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Textile glass reinforced plastics — Determination of void content — Loss on ignition, mechanical disintegration and statistical counting methods

Plastiques renforcés de verre textile — Détermination de la teneur en vide — Méthodes par perte au feu, par désintégration mécanique et par comptage statistique

Reference number
ISO 7822 : 1990 (E)

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 7822 was prepared by Technical Committee ISO/TC 61, *Plastics*.

Annex A of this International Standard is for information only.

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INTERNATIONAL STANDARD ISO 7822 : 1990 (E)

Textile glass reinforced plastics — Determination of void content — Loss on ignition, mechanical disintegration and statistical counting methods

1 Scope

This International Standard specifies three methods for the determination of the void content of textile glass reinforced plastics or composites, of which the constituents are of a solid nature.

1.1 Method A — Loss on ignition

This method is applicable to composites for which the effects of the loss on ignition test on the materials are known. Most matrix resins and textile glass fibre reinforcements fall into this class.

The method is not applicable to composites for which the effects of the loss on ignition test on the resin, the reinforcement, and any fillers are unknown. This may include silicone resins, which do not burn off completely, and fillers consisting of oxides, carbonates, etc., which may gain or lose weight. Note that separate weight loss tests on individual materials will usually, but not necessarily, give the same result as when all the materials are combined.

The accuracy of the method is ±2,5 % by volume.

1.2 Method B — Mechanical disintegration

This method is applicable if the composite can be disintegrated in such a way, for example by crushing in a press, that all the enclosed voids are connected with the outside of the composite material. The method is destructive and has limited application if the matrix material shows ductile behaviour under compression, unless it can be made more brittle in an artificial way (for example by cooling).

The method is especially suitable when the densities of the constituent materials are not known or not determinable.

The method neglects the influence of any volatile constituents that could evaporate during and after disintegration. In this connection, the conditioning shall be chosen with care. The method also does not take into account any cut or exposed voids at the surface of the sample.

The accuracy of the method is ±1 % by volume.

1.3 Method C — Statistical counting

This method is applicable to composites having a void content less than or equal to 1 % by volume.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 291 : 1977, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 1172 : 1975, *Textile glass reinforced plastics — Determination of loss on ignition.*

3 Principle

3.1 Method A — Loss on ignition

Determination of the densities of the resin, the reinforcement, filler(s) (if present) and the composite. Determination of the resin content and calculation of a theoretical composite density. Comparison with the measured composite density. The difference in densities indicates the void content.

NOTE — The density of the resin, in this method, is assumed to be the same in the composite as it is in a moulded mass. Although there is no realistic way of avoiding having to make this assumption, it is nevertheless not strictly correct. Differences in curing, heating, pressure and molecular forces arising from the reinforcement surface all make the density of the resin in the composite different from the bulk resin density.

Composites containing inorganic fillers require special care. An accurate determination of the filler content and density is required if the accuracy of this method is to be maintained.

3.2 Method B — Mechanical disintegration

Determination of the mass and volume, before and after disintegration, of a fibre reinforced plastic sample to obtain the void content by density difference.

3.3 Method C — Statistical counting

Superimposition of a square grid of 20 to 200 points on a micrographic section of the material to be tested. Statistically, the proportion of points of the grid which are superimposed on voids corresponds to the void content of the material. The counting method may be manual, or semi-automatic or automatic using suitable apparatus.

4 Apparatus

4.1 Method A

4.1.1 Micrometer, having an accuracy of ±1 µm.

4.1.2 Balance, having an accuracy of ±0,1 mg.

4.1.3 Muffle furnace, capable of being maintained at 625 °C ± 5 °C.

4.2 Method B

4.2.1 Disintegrator (in its simplest form a press), in which the test material is delaminated and crushed until all the voids are connected with the outside of the composite material. It is advisable, however, to use a closed-die unit made of hardened steel as shown in figure 1.

