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

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

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

Partie 1: Méthode par immersion, méthode du pycnomètre en milieu liquide et méthode par titrage

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Contents

Page

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 1183-1 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 1: Immersion method, liquid pyknometer method and titration 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 three methods for the determination of the density of non-cellular plastics in the form of void-free moulded or extruded objects, as well as powders, flakes and granules.

— **Method A: Immersion method**, for solid plastics (except for powders) in void-free form.

— **Method B: Liquid pyknometer method**, for particles, powders, flakes, granules or small pieces of finished parts.

— **Method C: Titration method**, for plastics in any void-free form.

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. This note is applicable to all three methods.

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 291:1997, *Plastics — Standard atmospheres for conditioning and testing*

ISO 472:1999, *Plastics — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and the following apply.

3.1
mass
quantity of matter contained in a body

NOTE It is expressed in kilograms (kg) or grams (g).

3.2
apparent mass
mass of a body obtained by measuring its weight using an appropriately calibrated balance

NOTE It is expressed in kilograms (kg) or grams (g).

3.3
density
ρ
the 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

The test atmosphere shall be in accordance with ISO 291. 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 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 Methods

5.1 Method A — Immersion method

5.1.1 Apparatus

5.1.1.1 Analytical balance or **instrument specifically designed for measurement of density**, accurate to 0,1 mg.

NOTE An automatically operating instrument may be used. The calculation of density may be done automatically using a computer.

5.1.1.2 Immersion vessel: a beaker or other wide-mouthed container of suitable size for holding the immersion liquid.

5.1.1.3 Stationary support, e.g. a pan straddle, to hold the immersion vessel above the balance pan.

5.1.1.4 Thermometer, graduated at 0,1 °C intervals, covering the range 0 °C to 30 °C.

5.1.1.5 Wire (if required), corrosion-resistant, of diameter not greater than 0,5 mm, for suspending specimens in the immersion liquid.

5.1.1.6 Sinker, of suitable mass to ensure complete immersion of the specimen, for use when the density of the specimen is less than that of the immersion liquid.

5.1.1.7 Pyknometer, with a side-arm overflow capillary, for determining the density of the immersion liquid when this liquid is not water. The pyknometer shall be equipped with a thermometer graduated at 0,1 °C intervals from 0 °C to 30 °C.

5.1.1.8 Liquid bath, capable of being thermostatically controlled to within ± 0,5 °C, for use in determining the density of the immersion liquid.

5.1.2 Immersion liquid

Use freshly distilled or deionized water or another suitable liquid containing not more than 0,1 % of a wetting agent to help in removing air bubbles. The liquid or solution with which the specimen comes into contact during the measurement shall have no effect on the specimen.

The density of immersion liquids other than distilled water need not be measured provided they are obtained from an accredited source and are accompanied by certificate.

5.1.3 Specimens

Specimens may be in any void-free form except for powder. They shall be of a convenient size to give adequate clearance between the specimen and the immersion vessel and should preferably have a mass of at least 1 g.

When cutting specimens from larger samples, proper equipment shall be used to ensure that the characteristics of the material do not change. 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.

5.1.4 Procedure

5.1.4.1 Weigh the specimen in air while suspended with a wire of maximum diameter 0,5 mm. Weigh specimens of mass less than or equal to 10 g to the nearest 0,1 mg. Weigh those of mass greater than 10 g to the nearest 1 mg. Record the mass of the specimen.

5.1.4.2 Immerse the specimen, still suspended by the wire, in the immersion liquid (5.1.2), contained in the immersion vessel (5.1.1.2) on the support (5.1.1.3). The temperature of the immersion liquid shall be 23 °C ± 2 °C (or 27 °C ± 2 °C). Remove any adhering air bubbles with a fine wire. Weigh the immersed specimen to the nearest 0,1 mg.

If the measurement is carried out in a temperature-controlled room, the temperature of the whole apparatus, including the immersion liquid, shall be within the range 23 °C ± 2 °C (or 27 °C ± 2 °C).

5.1.4.3 If necessary, determine the density of immersion liquids other than water as follows. Weigh the pyknometer (5.1.1.7) empty and then containing freshly distilled or deionized water at a temperature of 23 °C ± 0,5 °C (or 27 °C ± 0,5 °C). Weigh the same pyknometer, after cleaning and drying, filled with the immersion liquid [also at a temperature of 23 °C ± 0,5 °C (or 27 °C ± 0,5 °C)]. Use the liquid bath (5.1.1.8) to bring the water and immersion liquid to the correct temperature. Calculate the density ρ_{IL}, in grams per cubic centimetre, of the immersion liquid at 23 °C (or 27 °C), using the equation:

$$\rho_{IL} = \frac{m_{IL}}{m_W} \times \rho_W \qquad (1)$$

where

m_{IL} is the mass, in grams, of the immersion liquid;

m_W is the mass, in grams, of the water;

ρ_W is the density, in grams per cubic centimetre, of water at 23 °C (or 27 °C).

