



Designation: D 6226 – 98^{ε1}

Standard Test Method for Open Cell Content of Rigid Cellular Plastics¹

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^{ε1} NOTE—Editorially corrected Equation A2.5 in April 2002.

1. Scope

1.1 This test method covers cellular plastics, which are composed of membranes or walls of polymer separating small cavities or cells. These cells may be interconnecting (open cell), non-connecting (closed cell), or any combination of these types. This test method determines numerical values for open cells. It is a porosity determination, measuring the accessible cellular volume of a material. The remaining volume is that occupied by closed cells and cell walls. Since any conveniently sized specimen can only be obtained by some cutting operation, a fraction of the closed cells will be opened by specimen preparation and will be included as open cells, (see Note 2).

1.2 This test method provides good accuracy on predominantly highly open-celled materials. By not accounting for closed cells that were opened during specimen preparation, the accuracy decreases as the closed cell content increases and as the cell size increases.

1.3 The values as stated in SI units are to be regarded as the standard. The values in parentheses are given for reference only.

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—This test method and ISO 4590-1981 use the same basic principles but are significantly different in experimental detail.

NOTE 2—Two procedures for correcting for cells opened during specimen preparation are described in Appendix X1.1.

2. Referenced Documents

2.1 ASTM Standards:

D 618 Practice for Conditioning Plastics and Electrical Insulation Materials for Testing²

D 883 Terminology Related to Plastics²

D 2842 Test Method for Water Absorption of Rigid Cellular Plastics³

D 3576 Test Method for Cell Size of Rigid Cellular Plastics³

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁴

2.2 ISO Standard:

ISO 4590-1981 Cellular Plastics—Determination of Volume Percentage of Open and Closed Cells of Rigid Materials⁵

3. Terminology

3.1 Definitions:

3.1.1 Terms relating to plastics as given in Terminology D 883 shall be used where applicable.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *closed cell*—a cell totally enclosed by its walls and hence not interconnecting with other cells.

3.2.2 *open cell*—a cell not totally enclosed by its walls and open to the surface either directly or by interconnecting with other cells.

3.2.3 *volume of closed cells and cell walls*—inaccessible internal volume, consisting of an aggregate of solid polymer volume (cell walls, struts), filler volume, when applicable (solid particles or fibers), the volume of individual closed cells, and the volume of small cell groups interconnected by ruptured cell walls but otherwise inaccessible.

3.2.4 *uncorrected volume of open cells*—the aggregate measurement of both the internal porous volume of the material and the various irregular volumes accessible at the cut-cell surface of the test specimen.

3.3 Symbols:

3.3.1 *d*—specimen diameter, cm.

3.3.2 *h*—specimen height, cm.

3.3.3 *l*—specimen length, cm.

3.3.4 *O_v*—volume, percent open cells.

3.3.5 *V*—geometric volume of specimen, cm³.

¹ This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.22 on Cellular Materials—Plastics and Elastomers.

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² Annual Book of ASTM Standards, Vol 08.01.

³ Annual Book of ASTM Standards, Vol 08.02.

⁴ Annual Book of ASTM Standards, Vol 14.02

⁵ Available from American National Standards Institute, 11 W. 42nd St. 13th Floor, New York, New York 10036.

- 3.3.6 V_{CALIB} —volume of the calibration standard, cm^3 .
- 3.3.7 $V_{CHAMBER}$ —volume of the sample chamber, cm^3 .
- 3.3.8 V_{EXP} —expansion reference volume, cm^3 .
- 3.3.9 V_{SPEC} —specimen displacement volume, cm^3 , and,
- 3.3.10 w —specimen width, cm.

4. Summary of Test Method

4.1 This test method is based on a determination of porosity in which the accessible cellular volume of a cellular plastic is determined by application of Boyle’s Law, which states that the increase in volume of a confined gas results in a proportionate decrease in pressure. The apparatus consists of two chambers of known volume connected by a valve. One of the chambers, the calibrated sample chamber, is accessible for insertion of the test specimen and is connected to a source of high purity (at least 99.99 %) dry gas, such as nitrogen or helium. The pressure in the sample chamber is increased to a predetermined pressure and this value, P_1 , is noted. The valve between the two chambers is then opened and the second, lower Pressure, P_2 , is again noted. The ratio of the pressure change P_1/P_2 , is directly related to the volume of the sample chamber displaced by the specimen. The difference between this volume and the geometric volume of the specimen is a measure of the open-cell volume.

NOTE 3—The criteria for selecting a dry gas are that the gas shall not exhibit significant non-ideal behavior, dissolve the matrix, or readily diffuse into the matrix.

5. Significance and Use

5.1 This test method is intended to be used in specifications where porosity of cellular plastics has a direct bearing on their end use. For example, for thermal insulation applications, a high percentage of closed cells is necessary to prevent escape of gases and to promote low thermal conductivity. In flotation applications, high closed-cell content generally reduces water absorption.

