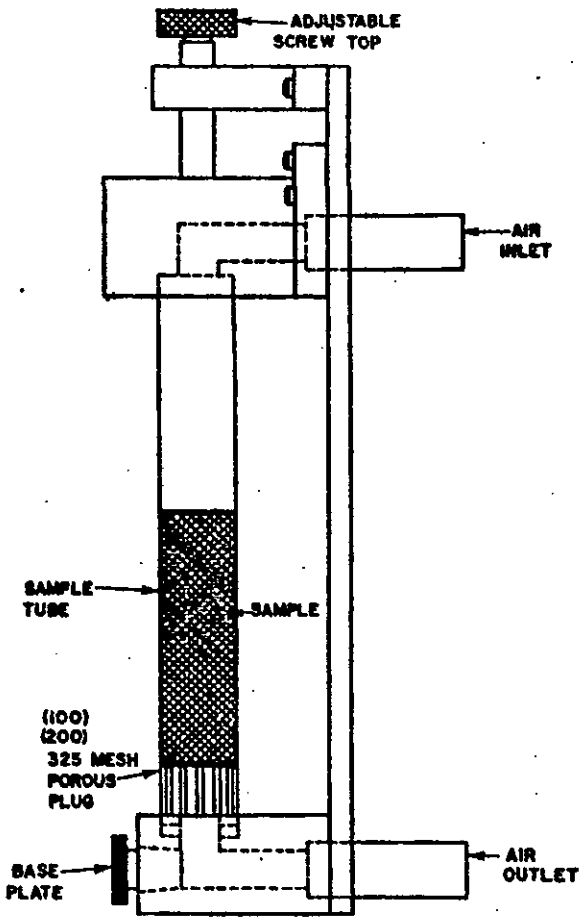


FIGURE 7. *Sample tube assembly, Picatinny Arsenal particle sizer.*

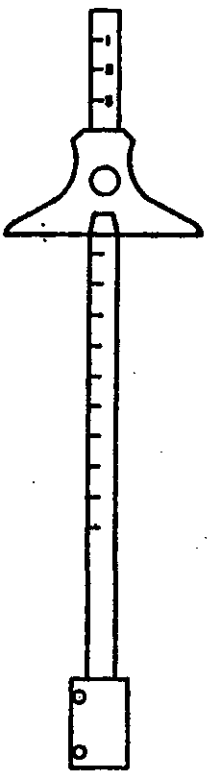