5.1.4.4 Calculate the density ρ_S, in grams per cubic centimetre, of the specimen at 23 °C (or 27 °C), using the equation:

$$\rho_S = \frac{m_{S,A} \times \rho_{IL}}{m_{S,A} - m_{S,IL}} \qquad (2)$$

where

$m_{S,A}$ is the apparent mass, in grams, of the specimen in air;

$m_{S,IL}$ is the apparent mass, in grams, of the specimen in the immersion liquid;

ρ_{IL} is the density of the immersion liquid at 23 °C (or 27 °C), in grams per cubic centimetre, as stated by the supplier or determined as specified in 5.1.4.3.

For specimens having a density less than that of the immersion liquid, the test may be performed in exactly the same way as described above, with the following exception: a sinker of lead or other dense material is attached to the wire, such that the sinker rests below the liquid level, as does the specimen, during immersion. The sinker may be considered as a part of the suspension wire. In this case, the upthrust exerted by the immersion liquid on the sinker shall be allowed for by using the following equation, rather than Equation (2), to calculate the density of the specimen:

$$\rho_S = \frac{m_{S,A} \times \rho_{IL}}{m_{S,A} + m_{K,IL} - m_{S+K,IL}} \qquad (3)$$

where

$m_{K,IL}$ is the apparent mass, in grams, of the sinker in the immersion liquid;

$m_{S+K,IL}$ is the apparent mass, in grams, of the specimen and sinker in the immersion liquid.

The bouyancy of the suspension wire in air is normally considered to be negligible, but for correction for air bouyancy see Clause 6.

5.1.4.5 Perform the test on a minimum of three specimens and calculate the mean result to three decimal places.

5.2 Method B — Liquid pyknometer method

5.2.1 Apparatus

5.2.1.1 **Balance**, accurate to 0,1 mg.

5.2.1.2 **Stationary support** (see 5.1.1.3).

5.2.1.3 **Pyknometer** (see 5.1.1.7).

5.2.1.4 **Liquid bath** (see 5.1.1.8).

5.2.1.5 **Dessicator**, connected to a vacuum system.

5.2.2 Immersion liquid

As specified in 5.1.2.

5.2.3 Specimens

Specimens of powders, granules or flakes shall be measured in the form in which they are received. The specimen mass shall be in the range of 1 g to 5 g.

5.2.4 Procedure

5.2.4.1 Weigh the pyknometer (5.2.1.3) empty and dry. Weigh a suitable quantity of the plastic material in the pyknometer. Cover the test specimen with immersion liquid (5.2.2) and remove all the air by placing the pyknometer in the desiccator (5.2.1.5) and applying a vacuum. Break the vacuum and almost completely fill the pyknometer with immersion liquid. Bring it to constant temperature [23 °C ± 0,5 °C (or 27 °C ± 0,5 °C)] in the liquid bath (5.2.1.4) and then complete filling exactly to the limit of the capacity of the pyknometer.

Wipe dry and weigh the pyknometer with the specimen and immersion liquid.

5.2.4.2 Empty and clean the pyknometer. Fill it with deaerated distilled or deionized water, remove any remaining air as above, and determine the mass of the pyknometer and its contents at the temperature of test.

5.2.4.3 Repeat the process with the immersion liquid if an immersion liquid other than water was used, and determine its density as specified in 5.1.4.3.

5.2.4.4 Calculate the density ρ_S, in grams per cubic centimetre, of the specimen at 23 °C (or 27 °C), using the following equation:

$$\rho_S = \frac{m_S \times \rho_{IL}}{m_1 - m_2} \tag{4}$$

where

m_S is the apparent mass, in grams, of the specimen;

m_1 is the apparent mass, in grams, of the liquid required to fill the empty pyknometer;

m_2 is the apparent mass, in grams, of the liquid required to fill the pyknometer containing the specimen;

ρ_{IL} is the density of the immersion liquid at 23 °C (or 27 °C), in grams per cubic centimetre, as stated by the supplier or determined as specified in 5.1.4.3.

5.2.4.5 Perform the test on a minimum of three specimens and calculate the mean result to three decimal places.

5.3 Method C — Titration method

5.3.1 Apparatus

5.3.1.1 **Liquid bath** (see 5.1.1.8).

5.3.1.2 **Glass cylinder**, of capacity 250 ml.

5.3.1.3 **Thermometer**, graduated at 0,1 °C intervals, with a range suitable for measuring the test temperature used.

5.3.1.4 **Volumetric flask**, capacity 100 ml.

5.3.1.5 **Flat-tipped glass rod stirrer**.

5.3.1.6 **Burette**, capacity 25 ml, of a design which enables it to be kept in the liquid bath (5.3.1.1), capable of dispensing 0,1 ml portions of liquid.

5.3.2 Immersion liquids

Required are two miscible liquids of different densities. One shall have a density just below that of the test material and the other a density higher than that of the test material. Densities of various liquids are given in Annex A as a guide. If necessary, carry out a rapid preliminary test in a few millilitres of the liquid.

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

5.3.3 Specimens

Specimens shall be in a suitable void-free form.

