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BSI Standards Publication

Rigid cellular plastics — Determination of the volume percentage of open cells and of closed cells (ISO 4590:2016)

National foreword

This British Standard is the UK implementation of EN ISO 4590:2016. It supersedes BS EN ISO 4590:2003 which is withdrawn.

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Plastiques alvéolaires rigides - Détermination du pourcentage volumique de cellules ouvertes et de cellules fermées (ISO 4590:2016)

Harte Schaumstoffe - Bestimmung des Volumenanteils offener und geschlossener Zellen (ISO 4590:2016)

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European foreword

This document (EN ISO 4590:2016) has been prepared by Technical Committee ISO/TC 61 “Plastics” in collaboration with Technical Committee CEN/TC 249 “Plastics” the secretariat of which is held by NBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 2017, and conflicting national standards shall be withdrawn at the latest by February 2017.

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The text of ISO 4590:2016 has been approved by CEN as EN ISO 4590:2016 without any modification.

Contents

Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 10, *Cellular plastics.*

This third edition cancels and replaces the second edition (ISO 4590:2002), which has been technically revised with the following changes:

— changes on Clause 2;

— introduction of a new test method based on the variation of the volume which is named 2b and is explained under 9.5 to 9.7;

— references to the test methods have been revised consequently and the cross references;

— some editorial updates have been introduced.

Introduction

The method 2b is included in order to update the basics of the method with the modern apparatus. This International Standard kept the same measurement equipment since the first version of 1981 and new test equipment has been included in accordance with the technical advances. The equipment, its performance and calibration, and the calculation of the new method are described in 9.5 to 9.9.

Rigid cellular plastics — Determination of the volume percentage of open cells and of closed cells

1 Scope

This International Standard specifies a general procedure for the determination of the volume percentage of open and of closed cells of rigid cellular plastics, by measurement first of the geometrical volume and then of the air-impenetrable volume of test specimens.

The procedure includes the correction of the apparent open-cell volume by taking into account the surface cells opened by cutting during specimen preparation. Three alternative methods (method 1, method 2a and method 2b), and corresponding apparatus, are specified for the measurement of the impenetrable volume.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
surface area
S
total surface area of the test specimen determined by measuring its geometrical dimensions

3.2
geometrical volume
V_g
volume of the test specimen determined by measuring its geometrical dimensions

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

3.4
impenetrable volume
V_i
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

ratio

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

Note 1 to entry: 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_0

apparent volume percentage of cells ω_r, corrected to take into account the surface cells opened by cutting during preparation of the test specimen

Note 1 to entry: 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_0

volume percentage remaining after accounting for the corrected volume percentage of open cells

$$\psi_0 = 100 - \omega_0$$

Note 1 to entry: This percentage includes the volume of the cell walls.

4 Principle

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

The impenetrable volume V_i is determined by either of two methods, namely

a) method 1 — by pressure variation (pyknometer), and

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.

The apparent volume percentage of open cells ω_r of the test specimen is calculated by plotting the curve $\omega_r = f(r)$ and extrapolating to $r = 0$, followed by calculation of the corrected volume percentage of open cells ω_0 and the corrected volume percentage of closed cells ψ_0.

5 Test specimens

5.1 Number

A minimum of three test specimens shall be prepared for each test. A total of three tests shall be carried out per test specimen.

5.2 Preparation

Cut test specimens out with a band saw and machine them if necessary, taking care that there is no deformation to the original cell structure other than at the surface. The specimens 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 shall be as follows.

— Method 1: Pressure variation (pyknometer) and method 2b

length: (25 ± 1) mm

width: (25 ± 1) mm

thickness: (25 ± 1) mm

— Method 2a: Volume expansion

length: (100 ± 1) mm

width: (30 ± 1) mm

thickness: (30 ± 1) mm

5.4 Sectioning of test specimens

All three 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.

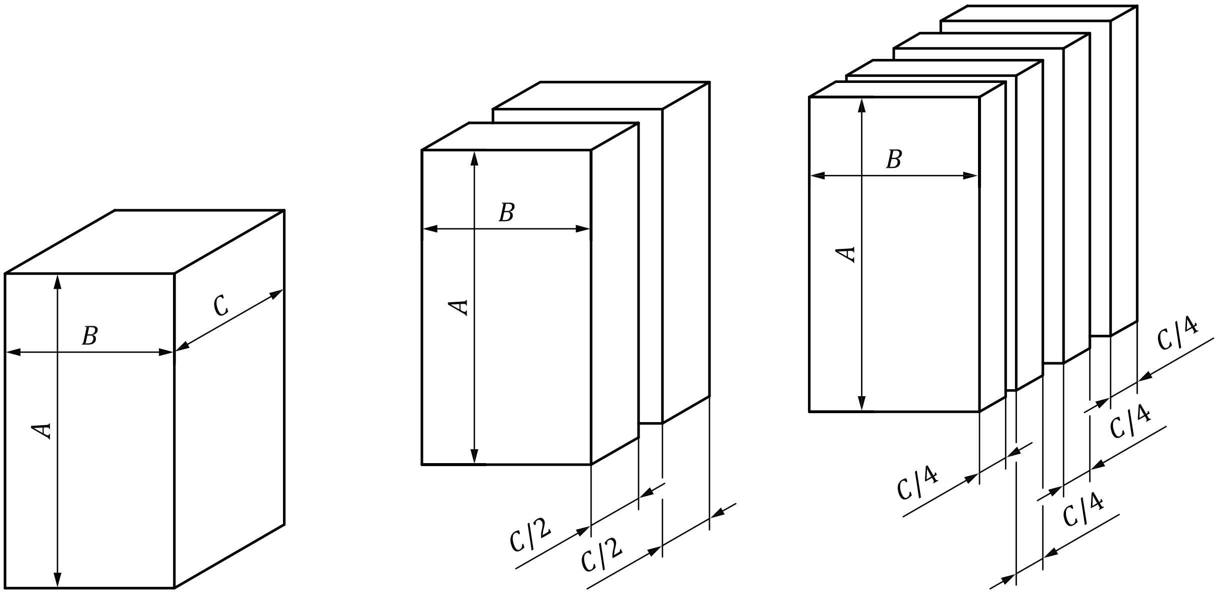
6 Conditioning and test atmospheres

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) %.

