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Cellular plastics -- Determination of volume percentage of open and closed cells of rigid materials

Plastiques alvéolaires -- Détermination du pourcentage volumique de cellules ouvertes et fermées des matériaux rigides

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International Standard 4590

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Cellular plastics — Determination of volume percentage of open and closed cells of rigid materials

Plastiques alvéolaires — Détermination du pourcentage volumique de cellules ouvertes et fermées des matériaux rigides

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4590 was developed by Technical Committee ISO/TC 61, *Plastics*, and was circulated to the member bodies in July 1978.

It has been approved by the member bodies of the following countries :

Austria	Greece	Poland
Belgium	Hungary	Romania
Brazil	Iran	South Africa, Rep. of
Bulgaria	Israel	Spain
Canada	Italy	Sweden
Czechoslovakia	Japan	Turkey
Egypt, Arab Rep. of	Korea, Rep. of	USA
Finland	Mexico	USSR
France	Netherlands	Yugoslavia
Germany, F.R.	New Zealand	

The member body of the following country expressed disapproval of the document on technical grounds :

United Kingdom

Printed in Switzerland

Cellular plastics — Determination of volume percentage of open and closed cells of rigid materials

1 Scope and field of application

This International Standard specifies a general method for the determination of the volume percentage of open and closed cells of rigid cellular plastics, by measurement first of the geometrical volume and then of the air impenetrable volume of test specimens. This method provides for correcting the apparent open cell volume by taking into account the surface cells opened by cutting during specimen preparation. Two alternative methods and corresponding apparatus are specified for the measurement of the impenetrable volume. The results obtained are to be used for comparison purposes only.

2 Reference

ISO 1923, *Cellular plastics and rubber — Determination of linear dimensions.*

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 surface area, S : The total surface area of the test specimen determined by measuring its geometrical dimensions.

3.2 geometrical volume, V_g : The volume of the test specimen determined by measuring its geometrical dimensions.

3.3 surface/volume ratio, r : The ratio $\frac{S}{V_g}$ for the test specimen.

3.4 impenetrable volume, V_i : The volume of the test specimen into which air cannot penetrate and from which gas cannot escape, under the test conditions.

3.5 apparent volume percentage of open cells, ω_r : The ratio

$$\frac{V_g - V_i}{V_g} \times 100$$

It includes the volume of the cells opened during cutting of the test specimen, and depends on the nature of the cellular plastic under test and on the surface/volume ratio r of the test specimen.

3.6 corrected volume percentage of open cells, ω_o : The apparent volume percentage of open cells ω_r, corrected to take into account the surface cells opened by cutting during preparation of the test specimens.

It is the limit of the apparent volume percentage of open cells ω_r as the surface/volume ratio r approaches zero.

3.7 corrected volume percentage of closed cells, ψ_o : Volume percentage remaining after accounting for corrected volume percentage of open cells :

$$\psi_o = 100 - \omega_o$$

This percentage includes the volume of the cell walls.

4 Principle

Determination of the surface area S and geometrical volume V_g of a number of test specimens, each having different geometrical surface/volume ratio r.

Determination of the impenetrable volume V_i by either of two methods, namely

a) method 1 — by pressure variation (pyknometer);

b) method 2 — by volume expansion.

The determination of the impenetrable volume V_i is based on the application of the Boyle-Mariotte law to a gas confined in an indeformable chamber, first in the absence and then in the presence of a test specimen.

Calculation of the apparent volume percentage of open cells ω_r of the test specimen, plotting of the curve $\omega_r = f(r)$ and extrapolation to $r = 0$, followed by calculation of the corrected volume percentage of open cells ω_o and the corrected volume percentage of closed cells ψ_o.

5 Test specimens

5.1 Number and shape

A minimum of three sets of test specimens, with each set consisting of three rectangular parallelepipeds (see figure 1) shall be prepared from each sample. The specimens of each of the three sets are to be designated r_1, r_2 and r_3.

5.2 Preparation

Test specimens are to be cut with a bandsaw and machined if necessary, with minimum deformation to the original cell structure. They shall be free of dust, voids and moulding skins.