FIGURE 8. *Sample height gauge, Picatinny Arsenal particle sizer.*

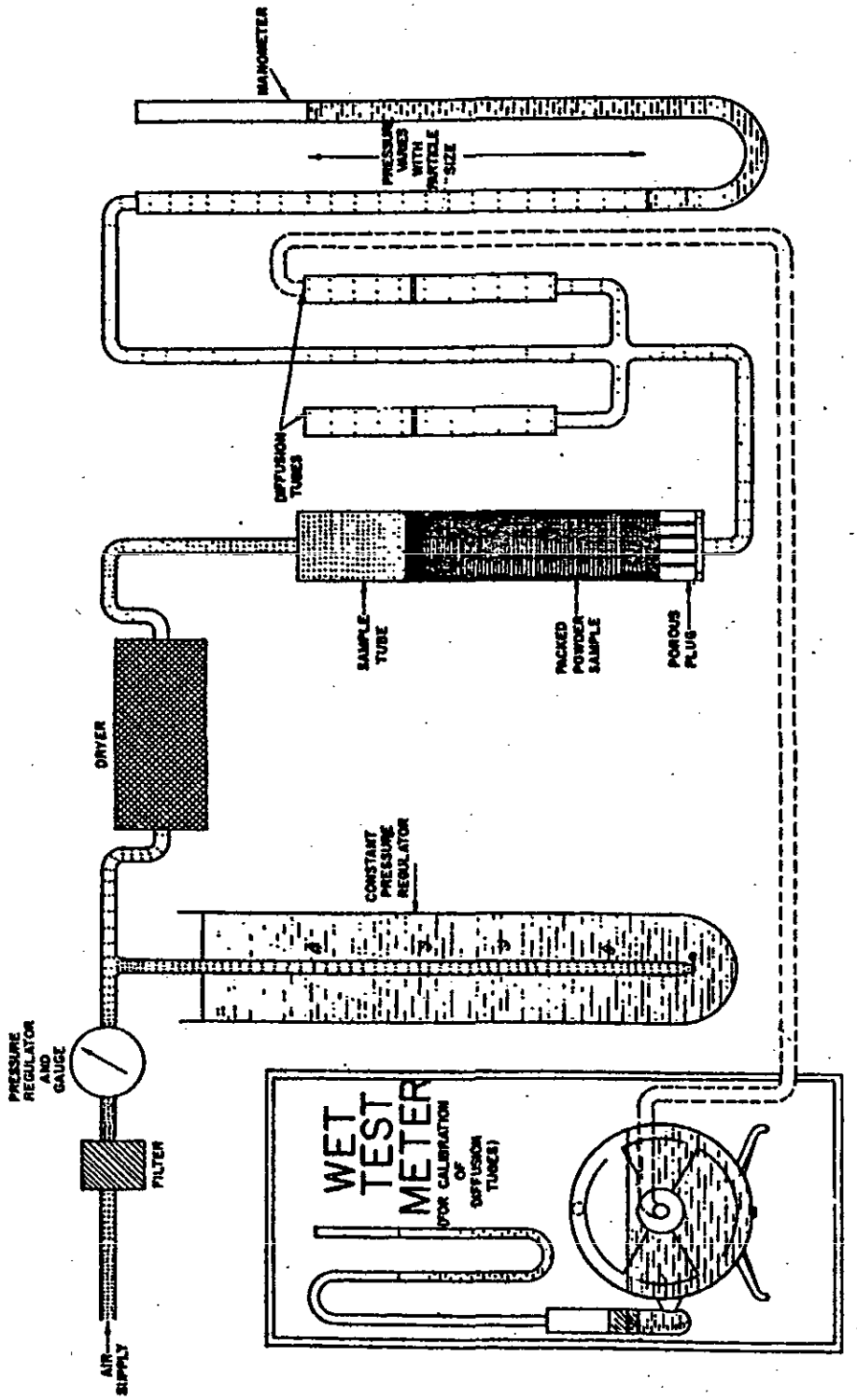
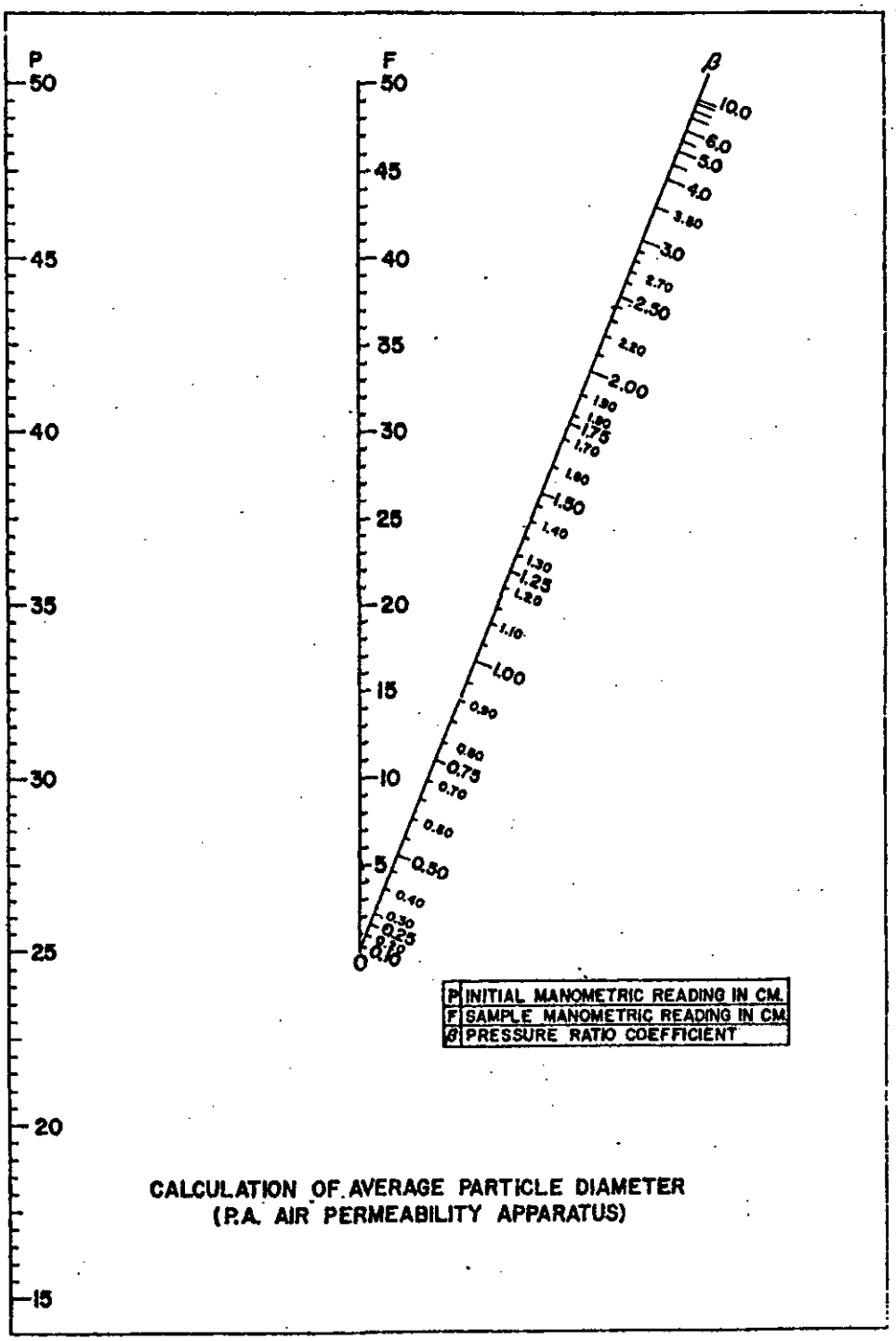


