Designation: E 1294 – 89 (Reapproved 1999)

Standard Test Method for Pore Size Characteristics of Membrane Filters Using Automated Liquid Porosimeter[1]

This standard is issued under the fixed designation E 1294; 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 This test method describes a procedure for measuring the pore size characteristics of membrane filters in the range of approximately 0.05 to 300 µm.

1.2 This test method uses the automated bubble point method described in ASTM Test Method F 316.

1.3 The liquid displacement technique used in this test method depends upon the capillary rise created by surface tension and uses the Washburn equation for calculating the pore diameter.

1.4 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

F 316 Test Method for Pore Size Characteristics of Membrane Filters for Use with Aerospace Fluids[2]

3. Summary of Test Method

3.1 A filter wet with liquid exhibits properties similar to those of array of liquid filled capillaries (see Note). The sample under test is thoroughly wetted with liquid of low surface tension and low vapor pressure and placed in a sample holder assembly. An increasing air pressure is applied upstream of the sample and as successively smaller pores empty, the air flow across the sample is recorded as a function of applied pressure. The point of first flow is identified as the bubble point (maximum pore size). This continues until the smallest detectable pore is reached. This information is then compared with the flow rate against applied pressure response for the dry sample. The pore size distribution is then obtained from wet and dry curves.

NOTE 1—This test method will not change the priorities of the membrane if the test liquids are chosen to be compatible with the material from which it is made.

4. Significance and Use

4.1 This test method may be used to determine the maximum pore size, minimum pore size, and mean flow pore size.

4.2 The pore size distributions by flow, number, and volume can be deduced from pressure versus flow data.

5. Apparatus

5.1 *Source of Compressed Air* up to 150 psi.

5.2 *Automated Liquid Porosimeter.*[3]

5.3 *Sample Holders*.

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

6. Reagents and Materials

6.1 *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.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Porofil.*[5]

6.3 *Isopropyl Alcohol*.

6.4 *Water*.

6.5 *Mineral Oil*.

7. Procedure

7.1 Follow the manufacturer's operating instructions to prepare the sample to introduce to the instrument for analysis.

7.2 Establish standard sampling procedures to obtain repeatable results.

8. Operating Instructions

8.1 Operating instructions shall be supplied by the manufacturer with every instrument. The instructions shall contain:

[1] This test method is under the jurisdiction of ASTM Committee E-48 on Biotechnology and is the direct responsibility of Subcommittee E48.03 on Unit Processes and Their Control.

Current edition approved March 31, 1989. Published May 1989.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] Coulter Porometer available from Coulter Electronics, Inc., 601 W. 20th Street, Hialeah, Florida 33010 U.S.A., has been found suitable for this purpose.

[4] "Reagent Chemicals, American Chemical Society Specifications,'' *American Chemical Society*, Washington DC. For suggestions on the testing of reagents no listed by the American Chemical Society, see" Analar Standards for Laboratory U.K. Chemicals," BDH Ltd., Poole, Dorset, and the "United States Pharmacopeia."

[5] Porofil wetting liquid, available from Coulter Electronics, Inc., has been found suitable for this purpose.

8.1.1 Brief instructions of the operating principles of the instrument,

8.1.2 Description of various systems,

8.1.3 Pore size range limitations,

8.1.4 Suggested maintenance procedures, and

8.1.5 Recommended spare parts.

9. Verification

9.1 Verification of the performance of the instrument function may be determined by using well characterized track-etched polycarbonate membrane filters[6] within the range of the instrument.

9.2 Interlaboratory comparisons within a particular industry should be made using these well characterized membranes.

10. Report

10.1 Report the following information:

10.1.1 Graph of raw data showing the pressure versus percent flow.

10.1.2 Tabulation of the raw data as well as the computed differential percent flow, cumulative percent flow, differential number percent, and cumulative number percent.

10.1.3 Graphs of differential and cumulative flow as well as number distributions plotted as pore size versus percent flow or percent number.

[6] Polycarbonate membrane filters, available from Nucleopore Corp., 7035 Commerce Circle, Pleasanton, CA 94566, have been found suitable for this purpose.

11. Precision and Bias

11.1 The precision of pore size analysis technique is not easy to verify. The mathematical model chosen to relate applied pressure to pore size is that of a right cylindrical pore. The best way to evaluate the precision is to evaluate the results from a series of track-etched polycarbonate membranes.[6] The manufacturer sizes them by electron microscopy, and uses ASTM porosimetry and bubble point tests to validate actual pore sizes.

11.2 *Precision*—The reproducibility of each instrument can be determined by repeatedly analyzing 0.1- and 10.0- µm polycarbonate membrane filters and determining the coefficient of variation. The results should not differ from the mean by more than the following amounts:

Pore Size Range	Repeatability: (same operator and apparatus), %	Reproducibility: (different operators and apparatus), %
10 µm	4	8
0.1 µm	2	4

Since the change in pore size per unit change in applied pressure is greatest at large pore sizes and diminished hyperbolically, repeatability and reproducibility increase accordingly with applied pressure.

11.3 *Bias*—The bias between two different instruments can be determined by comparing the mean flow pore sizes.