Designation: D 2856 – 94 (Reapproved 1998)

Standard Test Method for Open-Cell Content of Rigid Cellular Plastics by the Air Pycnometer[1]

This standard is issued under the fixed designation D 2856; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 Cellular plastics are composed of the 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 volume occupied by closed cells is considered to include cell walls. Since any conveniently sized specimen can only be obtained by some cutting operation, a fraction of the closed cells will be opened during sample preparation and will be included as open cells.

1.2 This test method consists of three procedures:

1.2.1 *Procedure A*, designed to correct for cells opened during sample preparation, by measuring cell diameter, calculating, and allowing for surface volume;

1.2.2 *Procedure B*, designed to correct for cells opened in sample preparation, by cutting and exposing new surface area equal to the surface area of the original sample dimension, and

1.2.3 *Procedure C*, which does not correct for cells opened during sample preparation and gives good accuracy on predominantly highly open-celled materials. 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 information 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.* Specific precautionary statements are given in Note 2, Note 4, and Note 8.

NOTE 1—This test method and ISO 4590-1981 use the same basic principles but are significantly different in experimental detail.

2. Referenced Documents

2.1 *ASTM Standards:*

D 618 Practice for Conditioning Plastics and Electrical Insulation Materials for Testing[2]

D 883 Terminology Relating to Plastics[2]

D 3576 Test Method for Cell Size of Rigid Cellular Plastics[3]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[4]

2.2 *ISO Standard:*

ISO 4590-1981 Cellular Plastics—Determination of Volume Percentage of Open and Closed Cells of Rigid Materials[5]

3. Terminology

3.1 *Definition:*

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

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

3.2.2 *volume of closed cells*—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.

3.2.3 *corrected volume of open cells*—the internal porous volume.

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.

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[1] This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D 20.22 on Cellular Plastics.

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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, NY 10036.

3.3 *Symbols:*

A = total geometric surface area of specimen, cm^2,
h = specimen height, cm,
l = specimen length, cm,
O_c = volume, percent open cells,
t = average chord length found in cell size determination, cm,
V = geometric volume of specimen, cm^3,
V_s = volume of the surface cells opened by sample preparation, cm^3,
V_1 = displacement volume of the specimen, cm^3,
V_2 = displacement volume after trisection, cm^3, and
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 decrease in volume of a confined gas results in a proportionate increase in pressure. The apparatus consists of two cylinders of equal volume with an accessible chamber provided in one of the cylinders for insertion of the test specimen. Pistons in both cylinders permit volume changes. The pressures are increased equally by decreasing both volumes when a specimen is present in the specimen chamber. The volume change for the specimen cylinder is smaller than for the empty reference chamber, and corresponds to the displacement volume of the specimen. The difference between this volume and the geometric volume of the specimen is a measure of the open-cell volume.

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.

6. Apparatus

6.1 *Air Pycnometer*.

NOTE 2—**Warning:** Beckman no longer manufactures or services the Beckman Pycnometer around which this test method was written. It is imperative that alternate equipment be investigated and data made available.

6.2 *Cutting Device*, for sample 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/centimetre (10 teeth/inch).

6.3 *Vernier Calipers*, or dial micrometer measuring device, capable of measuring specimens to the nearest 0.1 cm (0.04 in.).

6.4 *Razor Blades*, single-edge, new.

7. Principle of Operation of the Air Pycnometer

NOTE 3—The "half-atmosphere" method for operation of the air pycnometer is described in this test method. It subjects the specimen to a minimum pressure of approximately 0.05 MPa (7.4 psi). This rarely distorts the specimen, although occasional drifting of the pressure may be evidence that some gas is being withdrawn from the specimen, or that cells are rupturing. This is a modification of the "two-atmosphere" method described in the instruction manual for the air pycnometer instrument.

7.1 Fig. 1 depicts two chambers assumed (for illustrative purposes) to be of equal volume, with no specimen in the specimen chamber. In order to maintain equal pressure on both sides of the differential pressure indicator, any movement of one piston from Position 1 must be duplicated by a similar movement of the second piston. If both pistons are advanced to Position 2, with a specimen, V_x; inserted in Chamber B, and the coupling valve closed, the pressure on both chambers will not be equal. However, the pressure can be equalized by withdrawing Piston B an amount proportional in volume to V_x (from Position 2 to Position 3). The distance, d_x, from Position 2 to Position 3 is calibrated to read directly in cubic centimetres, and a digital counter is employed to indicate this distance.