7 Measurement of surface area S and geometrical volume V_g

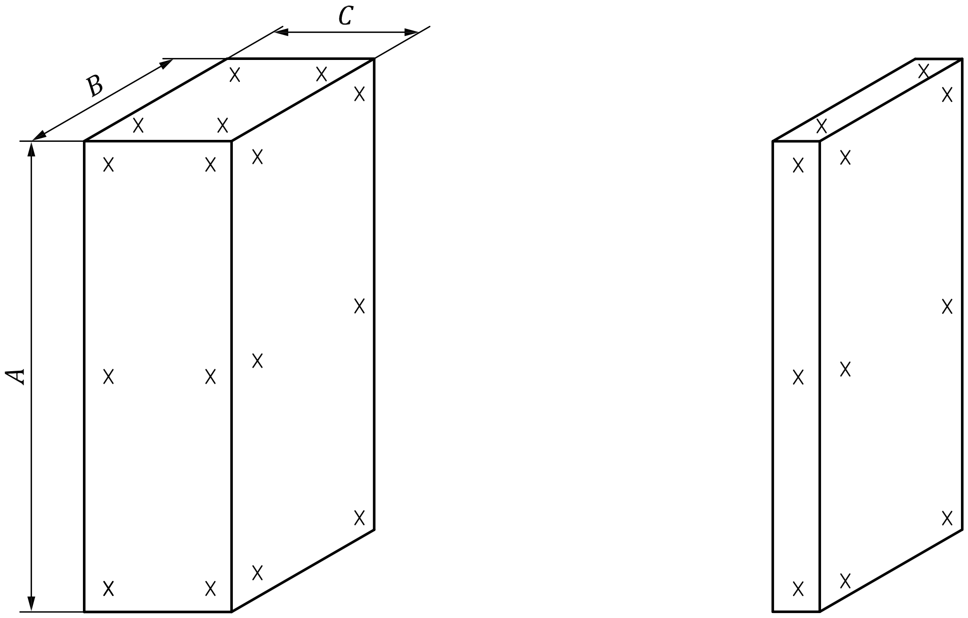
7.1 Determine the linear dimensions of each test specimen in accordance with ISO 1923, except that measurements shall be made to the nearest 0,05 mm. The locations of the measurement points shall be as shown in Figure 2.

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.



(r1) **(r2)** **(r3)**

Figure 1 — Pattern for cutting test specimens



a) Thick specimen **b) Thin specimen**

Figure 2 — Locations of measurement points

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

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

8.1 Principle of method 1

The following characteristics are determined for an atmospheric pressure p_{amb} and a pressure reduction p_{e} in the test chamber in relation to p_{amb}:

a) the corresponding change in volume $\delta V_{\mathrm{A}1}$ of the test chamber in the absence of a test specimen; this determination constitutes the calibration of the apparatus;

b) the corresponding change in volume $\delta V_{\mathrm{A}2}$ of the test chamber in the presence of a test specimen.

The impenetrable volume V_{i} of the test specimen is given by Formula (1):

$$V_{\mathrm{i}} = \frac{\delta V_{\mathrm{A}1} - \delta V_{\mathrm{A}2}}{-p_{\mathrm{e}}} p_{\mathrm{B}} \qquad (1)$$

where

p_{B} is equal to $p_{\mathrm{amb}} + p_{\mathrm{e}}$.

In practice (see 8.2.2), V_{i} is calculated from the equivalent Formula (2):

$$V_{\mathrm{i}} = \frac{l_1 - l_2}{-Kp_{\mathrm{e}}} p_{\mathrm{B}} \qquad (2)$$

where

l_1 is the pyknometer scale reading corresponding to $K\delta V_{\mathrm{A}1}$;

l_2 is the pyknometer scale reading corresponding to $K\delta V_{\mathrm{A}2}$;