FIGURE 9. *Line diagram, Picatinny Arsenal particle sizer.*

NOMOGRAM I FOR $\beta = \sqrt{\frac{F}{P-F}}$

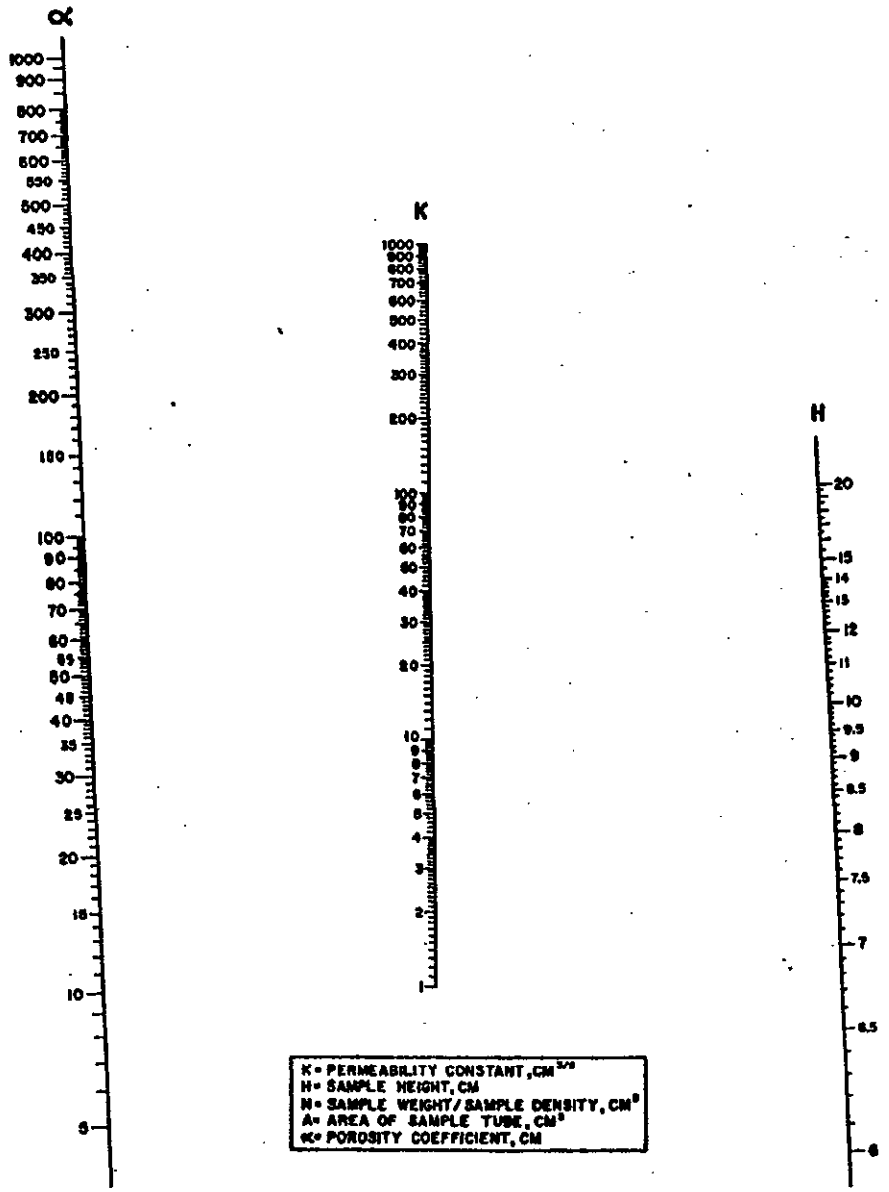


CALCULATION OF AVERAGE PARTICLE DIAMETER
(P.A. AIR PERMEABILITY APPARATUS)

FIGURE 10.

NOMOGRAM IIA FOR $\frac{KHN}{(AH-N)^{1/2}} = \alpha$

WHERE A=2 AND N=10



CALCULATION OF AVERAGE PARTICLE DIAMETER
(F.A. AIR PERMEABILITY APPARATUS)

FIGURE 11.