8. Sampling and Test Specimen Preparation

8.1 The test specimen shall be a cube having a nominal dimension of 2.50 by 2.50 by 2.50 cm (0.984 by 0.984 by 0.984 in.). Unless otherwise agreed upon, at least five specimens, selected at random, shall be tested. All specimens having obvious defects shall be eliminated.

NOTE 4—**Caution:** Cylindrical-shaped specimens will give erroneous results by Procedure A or B of this test method.

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

8.3 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 Number 0 or finer sandpaper. Resulting dust shall be blown from the specimen.

9. Conditioning

9.1 Condition specimens at standard laboratory atmosphere, 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5% relative humidity for a minimum of 24 h.

9.2 Since this test method depends on very accurate measurement of air volumes, the temperature of the environment, the apparatus, the specimen, and the specimen cup must be kept constant within 2°C and the relative humidity controlled within 5% during the entire test.

PROCEDURE A—DETERMINATION OF OPEN-CELL CONTENT USING CELL DIMENSIONS TO CORRECT FOR CELLS CUT BY SPECIMEN PREPARATION

10. Procedure

10.1 Calibrate the air pycnometer by the procedure outlined in Annex A1.

10.2 Measure and record the length, l, height, h, and width, w, of the specimen to the nearest 0.003 cm (0.001 in.).

NOTE 5—Some pycnometers are equipped with a purge valve, as well as a coupling valve; others have no purge valve. In the following instructions, ignore any references to the purge valve if the pycnometer has none.

