Designation: D 2873 – 94 (Reapproved 1999)ε1

Standard Test Method for Interior Porosity of Poly(Vinyl Chloride) (PVC) Resins by Mercury Intrusion Porosimetry[1]

This standard is issued under the fixed designation D 2873; 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.

ε[1] NOTE—Inch-pound units were deleted editorially in April 2000.

1. Scope

1.1 This test method describes a procedure for measuring the interior pore volume and the apparent pore diameter distribution of porous poly(vinyl chloride) resins. The measurements are made by forcing mercury under increasing pressure through a graduated penetrometer into the open pores of the resin samples. The volume of mercury forced into the pores is defined from the change of the mercury volume in the penetrometer; the apparent pore diameter distribution can be defined from incremental volume changes with increasing pressure.

1.2 **Warning**—This standard includes the use of an OSHA-designated hazardous chemical (Mercury). For specific hazard information and guidance relative to use, consult the health and safety documents provided by the supplier, for example, the material safety data sheet.

1.3 The values stated in SI units are to be regarded as the standard.

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.*

NOTE 1—There are no ISO standards covering the primary subject matter of this test method.

2. Referenced Documents

2.1 *ASTM Standards:*

D 883 Terminology Relating to Plastics[2]

D 1600 Terminology of Abbreviated Terms Relating to Plastics[2]

D 2396 Test Method for Powder-Mix Time of Poly(Vinyl Chloride) (PVC) Resins Using a Torque Rheometer[3]

3. Terminology

3.1 *Definitions*—Definitions are in accordance with Terminologies D 883 and D 1600 unless otherwise indicated.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *interior pore volume*—change in the volume of mercury observed above an applied pressure at which a demarcation exists between interior pores and voids between resin particles, and a maximum applied pressure. It is expressed in cubic centimeters per gram. For many resins with a medium size near 100 µm, such a demarcation exists at or near 390 kPa, corresponding to an apparent pore diameter of approximately 3.1 µm. A maximum applied pressure of 35 000 kPa has been found sufficient with most vinyl resins; the use of 21 000 kPa yields slightly lower values with equivalent precision.

4. Significance and Use

4.1 This test method is intended to compare differences in the total interior pore volume of porous vinyl resins. In general, in certain formulations, resins of higher porosity are better dry-blending resins; thus the interior porosity measurement defines one of the criteria useful for the definition of the dry-blend properties of vinyl resins.

5. Limitations

5.1 Only those pores open to the outside surface of the resin sample are filled with the mercury. The apparent pore diameter distribution that may be defined from the data may not be physically significant if there are large openings within the sample which are connected to the surface by narrow pores.

5.2 The pressure applied limits the extent of the open pores filled; thus, at approximately 35 000 kPa the minimum diameter pore penetrated is about 0.035 µm, while at 21 000 kPa the minimum diameter is 0.058 µm.

NOTE 2—The smallest pore diameter entered by the mercury under pressure is stated by:

$$D = 1207 / P_{kPa} \quad (1)$$

where:

D = diameter of the pore, µm, and

P = absolute pressure.

[1] This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials.

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[2] *Annual Book of ASTM Standards,* Vol 08.01.

[3] *Annual Book of ASTM Standards,* Vol 08.02.

5.3 This test method is applicable to poly(vinyl chloride) resins that show a relatively distinct end point in a powder-mix test such as Test Method D 2396. Application to other resins requires supplementary definition of the demarcation between interior pores and voids between particles.

6. Apparatus

6.1 *Mercury Intrusion Porosimeter*, equipped with a penetrometer capable of providing precise definition of volume change of mercury at pressure increments between atmospheric pressure and 21 000 kPa (preferably 35 000 kPa).[4]

6.2 *Vacuum Pump*, capable of evacuating the apparatus to < 50 µm pressure (7 mPa).

6.3 *Vacuum Gage*, reading up to 1000 µm Hg (130 mPa).

6.4 *Analytical Balance*, capable of measuring to ±0.0001 g.

6.5 *Camel's-Hair Brush*, or equivalent.

6.6 *Silicone Grease* (high vacuum).

6.7 *Isopropanol*, reagent grade, or suitable hydraulic liquid.

6.8 *Mercury*, preferably purified.

7. Procedure

7.1 From an excess of the test resin weighed in a suitable dish, carefully transfer an appropriate weight of the resin powder to the chamber of the sample holder or penetrometer. Reweigh the sample dish and remaining powder and obtain the sample weight by difference. All weights should be within ±0.0001 g.

7.2 The sample size to be used is dependent on the porosity of the resin and the applicable volume of the instrument used. Best results are obtained using the largest sample size consistent with the apparatus employed. For many general purpose dry blend resins the total interior pore volume ranges from 0.25 to 0.30 cm^3/g. The volatiles content of the sample tested should be less than 0.1 weight %.

7.3 Place the penetrometer containing the sample in the appropriate chamber following the directions provided in the instruction manual of the apparatus used. Apply silicone grease to the penetrometer joints to ensure a good seal.

7.4 Evacuate the apparatus using the pump until the vacuum gage reaches <50 µm. Use of a cold trap will minimize volatiles entering the pump.

7.5 Following the operating instructions provided with the instrument, allow the mercury to come in contact with the penetrometer or sample cell. Carefully open the specified valve to the atmosphere until the pressure of the gage indicates 50 kPa. Under this condition, the mercury will completely fill the penetrometer or sample cell and completely envelop the resin particles. Allow the excess mercury to drain out of the penetrometer or sample chamber.