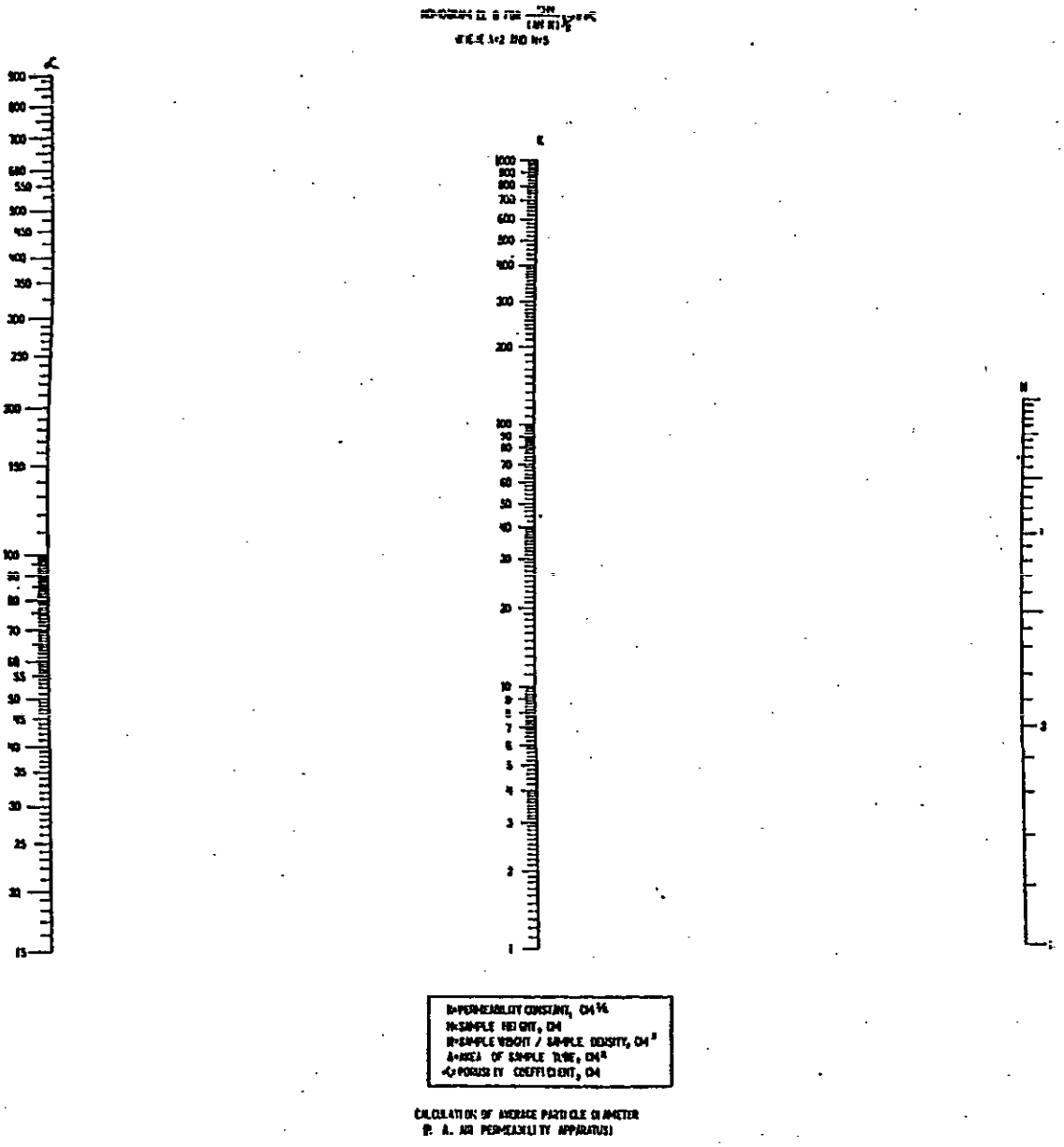


FIGURE 12.

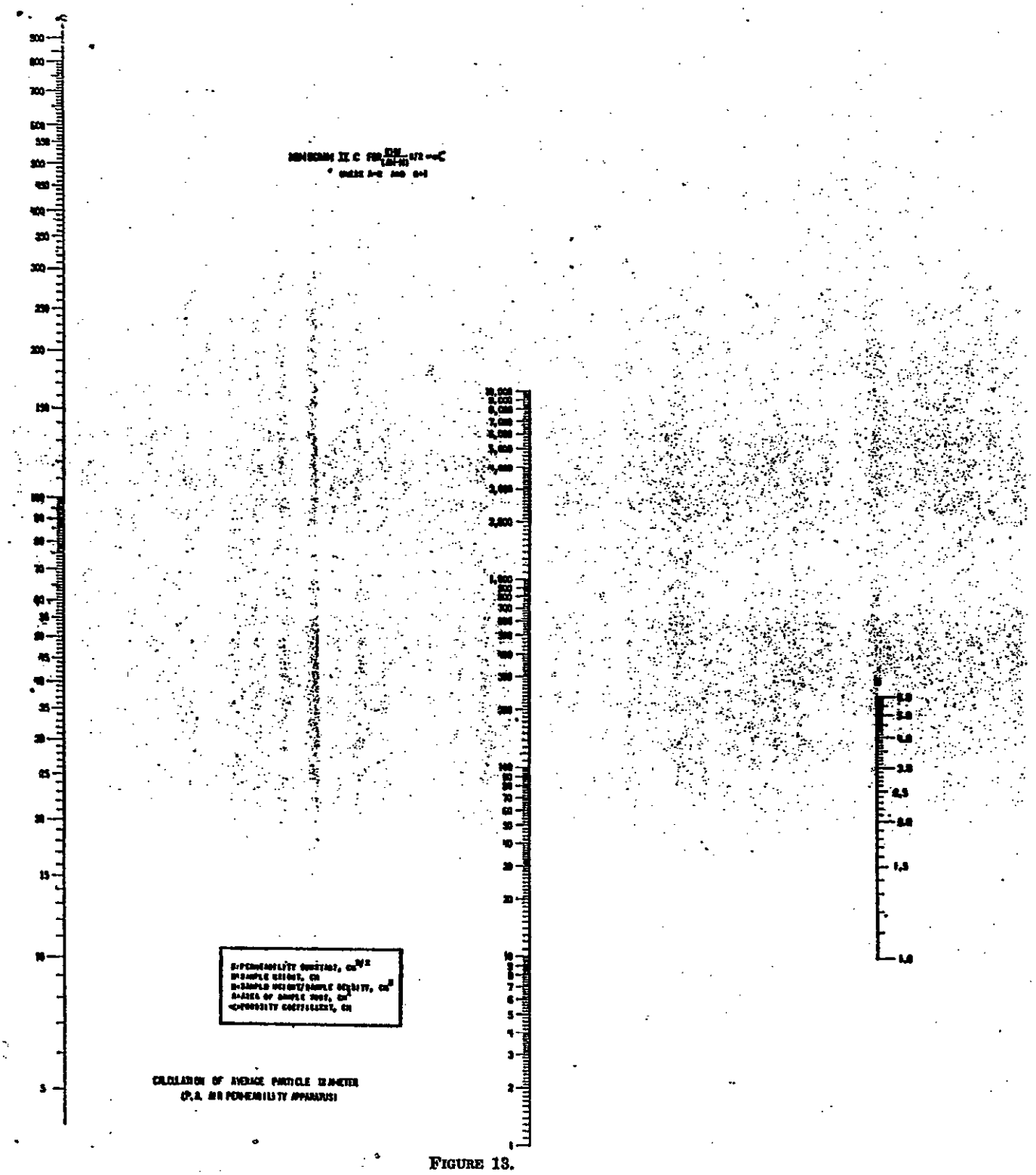


FIGURE 13.