5.2 Before proceeding with this test method, reference should be made to the specification of the material being tested. Any test specimen preparation, conditioning, or dimensions, or both, and testing parameters covered in the materials specification shall take precedence over those mentioned in this test method. If there are no material specifications, then the default conditions apply.

6. Apparatus

6.1 *Gas Pycnometer*—A schematic diagram of the pycnometer apparatus is shown in Fig. 1. It should be constructed from metal and have the following features:

- 6.1.1 *Sample Chamber* ($V_{CHAMBER}$), having a volume approximately between 30 and 150 cm^3 (1.8 and 9.2 in.^3), calibrated to the nearest 0.1 cm^3 (0.006 in.^3).
- 6.1.2 *Expansion Reference Volume* (V_{EXP}), a precisely calibrated volume known to the nearest 0.1 cm^3 (0.006 in.^3).
- 6.1.3 *Gage Pressure Transducer*, capable of measuring 0 to 175 kPa (0 to 25 psig) with minimum volume displacement and linear within 0.1%.
- 6.1.4 *Pressure Relief Valve*, to avoid over pressurization of the transducer.

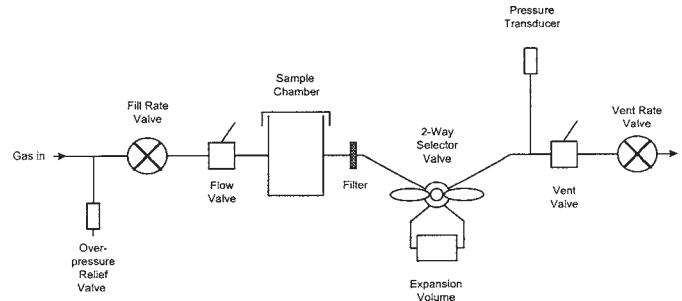


FIG. 1 Schematic Diagram of Gas Pycnometer

6.1.5 *Filter*, to prevent powder from contaminating the transducer and selector valves.

6.1.6 *Input Flow Control Toggle and Needle Valves*, or alternative means to control pressurization.

6.1.7 *Output Flow Control Toggle and Needle Valves*, or alternative means to vent the gas.

6.1.8 *Two-Way Selector Valve*, to connect the reference volume to the sample chamber.

6.1.9 *Nonporous Calibration Standard*, (for example a stainless steel sphere) of known volume which fills from 1/3 to 2/3 of the sample chamber.

6.1.10 *Digital Meter*, for reading the pressure from the transducer to 0.007 kPa (0.001 psig).

6.1.11 *Sample Chamber Closure*, with O-ring seal.

6.1.12 *Calibration Procedure*, for the gas pycnometer is outlined in A2.2

6.2 *Cutting Device*, for specimen preparation, such as a bandsaw or hobby jigsaw, the blade of which must be capable of producing a smooth cut. This will require a blade with at least four teeth/cm (10 teeth/in.). A cellular hole cutter can also be used.

6.3 *Vernier Calipers*, or micrometre measuring device, capable of measuring specimens to the nearest 0.003 cm (0.001 in.).

7. Sampling and Test Specimen Preparation

7.1 The standard test specimen is two cubes having a nominal dimension of 2.5 by 2.5 by 2.5 cm (1.0 by 1.0 by 1.0 in.), (see Note 4). An alternative configuration is two cylinders with nominal cross sectional areas of 6.25 cm^2 (0.97 in.^2) by 2.5 cm (1.0 in.) in height, (see Note 5). It may be practical in some instances (for example, when using smaller sized sample chambers or when there is a limited amount of material) to use only one cube or one cylinder. However the specimen displacement volume (V_{SPEC}) should be at least 15 % of the sample chamber volume.

NOTE 4—The analyst should not substitute a single 5 by 2.5 by 2.5 cm rectangular prism for the two cubes because the surface areas are not equal.

NOTE 5—The cylindrical configuration is not recommended for anisotropic materials.

7.2 Unless otherwise agreed upon, at least three sets of two cubes or two cylinders, selected at random, shall be tested. All specimens having obvious defects shall be omitted.

7.3 Sample selection on commercially available materials shall be by agreement between the supplier and the user.

7.4 Test specimens shall be machined or sawed from the sample so as to have smooth surfaces. All machined or sawed surfaces may be further smoothed by slicing techniques or sanding with a number 400 or finer sandpaper. Resulting dust should be blown from the specimens.

8. Conditioning

8.1 Condition specimens at standard laboratory atmosphere $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) and $50 \pm 5\%$ relative humidity for a minimum of 24 h.