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

8.2 Description of apparatus for method 1

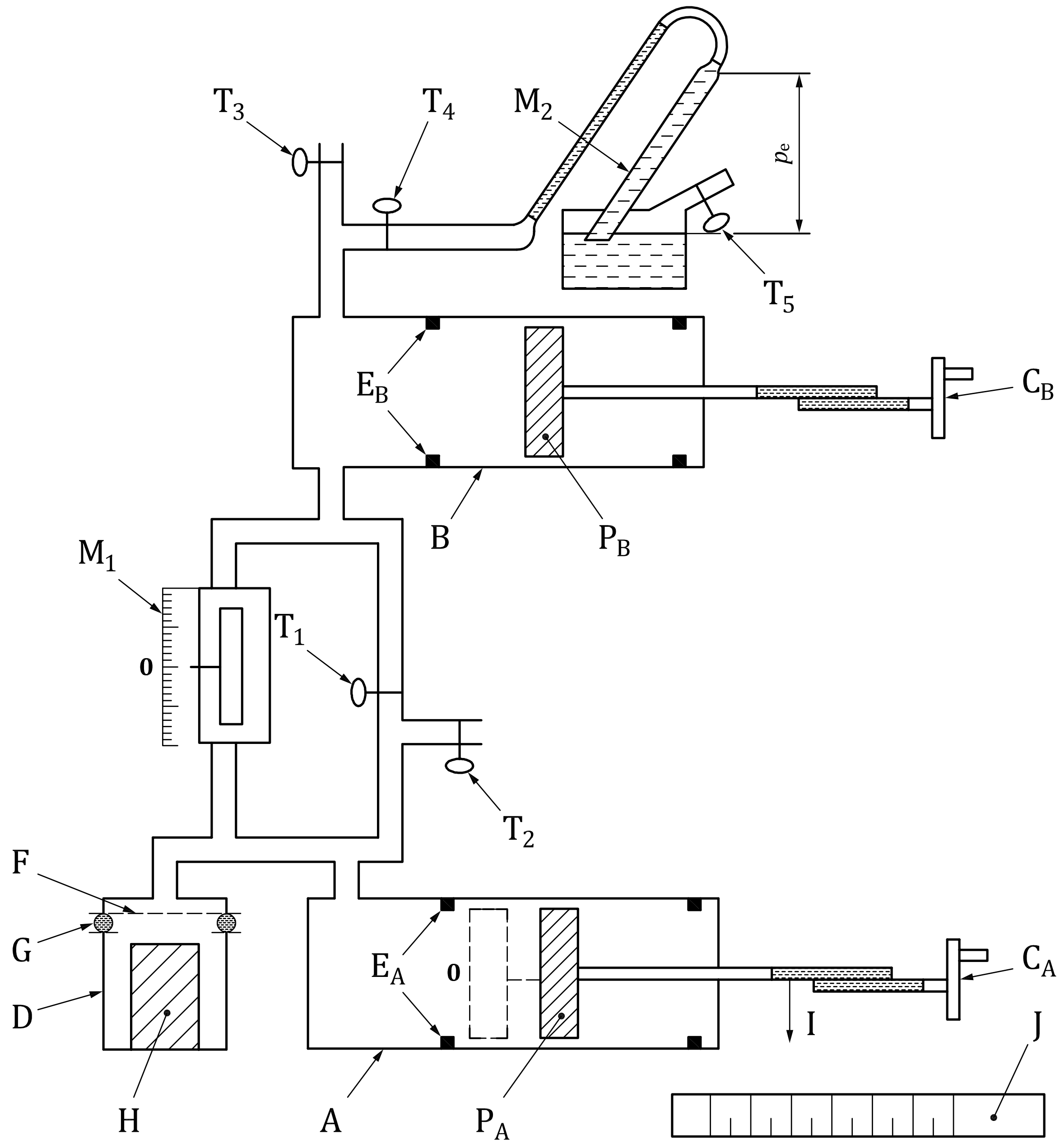
8.2.1 The apparatus consists of an air pyknometer that permits instant reading of the difference between internal pressure and atmospheric pressure. A schematic diagram of the apparatus is shown in Figure 3. It consists essentially of the following items:

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 airtight 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 airtight 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 piston P_{A} by means of crank C_{A}.



Key

A	test chamber	H	test specimen
B	reduced-pressure chamber	I	indicator
C_A, C_B	cranks	J	scale
D	measurement chamber	M_1, M_2	differential manometers
E_A, E_B	endpoints for displacement of pistons	P_A, P_B	pistons
F	filter	T_1 to T_5	valves
G	airtight joint		

Figure 3 — Schematic diagram of apparatus for determination of impenetrable volume V_i by method 1

The indicator I of the displacement of 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 obtained 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 on 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 ±0,5 mmH_2O).

The pressure in chamber B is adjustable (when valves T_1 and T_3 are closed) by moving piston P_B by means of 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:

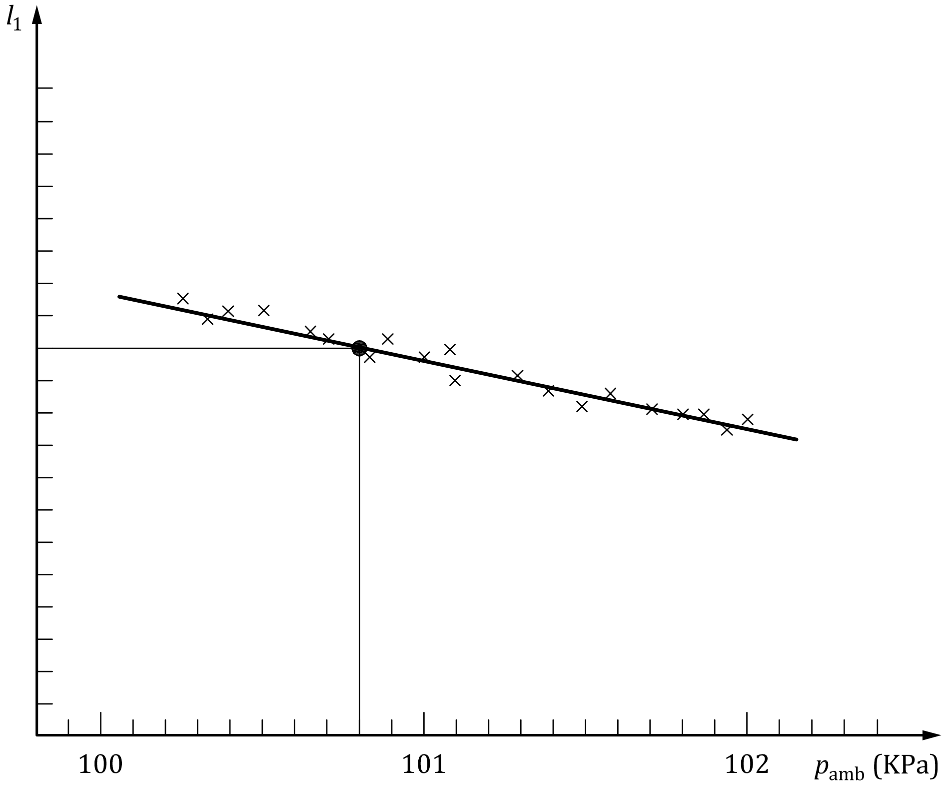
$$p_e = p_B - p_{amb} \tag{3}$$

8.3 Calibration of pyknometer apparatus

Determine, in accordance with 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}.

NOTE 1 In order to eliminate the need to determine l_1 each time the barometric pressure p_{amb} changes, it can 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 4 by repeating the calibration procedure over a period of several days over which p_{amb} varies.

NOTE 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'.



Key

l_1	scale reading
p_{amb} (KPa)	atmospheric pressure

Figure 4 — Calibration graph for method 1 (p_e = −200 mmH$_2$O)

8.4 Procedure for method 1

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

Determine the atmospheric pressure p_{amb} to the nearest 10 Pa[1].