7.6 With the penetrometer or sample chamber in the proper position for pore volume measurements, obtain penetration volume readings at the pressures (kPa) in the following list. With instruments providing a direct reading in absolute pressure, the corresponding absolute pressures are employed. At an absolute pressure of 12 kPa, pores or voids between resin particles of 100 µm in diameter are filled with mercury.

kPa		
35	860	7 900
70	1210	10 000
140[A]	1700	14 000
240[A]	2400	21 000[A]
310[A]	3100	28 000
590[A]	4500	35 000[A]
700	5900	

[A] For some general-purpose dry blend resins, measurements at these pressures are sufficient to define the total interior pore volume.

7.6.1 The pressures shown represent the minimum required to define the apparent pore volume distribution curve. Measurements at pressures below atmospheric provide a relative definition of interparticle voids and are necessary to define the demarcation between interior pores and exterior voids of some resins, particularly of porous resins with a median particle size significantly less than 100 µm.

7.7 Follow the instructions in the manual to release the pressure in the apparatus. Remove the penetrometer or sample holder and remove and dispose of the mercury-contaminated resin in a container for chemical waste.

7.8 Carefully clean the penetrometer or sample holder assembly prior to reuse. Washing with toluene followed by a rinse with acetone will remove silicone lubricant contamination.

8. Calculation

8.1 The data are first treated to convert the pressure readings to total absolute pressure. These conversions will not be necessary with instruments that provide absolute pressure readings directly.

8.2 To calculate total absolute pressure for the gage readings, add the atmospheric pressure to the gage pressure reading. Subtract the head pressure of mercury coresponding to the appropriate penetrometer range (Note 2). A typical chart for use with a 0.20-cm^3 penetrometer is shown in Table 1. Record the value so obtained as total absolute pressure.

NOTE 3—In some earlier instruments in which the penetrometer is placed upright in the pressure chamber, the mercury head pressure is added rather than subtracted.

8.3 Plot the penetrometer readings versus the total absolute pressure on four phase semilog graph paper using a French curve to connect the points. This curve represents a profile of the apparent internal pore size distribution. For curves that show an essentially flat portion near 390 kPa, that is the curve is enearly horizontal to the absolute pressure axis at this pressure, a simple treatment of the data is applicable. From the curve, read the penetrometer stem reading (in cm^3) at 390 kPa and at 21 000 kPa or, preferably, at 35 000 kPa. Calculate the total interior pore volume using the following equation:

$$\text{Interior pore volume, } cm^3/g = (V_2 - V_1)/S. \quad (2)$$

where:

V_2 = volume at 35 000 kPa, (or 21 000 kPa if the lower pressure is used),
V_1 = volume at 390 kPa, and
S = sample weight, g.

8.4 For samples that show an appreciable volume change near 390 kPa on the apparent pore volume distribution curve,

[4] Mercury porosimeters manufactured by Super Pressure, formerly American Instrument Corp. and Micrometrics Instrument Corp., have been found suitable.

TABLE 1 Mercury Head Pressure Correction Applicable to 0.20-cm³ Penetrometer

NOTE 1—Pentrometer length: 24.2 cm
Graduations: 0 to 0.20 cm^3(100 divisions covering the 13.75-cm length of the capillary stem).

Penetrometer Readings, cm^3	Mercury Head Pressure, kPa	Penetrometer Readings, cm^3	Mercury Head Pressure, kPa	Penetrometer Readings, cm^3	Mercury Head Pressure, kPa	Penetrometer Readings, cm^3	Mercury Head Pressure, kPa
0.000–0.003	31.7	0.051–0.057	26.9	0.104–0.110	22.1	0.156–0.163	17.2
0.004–0.012	31.0	0.058–0.065	26.2	0.111–0.117	21.4	0.164–0.170	16.5
0.013–0.019	30.3	0.066–0.072	25.5	0.118–0.125	20.7	0.171–0.178	15.9
0.020–0.027	29.6	0.073–0.080	24.8	0.126–0.133	20.0	0.179–0.185	15.2
0.028–0.034	29.0	0.081–0.086	24.1	0.134–0.140	19.3	0.186–0.192	14.5
0.035–0.042	28.3	0.087–0.095	23.4	0.141–0.148	18.6	0.193–0.200	13.8
0.043–0.050	27.6	0.096–0.103	22.8	0.149–0.155	17.9		

additional data at pressures below atmospheric may be required. A plot of the differential pore volume in cubic centimeters per gram versus the pore diameter in microns will, with some resins, indicate a minimum point in advance of the major interior absorption region which can be attributed to a demarcation between external voids and interior porosity. If this demarcation point can be established, the pore volume reading at the corresponding absolute pressure and the volume reading at the maximum applied pressure (kPa) can be used to define the total interior pore volume as described by the equation in 8.3.

9. Report

9.1 Report the total interior pore volume to the nearest 0.001 cm^3/g, indicating the maximum applied pressure used.

10. Precision and Bias [5]

10.1 The following should be used for judging the acceptability of results (95 % confidence limits):

10.1.1 *Repeatability*—Duplicate results by the same operator should not be considered suspect unless they differ by more than 7.1 %, relative.

10.1.2 *Reproducibility*—The average result of two determinations reported by one laboratory should not be considered suspect unless it differs from that of another laboratory by more than 9.5 %, relative.

11. Keywords

11.1 interior pore volume; PVC resin; resin porosity

[5] Round-robin data for this test method may be obtained from ASTM Headquarters, Request RR: D-20-1007.