ASTM INTERNATIONAL

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Standard Test Methods for
Pore Size Characteristics of Membrane Filters by Bubble Point and Mean Flow Pore Test[1]

This standard is issued under the fixed designation F316; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the determination of two of the pore size properties of membrane filters with maximum pore sizes from 0.1 to 15.0 μm.

1.2 Test Method A presents a test method for measuring the maximum limiting pore diameter of nonfibrous membranes. The limiting diameter is the diameter of a circle having the same area as the smallest section of a given pore (Fig. 1).

1.3 Test Method B measures the relative abundance of a specified pore size in a membrane, defined in terms of the limiting diameter.

1.4 The analyst should be aware that adequate collaborative data for bias statements as required by Practice D2777 is not provided. See the precision and bias section for details.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]
D1129 Terminology Relating to Water

D1193 Specification for Reagent Water
D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
E128 Test Method for Maximum Pore Diameter and Permeability of Rigid Porous Filters for Laboratory Use

3. Terminology

3.1 *Definitions*—For definitions of other terms used in these test methods, refer to Terminology D1129.

3.2 *Definitions of Terms Specific to This Standard:*
3.2.1 *pore size*—capillary equivalent pore diameter.

3.2.2 *limiting pore diameter*—diameter of a circle having the same area as the smallest section of a given pore.

TEST METHOD A—MAXIMUM PORE SIZE

4. Summary of Test Method

4.1 The bubble point test for maximum pore size is performed by prewetting the filter, increasing the pressure of gas upstream of the filter at a predetermined rate and watching for gas bubbles downstream to indicate the passage of gas through the maximum diameter filter pores.

4.2 The pressure required to blow the first continuous bubbles detectable by their rise through a layer of liquid covering the filter is called the "bubble point", and is used to calculate maximum pore size.

5. Significance and Use

5.1 This test method may be used to:
5.1.1 Determine the maximum pore size of a filter,
5.1.2 Compare the maximum pore sizes of several filters, and
5.1.3 Determine the effect of various processes such as filtration, coating, or autoclaving on the maximum pore size of a membrane.

5.2 Membrane filters have discrete pores from one side to the other of the membrane, similar to capillary, tubes. The bubble point test is based on the principle that a wetting liquid is held in these capillary pores by capillary attraction and surface tension, and the minimum pressure required to force
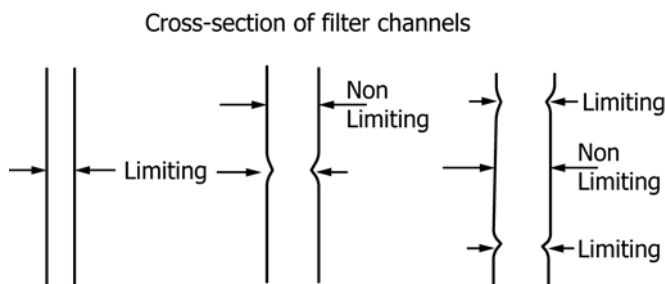

Cross-section of filter channels

FIG. 1 Examples of Limiting Diameters

liquid from these pores is a function of pore diameter. The pressure at which a steady stream of bubbles appears in this test is the bubble point pressure. The bubble point test is significant not only for indicating maximum pore size, but may also indicate a damaged membrane, ineffective seals, or a system leak.

5.3 The results of this test method should not be used as the sole factor to describe the limiting size for retention of particulate contaminants from fluids. The effective pore size calculated from this test method is based on the premise of capillary pores having circular cross sections, and does not refer to actual particle size retention. See Test Method E128 for additional information.

6. Apparatus

6.1 *Filter Holder,* as shown in Fig. 2, consisting of a base A, a locking ring B, O-ring seal C, support disk D, and gas inlet E. The support disk shall be 2-ply construction, consisting of a 100 by 100 mesh or finer screen and a perforated metal plate for rigidity. The diameter of the test filter may be either 25 or 47 mm, as appropriate to the holder being used for the test.

6.2 *Manifold,* as shown in Fig. 3, a micrometric flow control valve capable of providing a linear rise in pressure and a gas ballast of at least 16 000-cm³ capacity.

Note 1—For less accurate determinations, the simplified apparatus shown in Fig. 4 may be used.

6.3 *Pressure Gages* (and mercury manometer if required), covering the range of pressures needed for the pore sizes under investigation (see Table 1).

6.4 *Metal Punch,* used to cut a suitable size filter from the test sheet to fit the test filter holder.
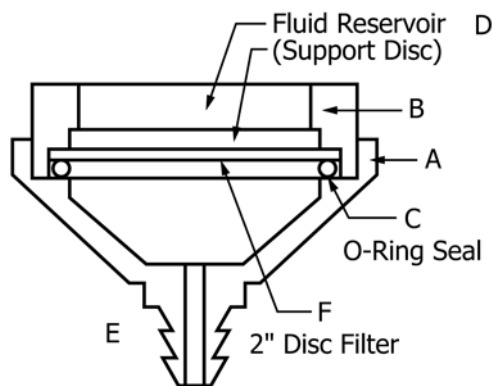

FIG. 2 Filter Holder

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[3] Other grades may be used provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

7.2 *Water,* conforming to Specification D1193, Type IV or higher purity.

7.3 *Denatured Alcohol.*

7.4 *Petroleum Distillate,* with surface tension of 30 dynes/cm at 25°C.

7.5 *Mineral Oil,* such as USP liquid petrolatum heavy, with surface tension of 34.7 dynes/cm at 25°C.

7.6 *1,1,2-trichloro-l,2,2-trifluoroethane (Freon TF®),* available from commercial chemical supply houses.