8.4.2 Verify the zero readings of 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 Annex A (see A.4, A.5 and A.6).

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[1] 10 Pa ≈ 1 mmH$_2$O

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 shall be stable. If such is not the case, there exists one of the anomalies discussed in Annex A (see A.4, A.5 and A.6), namely rupture of cell walls, test specimen deformation or rapid variation of p_{amb}.

In the case of test specimens of new types of cellular material, preliminary determinations shall 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 the pressure reduction shall be used for which l still varies directly as p_e and which permits a stable equilibrium to be achieved. The apparatus shall be re-calibrated, in accordance with 8.3, using that value of p_e.

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 progressively bring 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_1, 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 Formula (4):

$$V_i = \frac{l_1 - l_2}{-Kp_e} p_B \tag{4}$$

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 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 the size of a chamber 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 2a

9.2.1 The apparatus consists of a glass-tubing manometer assembly as shown schematically in Figure 5. The test specimen chamber K is provided with a ground-glass cap L such that a gastight 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 chamber K is brought to atmospheric pressure prevailing at the time of test by means of the valve T_5. 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 fluctuations in ambient temperature, the whole apparatus shall be enclosed in a draughtproof case R, fitted with a transparent front panel and a trap door S through which test specimens can be introduced into the chamber K.

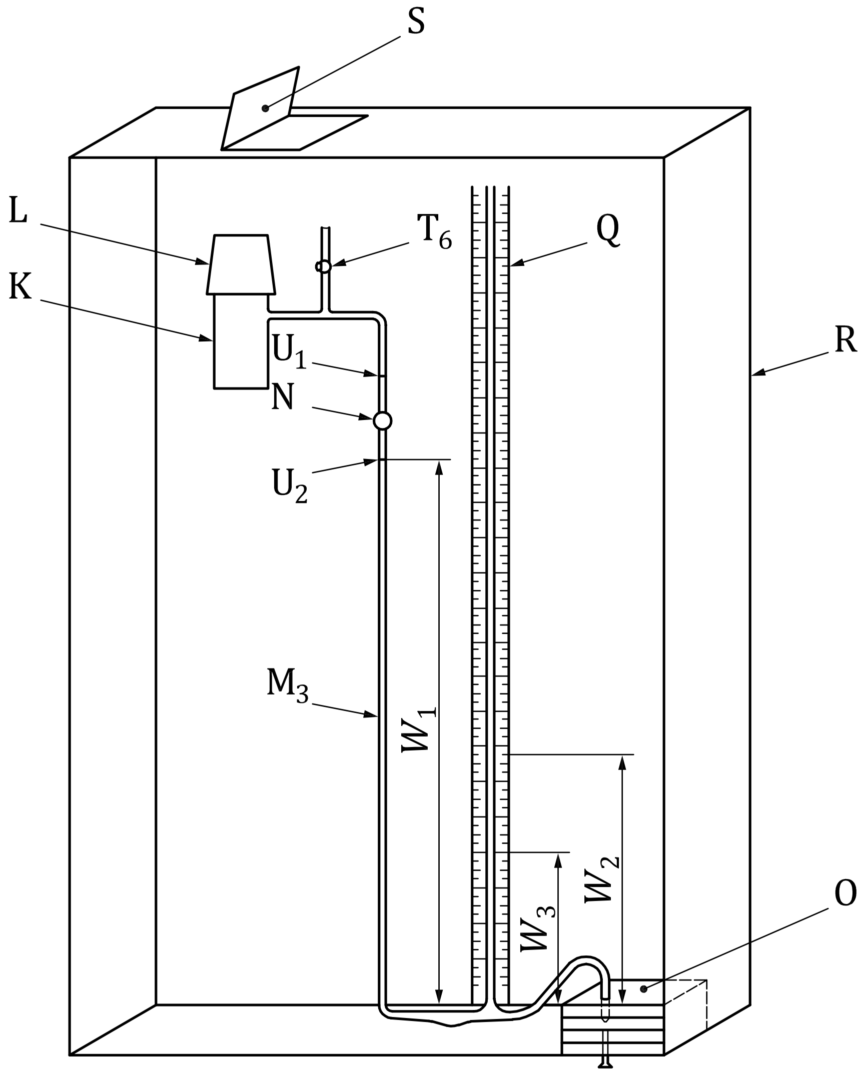
NOTE Several models of such apparatus have been constructed and used successfully, observing the following parameters:

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.



Key

K	test specimen chamber	R	draughtproof case
L	ground-glass cap	S	trap door
M_3	manometer	T_6	valve
N	expansion bulb	U_1, U_2	marks
O	reservoir	W_1, W_2, W_3	liquid levels
Q	scale		

Figure 5 — Schematic diagram of apparatus for determination of impenetrable volume V_i by method 2a

9.3 Calibration of apparatus for method 2a

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 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 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 — To meet the required stability condition for V_K (see A.1), it is imperative that the 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