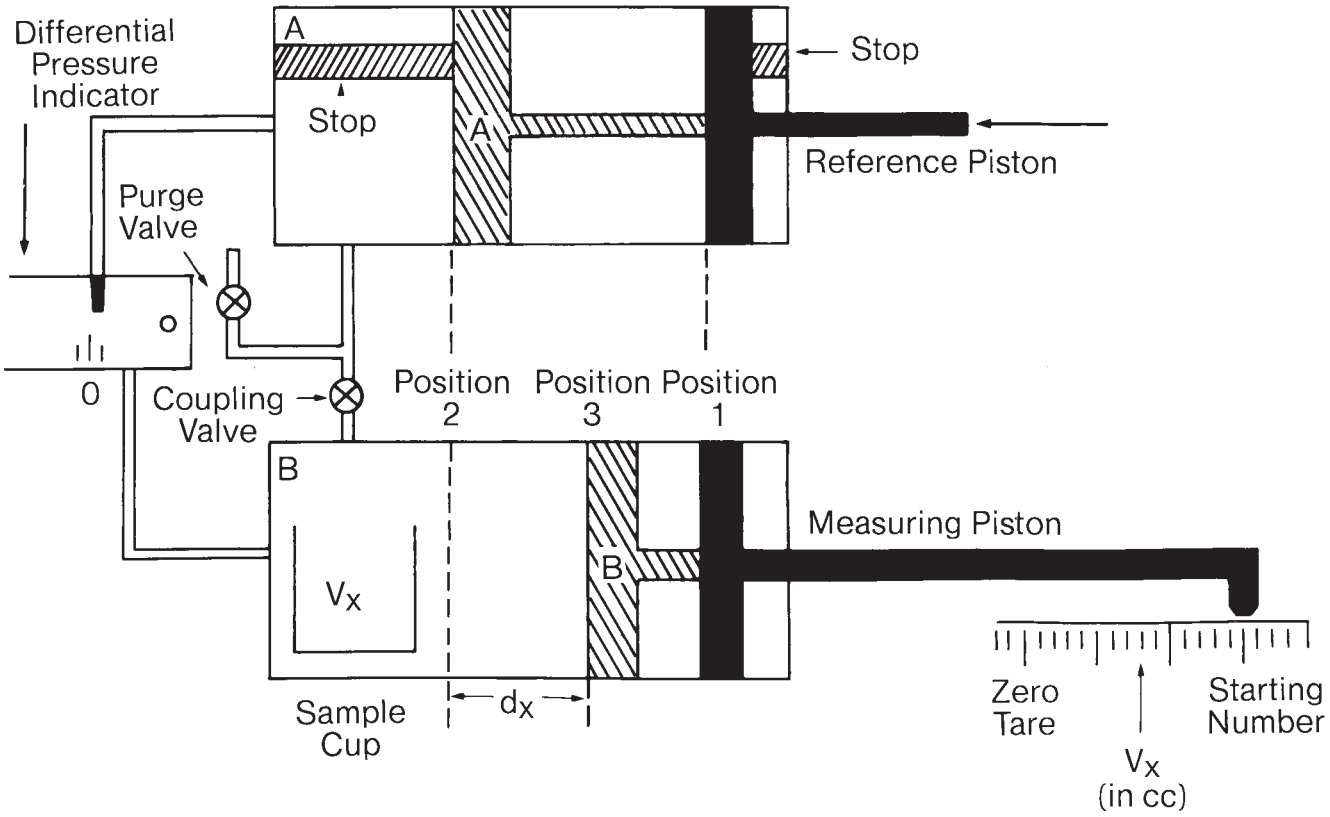

FIG. 1 Air Pycnometer—Piston Action

10.3 With the coupling valve open and purge valve closed, turn the reference handwheel clockwise to the inner stop. Turn the measuring handwheel so that the counter registers the estimated specimen volume, in cubic centimetres.

10.4 Insert the specimen into the specimen cup and seal the cup tightly by means of the lever provided on the instrument.

10.5 Turn both handwheels counter-clockwise, simultaneously, until the reference handwheel encounters its other stop and the counter for the measuring handwheel registers slightly higher than the "starting number." (This starting number is different for each instrument and must be furnished by the instrument manufacturer). Then, carefully turn the measuring handwheel clockwise to the starting number (Note 6). Wait 1 min for temperature and pressure equilibration (Note 7), then close the coupling valve.

NOTE 6—When making final adjustments of the measuring handwheel, always approach the desired setting in the clockwise direction, for maximum precision.

NOTE 7—A shorter time period here as well as in 11.7 may be permissible for some materials. For others, a shorter time may decrease precision.

10.6 Keep the pointer of the differential pressure indicator on scale during this next step. Turn both handwheels clockwise, simultaneously, until the reference handwheel again reaches the inner stop and the scale pointer is on zero.

10.7 Wait 1 min (Note 6 and Note 7). Then, carefully readjust the pointer to zero, using the measuring handwheel (Note 8).

10.8 Record the counter reading, V_x, apply the calibration correction, and record the resulting specimen displacement volume as V_1.

NOTE 8—**Caution:** If rapid drifting of the pointer occurs during either of the two 1-min waiting periods, the cells may be rupturing. Under these conditions, an accurate open-cell content cannot be determined.

10.9 Open the coupling valve. Remove and clean the specimen cup.

10.10 Determine the average chord length, t, of the cellular material by Test Method D 3576.

11. Calculation

11.1 Calculate the geometric volume, V, of each specimen from its individual measurements of length, width, and height. The volume, V, in cubic centimetres will be: $V = l \times w \times h$.

11.2 Calculate the total surface area, A, of each specimen in square centimetres as follows: $A = 2(lw + lh + hw)$.

11.3 Calculate the volume occupied by the surface cells, V_s, using A from 10.2 and t from 10.10 as follows: $V_s = (A \times t)/1.14$.

11.4 Calculate the volume, percent open cells, O_c, of each specimen as follows: $O_c = [(V - V_1 - V_s)/V] \times 100$.

PROCEDURE B—DETERMINATION OF OPEN-CELL CONTENT, CORRECTING FOR CELLS OPENED IN SPECIMEN PREPARATION BY CUTTING THE TEST SPECIMEN INTO EIGHT SMALLER PIECES

12. Procedure

12.1 Specimen preparation and measurement of the sample volumes are the same as in Procedure A, 10.1-10.8.

12.2 Cut the specimen with a razor blade along planes parallel to the sides of the specimen. This step will involve three cuts, as shown in Fig. 2.

12.3 Repeat 10.3-10.8 to determine the pycnometer volume of the eight sections together. Apply the correction factor and record this result as V_2.

NOTE 9—Caution should be taken to ensure that cuts are made as clean as possible, parallel to the original cut surfaces made during specimen preparation. Specimens in which an appreciable amount of solid matter has broken away upon cutting should be discarded.

13. Calculation

13.1 Calculate the geometric volume, V, in cubic centimetres, of each specimen before sectioning from its individual measurements of length, width, and height as follows: $V = l \times w \times h$.

