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Plastics — Methods for determining the density of non-cellular plastics —

Part 2:
Density gradient column method

Plastiques — Méthodes de détermination de la masse volumique des plastiques non alvéolaires —

Partie 2: Méthode de la colonne à gradient de masse volumique

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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.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This second edition cancels and replaces the first edition (ISO 1183-2:2004), which has been technically revised. The main changes compared to the previous edition are as follows:

— normative references have been changed to undated;

— text has been revised editorially.

A list of all parts in the ISO 1183 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Plastics — Methods for determining the density of non-cellular plastics —

Part 2:
Density gradient column method

WARNING — The use of this document may involve hazardous materials, operations or equipment. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this document to establish appropriate health and safety practices.

1 Scope

This document specifies a gradient column method for the determination of the density of non-cellular moulded or extruded plastics or pellets in void-free form. Density gradient columns are columns containing a mixture of two liquids, the density in the column increasing uniformly from top to bottom.

NOTE   Density is frequently used to follow variations in physical structure or composition of plastic materials. Density can also be useful in assessing the uniformity of samples or specimens. The density of plastic materials can depend upon the choice of specimen preparation method. When this is the case, precise details of the specimen preparation method are intended to be included in the appropriate material specification.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 1183-1, *Plastics — Methods for determining the density of non-cellular plastics — Part 1: Immersion method, liquid pycnometer method and titration method*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
density
ρ
ratio of the mass m of a sample to its volume V (at the temperature T) expressed in kg/m^3, kg/dm^3 (g/cm^3), or kg/l (g/ml)

Note 1 to entry: The following terms, based upon ISO 80000-4[1], are given in Table 1 for clarification.

Table 1 — Density terms

Term	Symbol	Formulation	Units
Density	ρ	m/V	kg/m^3 kg/dm^3 (g/cm^3) kg/l (g/ml)
Specific volume	v	V/m (= $1/\rho$)	m^3/kg dm^3/kg (cm^3/g) l/kg (ml/g)

4 Conditioning

Conditioning and testing shall be in accordance with ISO 291 or the appropriate material standard. In general, conditioning specimens to constant temperature is not required, because the determination itself brings the specimen to the constant temperature of the test.

Specimens which change in density during the test to such an extent that the change is greater than the required accuracy of the density determination shall be conditioned prior to measurement in accordance with the applicable material specification. When changes in density with time or atmospheric conditions are the primary purpose of the measurements, the specimens shall be conditioned as described in the material specification and, if no material specification exists, then as agreed upon by the interested parties.

5 Method

5.1 Apparatus

5.1.1 Density gradient column, consisting of a suitable graduated column, not less than 40 mm in diameter, with a cover. The height of the column shall be compatible with the accuracy required. A graduation interval of 1 mm for the scale on the column is normal.

5.1.2 Liquid bath, capable of being thermostatically controlled to within ±0,1 °C or ±0,5 °C, depending on the sensitivity required (see Annex B).

5.1.3 Calibrated glass floats, covering the density range in which measurements are to be made and approximately evenly distributed throughout this range.

These may be purchased from an accredited source or prepared as described in 5.4.1.

5.1.4 Balance, accurate to ±0,1 mg.

5.1.5 Siphon or pipette assembly, for filling the gradient column (5.1.1), as shown in Figure B.1 or B.2, or any other suitable device.

5.2 Immersion liquids

Required are two miscible liquids of different densities, freshly distilled in the case of pure liquids. The densities of various liquids are given in Annex A as a guide.

The liquid with which the specimen comes into contact during the measurement shall have no effect on the specimen.

Prepare the mixture of liquids as specified in 5.4.1.2.

5.3 Specimens

Specimens shall consist of pieces of the material cut to any convenient shape for ease of identification. The dimensions of each piece shall be chosen to permit accurate measurement of the position of the centre of the piece.

When cutting specimens from larger samples, care shall be taken to ensure that the characteristics of the material are not changed due to excessive heat generation. The surface of the specimen shall be smooth and free from cavities to minimize the entrapment of air bubbles upon immersion in the liquid, otherwise errors will be introduced.

NOTE   Specimens of less than 5 mm in diameter are normally suitable.