$$\frac{W_2 - W_3}{W_1 - W_3} \qquad (5)$$

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

$$\frac{W_2 - W_3}{W_1 - W_3}\left(V_K + \delta V_K\right) = V_C \qquad (6)$$

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

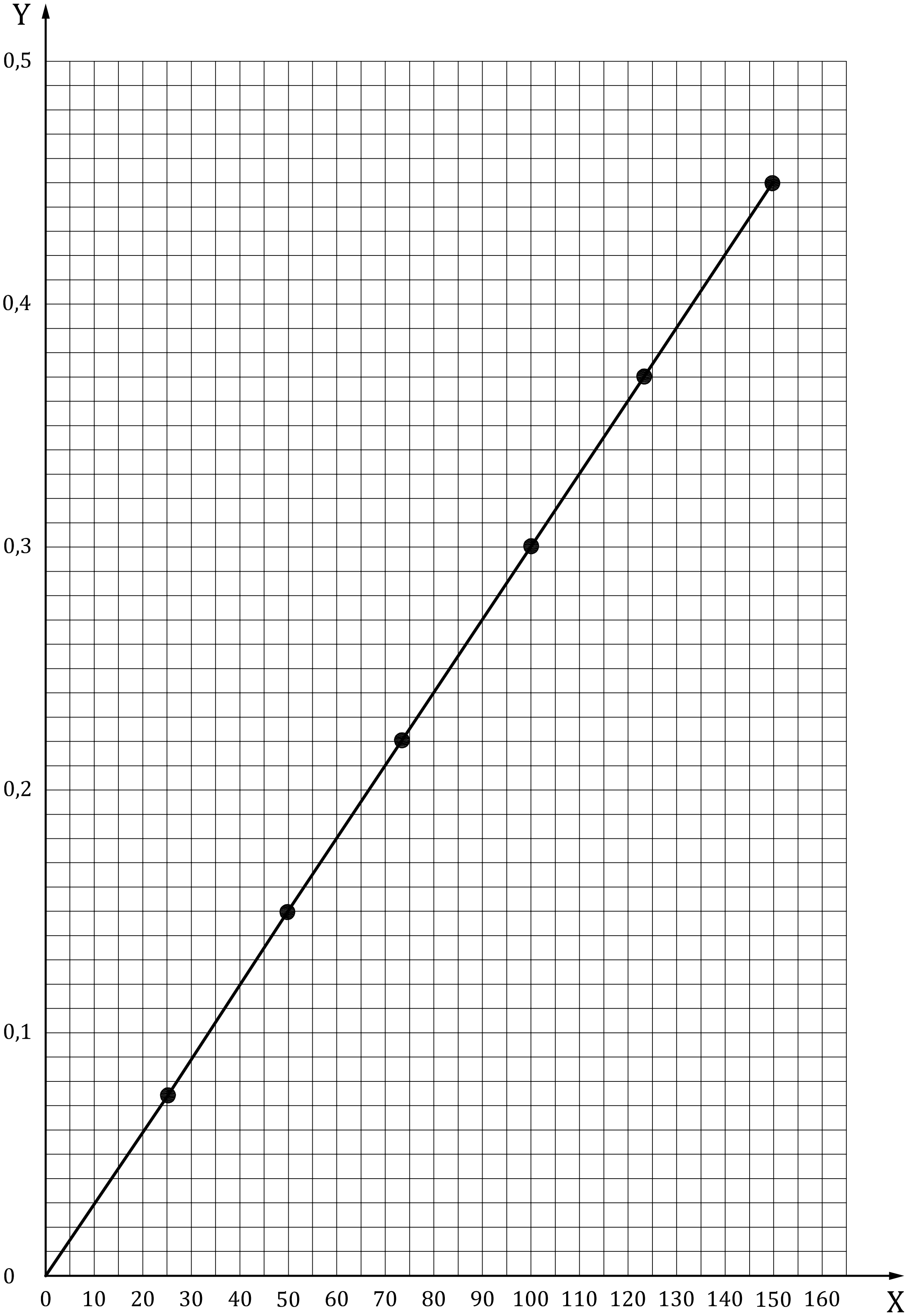
$$\frac{W_2' - W_3'}{W_1' - W_3'}\left(V_K + \delta V_K\right) = V_C' \qquad (7)$$

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

$$\frac{W_2 - W_3}{W_1 - W_3} \qquad (8)$$

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

This graph (see Figure 6) will be used for the determination of the impenetrable volume V_i of the test specimens.



Key

X volume V_c of calibrated standard (cm^3)

Y (W_2-W_3)/(W_1-W_3)

Figure 6 — Calibration graph for method 2

9.4 Procedure and calculation for method 2a

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).