7.7 *Clean Gas Pressure Source,* with regulation (filtered air or nitrogen).

Note 2—Table 1 lists the nominal surface tension of these liquids at 25°C. Table 2 lists the simplified maximum pore size formulas based on these values, where the liquid completely wets the membrane.

8. Procedure

8.1 Wet the test membrane completely by floating it on a pool of the liquid. Use a vacuum chamber to assist in wetting the filter, if needed.

8.2 Place the wet membrane in the filter holder.

8.3 Close the filter holder and apply slight gas pressure to eliminate possible liquid back flow.

8.4 Cover the perforated metal plate with 2 to 3 mm of test liquid.

8.5 Increase the gas pressure slowly. Record the lowest pressure at which a steady stream of bubbles rises from the central area of the liquid reservoir.

Note 3—Faulty sealing may cause erroneous bubbling from the sealing edge of the liquid reservoir. Be sure to record the bubble point pressure with bubbles from the central area of the reservoir (see Fig. 5).

9. Calculation

9.1 If the test liquid is known to wet the membrane completely, calculate the maximum pore size from the following equation:

$$d = C\gamma/p \qquad (1)$$

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[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

FIG. 3 Manifold for Bubble Point Testing
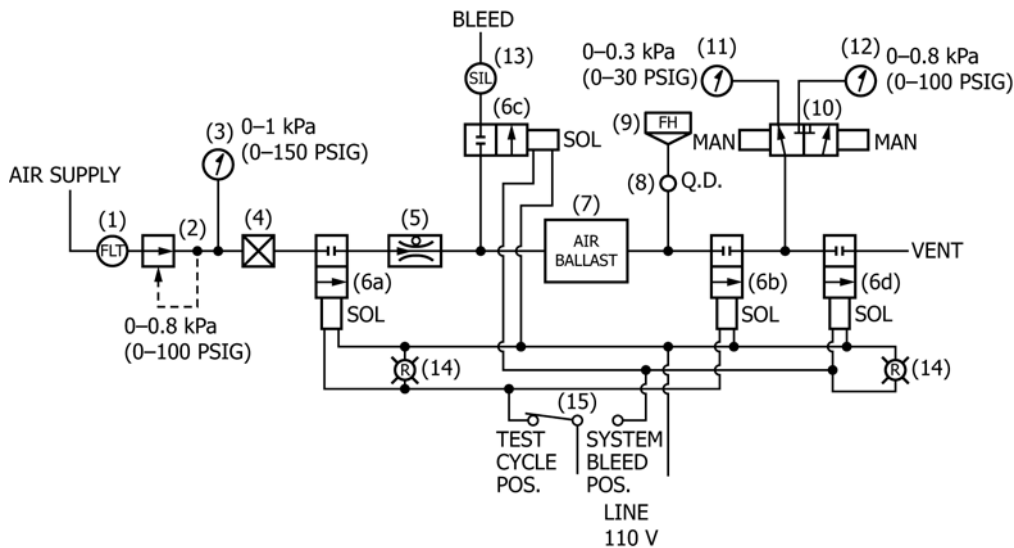
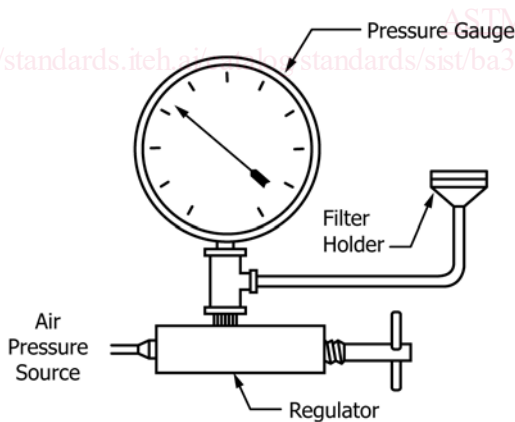| Key	Quantity	Component
1	1	Filter
2	1	Pressure regulator
3	1	Pressure gage
4	1	Valve shutoff, manual
5	1	Valve, flow control, manual
6	4	Valve, solenoid, nc
7	1	Air ballast
8	1	Quick disconnect fitting
9	2	Open filter holder, 47 mm
10	1	Valve, 3-way, manual
11	1	Test gage, 0-0.3 kPa (0-30 psig)
12	1	Test gage, 0-0.8 kPa (0-100 psig)
13	1	Exhaust silencer
14	2	Pilot light, red, elec.
15	1	Switch, spdt, elec.

FIG. 4 Test Setup (Simplified)

where:

d = limiting diameter, µm,
γ = surface tension, mN/m, (dynes/cm),
p = pressure, Pa or cm Hg, and
C = constant, 2860 when p is in Pa, 2.15 when p is in cm Hg, and 0.415 when p is in psi units.

NOTE 4—The fluid must completely wet the membrane filter with the contact angle being zero. If the contact angle is greater than zero, the calculated effective pore size will be larger than the actual effective pore size rating.

10. Reporting Results

10.1 Record the minimum pressure for gas passage as indicated by continuous bubbles. Record the maximum pore size calculated, along with identification of the membrane tested and the liquid used.

TEST METHOD B—DETERMINATION OF PORE SIZE DISTRIBUTION

11. Summary of Test Method

11.1 A fluid-wet filter will pass air when the applied air pressure exceeds the capillary attraction of the fluid in the pores. Smaller pores will exhibit similar behavior at higher pressures. The relationship between pore size and pressure has been established, as indicated in Table 2.

11.2 By comparing the gas flow rates of both a wet and dry filter at the same pressures, the percentage of the flow passing through the filter pores larger than or equal to the specified size may be calculated from the pressure-size relationship. By