Hot-wire cutting shall not be used.

5.3 Dimensions

The required test specimen dimensions depend on the specific method used to measure the impenetrable volume V_i. Initial specimen sizes are to be cut as follows :

Method 1 : Pressure variation (pyknometer)

length : 40 mm
width : 30 mm
thickness : 20 mm

Method 2 : Volume expansion

length : 100 mm
width : 30 mm
thickness : 30 mm

5.4 Sectioning of test specimens

Both methods require that specimens r_2 and r_3 of each set be further sectioned as shown in figure 1 to provide a range of surface/volume ratios for testing.

6 Conditioning and testing atmospheres

The test specimens shall be conditioned for not less than 16 h at 23 ± 2 °C and 50 ± 5 % relative humidity prior to testing. It is important that the test be conducted at 23 ± 2 °C and preferably at controlled and moderate humidity, i.e. 50 ± 5 % relative humidity.

7 Measurement of surface area S and geometrical volume V_g

7.1 Determine the linear dimensions of each test specimen according to ISO 1923, except that measurements shall be made to the nearest 0,05 mm. Location of the measurement points shall be as shown in figure 2.

7.2 Calculate the average linear dimensions, the surface area S, and the geometrical volume V_g, retaining all significant figures for test specimens r_1 (one parallelepiped), r_2 (two parallelepipeds) and r_3 (four parallelepipeds). Round off the final values for surface area S to the nearest 0,01 cm^2 and for the geometrical volume V_g to the nearest 0,01 cm^3.

8 Determination of impenetrable volume V_i by method 1 : Pressure variation (pyknometer)

NOTE — The impenetrable volume V_i is to be determined according to either method 1 or method 2. The principle, description of apparatus, calibration, procedure and calculation for these two methods for determining V_i are specified in this clause and clause 9, respectively.

8.1 Principle of method 1

Determination of the following characteristics for an atmospheric pressure p_{amb} and a pressure reduction p_e in the test chamber in relation to p_{amb} :

a) the corresponding volume change δV_{A1} of the test chamber in the absence of a test specimen; this determination constitutes the calibration of the apparatus;

b) the corresponding volume change δV_{A2} of the test chamber in the presence of a test specimen.

The impenetrable volume V_i of the test specimen is given by the equation

$$V_i = \frac{\delta V_{A1} - \delta V_{A2}}{- p_e} p_B$$

where $p_B = p_{amb} + p_e$

In practice (see 8.2.2), V_i is calculated from the equivalent equation

$$V_i = \frac{l_1 - l_2}{K p_e} p_B$$

where

l_1 is a pyknometer scale reading corresponding to $K \delta V_{A1}$;

l_2 is a pyknometer scale reading corresponding to $K \delta V_{A2}$;

K is a constant relating the pyknometer scale readings to volume change in the chamber.

8.2 Description of apparatus for method 1

8.2.1 The apparatus consists of an air pyknometer that permits instant reading of the difference between internal pressure and atmospheric pressure. A schematic diagram of the apparatus is shown in figure 3. It consists essentially of the following items :

a) test chamber A, including a removable measurement chamber D of volume approximately 50 cm^3, which fits to the main part of chamber A by means of an appropriate mechanical device, a filter F and an air-tight circular joint G, to ensure impermeability and reproducibility of the geometrical volume of this part of the test chamber;

b) chamber B to create the reduced pressure.

8.2.2 The two chambers A and B are linked in parallel by means of tubing fitted with a valve T_1, which can connect or disconnect them, and a differential manometer M_1. The tubing can be connected directly to atmosphere by means of valve T_2.

When chamber D is connected to chamber A by means of the air-tight joint G and the valve T_1 is closed, the volume V_A of the combined chambers (including the free volume of the chambers and of the tubing connected to the manometer M_1 and to the valve T_1) can be modified by moving the piston P_A by means of the crank C_A.

The indicator I of the displacement of the piston P_A permits reading directly on a scale J, with a precision of 0,25 %, a value l which has been precalibrated by the manufacturer to some corresponding change δV_A, starting from an initial reference value V_0.