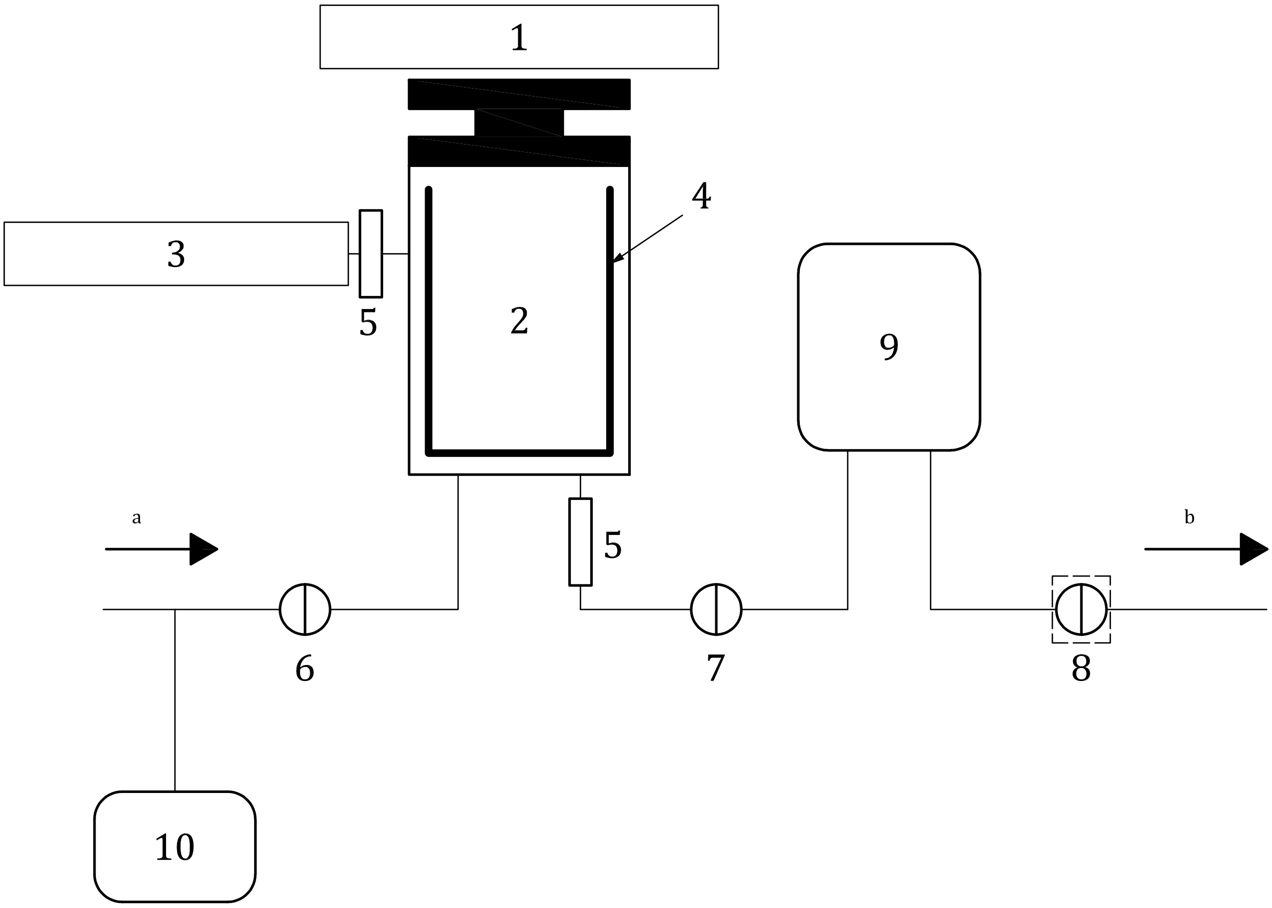
9.4.2 Calculate the ratio

$$\frac{W_2 - W_3}{W_1 - W_3} \tag{9}$$

obtained with the test specimen and read from the calibration graph (see Figure 6) the corresponding value of the impenetrable volume V_i from the abscissae.

9.5 Description of apparatus for method 2b

9.5.1 **Apparatus**. In Figure 7, a schematic diagram of the equipment is shown. The equipment shall have the following features:



Key

1	sample chamber closure
2	sample chamber
3	pressure transducer
4	sample chamber cap
5	filter
a	Gas in.
6	fill rate valve
7	two-way selector valve
8	vent rate valve
9	expansion chamber
10	over pressure relief valve
b	Gas out.

Figure 7 — Schematic diagram of the apparatus for method 2b

9.5.1.1 Sample chamber, having a known volume ($V_{chamber}$) between 30 cm^3 and 150 cm^3.

9.5.1.2 Expansion chamber, with a volume (V_{exp}) which produces an appreciable pressure drop.

9.5.1.3 Gage pressure transducer, capable of measuring 0 kPa to 175 kPa.

9.5.1.4 Pressure relief valve, to avoid over pressurization of the gage pressure transducer.

9.5.1.5 Filter, to prevent powder from contaminating the gage pressure transducer and selector valves.

9.5.1.6 Input flow control toggle and needle valves, or alternative means to control pressurization.

9.5.1.7 Output flow control toggle and needle valves, or alternative means to vent the gas.

9.5.1.8 Two-way selector valve, to connect the expansion chamber to the sample chamber.

9.5.1.9 Nonporous calibration standard, (for example, a stainless steel sphere) of known volume which fills from 1/3 to 2/3 of the sample chamber.

9.5.1.10 Digital meter, for reading the pressure from the transducer to at least 0,01 kPa.

9.5.1.11 Sample chamber closure.

9.5.1.12 Sample chamber cap.

9.6 Calibration of apparatus for method 2b

Through this calibrating method, $V_{chamber}$ and V_{exp} are determined.

A pressure between 0 kPa and 152 kPa shall be used for the calibration.

A measurement without any test specimen in the sample chamber but with sample chamber cap at regular test pressure (normally 20 kPa) is realized; follow the same procedure as for test procedure (see 9.7). The values p_1 and p_2 are determined.

Afterwards, the calibration standard (9.5.1.9) is introduced to the sample chamber cap. Testing this calibration standard at the same conditions new values for the pressures are obtained, p_1' and p_2'.

Through the values of p_1, p_2, p_1' and p_2' and the volume of the calibration standard, the $V_{chamber}$ and V_{exp} are determined as follows:

- $p_1V_{chamber} = p_2 (V_{chamber} + V_{exp})$;
- $p_1' (V_{chamber} - V_{cal}) = p_2'(V_{chamber} - V_{cal} + V_{exp})$;
- $V_{exp} = V_{chamber}*(p_1 - p_2)/p_2$;
- $p_1'*(V_{chamber} - V_{cal}) = p_2' (V_{chamber} - V_{cal}) + p_2' *V_{chamber} *(p_1 - p_2)/p_2$;
- $V_{chamber} = V_{cal}(p_1' - p_2')/[(p_1' - p_2') - (p_1 - p_2)* p_2'/p_2]$.

9.7 Test procedure for method 2b

9.7.1 Measure the length, height and width of the test specimens in accordance with Clause 7. Then weight the test specimens.

9.7.2 With opened equipment, empty sample chamber (only with the sample chamber cap inside) and all valves opened, a zero pressure in comparison with the air pressure shall be determined.

9.7.3 Then the flow valve shall be closed.

9.7.4 Afterwards, operate the two-way valve so that it isolates the expansion chamber (reference) from the rest of the system.

9.7.5 Finally open the vent valve.

9.7.6 Introduce the test specimen in the sample chamber cap and close the sample chamber.

9.7.7 The air and vapors trapped within pores will be removed from the test specimen by a prolonged purge when all valves are opened. The number and time of purges shall be registered.

It is recommended to use a pressure for the purges between 15 kPa and 20 kPa.

9.7.8 Once the purges are done, maintaining the gas flow, all valves of the system shall be opened and the pressure reading of the gage pressure transducer shall be zero.

9.7.9 Close the two-way valve and the vent valve so that it again isolates the expansion volume (reference) chamber from the rest of the system.

9.7.10 Open the flow valve to reach the set pressure for the sample chamber.

It is recommended to use as set pressure in the sample chamber of 20 kPa. In some cases, it is desirable to use lower pressures to avoid that the pressure distorts the test specimen. At any case, the test pressure shall be noted.

9.7.11 Close the flow valve and allow the pressure to stabilize (with 15 s is time enough for most test specimens). Record the final pressure as p_1.