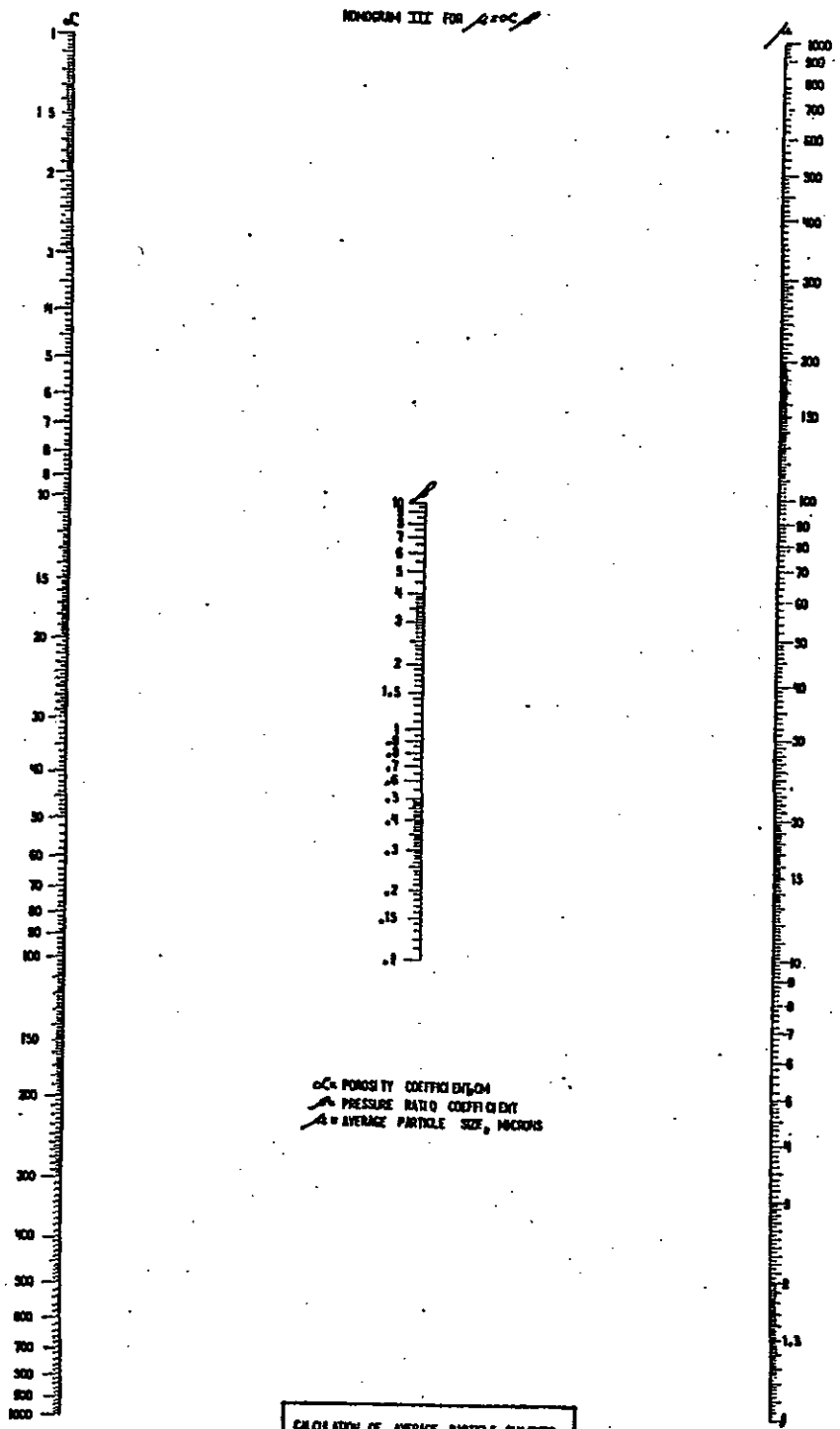


FIGURE 14.

