

INTERNATIONAL STANDARD

**ISO
7822**

First edition
1990-09-01

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)



Documents

Document Sharing Hub

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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.

© ISO 1990

All rights reserved. No part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

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 $\pm 2,5$ % 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 $\pm 1 \mu\text{m}$.

4.1.2 **Balance**, having an accuracy of $\pm 0,1 \text{ mg}$.

4.1.3 **Muffle furnace**, capable of being maintained at $625^\circ\text{C} \pm 5^\circ\text{C}$.

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.

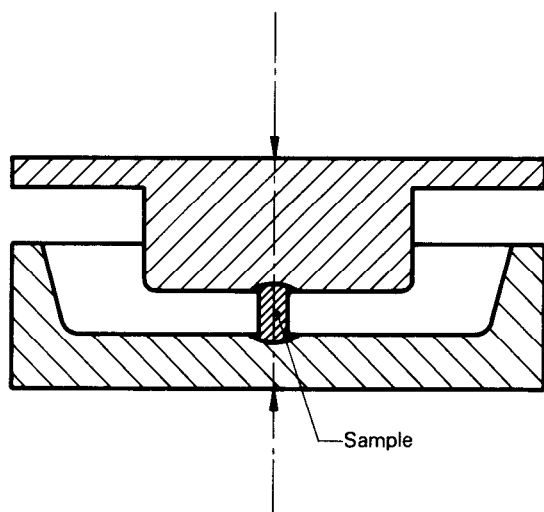


Figure 1 — Disintegration die

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

4.2.3 **Balance**, with an accuracy of $\pm 0,1 \text{ mg}$.

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 $\times 400$, 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 pycnometer and the disintegration procedure used. When an air pycnometer is used, quantities of about 25 g or 12 000 mm³, 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 parallelepiped 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