NOTE — The relationship between l and δV_A is defined by a proportionality constant K ($l = K \delta V_A$) as provided by the equipment manufacturer or by calibration from standard volumes. The proper value for K is achieved only if the zero reading on scale J is previously adjusted during the setting up of the air pyknometer in accordance with the manufacturer's instructions. The value of K for one commercially available air pyknometer is 2,0.

8.2.3 Chamber B can be connected directly to the atmosphere by means of valve T_3. Moreover, it is connected by means of tubing and valve T_4 to a differential manometer M_2 which indicates the pressure reduction that can be imposed at any time to the internal volume of chamber B with respect to the ambient atmosphere. The manometer M_2 shall permit the reading of the pressure reduction to 0,25 % (i.e., a pressure reduction p_e of $-$ 200 mmH_2O shall be read to within $\pm$ 0,5 mmH_2O).

The pressure in chamber B is adjustable (when valves T_1 and T_3 are closed) by moving the piston P_B by means of the crank C_B. The difference p_e (negative in the procedure for method 1) between the pressure p_B in chamber B and the atmospheric pressure p_{amb} is indicated on the manometer M_2 when valve T_4 is open :

$$p_e = p_B - p_{amb}$$

8.3 Calibration of pyknometer apparatus

Determine, according to the test procedure specified in 8.4 and for the atmospheric pressure p_{amb} prevailing at the moment of test, the reading l_1 corresponding to a pressure change $p_e = -$ 200 mmH_2O in relation to p_{amb}.

NOTES

1 In order to eliminate the need for determining l_1, each time the barometric pressure p_{amb} changes, it may be desirable to establish a calibration curve of $l_1 = f(p_{amb})$ for a given value of p_e. This can be accomplished as shown in figure 6 by repeating step 8.3 over a period of several days over which p_{amb} varies.

2 If it is desired, for some cellular materials, to determine the impenetrable volume of the test specimens at another pressure reduction p'_e, for example $-$ 300 mmH_2O, it will be necessary to plot a calibration curve for p'_e.

8.4 Procedure for method 1

8.4.1 Prior to testing, move piston P_A and P_B along the whole available distance to change completely the air in chambers A and B and the tubing. In this case, all the valves should be open. In order to obtain greater homogeneity between internal and external environments, it is advisable to repeat this operation several times.

Determine the atmospheric pressure p_{amb} to the nearest 10 Pa*.

8.4.2 Verify the zero readings of the manometers M_1 and M_2.

8.4.3 Place chamber D (containing the test specimen, if applicable) in position.

8.4.4 Again change the air in the apparatus by moving pistons P_A and P_B in the appropriate way.

8.4.5 Adjust piston P_A so as to obtain a reading $l = 0$ on scale J. Position piston P_B to enable the desired pressure reduction to be achieved.

8.4.6 Close valves T_3, T_2 and then T_1. Wait a few seconds. Both manometers M_1 and M_2 should indicate zero. If such is not the case, re-open valves T_1, T_3 and T_2, repeat the operation specified in 8.4.4 and then proceed in accordance with 8.4.5. If the manometers continue to show instability, measurements are impossible due to anomalies discussed in the annex (see clauses A.4, A.5 and A.6).

8.4.7 When the differential manometers are stable, lower the internal pressure by progressively moving piston P_B and almost simultaneously piston P_A to maintain the indicator on manometer M_1 close to zero, while observing the pressure reduction on manometer M_2.

Never move piston P_A backwards during this operation.

8.4.8 Proceed as specified in 8.4.7 until the pressure reduction $p_e = -$ 200 mmH_2O. The equilibrium must be stable. If such is not the case, there exists one of the anomalies discussed in the annex (see clauses A.4, A.5 and A.6), namely rupture of cell walls, test specimen deformation or rapid variation of p_{amb}.

NOTE — In the case of test specimens of new types of cellular materials, preliminary determinations should be performed using several values of pressure reduction p_e, chosen in arithmetic progression (for example, $-$ 100 mmH_2O, $-$ 200 mmH_2O, $-$ 300 mmH_2O, etc.). During the test, the highest value of pressure reduction should be used for which l still varies directly as p_e, and which permits a stable equilibrium to be achieved. The apparatus should be re-calibrated, in accordance with 8.3, using that value of p_e.