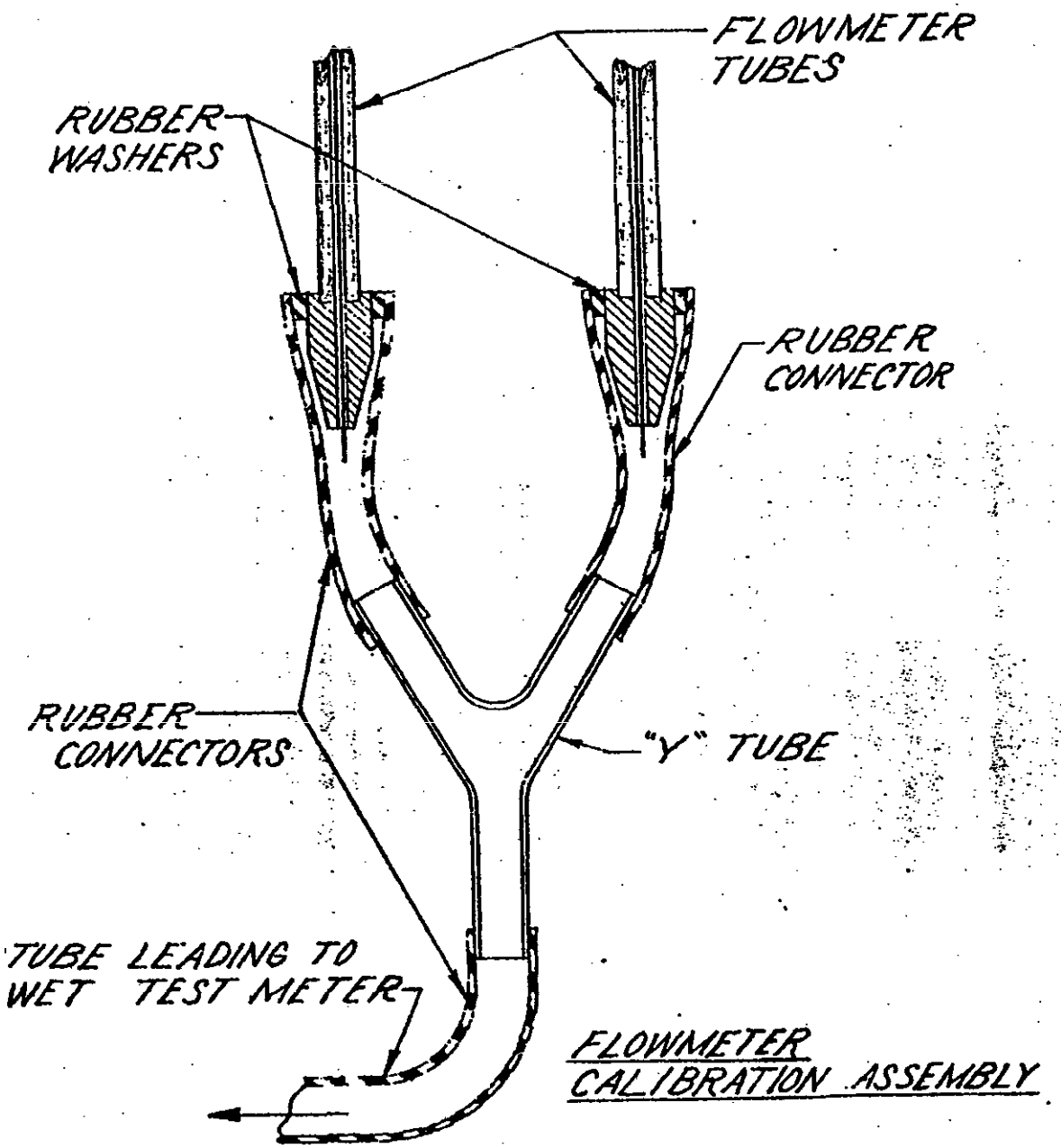


FIGURE 15.

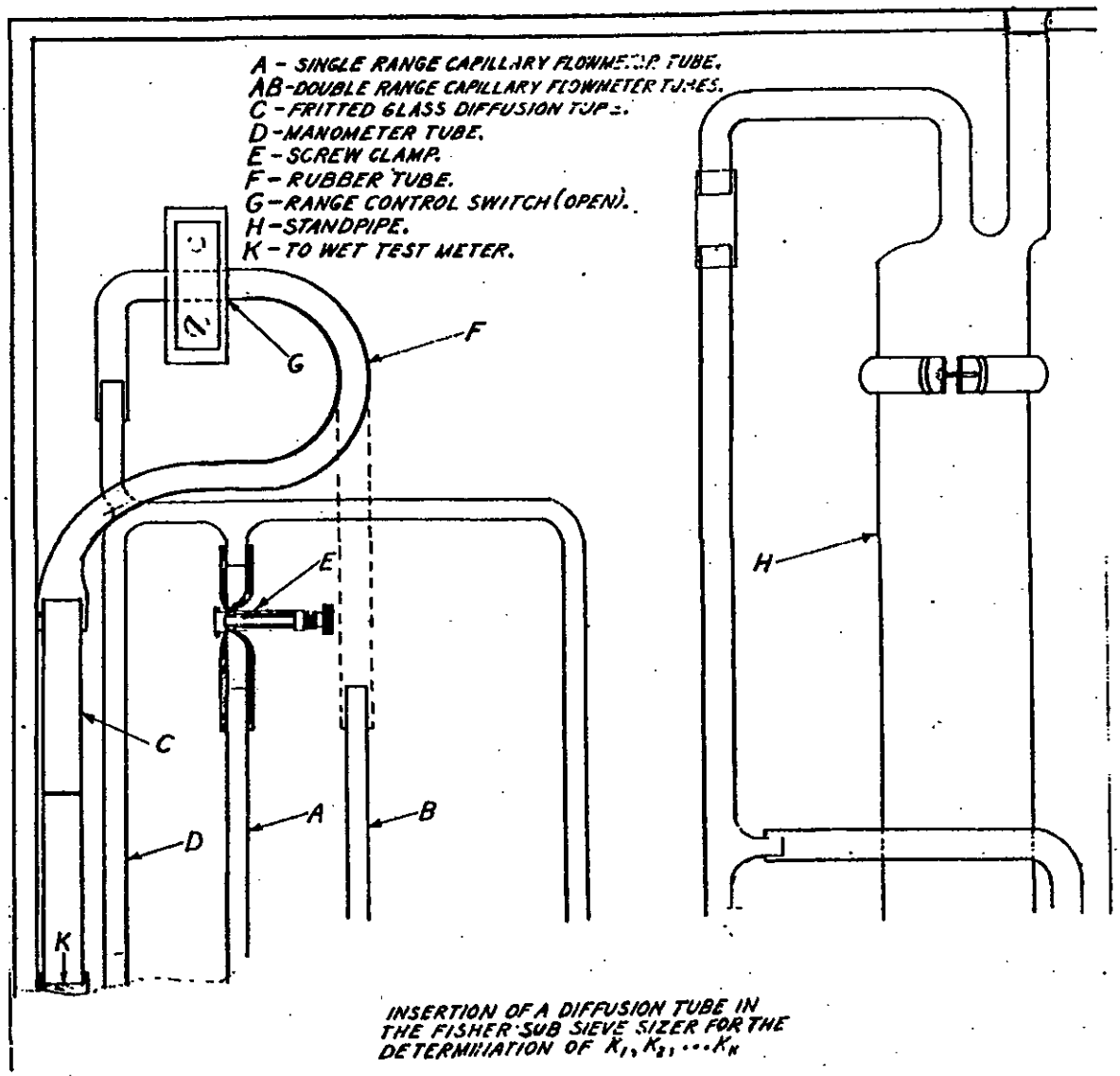


FIGURE 16.