13.2 Calculate the open-cell content, O_c, of each specimen expressed as a percentage of the calculated volume, V, where V_1 is the displacement volume of the specimen, and V_2 is the displacement volume after cutting along three planes as determined by the air pycnometer, as follows: $O_c = [1 - (2V_1 - V_2)/V] \times 100$.

NOTE 10—These calculations are based upon a cube. They assume the cells to be of uniform size. In cases of extremely low open-cell content, negative numbers may result due to the irregular size of cells in the sample.

PROCEDURE C—DETERMINATION OF OPEN-CELL CONTENT WITHOUT CORRECTING FOR SURFACE CELLS OPENED BY CUTTING

14. Procedure

14.1 Use this procedure when an approximate open-cell content is desired, or when excessive instrument eliminates the possibility of using Procedures A and B.

14.2 Sample preparation and measurement of the specimen volumes are the same as in Procedure A, 10.2-10.8.

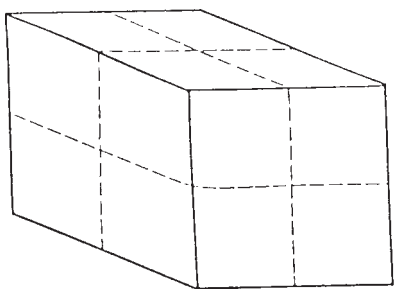

FIG. 2 Specimen Cutting Diagram

NOTE 11—When the indicator drift is extremely rapid, that is, when considerable drift occurs after 60 s of equilibration, the procedure for measuring volume on the remaining specimens should be as follows: Turn both measuring and reference handwheels in a clockwise direction, keeping the pressure in each compartment approximately equal, as shown by the differential pressure indicator. When the reference handwheel reaches its stop, adjust the measuring handwheel until the pressures are equal and the indicator reads zero. Immediately read the counter. Record this volume as V_1 after making the zero correction. (Annex A1, Note A1.5)

15. Calculation

15.1 Calculate the geometric volume, V, in cubic centimetres of each specimen from the measurements of length, width, and height as follows: $V = l \times w \times h$.

15.2 Calculate the open-cell content, O_c, of each specimen expressed as the percentage of the calculated volume, V, as follows: $O_c = [(V - V_1)/V] \times 100$.

16. Report

16.1 Report the following information:

16.1.1 Complete sample identification including cellular plastic source, manufacturer, lot number, and date of production, when known;

16.1.2 The procedure (A, B, or C) used, the number of specimens, conditioning of samples, and atmosphere of testing, if other than recommended, and

16.1.3 The date of testing.

16.2 Report the results as the average of all the specimens tested for each sample.

17. Precision and Bias [6]

17.1 *Precision*—The data in Table 1, Table 2, and Table 3 are from a round robin conducted in 1981. Because tests were conducted in only three laboratories for Procedure A, the data in Table 1 should be used cautiously. Tests were conducted in five laboratories for Procedures B and C. Each test result was the average from five specimens. Each laboratory reported one test result per material. Therefore:

$$S_r = S_i/(5)1 \,/\, 2 \qquad (1)$$

where:

S_i = standard deviation based on five specimens per cell.

17.1.1 In Table 1, Table 2, and Table 3 for the material indicated:

17.1.1.1 S_r is the within-laboratory standard deviation;

17.1.1.2 S_L is the square root of the variance between laboratories;

17.1.1.3 $I_r = 2.83\ S_r$ (I_r may be used as shown in 17.1.2), and

17.1.1.4 $I_R = 2.83\ (S_r^2 + S_L^2)^{1/2}$ (I_R may be used as shown in 17.1.3).

17.1.2 *Repeatability*—In comparing two averages (of five specimens each) for the same material obtained on the same equipment by the same operator on the same day, the averages should be judged not equivalent if they differ by more than the I_r for the material.

[6] Supporting data are available from ASTM Headquarters. Request RR: D20 – 1097.