* 10 Pa ≈ 1 mmH_2O

8.4.9 Note the value of l_1 or l_2 corresponding to the pressure reduction p_e. Then open valve T_1 and bring progressively the pyknometer apparatus to atmospheric pressure by means of piston P_B and, if necessary, piston P_A. When the reading on manometer M_2 is equal to zero, open all valves. Never return to atmospheric pressure too abruptly.

8.4.10 Repeat twice the operations from 8.4.5 to 8.4.9. Generally the first two values of l_2 (or of l_1) will be appreciably different. Suppose that the second value is lower than the first. If the third value obtained lies between the first two and does not differ from the second by more than the precision in reading l, calculate l_2 (or l_1) as the average of the last two readings.

If these two conditions are not met and particularly if the third reading is still lower than the second, carry out fresh measurements as above until two measurements do not differ by more than the "reading" error.

8.5 Calculation for method 1

Calculate the impenetrable volume V_i from the equation

$$V_i = \frac{l_1 - l_2}{-Kp_e} p_B$$

where

l_1 is the value corresponding to the atmospheric pressure p_{amb} prevailing at the time of test;

$p_B = p_{amb} + p_e$, is expressed in conventional millimetres of water.

9 Determination of impenetrable volume V_i by method 2 : Volume expansion

9.1 Principle of method 2

In accordance with the Boyle-Mariotte law, an increase in volume of a confined gas results in a proportionate decrease in pressure. If a chamber size is increased equally with and without a test specimen in the chamber, the pressure drop will be less for the empty chamber. In this method the relative pressure drop, previously calibrated to standard volumes, is determined from the difference in scale readings of a manometer tube open to atmospheric pressure.

The impenetrable volume V_i is seen by the chamber as a smaller apparent standard volume as the percentage of open cells increases.

9.2 Description of apparatus for method 2

9.2.1 The apparatus consists of a glass tubing manometer assembly as shown schematically in figure 4. The test specimen chamber K is provided with a ground glass cap L such that a gas-tight seal can be obtained by applying vacuum grease to the joint. The chamber K is connected via an expansion bulb N to a manometer M_3 filled with water containing a few drops of a surfactant and a colorant. The liquid level in the manometer is adjusted by means of a reservoir O. (This can be controlled by using a syringe.) The gas in the chamber K is brought to atmospheric pressure prevailing at the time of test by means of the valve T_6. A scale Q, graduated in millimetres, is attached to the open arm of the manometer M_3.

9.2.2 In order to avoid errors due to fluctuation in ambient temperature, the whole apparatus is enclosed in a draft-proof case R, fitted with a transparent front panel and a trap door S through which test specimens can be introduced into the chamber K.

NOTE — Several models of such apparatus have been constructed and used successfully, observing the following parameters :

a) volume V_K of the chamber K and glass tubing to mark U_1 : 310 cm^3

b) volume V_N of the expansion bulb between marks U_1 and U_2 : 10,5 cm^3

c) height of mark U_2 above the bottom of the manometer : at least 650 mm

d) minimum internal diameter of the glass tubing : 10 mm.

9.3 Calibration of volume expansion apparatus

9.3.1 Six calibrated standards are required (for example brass cylinders) having volumes up to 150 cm^3, known with an accuracy of 0,1 cm^3.

9.3.2 With valve T_6 open, adjust the liquid level in the manometer M_3 to mark U_2 and note to the nearest millimetre the corresponding level W_1 on the open arm of the manometer.

9.3.3 Raise the liquid level up to the mark U_1. Close the valve T_6. Let the volume of the chamber K (including the tubing up to U_1) be V_K and the atmospheric pressure prevailing at that moment be p_{amb}.