5.4 Procedure

5.4.1 Preparation and calibration of glass floats

5.4.1.1   The glass floats (5.1.3) may be produced by any convenient method. They shall be approximately spherical, of diameter not greater than 5 mm and fully annealed.

5.4.1.2   To prepare the glass floats for use, prepare a series of mixtures of about 500 ml of the two immersion liquids (5.2) covering the density range to be used in the density gradient column (5.1.1). With the floats and column at ambient temperature, place the floats carefully into these mixtures.

Adjust selected floats to match approximately the densities of the mixtures:

a)   either by rubbing the float on a glass plate covered with a thin slurry of silicon carbide of particle size less than 38 μm (400 mesh) or another suitable abrasive;

b)   or by etching the float with hydrofluoric acid.

5.4.1.3   Determine the exact density of each glass float calibrated as above by placing it in a mixture of two suitable liquids (5.2) in the bath (5.1.2) maintained at $(t \pm 0,1)$ °C, where t is 23 °C or 27 °C (whichever will be used for the density gradient column). If the float sinks, add the denser of the two liquids (if the float rises, add the less dense) and stir gently to homogenize. Allow the mixture to stabilize. If the float still moves, adjust the density of the mixture again. Repeat this procedure until the float remains stationary for at least 30 min.

5.4.1.4   For each float, determine, to the nearest 0,000 1 g/ml, the density of the solution in which the float remained in equilibrium, using the pycnometer method (method B) described in ISO 1183-1 or any other suitable method. Apply the buoyancy correction described in ISO 1183-1, if necessary. Record this density as the density of the float.

Calibrated glass floats may also be purchased from accredited manufacturers.

5.4.2 Preparation of density gradient column

Methods for preparing the density gradient column are not specified in this document, but examples of two methods are given in Annex B.

5.4.3 Measurement of density

Wet three test specimens with the less dense of the two liquids used in the column and gently place them in the column. Allow the column and specimens to reach equilibrium, which will require 10 min or more. Films less than 0,05 mm thick require at least 1,5 h to settle. Rechecking thin-film specimens after several hours is advisable.

NOTE 1   One of the most common sources of error in the determination is air bubbles.

NOTE 2   Suitable methods for removing air bubbles from the specimens are, for example, a fine wire carefully manipulated or applying a vacuum to the column.

Old specimens can be removed without destroying the gradient by collecting them in a wire screen basket attached to a long wire. The basket is pulled up very slowly from the bottom of the column and, after cleaning, returned to the bottom of the column. It is essential that this be done at a slow enough rate (approximately 10 mm length of column per minute) in order not to disturb the density gradient. It can conveniently be done using a clock motor. After cleaning the column, recheck the calibration and replot density versus height.

5.4.4   Calculations

The densities of specimens may be determined graphically or by calculation from the levels at which they settle, as follows:

a)   Graphical method

Make a plot of float density versus float height on a chart large enough to be read accurately to within ±0,000 1 g/cm^3 and ±1 mm. Find the height of each specimen on the chart and read off the corresponding density.

b)   Calculation method

Calculate the density $\rho_{S,x}$ of each specimen by interpolation, using the Formula (1):

$$\rho_{S,x} = \rho_{F1} + \frac{(x-y) \times (\rho_{F2} - \rho_{F1})}{z - y} \tag{1}$$

where

ρ_{F1} and ρ_{F2}	are the densities of the two floats at the lower and higher ends, respectively, of the density range;
x	is the distance of the specimen above an arbitrary level;
y and z	are the distances above the same arbitrary level of the two floats of density ρ_{F1} and ρ_{F2}, respectively.

Method b) does not reveal calibration errors. These can only be detected by using method a), the graphical method. Method b) can be used when the calibration is known to be linear within the range being used.

If the relationship between float position and density is not linear, a second order polynomial may be used for interpolation of density.

Corrections for buoyancy, if required, can be calculated as described in ISO 1183-1.

6   Test report

The test report shall include the following information:

a)   a reference to this document, i.e. ISO 1183-2:2019;

b)   all details necessary for complete identification of the material tested, including the specimen preparation method and pretreatment, if applicable;

c)   the immersion liquids used;

d)   the value of the density determined for each of the three specimens and the arithmetic mean of these values;

e)   the temperature of the determination;

f)   details of any buoyancy corrections made;

g)   the date of the determination.

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