5.3.4 Procedure

5.3.4.1 By means of the volumetric flask (5.3.1.4), accurately measure 100 ml of the less dense immersion liquid (see 5.3.2) into the clean, dry 250 ml glass cylinder (5.3.1.2). Put the cylinder into the liquid bath (5.3.1.1) controlled at 23 °C ± 0,5 °C (or 27 °C ± 0,5 °C).

5.3.4.2 Place the pieces of the test specimen in the cylinder. They shall fall to the bottom and be free of air bubbles. Allow the cylinder and its contents to stabilize at the bath temperature, stirring at intervals.

NOTE It is recommended that the thermometer (5.3.1.3) be kept permanently in the liquid. This makes it possible to check that thermal equilibrium is attained at the time of measurement and, in particular, that the heat of dilution has been dissipated.

5.3.4.3 When the temperature of the liquid is 23 °C ± 0,5 °C (or 27 °C ± 0,5 °C), add the more dense immersion liquid millilitre by millilitre from the burette (5.3.1.6). Stir the liquid after each addition by means of the glass rod (5.3.1.5), held vertically, and avoid producing air bubbles.

After each addition of the more dense liquid and mixing, observe the behaviour of the pieces of test specimen.

At first, they will fall rapidly to the bottom but, as more of the more dense liquid is added, their rate of fall will become slower. At this point, add the more dense liquid in 0,1 ml amounts. Note the total amount of more dense liquid added when the lightest pieces of specimen become suspended within the liquid, at the level to which they are brought by stirring, without moving up or down for at least 1 min. At this point in the titration, note the amount of more dense liquid required. The density of the liquid mixture at this point corresponds to the lower limit of the density of the test specimen.

Add more of the more dense liquid until the heaviest pieces of specimen remain at a constant level within the liquid for at least 1 min. Note the amount of more dense liquid required.

For each pair of liquids, establish the relationship between the amount of more dense liquid added and the density of the resulting mixture, and plot the relationship in the form of a graph.

The density of the liquid mixture at each point on the graph can be determined by a pyknometer method.

6 Correction for buoyancy in air

Where weighings are made in air, the values of the "apparent masses" obtained shall be corrected to compensate for the effect of the air buoyancy on the specimen and on any weights used. This will be the case if the accuracy of the results is to be between 0,2 % and 0,05 %.

The true mass m_T, in grams, is calculated using the equation:

$$m_T = m_{APP} \times \left(1 + \frac{\rho_{Air}}{\rho_S} - \frac{\rho_{Air}}{\rho_L} \right) \tag{5}$$

where

m_{APP} is the apparent mass, in grams;

ρ_{Air} is the density, in grams per cubic centimetre, of air (approximately 0,001 2 g/cm^3 at 23 °C and 27 °C);

ρ_S is the density, in grams per cubic centimetre, of the specimen at 23 °C (or 27 °C);

ρ_L is the density, in grams per cubic centimetre, of the weights used.

For improved accuracy, the dependence of the density of air on pressure and temperature can be taken into account:

$$\rho_{Air} = \frac{0,001\,31}{(1 + 0,003\,67 \times t)} \times \frac{1}{P} \tag{6}$$

where

t is the test temperature, in degrees Celsius;

P is the atmospheric pressure, in bars.

7 Test report

The test report shall include the following information:

a) a reference to this International Standard;

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

c) the method used (A, B or C);

d) the immersion liquid(s) used;

e) the test temperature;

f) the individual values and arithmetic mean of the density;

g) a statement as to whether any buoyancy correction was made and, if so, what kind of correction.

Annex A
(informative)

Liquid systems suitable for use in Method C

WARNING — Some of the following chemicals may be hazardous.

Table A.1 — Liquid systems for Method C

System	Density range g/cm^3
Methanol/benzyl alcohol	0,79 to 1,05
Isopropanol/water	0,79 to 1,00
Isopropanol/diethylene glycol	0,79 to 1,11
Ethanol/water	0,79 to 1,00
Toluene/carbon tetrachloride	0,87 to 1,60
Water/aqueous solution of sodium bromide [a]	1,00 to 1,41
Water/aqueous solution of calcium nitrate	1,00 to 1,60
Ethanol/aqueous solution of zinc chloride [b]	0,79 to 1,70
Carbon tetrachloride/1,3-dibromopropane	1,60 to 1,99
1,3-Dibromopropane/ethylene bromide	1,99 to 2,18
Ethylene bromide/bromoform	2,18 to 2,89
Carbon tetrachloride/bromoform	1,60 to 2,89
Isopropanol/methylglycol acetate	0,79 to 1,00

[a] A density of 1,41 is equivalent to a mass fraction of about 40 % sodium bromide.

[b] A density of 1,70 is equivalent to a mass fraction of about 67 % zinc chloride.

The following may also be used in various mixtures:

	Density (g/cm^3)
n-Octane	0,70
Dimethylformamide	0,94
Tetrachloroethane	1,60
Ethyl iodide	1,93
Methylene iodide	3,33

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