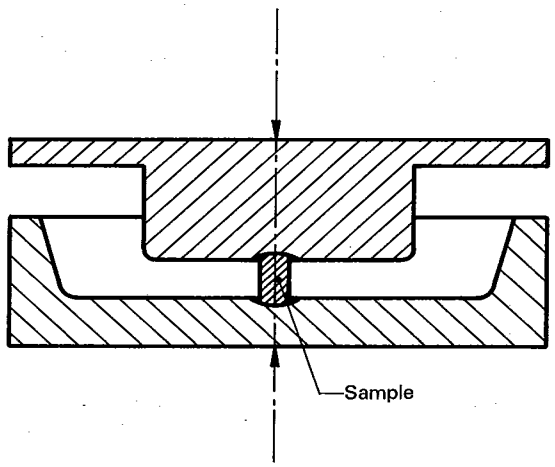


Figure 1 — Disintegration die

4.2.2 Air or gas pyknometer, suitable for measuring the volumes of the original material and the disintegrated material with an accuracy better than ±0,1 %. (See figure 2 for the principle of an air pyknometer.)

4.2.3 Balance, with an accuracy of ±0,1 mg.

4.2.4 Deep-freezing unit (if required), such as an insulated box or bottle, supplied with solid carbon dioxide or liquid nitrogen.

4.2.5 Suitable film material, such as aluminium or polyethylene.

4.3 Method C

4.3.1 Microscope, having a magnifying power up to ×400, and fitted with either counting-grid oculars or a device allowing the simultaneous viewing of a sample and a counting grid placed beside the microscope on the table.

4.3.2 Equipment for the preparation of micrographic sections, comprising

a) a cutting machine;

b) a polishing machine;

c) a mould for encasing the section in transparent resin;

d) an ultrasonic cleaning bath.

5 Preparation and number of test specimens

5.1 Methods A and B

5.1.1 The test specimens shall be representative of the composite to be examined. The quantity and form will depend on the pyknometer and the disintegration procedure used. When an air pyknometer is used, quantities of about 25 g or 12 000 mm^3, preferably in the form of strips about 38 mm long, about 10 mm wide and of the thickness of the composite, are needed for each determination.

5.1.2 From the composite to be examined, cut a series of at least five strips of appropriate size and mass. The pieces shall be clean and dry.

5.2 Method C

5.2.1 The specimens shall have the shape of a parallepiped 40 mm long and 10 mm wide, the thickness depending on the thickness of the structure from which the specimens have been cut, with a maximum value of 15 mm. The number of specimens to be taken will depend on the number of sections needed and on the nature of the structure to be tested; each specimen can give several sections.

5.2.2 Prepare a total of at least five sections from among the specimens. The polishing operation and the encasing of the sections in cold-curing resin shall be carried out in accordance with the procedures used in micrography. (See annex A.)

6 Atmosphere for conditioning and testing

6.1 Conditioning of the specimens

The test specimens shall be conditioned in one of the standard atmospheres specified in ISO 291, for at least 16 h, unless otherwise stipulated.

6.2 Test atmosphere

The specimens shall be tested in the standard atmosphere selected for conditioning (see 6.1).

7 Procedure

7.1 Method A

7.1.1 General

Densities calculated from mass and volume measurements are acceptable if the specimens are smooth, uniform, and of such

shape that the volume can be calculated accurately from the dimensions.

7.1.2 Density of the composite

7.1.2.1 The volume of each specimen shall be not less than 2 cm^3. Make dimensional measurements with the micrometer (4.1.1) at all edges (12 in all for a six-sided rectangular block). Use the averages for each dimension to calculate the volume.

7.1.2.2 The tolerance on the precision of the micrometer measurements shall be ± 1 µm. With maximum tolerance build-up on a small specimen, this could result in an error in the calculated volume of 0,6 %. For larger specimens, and with some measurements being in error on the plus side and some on the minus side, the error in the calculated volume ought not to exceed 0,2 %.

7.1.2.3 Calculate the density of each test specimen by dividing the mass by the volume; express the density in grams per cubic centimetre.