8.2 Since this test method depends on very accurate measurement of gas pressures, the temperature of the environment, the apparatus, the specimen, and the sample cup must be kept constant within $\pm 2^\circ\text{C}$.

8.3 The temperature at which the calibration is performed shall be within $\pm 2^\circ\text{C}$ of the temperature at which the analysis is performed.

9. Procedure

9.1 Measure and record the length, l , height, h , and width, w , of the specimens to the nearest 0.003 cm (0.001 in.). If using cylindrical specimens measure the height, h , and diameter, d .

9.2 Close the flow valve if not already closed.

9.3 Operate the two-way selector valve so that it isolates the expansion volume (reference) chamber from the rest of the system.

9.4 Open the vent valve if not already open.

9.5 Remove the sample chamber cap if not already open and remove any previous specimen.

9.6 Insert the new specimen and securely replace the sample chamber cap.

9.7 Air, and vapors trapped within pores, crevices, or among the pieces of the specimen will be removed from the specimen by a prolonged purge when all valves are opened. Report the time used. However, these impurity gases are much more rapidly removed by alternately increasing and decreasing the gas pressure in the sample chamber.

9.8 Close the vent valve and open the flow valve. Allow the pressure to rise to 20 kPa (2.9 psig), then close the flow valve and open the vent valve. When the pressure has fallen to less than 3 kPa (0.4 psig), close the vent valve. This purging process should be repeated at least twice more. The vent valve must remain open at the completion of the purge process. Report the number of purge cycles used.

9.9 Operate the two-way selector valve so that it connects the expansion volume (reference) chamber with the rest of the system. Allow the pressure to fall to a steady value, correcting the pressure display with the zero control as necessary.

9.10 Operate the two-way selector valve so that it again isolates the expansion volume (reference) chamber from the rest of the system, ensuring that the pressure display does not shift from zero. If a shift occurs, repeat 9.9.

9.11 Close the vent valve neglecting any change in pressure.

9.12 Open the flow valve and fill the chamber to 20 kPa (2.9 psig) as shown on the pressure indicator.

NOTE 6—It may be desirable in some instances to use pressures less than 20 kPa for the purging and testing steps. The applied pressure should not distort the specimen. If other pressures are used, they shall be noted on the test report.

9.13 Close the flow valve and allow the pressure to stabilize or wait a fixed period of time (10 to 15 s give best results for most samples) and record the final pressure as P_1 . Report the time used if the pressure did not stabilize.

9.14 Immediately operate the two-way selector valve so that it again connects the expansion volume (reference) chamber with the rest of the system and allow the pressure to stabilize or wait the same fixed period of time as in 9.13. Record the final pressure as P_2 .

NOTE 7—If the pressure reading drifts continuously to lower pressures, the cells may be rupturing or the test gas may be diffusing through the cell walls. Under these conditions, an accurate open-cell content cannot be determined.

9.15 Open the vent valve and allow the pressure to fall to 0 kPa (0 psig)..

9.16 Return to 9.9 if multiple determinations are to be made on this specimen.

9.17 Remove the specimen from the sample chamber.

9.18 If the instrument is to remain idle for an extended period (days), close the sample chamber and shut off the gas supply.

9.19 Calculate the specimen volume from the general equation:

$$V_{SPEC} = V_{CHAMBER} - V_{EXP} / [(P_1/P_2) - 1]. \quad (1)$$

NOTE 8—Several companies produce both manually operated and automatically operated gas pycnometers which meet the specifications described herein. Please refer to the specific operating manuals for additional details on operation and calibration.

10. Calculation

10.1 Calculate the geometric volume, V , in cubic centimetres of the specimen by adding together the volumes of its component specimens determined from their measurements of length, width, and height as follows:

$$\text{If two cubes: } V = (l_1 \times w_1 \times h_1) + (l_2 \times w_2 \times h_2) \quad (2)$$

$$\text{Or if two cylinders: } V = [(\pi \times d_1^2 \times h_1)/4] + [(\pi \times d_2^2 \times h_2)/4]. \quad (3)$$

10.2 Calculate the open-cell content, O_v , of each specimen expressed as the percentage of the calculated volume, V , as follows:

$$O_v = [(V - V_{SPEC})/V] \times 100 \quad (4)$$

10.3 If desired the percent volume occupied by the closed cells and cell walls, CW_v , can be calculated from the equation:

$$CW_v = 100 - O_v \quad (5)$$

10.4 When the specific gravity of the material in its solid form is known the percent volumes occupied by the cell walls, W_v , and the closed cells, C_v , can be calculated individually. The cell wall volume percentage is calculated as follows:

$$W_v = [m / (sg \times V)] \times 100 \quad (6)$$

where:

m = the mass of the specimen, g,

sg = the specific gravity of the material in solid form, g/cm^3 , and

V = the geometric volume of the specimen, cm^3 .