9.7.12 Immediately operate the two-way selector valve so that it again connects the expansion chamber (reference) with the sample chamber and allow the pressure to stabilize. Record the final pressure as p_2.

9.7.13 Finally open the vent valve and allow the pressure to fall to zero.

9.7.14 The impenetrable volume V_i of the test specimen can be calculated from Formula (10):

$$V_i = (p_2 \cdot V_{\text{chamber}} + p_2 \cdot V_{\text{exp}} - p_1 \cdot V_{\text{chamber}})/(p_2 - p_1) \tag{10}$$

9.8 Test sequence for method 2b

Three tests specimens of (25 × 25 × 25) mm are prepared. This will be the set r_1. Once analysed, in accordance with the procedure described in 9.7, these will be cut in two equal half parts and then become the set r_2. Repeat the analysis in accordance with 9.7. Afterwards, all the parts are divided again in two equal parts having the set r_3 and analysed according to 9.7. In this way:

- set 1: formed by three test specimens of (25 × 25 ×25) mm. Each test specimen is analysed separately;
- set 2: formed by 6 test specimens of (25 × 25 × 12,5) mm. Each two test specimens coming from the same r_1 specimen are analysed together;
- set 3: formed by 12 test specimens of (25 × 25 × 6,25) mm. Each four test specimens coming from the same r_1 test specimen are analysed together.

Measuring the sets of test specimens in this way, the posterior extrapolation of the ratio S/V to zero allows the evaluation of the cut during the test specimens preparation process.

The dimensions of each test specimen shall be determined separately in accordance with Clause 7. The average dimensions of each set, formed by one, two or four test specimens, are calculated. These averages are used for the calculation of the surfaces and geometric volumes. The test specimens of the set r_1 are weighted separately. Each group of two test specimens coming from r_2 and each group of four test specimens coming from r_3 are weighted together.

Once measured and weighted, the test specimens are analysed in accordance with the procedure described in 9.7, having the percentage of closed/open cells.

The obtained data will be treated as described in 9.9.

9.9 Calculations and expression of results for method 2b

9.9.1 Geometric volume, V_g, in cm^3:

- For r_1 $V_g = (A \times B \times C)$
- For r_2 $V_g = 2 \times [(A_1 + A_2)/2 \times (B_1 + B_2)/2 \times (C_1 + C_2)/2]$
- For r_3 $V_g = 4 \times [(A_1 + A_2 + A_3 + A_4)/4 \times (B_1 + B_2 + B_3 + B_4)/4 \times (C_1 + C_2 + C_3 + C_4)/4]$

9.9.2 Surface area, S, in cm^2:

- For r_1 $S = 2\ (A \times B + A \times C + B \times C)$
- For r_2 $S = 4\{[(A_1 + A_2)/2] \times [(B_1 + B_2)/2] + [(A_1 + A_2)/2] \times [(C_1 + C_2)/2] + [(B_1 + B_2)/2] \times [(C_1 + C_2)/2]\}$
- For r_3 $S = 8\ \{[(A_1 + A_2 + A_3 + A_4)/4] \times [(B_1 + B_2 + B_3 + B_4)/4] + [(A_1 + A_2 + A_3 + A_4)/4] \times [(C_1 + C_2 + C_3 + C_4)/4] + [(B_1 + B_2 + B_3 + B_4)/4] \times [(C_1 + C_2 + C_3 + C_4)/4]\}$

9.9.3 Volume percentage of open cells, ω_r

$$\omega_r = [(V_g - V_i)/V_g] \times 100 \tag{11}$$

9.9.4 Volume percentage of closed cells and cell walls, ψ_r:

$$\psi_r = 100 - \omega_r \tag{12}$$

10 Correction for specimen surface cells opened during specimen preparation

10.1 For the pressure-variation method (see Clause 8)

After V_i has been determined for each of the (at least) three specimens, bisect each specimen three times along its three centre planes to give eight cubes. Determine the impenetrable volume of each set of eight cubes following 8.4.5 to 8.4.9 and record the average volume as V_d.

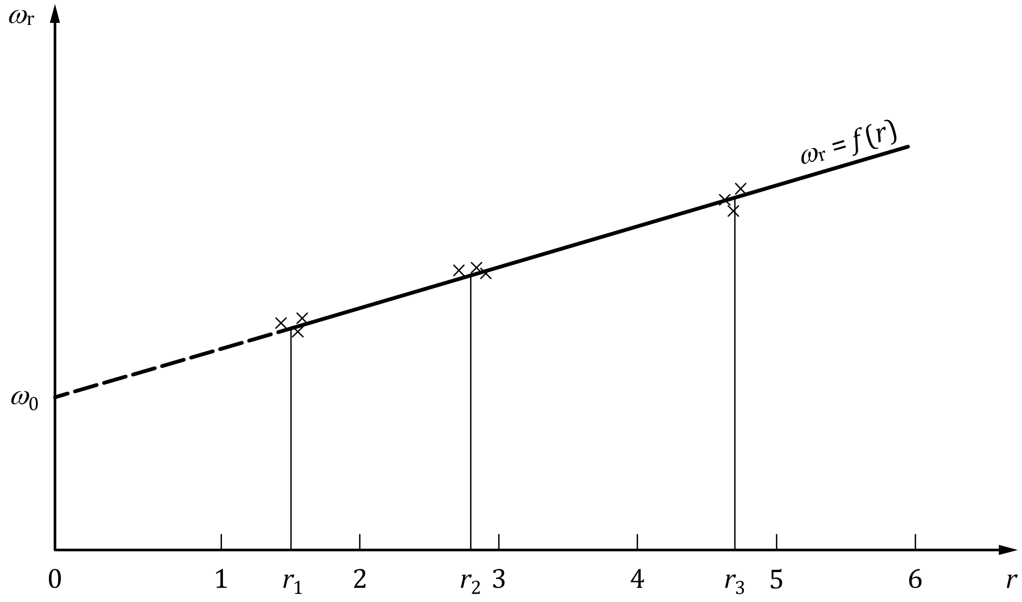
10.2 For the volume-expansion method (see Clause 9)

Determine the apparent volume percentage of open cells in the test specimens, ω_r, corresponding to various values of r (= S/V_g).