9.3.4 Lower both liquid levels by withdrawing the liquid until the level in the closed arm reaches the mark U_2, corresponding to an expansion δV_K. Perform this operation slowly, controlling the speed so that the liquid level passes from mark U_1 to mark U_2 in 60 ± 1 s. Wait 30 ± 1 s to allow the liquid still on the wall of the expansion bulb N to rejoin the manometric liquid, constantly keeping the liquid level at mark U_2. At the end of this time, read the liquid level W_2 in the open arm of the manometer, rounding to the nearest millimetre. Then slowly open valve T_6, set the liquid at mark U_1 and repeat the previous operations until two successive identical readings, rounded to the nearest millimetre, are obtained.

9.3.5 Remove the cap L, insert in the test chamber K a calibrated standard of known volume V_c and replace the cap.

IMPORTANT NOTE — To meet the required stability condition for V_K (see the annex, clause A.1), it is imperative that cap L is always placed in the same position on the chamber K because even a small variation in the position of the cap on the chamber can produce a significant variation in the initial volume.

Repeat the operations specified in 9.3.3 and 9.3.4, and record to the nearest millimetre the level W_3 on the open arm of the manometer.

9.3.6 Calculate the ratio

$$\frac{W_2 - W_3}{W_1 - W_3}$$

where

W_1 is the reading of the initial level;

W_2 and W_3 are, respectively, the manometric readings after expansion for the test chamber K without and with the calibrated standard present.

Then

$$\frac{W_2 - W_3}{W_1 - W_3}(V_K + \delta V_K) = V_c$$

9.3.7 Repeat the operations specified in 9.3.2 to 9.3.5 using other calibrated standards having volumes V'_c, V''_c, etc.

For V'_c, the readings will be W'_1, W'_2, W'_3 and

$$\frac{W'_2 - W'_3}{W'_1 - W'_3}(V_K + \delta V_K) = V'_c$$

Plot these results on a graph having, as abscissae, the values of V_c, V'_c, etc., and for the ordinates the corresponding values of the ratio

$$\frac{W_2 - W_3}{W_1 - W_3}$$

The graph should be a straight line passing through the origin.

This graph (see figure 5) will be used for the determination of the impenetrable volume V_i of the test specimens.

9.4 Procedure and calculation for method 2

9.4.1 Using a test specimen in place of a calibrated volume standard, follow the same procedure as for the calibration (see 9.3).

9.4.2 Calculate the ratio

$$\frac{W_2 - W_3}{W_1 - W_3}$$

obtained with the test specimen and read from the calibration graph (see figure 5) the corresponding value of the impenetrable volume V_i from the abscissae.

10 Correction for surface cells opened by cutting

Determine the apparent volume percentage of open cells of test specimens, ω_r, corresponding to various values of $r = S/V_g$. To do this, use at least three test specimens for each of three values of r (consisting of one parallelepiped for r_1, two parallelepipeds for r_2, and four parallelepipeds for r_3). These values will be used for plotting the straight line $\omega_r = f(r)$ and its extrapolation for $r = 0$ which gives the desired ω_o.

The cutting pattern for the different values of r is shown in figure 1; an example of the plotting of the straight line $\omega_r = f(r)$ is shown in figure 7.

NOTE — Should this straight line intercept the ordinate below the origin, either the apparatus is not working properly or the test procedure has not been followed properly.

11 Expression of results

11.1 Apparent volume percentage of open cells

Calculate the apparent volume percentage of open cells ω_r of the test specimens from the equation

$$\omega_r = \frac{V_g - V_i}{V_g} \times 100$$

where

V_g is the geometrical volume, in cubic centimetres, of the test specimens determined in accordance with 7.2;

V_i is the impenetrable volume, in cubic centimetres, of the test specimens determined in accordance with either method 1 (8.5) or method 2 (9.4.2).

11.2 Corrected volume percentage of open cells

Plot the curve $\omega_r = f(r)$ and, by extrapolating to $r = 0$, determine the corrected volume percentage of open cells ω_o.

11.3 Corrected volume percentage of closed cells

Calculate the corrected volume percentage of closed cells ψ_o from the equation

$$\psi_o = 100 - \omega_o$$

12 Test report

The test report shall include the following information :

a) reference to this International Standard;

b) identification and description of the cellular material;