7.1.3 Density of the glass reinforcement

Most of the glass fibre reinforcements used are of the following types, having the indicated approximate density, expressed in grams per cubic centimetre:

E glass :	2,47 to 2,75
C glass :	2,49
A glass :	2,50
S glass :	2,49
D glass :	2,16
R glass :	2,58

Care shall be taken to use the exact density.

7.1.4 Density of the resin

Density measurements supplied by the resin manufacturer are acceptable if they are certified for each batch.

7.1.5 Resin content of composite

Determine the resin content of the composite in accordance with ISO 1172. The loss on ignition in that method is the resin content of the sample expressed as a percentage by mass.

7.2 Method B

Determine the volume V_1 of the test strips, using the pyknometer (4.2.2), and their mass m_1, using the balance (4.2.3). Then place the strips breadthwise between the platens of a press or in a closed die (see 4.2.1) and crush them. If the composite contains a matrix material too ductile to be disintegrated at room temperature, place the test strips in the deep-freezing unit (4.2.4) for a few minutes, cooling them with solid carbon dioxide or liquid nitrogen, depending on the material being tested. To prevent loss of material, enclose the sample in the film material (4.2.5). Inspect the crushed material and, if necessary, crush it further until a completely disintegrated material is obtained. After conditioning, determine the volume V_2 and mass m_2.

7.3 Method C

7.3.1 Determination of the parameters necessary to make the measurements

7.3.1.1 Determine the order of magnitude of the void content φ_v by a rapid preliminary examination, using the microscope (4.3.1), by applying one of the grids to one of the sections three times, examining a different "field" each time.

7.3.1.2 Select a relative error e_v and determine the total number of points to be observed P by means of the graph in figure 3. In the case of manual counting, choose the relative error so that the number of points to be counted $P\varphi_v$ is less than 100.

7.3.1.3 Select a grid, bearing in mind that the lower the void content, the greater should be the total number of points on the grid.

7.3.1.4 Calculate

a) the total number of fields:

$$N_2 = \frac{P}{N}$$

where N is the number of points on the chosen grid;

b) the number of fields for each section:

$$N_3 = \frac{\text{area of the section}}{\text{area covered by the grid}}$$

c) the number of sections n to be made:

$$n = \frac{N_2}{N_3}$$

7.3.2 Measurement of the void content

Count the total number N_1 of points of the grid that are superimposed on a void in the N_2 fields of the n microscopic sections.

Fields distributed over the same section shall not overlap each other.

8 Expression of results

8.1 Method A

Calculate the void content φ_v, expressed as a percentage by mass, using the formula

$$\varphi_v = \frac{100\,(\varrho_c - \varrho_{mc})}{\varrho_c}$$

where

ϱ_{mc} is the measured density, in grams per cubic centimetre, of the composite;

ϱ_c is the theoretical density, in grams per cubic centimetre, of the composite;

$$\varrho_c = \frac{100}{\frac{w_r}{\varrho_r} + \frac{w_f}{\varrho_f}}$$

w_f being the textile glass fibre content, expressed as a percentage by mass, of the composite;

w_r being the resin content, expressed as a percentage by mass, of the composite;

ϱ_f being the density, in grams per cubic centimetre, of the textile glass fibre;

ϱ_r being the density, in grams per cubic centimetre, of the resin.

8.2 Method B

Calculate the void content φ_v, expressed as a percentage by volume, using the formula

$$\varphi_v = 100 \left(1 - \frac{\varrho_1}{\varrho_2}\right)$$

where

ϱ_1 is the density, expressed in grams per cubic centimetre, of the composite to the nearest 0,001 g/cm^3, given by the equation

$$\varrho_1 = \frac{m_1}{V_1}$$

ϱ_2 is the density, expressed in grams per cubic centimetre, of the disintegrated material to the nearest 0,001 g/cm^3, given by the equation

$$\varrho_2 = \frac{m_2}{V_2}$$

8.3 Method C

Calculate the void content φ_v, expressed as a percentage by volume, using the formula

$$\varphi_v = \frac{N_1}{P}$$

where

N_1 is the total number of void points counted;

P is the total number of points examined.