TABLE 1 Open Cell, Procedure A

Material	Average, %, Open Cell	S_r, %, Open Cell	SL, %, Open Cell	I_r, %, Open Cell	I, %, Open Cell
EXT PS (NBS GM53)	−0.23	0.17	4.01	0.48	11.36
EXT PS	1.80	0.20	2.07	0.57	5.89
TRIMER PUR (NBS GM 43)	4.06	0.21	3.07	0.59	8.71
PUR	4.24	0.33	2.75	0.93	7.84
EXP PS	9.61	0.16	0.48	0.45	1.43

TABLE 2 Open Cell, Procedure B

Material	Average, %, Open Cell	S_r, %, Open Cell	SL, %, Open Cell	I_r, %, Open Cell	I, %, Open Cell
EXT PS	0.71	0.54	1.07	1.53	3.39
EXP PS (NBS GM53)	1.97	0.38	0.55	1.08	1.89
TRIMER PUR (NBS GM43)	3.54	0.40	1.39	1.13	4.09
PUR	4.43	0.50	1.14	1.42	3.52
EXP PS	7.99	0.42	0.43	1.19	1.70

TABLE 3 Open Cell, Procedure C

Material	Average, %, Open Cell	S_r, %, Open Cell	SL, %, Open Cell	I_r, %, Open Cell	I, %, Open Cell
EXT PS	7.13	0.20	1.96	0.57	5.58
EXT PS (NBS GM53)	10.27	0.17	1.16	0.48	3.32
TRIMER PUR (NBS GM 43)	12.06	0.21	0.89	0.59	2.59
PUR	12.21	0.29	0.83	0.82	2.49
EXP PS	12.50	0.26	0.96	0.74	2.81
MF	98.97	0.11	0.58	0.31	1.67

17.1.3 *Reproducibility*—In comparing two averages (of five specimens each) for the same material obtained by different operators on different equipment on different days, the averages should be judged not equivalent if they differ by more than the I_R for the material.

17.1.4 The judgments in 17.1.2 and 17.1.3 will be corrected in approximately 95% of such comparisons.

NOTE 12—For further information, see Practice E 691.

17.2 *Bias*—No statement of bias is possible because there is no accepted standard for these procedures.

18. Keywords

18.1 cell wall; cellular plastics; closed cell; open cell; pycnometer

ANNEX

(Mandatory Information)

A1. PROCEDURE FOR OPERATING AND CALIBRATING THE AIR PYCNOMETER

A1.1 With both the purge valve and the coupling valve open, run both pistons in and out of the cylinders seven or eight times to purge the instrument, ending with both handwheels in the clockwise position.

NOTE A1.1—The zero check procedure is identical to the measurement and calibration procedure, but is made with the sample cup clean and empty. The zero check is used to verify the zero offset of the instrument. The calibration check verifies the accuracy of the unit.

A1.2 Close the purge valve.

NOTE A1.2—Some pycnometers are not equipped with a purge valve. If one is not present, omit A1.1 and A1.2 starting the calibration procedure with A1.3.

A1.3 Place a calibration ball in the sample chamber, locking the sample cup firmly in position. Adjust the measuring handwheel so that the readout is approximately the volume of the calibration ball.

NOTE A1.3—Calibration balls are provided in two sizes with the pycnometer: A38.1-mm (1.5-in.) calibration ball having a volume of 28.96 ± 0.15 cm^3 and a 25.4-mm (1.0-in.) calibration ball having a volume of 8.58± 0.15 cm^3. The calibration ball chosen should be that closest in volume to the anticipated volumes of the test specimens.

A1.4 Simultaneously rotate the reference handwheel and the measuring handwheel counterclockwise to their respective stops. Rotate the measuring handwheel clockwise to the starting number which is clearly marked on the pycnometer. Wait 1 min, then close the coupling valve.

A1.5 Simultaneously rotate both handwheels clockwise until the reference handwheel rests against the stop, keeping the pointer on scale during this process.

NOTE A1.4—When making final adjustments of the counter and the differential pressure indicator, always approach the final setting from the same direction.

A1.6 Repeat A1.3 and A1.5 several times until at least five values for the calibration ball are obtained. Average these values and obtain a correction factor based upon the difference between the known volume of the calibration ball and the determined volume.

NOTE A1.5—The zero will vary slightly from day to day, probably due to temperature variations. Take at least five determinations and establish the average. Determine this in exactly the same manner as for a specimen, except place the calibration ball in the sample cup. If a negative correction factor is obtained, add a corresponding amount to the subsequent volume readings on specimens. Similarly, if the determined volume of the calibration ball is greater than the known volume, subtract a corresponding amount from the subsequent volume readings for the specimens.