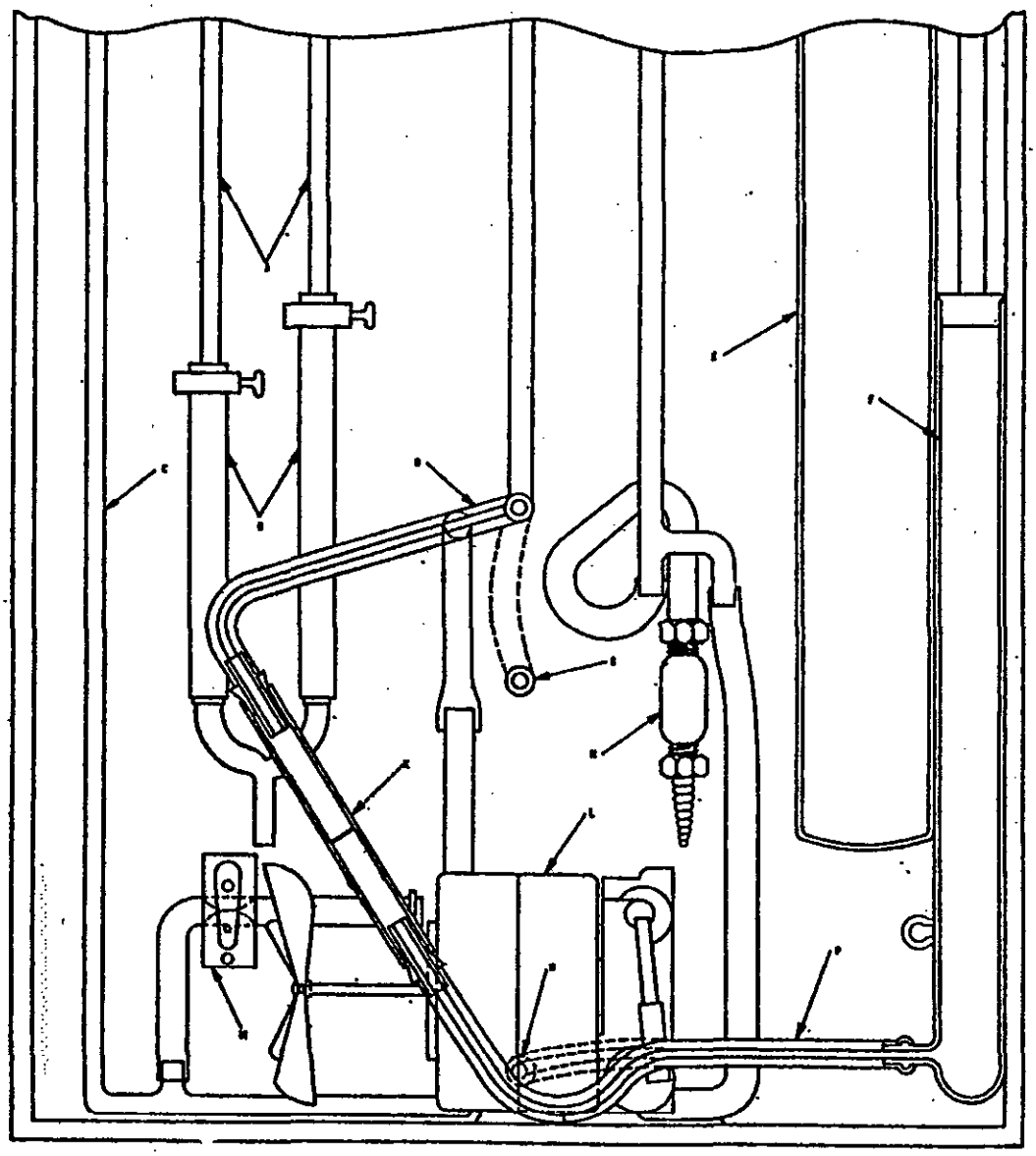


FIGURE 17.

