BRITISH STANDARD

BS EN ISO 1183-3:1999
Incorporating Corrigendum No. 1

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

Part 3: Gas pyknometer method

The European Standard EN ISO 1183-3:1999 has the status of a British Standard

ICS 83.080.01

BSI British Standards

National foreword

This British Standard is the English language version of EN ISO 1183-3:1999. It is identical with ISO 1183-3:1999.

The UK participation in its preparation was entrusted to Technical Committee PRI/21, Testing of plastics, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Summary of pages

This document comprises a front cover, an inside front cover, the EN ISO title page, the EN ISO foreword page, the ISO title page, pages ii to iv, pages 1 to 7 and a back cover.

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EUROPEAN STANDARD
NORME EUROPÉENNE
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EN ISO 1183-3

September 1999

ICS 83.080.01

English version

Plastics — Methods for determining the density of non-cellular plastics — Part 3: Gas pyknometer method

(ISO 1183-3:1999)

Plastiques —
Méthodes pour déterminer la masse volumique des plastiques non alvéolaires —
Partie 3: Méthode du pycnomètre à gaz
(ISO 1183-3:1999)

Kunststoffe —
Bestimmung der Dichte von nicht verschäumten Kunststoffen —
Teil 3: Gas Pyknometer Verfahren
(ISO 1183-3:1999)

This European Standard was approved by CEN on 22 August 1999.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Ref. No. EN ISO 1183-3:1999 E

Foreword

The text of the International Standard ISO 14126:1999 has been prepared by Technical Committee ISO/TC 61 “Plastics” in collaboration with Technical Committee CEN/TC 249 “Plastics”, the secretariat of which is held by IBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 2000, and conflicting national standards shall be withdrawn at the latest by March 2000.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 1183-3:1999 was approved by CEN as a European Standard without any modification.

NOTE Normative references to International Standards are listed in Annex ZA (normative).

INTERNATIONAL STANDARD

ISO 1183-3

First edition
1999-09-15

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

Part 3:
Gas pyknometer method

Plastiques — Méthodes pour déterminer la masse volumique des plastiques non alvéolaires —

Partie 3: Méthode utilisant un pycnomètre à gaz

Reference number
ISO 1183-3:1999(E)

Contents

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 3.

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 1183-3 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*. Together with the other parts (see below), it 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, pyknometer method and titration method;*

— *Part 2: Density gradient column method;*

— *Part 3: Gas pyknometer method.*

Annex A of this part of ISO 1183 is for information only.

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Introduction

This part of ISO 1183 is one of a series dealing with methods of measuring the density of solid non-cellular plastics. The values obtained using this part of ISO 1183 are expected to be comparable to those obtained using the other parts.

Density measurements may be used to investigate variations in the physical structure or the molecular order of materials. Such measurements are widely used to determine the degree of crystallinity of polymers. In addition, they may be used to determine the amount of filler present.

The density of a plastic material may depend on any conditioning or thermal treatment which the material has undergone.

The physical structure of a polymer can change with time and temperature. Its volume is also a temperature-dependent property. This means that the density may vary with time and/or temperature.

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 method for the determination of the density or the specific volume of solid non-cellular plastics of any shape which do not contain closed pores.

2 Terms, definitions, symbols, units and abbreviated terms

For the purposes of this part of ISO 1183, the following terms, definitions, symbols, units and abbreviated terms apply:

2.1
test material
material to be tested

2.2
test specimen
that part of the test material actually subjected to the test

2.3
mass
m
quantity of matter contained in a body

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

2.4
weight
W
force produced by gravity acting on a mass

NOTE 1 Since gravity varies with location, so does weight.

NOTE 2 Weight is expressed in newtons (N).

2.5
apparent mass
m_{app}
mass of a body obtained by measuring its weight using an appropriately calibrated balance

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

2.6
volume
V
size of a body in three-dimensional space, excluding pores

NOTE 1 Due to thermal expansion, volume varies with temperature T.

NOTE 2 Volume is expressed in cubic metres (m^3), litres (l), cubic centimetres (cm^3) or millilitres (ml).

2.7
density
ρ
mass per unit volume of a material at a given temperature T

NOTE 1 Density is calculated using the equation

$$\rho^T = \frac{m_{app}}{V} \quad \text{at constant } T \tag{1}$$

NOTE 2 It is expressed in kilograms per cubic metre (kg/m^3), kilograms per cubic decimetre (kg/dm^3), grams per cubic centimetre (g/cm^3), kilograms per litre (kg/l) or grams per millilitre (g/ml).

2.8
specific volume
v
volume per unit mass of a material at a given temperature T

NOTE 1 Specific volume is calculated using the equation

$$v^T = \frac{V^T}{m_{app}} = \frac{1}{\rho^T} \quad \text{at constant } T \tag{2}$$

NOTE 2 It is expressed in cubic metres per kilogram (m^3/kg), cubic decimetres per kilogram (dm^3/kg), cubic centimetres per gram (cm^3/g), litres per kilogram (l/kg) or millilitres per gram (ml/g).

NOTE 3 Density is to be distinguished from specific gravity, which is the ratio of the weight of a given volume of a material to that of an equal volume of water at a stated temperature T.

3 Principle

3.1 The volume of a specimen of known apparent mass is determined by measuring the change of gas volume within a pyknometer upon introducing the specimen. The volume change may be obtained either directly by means of a movable piston or indirectly by measuring the change of the pressure within the pyknometer and calculating the volume using the pressure-volume relationship for ideal gases. The volume obtained by this procedure is related to the solid alone without its pores. Density is calculated using eq. (1) given above.