9 Precision

The precision of this test method is not known because inter-laboratory data are not available. This method may not be suitable for use in specifications or in the case of disputed results as long as these data are not available.

10 Test report

The test report shall include the following particulars:

a) a reference to this International Standard and the method used (A, B or C);

b) an identification and description of the material tested;

c) a description of the sampling;

d) the number of determinations;

e) the results of each individual determination and the average void content as a percentage by volume;

f) any additional observations;

g) the following additional information, depending on the method used:

1) Method A

— the density of each of the specimens,

— the percentage content by mass and the theoretical density of the resin and the textile glass fibre reinforcement;

2) Method B

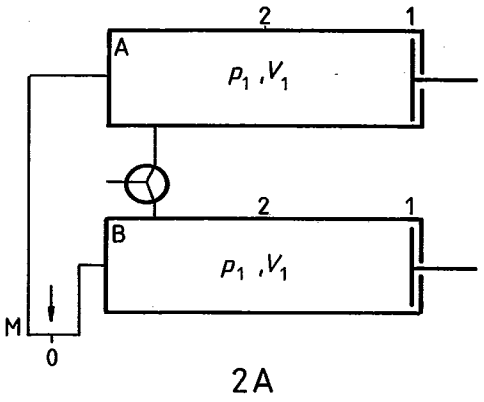
— the type of pyknometer used,

— a description of the method of freezing, if used;

3) Method C

— the number of sections made and their positions in the test specimens,

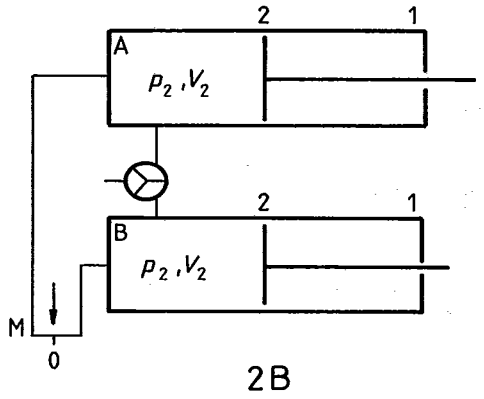
— the measurement parameters e_v and P.



2A

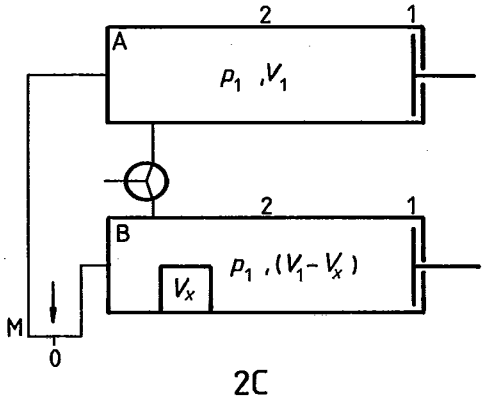
2A The apparatus has two identical chambers, each containing a piston.

Originally, the pressure p_1 is atmospheric pressure.



2B

2B With the chambers closed, piston A is moved from position 1 to position 2; in order to equalize pressures (differential manometer M = 0), piston B must be moved the same distance, i.e. from position 1 to position 2.

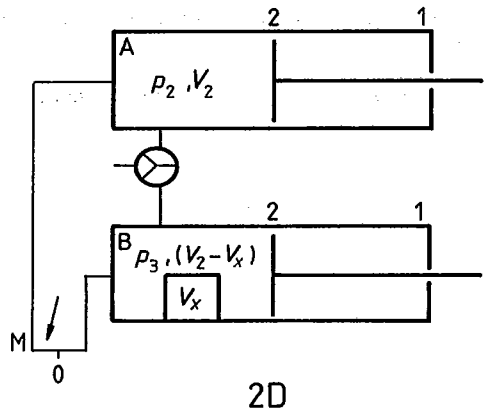


2C

2C Same starting conditions as 2A.