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 to $r = 0$ which gives the desired ω_0.

The cutting pattern for the different values of r is shown in Figure 1; an example of the straight line $\omega_r = f(r)$ is shown in Figure 8.

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.



Key

ω_r apparent volume percentage of open cells

r geometrical-surface/volume ratio (S/V_g)

Figure 8 — Graph for determining the correction factor for cells opened during test specimen preparation

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 Formula (13):

$$\omega_r = \frac{V_g - V_i}{V_g} \times 100 \tag{13}$$

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 (see 8.5), method 2a (see 9.4.2) or method 2b (see 9.7.14).

11.2 Corrected volume percentage of open cells

11.2.1 For the pressure-variation method (see Clause 8).

$$\omega_0 = \frac{V_g - 2V_i + V_d}{V_g} \times 100 \quad (14)$$

11.2.2 For the volume-expansion method (see Clause 9).

Plot the curve $\omega_r = f(r)$ and, by extrapolating to $r = 0$, determine the corrected volume percentage of open cells ω_0.

11.2.3 Corrected volume percentage of closed cells

Calculate the corrected volume percentage of closed cells Ψ_0 from Formula (15):

$$\Psi_0 = 100 - \omega_0 \quad (15)$$

12 Precision

12.1 The precision of the pressure-variation method (method 1) is shown in Table 1. The data are from a round robin conducted in 1981 (see the Note). Tests were conducted in five laboratories. Each test result was the average from five specimens. Each laboratory reported one result per material.

NOTE Data are available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428, USA. Request research report RR D20-1099.

Table 1 — Percentage of open cells including correction for surface cells opened during specimen preparation — Pressure-variation method (method 1)

Material	Average %	s_r %	s_L %	I_r %	I_R %
Ext PS	0,71	0,54	1,07	1,53	3,39
Ext 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

where

s_r is the within-laboratory standard deviation;

s_L is the square root of the variance between laboratories;

$I_r = 2{,}83 s_r$ (see repeatability below);

$I_R = 2{,}83\sqrt{(s_r^2 + s_L^2)}$ (see reproducibility below).

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

The accuracy of this method cannot be stated because there are no established reference materials for the characteristic being determined.

12.2 The precision of method 2a and 2b is not known, and data obtained by these methods should not be used in resolving disputes between suppliers and users.

13 Test report

The test report shall include the following information:

a) a reference to this International Standard, i.e. ISO 4590:2016;

b) all details necessary for complete identification of the cellular material tested;

c) the procedure used for the determination of impenetrable volume V_i, i.e. method 1 (pyknometer) or method 2a or method 2b (volume expansion);

d) the individual results and the mean values of the corrected volume percentage of open cells ω_0 and of closed cells ψ_0;

e) when applicable, the direction of the greatest dimension A of the test specimens (see Figure 1) in relation to any anisotropy of the material;

f) any deviation from the method specified;

g) the date of testing;

h) all details necessary to identify the test facility.

Annex A
(normative)

Notes on procedure

A.1 Stability of reference volume

It is essential that the reference volume of the test chamber is constant. This volume affects δV_{A1} and δV_{A2} (method 1) and $V_K + \delta V_K$ (method 2).

Should the reference value not be identical in the measurements with and without the test specimen, the error in determining V_i, which is determined by difference, could become important.

A.2 Influence of atmospheric pressure

The atmospheric pressure p_{amb} normally should not vary by more than 100 Pa during the period of testing with and without specimens.

In the case of method 1, the use of the calibration graph $l_1 = f(p_{amb})$ (see Figure 4) permits correcting for such variation in p_{amb}, if it exists.

On the contrary, method 2 requires verification of the stability of p_{amb} during the complete test period.

A.3 Choice of the value of p_e (method 1) or δVK (method 2)

The precision of the test method increases with an increase in p_e (or δV_K).

On the other hand, it is necessary to use p_e (or δV_K) values sufficiently low to maintain V_i constant during the test and to avoid rupture of cell walls due to pressure variations.

The most appropriate values depend on the nature of the cellular material involved. For method 1, it has been found that 200 mmH_2O is a satisfactory value for most cellular plastics.

A.4 Influence of temperature

Because the Boyle-Mariotte law assumes constant temperature, it is necessary to operate in a room with temperature control. The same requirement applies if liquid differential manometers are utilized.

The apparatus and test specimens shall be conditioned in the controlled-temperature room for a sufficient period to reach equilibrium before testing.

For the same reason, it is necessary to avoid any heating or cooling of the test chamber between two measurements, for instance, because of an abrupt shift of the test chamber from reduced pressure to atmospheric pressure.

A.5 Influence of humidity

It is advisable to work under controlled and moderate humidity conditions [for example (50 ± 5) % relative humidity]. The effect of humidity variations can be detected during the measurements (instability of the equilibrium in the initial conditions of p_e or δV_K or lack of repeatability between two successive measurements). The presence of moisture in test specimens is revealed by behaviour similar to that described in A.6.

A.6 Influence of gas occluded in test specimens

Test specimens having in their cells gases other than normal air at atmospheric pressure behave during the pressure-variation cycle as if their impenetrable volume varies with time. In method 1, this can, for example, cause instability of δV_{A2} at a given p_{e}.

Diffusion of blowing-agent gases, infusion of air by permeation through the cell walls or the presence of moisture can cause this problem.

This can generally be detected by determining, prior to the beginning of the test, whether the initial equilibrium can be maintained with the test chamber isolated from the atmosphere.

If drift or instability occurs, this can be corrected in some cases by cutting the test specimens at least one week before the test measurements are made. In other cases, where the diffusion rate is slower, correction can be made by determining V_{i} at various times for each specimen, plotting V_{i} versus the square root of elapsed time, and extrapolating the resulting straight line back to zero time. V_{i} at zero time will be free of the effect from occluded gases in the cells.

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