



Designation: ~~D6226-05~~ Designation: D6226 - 10

Standard Test Method for Open Cell Content of Rigid Cellular Plastics¹

This standard is issued under the fixed designation D6226; 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 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 ~~brackets~~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~~4590 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:²

D883 Terminology Relating to Plastics

D2842 Test Method for Water Absorption of Rigid Cellular Plastics

D3576 Test Method for Cell Size of Rigid Cellular Plastics

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 ISO Standard:

ISO ~~4590-1981~~ISO 4590 Cellular Plastics—Determination of Volume Percentage of Open and Closed Cells of Rigid Materials³

3. Terminology

3.1 Definitions:

3.1.1 Terms relating to plastics as given in Terminology D883 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.

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

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

*A Summary of Changes section appears at the end of this standard.

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, cm^3 .
- 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 shall 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 shall have the following features:

6.1.1 *Sample Chamber* (V_{CHAMBER}), having a volume approximately between 30 and 150 cm^3 [1.8 (1.8 and 9.2 in.³)], calibrated to the nearest 0.1 cm^3 [0.006 in.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 (0.006 in.³)].

6.1.3 *Gage Pressure Transducer*, capable of measuring 0 to 175 kPa [0 (0 to 25 psig) psig] with minimum volume displacement and linear within 0.1%.

6.1.4 *Pressure Relief Valve*, to avoid over pressurization of the transducer.

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.

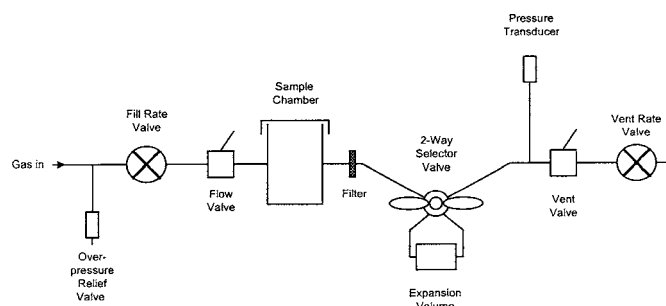


FIG. 1 Schematic Diagram of Gas Pycnometer

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

6.1.10 *Digital Meter*, for reading the pressure from the transducer to 0.007 kPa [~~0.001 psig~~](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 4 teeth/cm [~~10 teeth/in.~~](10 teeth/in.). A cellular hole cutter can also be used.

6.3 *Vernier Calipers*, or micrometer measuring device, capable of measuring specimens to the nearest 0.003 cm [~~0.001 in.~~](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(1.0 by 1.0 by 1.0 in.)~~in.), (see Note 4). An alternative configuration is two cylinders with nominal cross sectional areas of 6.25 cm² [~~0.97 in.~~] by 2.5 cm [~~1.0 in.~~] in height, (see (0.97 in.) by 2.5 cm (1.0 in.) in height, (see Note 5). It is possible that it will 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}) shall be at least 15 % of the sample chamber volume.

NOTE 4—The analyst shall 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. It is permissible for machined or sawed surfaces to be further smoothed by slicing techniques or sanding with a number 400 or finer sandpaper. Resulting dust shall be blown from the specimens.

8. Conditioning

8.1 Condition specimens at standard laboratory atmosphere $23 \pm 2^\circ\text{C}$ [~~73.4(73.4 \pm 3.6^\circ\text{F})~~3.6°F] and $50 \pm 5\%$ 10 % 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.

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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.~~](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~~](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~~](0.4 psig), close the vent valve. This purging process shall 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~~](2.9 psig) as shown on the pressure indicator.

NOTE 6—It will be desirable in some instances to use pressures less than 20 kPa for the purging and testing steps. The applied pressure shall 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}~~ (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/cm³, and

V = the geometric volume of the specimen, cm³.

10.5 The closed cell volume percentage can be then calculated with the equation:

$$C_v = 100 - O_v - W_v \quad (7)$$

11. Report

11.1 Report the following information:

11.1.1 Identification and description of the cellular material,

11.1.2 The date of manufacture and lot number, if known,

11.1.3 The number of specimens, conditioning of specimens, and gas of testing, if other than recommended, and

11.1.4 The date of testing.

11.2 Report the open cell percentage as the average of all specimens tested for the material.

11.3 If requested, report the closed cell and cell wall percentages as the average of all specimens tested for the material.

12. Precision and Bias

12.1 Table 1 is based on a round robin conducted in 2004 in accordance with Practice E691, involving four materials tested by six laboratories. For each material, all of the samples were prepared at one source, but the individual specimens were prepared at the laboratories that tested them. Each laboratory obtained five test results for each material. Precision, characterized by repeatability (S_r and r) and reproducibility (S_R and R) have been determined as shown in Table 1. (**Warning**—The explanation of r and R are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Table 1 should not be applied to acceptance or rejection of materials, as these data apply only to the materials tested in the round robin and are unlikely to be rigorously representative of other lots, formulations, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E691 to generate data specific to their materials and laboratory.)

NOTE 9—The precision data presented in Table 1 was obtained using the test conditions defined in this test method. If a material specification defines other test conditions, this precision data shall not be assumed to apply.