Sample of volume V_x is inserted in chamber B.

Figure 2 — Principle of the gas-comparison pyknometer for measurement of the true volume of materials with open pores



2D

2D With the chambers closed, piston A is moved from 1 to 2; due to V_x, when piston B is moved from 1 to 2, the pressures are not equalized.

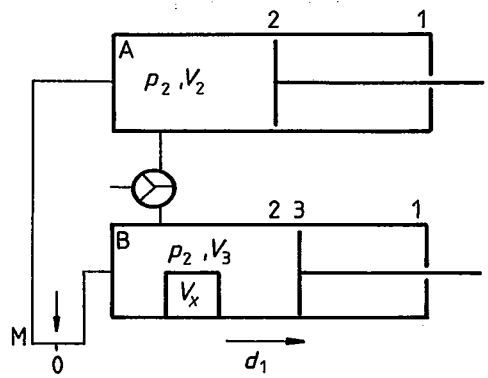
Pressure p_3 in chamber B will be higher than p_2.

$$p_1(V_1 - V_x) = p_3(V_2 - V_x)$$

$$p_3 = \frac{p_1(V_1 - V_x)}{V_2 - V_x} = \frac{p_1V_1 - p_1V_x}{\frac{p_1V_1 - p_2V_x}{p_2}} = p_2\frac{p_1V_1 - p_1V_x}{p_1V_1 - p_2V_x}$$

$$p_1V_x < p_2V_x$$

$$p_3 > p_2$$

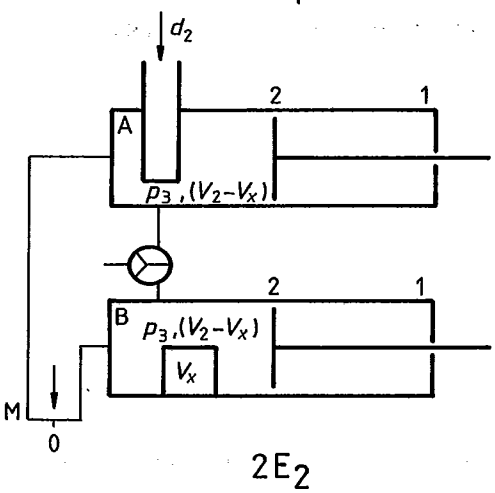


$2E_1$

2E The pressures are equalized

— either keeping p_2 constant, i.e. by moving piston B to position 3 at a distance from position 2 proportional to V_x (see $2E_1$);

— or keeping p_3 constant, i.e. by introducing into chamber A a calibrated volume equivalent to V_x; the displacement d_2 is proportional to V_x (see $2E_2$).



$2E_2$

Typically, the sample volume is about 20 cm³.

The precision attainable with the available instruments varies from 0,001 cm³ to 0,05 cm³.

A = Chamber A	0 = Equilibrium position
B = Chamber B	1 = Position 1 of the piston
M = Manometer	2 = Position 2 of the piston
p = Pressure	3 = Position 3 of the piston
V = Volume	d_1 = Piston displacement
x = Unknown	d_2 = Calibrated volume displacement

Figure 2 (*concluded*)