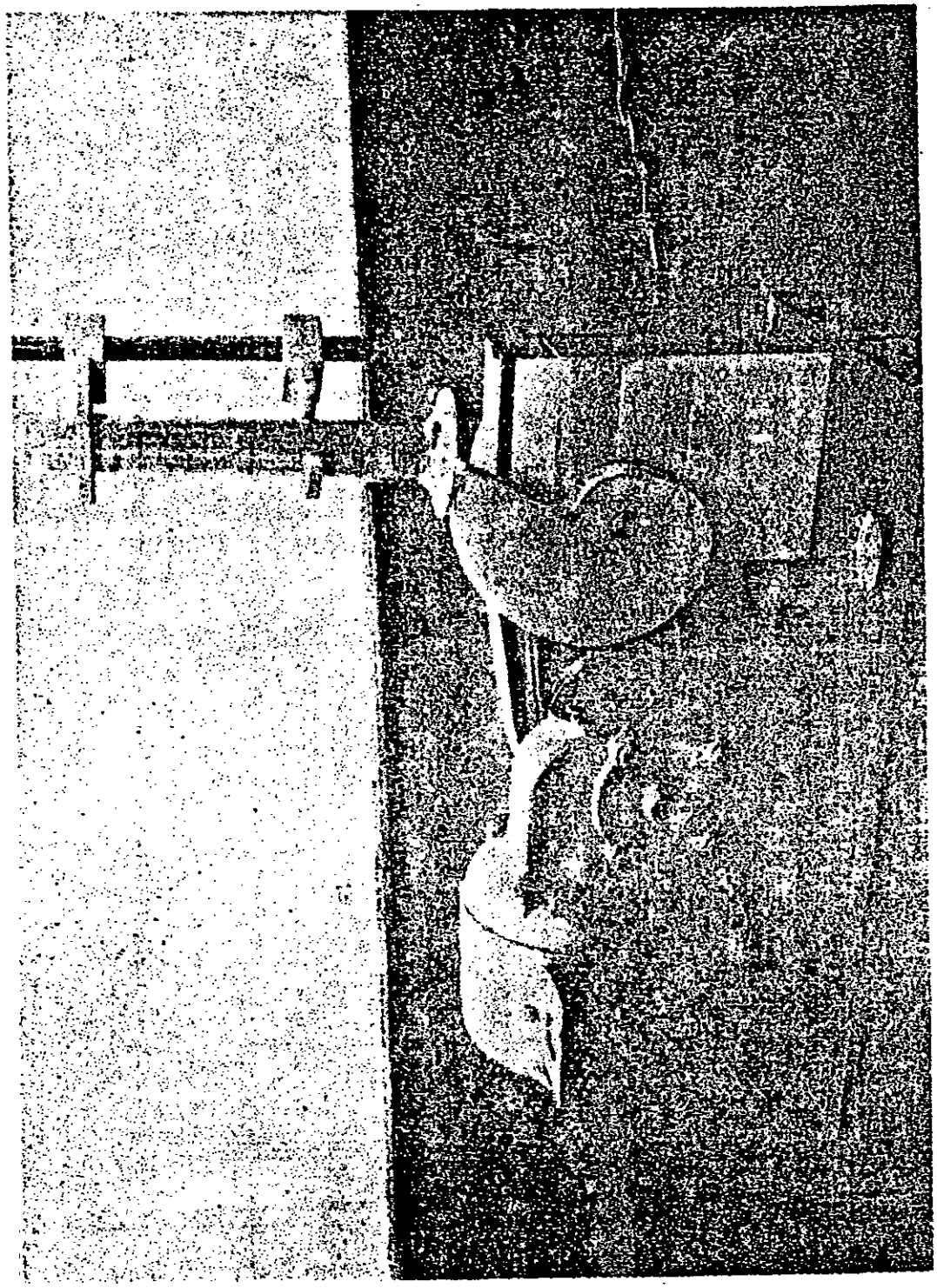
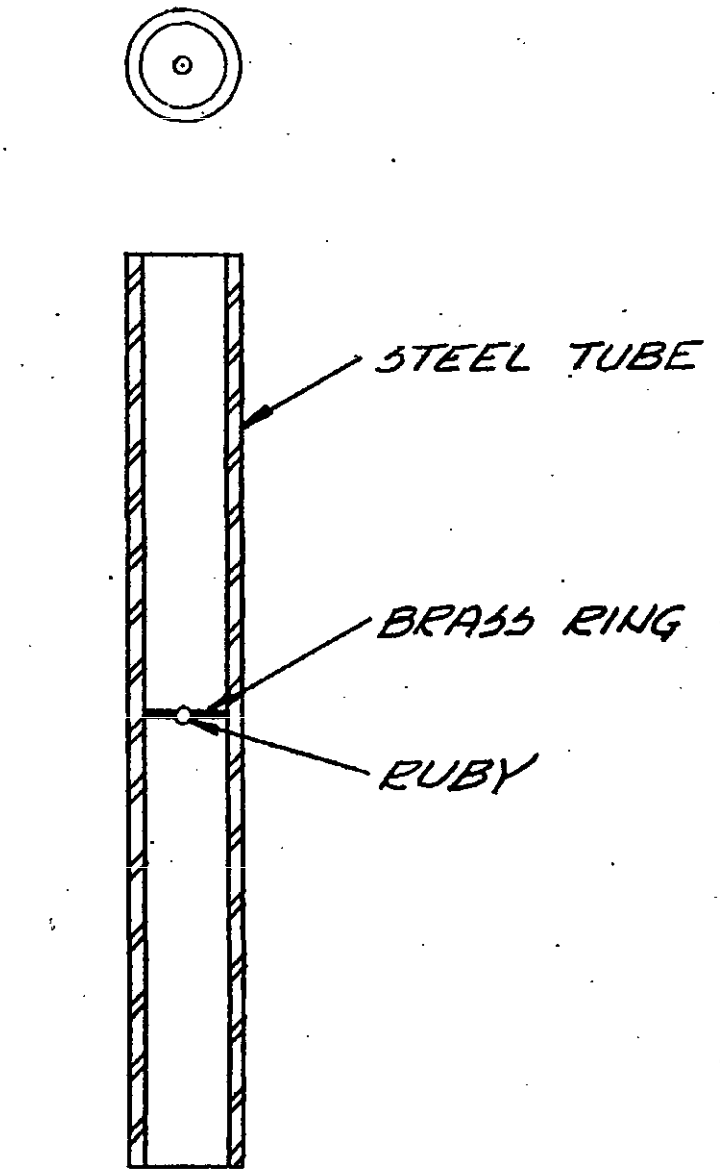


FIGURE 18.



CALIBRATOR

FIGURE 19.