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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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.

ISO 1183-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

Together with the other parts (see below), this part of ISO 1183 cancels and replaces ISO 1183:1987, which has been technically revised.

ISO 1183 consists of the following parts, under the general title *Plastics — Methods for determining the density of non-cellular plastics*:

— *Part 1: Immersion method, liquid pyknometer method and titration method*

— *Part 2: Density gradient column method*

— *Part 3: Gas pyknometer method*

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

Part 2: Density gradient column method

WARNING — The use of this part of ISO 1183 may involve hazardous materials, operations or equipment. This part of ISO 1183 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 part of ISO 1183 to establish appropriate health and safety practices and to determine the applicability of any regulatory limitations prior to use.

1 Scope

This part of ISO 1183 specifies a gradient column method for the determination of the density of non-cellular moulded or extruded plastics 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 This part of ISO 1183 is applicable to pellets as long as they are void-free. Density is frequently used to follow variations in physical structure or composition of plastic materials. Density may also be useful in assessing the uniformity of samples or specimens. Often the density of plastic materials will depend upon the choice of specimen preparation method. When this is the case, precise details of the specimen preparation method will have to be included in the appropriate material specification.

2 Normative references

The following referenced documents are indispensable for the application 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 31-3, *Quantities and units — Part 3: Mechanics*

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

ISO 1183-1:2004, *Plastics — Methods for determining the density of non-cellular plastics — Part 1: Immersion method, liquid pyknometer method and titration method*

3 Terms and definitions

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

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 The following terms, based upon ISO 31-3, are given here 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.

NOTE 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 pyknometer method (method B) described in ISO 1183-1:2004 or any other suitable method. Apply the buoyancy correction described in ISO 1183-1:2004, Clause 6, if necessary. Record this density as the density of the float.

NOTE 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 part of ISO 1183, 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 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 equation:

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

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.

NOTE 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 1183-1:2004, Clause 6.

6 Test report

The test report shall include the following information:

a) a reference to this part of ISO 1183;

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.