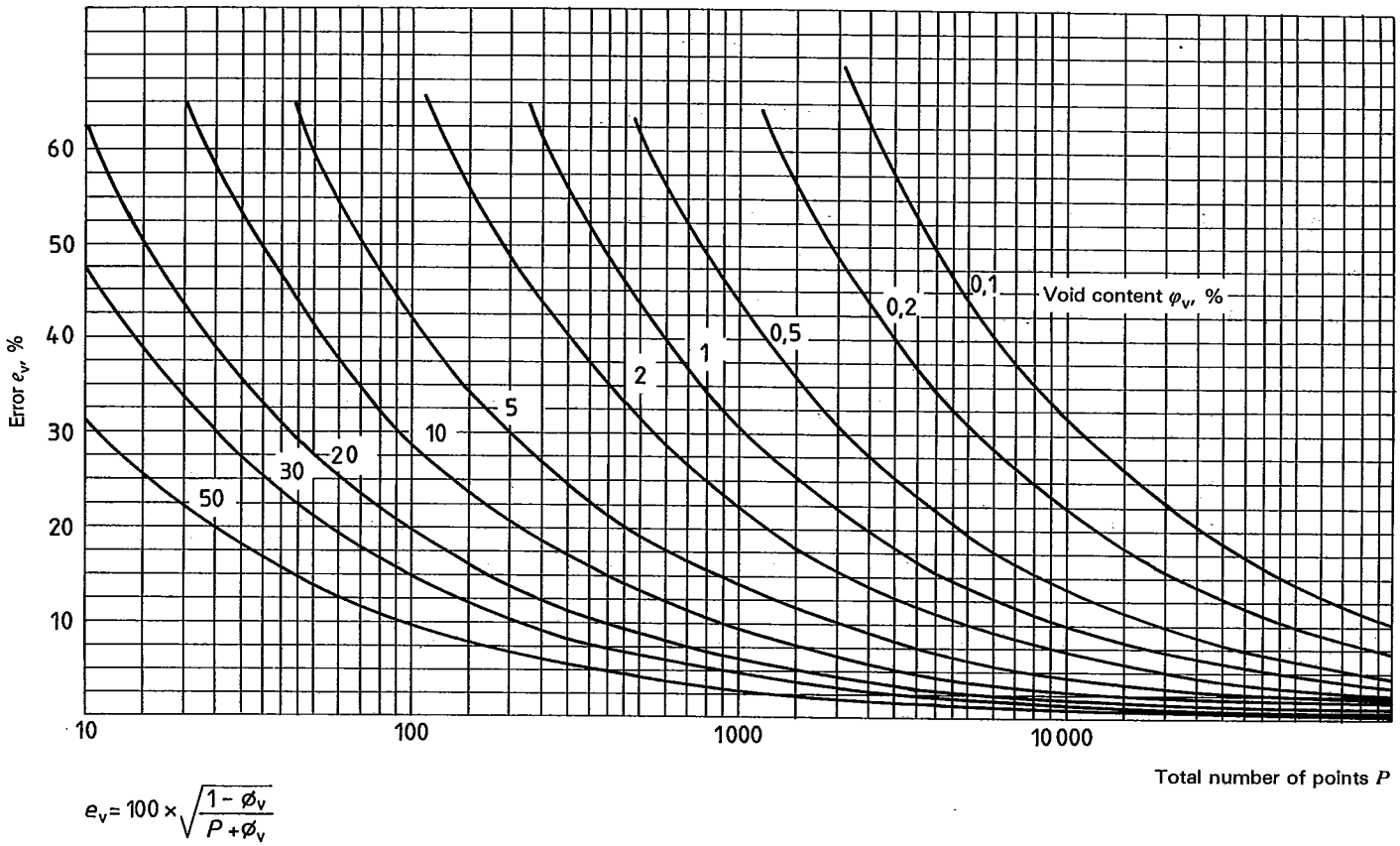


Figure 3 — Measurement error versus the total number of points for different void content

Annex A
(informative)

Polishing the sections

The general conditions for the polishing of the sections are the following:

Speed: 200 rpm

Force: about 5 N (500 gf) (for ø 25 mm sections)

Time	Grain	Remark
3 min	400 (35 µm)	Copious watering
5 min	800 (22 µm)	Copious watering
15 min	12-H alumina (Al_2O_3)	The alumina is used slightly diluted, with an almost pasty consistency.

These conditions may be varied, depending on the type of reinforced plastic, in order to remove all the scratches from the face being polished.

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Descriptors : plastics, glass reinforced plastics, tests, determination, void fractions, density (mass/volume).

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