3.2 The smaller the molecules of the gas the narrower the pores will be that can be penetrated.

3.3 Preferably gases of low affinity of adsorption onto the test material should be used.

3.4 Particularly for pressure-measuring pyknometers the accuracy of the method depends on the applicability of the ideal gas law (Boyle — Mariotte law).

NOTE For high-accuracy measurements, helium should preferably be used because it behaves most like an ideal gas. It can penetrate into pores at least as small as 1 µm diameter and has a low tendency to adsorb onto the surface of the test material.

4 Apparatus and materials

4.1 Analytical balance, accurate to 0,1 mg.

4.2 Gas pyknometer, of suitable cell volume, accurate to 0,01 % of the cell volume.

NOTE Accuracy is improved if the test specimen fills as much of the cell as possible. An example of a two-chamber pressure-type pyknometer is given in Annex A, together with a procedure for its calibration and operation.

4.3 Measurement gas, preferably helium with a purity of 99,99 % or better for highest accuracy, or other non-corrosive and non-adsorbing gas, e.g dry air, at a pressure of up to 300 kPa.

4.4 Thermostatically controlled bath or enclosure, capable of maintaining the desired test temperature T (preferably 23 °C) to the nearest 1 °C. Alternatively, a gas pyknometer with a suitable built-in temperature control may be used.

5 Test specimens

5.1 If not excluded by the conditioning procedure, dry test specimens to constant mass before carrying out any volume measurements. Take care to choose suitable drying conditions to prevent changes in density of the test material.

5.2 Test materials consisting of powder, granules, pellets or flakes can be tested as received. Other materials may be cut to any shape convenient for the size of the pyknometer cell used. Take care to avoid changes in density resulting from compressive stresses on the material during cutting.

Prepare specimens containing closed pores in a suitable way, e.g. by grinding.

5.3 Specimens whose change in density on conditioning may be greater than the accuracy required of the density determination shall be conditioned before testing in accordance with the relevant material standard. Conditioning at a particular humidity or to a constant degree of crystallinity may be required.

5.4 When changes in density as a function of time or the ambient conditions are the main purpose of the determination, condition the specimens as agreed by the interested parties.

6 Calibration

Set the pyknometer to the desired temperature T, preferably 23 °C. Adjust the volumes of the pyknometer cells to the desired values or measure the volumes of the empty cells. Determine the mass of a calibration specimen of known density to the nearest 0,1 mg or use a calibration specimen of known volume. Introduce the calibration specimen into the measurement cell. Purge for 3 min with the measurement gas to replace the air and any humidity which may be adsorbed on the surface of the specimen. If necessary, allow additional time for temperature equilibrium to be established. When the preset temperature has been reached, measure the change in volume or pressure produced by introduction of the specimen in accordance with the principles of operation of the particular type of pyknometer used. Determine the calibration factor k_c from equation (3a) or (3b).

NOTE When using a pressure-type apparatus, the volume of the specimen can be calculated from the pressure change using the pressure-volume relationship for ideal gases (Boyle-Mariotte law). This may be done automatically by some pyknometers.

$$k_c = \frac{V_c}{V_c^o} \tag{3a}$$

$$k_c = \frac{V_c \cdot \rho_c^o}{m_c} \tag{3b}$$

where

V_c is the measured volume of the calibration specimen;

V_c^o is the known volume of the calibration specimen;

ρ_c^o is the known density of the calibration specimen;

m_c is the mass of the calibration specimen.

Recalibrate the pyknometer if the cell volume or temperature is changed, a different measurement gas is used or the pressure of the measurement gas is changed significantly.

7 Procedure and calculation

Repeat the procedure described in the Clause **6** using the test specimen. Calculate the density using the equation

$$\rho_s^T = \frac{m_s}{V_s^T} \times k_c \qquad (4)$$

where

m_s is the mass of the test specimen;

$V_s T$ is the volume of the test specimen at temperature T.

Carry out the determination on three or more test specimens of the same material.

8 Precision

The precision of this test method is not known because inter-laboratory data are not available. When such data become available, a precision statement will be added at the following revision.

NOTE The reproducibility can be expected to be better than approximately 0,2 %, while the repeatability can be expected to be better than approximately 0,1 %.

9 Test report

The test report shall include the following details:

a) a reference to this part of ISO 1183;

b) all details necessary for the complete identification of the material tested;

c) the arithmetic mean density for all the specimens tested, and the standard deviation of the mean;

d) the number of specimens tested and the mass of each;

e) details of any conditioning procedure used;

f) the measurement gas used and its purity;

g) the test temperature;

h) the calibration material used;

i) the type of pyknometer used and its manufacturer;

j) details of any operation not specified in this part of ISO 1183 as well as details of any incident likely to have affected the results;

k) the date of the test.

Annex A (informative)
Two-chamber pressure-type pyknometer

A.1 Apparatus

The apparatus is composed of two inter-connected cells [a measurement cell (volume V_{meas}) and an expansion cell (volume V_{exp})], valves for the gas inlet (V_1) and outlet (V_3) and a valve (V_2) separating the two cells, as shown in Figure A.1 a). The measurement cell is fitted with a pressure sensor. The pyknometer may be operated manually or automatically.

A.2 Calibration

Before starting the calibration procedure, the apparatus is purged by opening all the valves and sweeping both cells with gas, leaving the two cells full of gas at atmospheric pressure. The readout of the pressure sensor is then set to zero. These preliminary operations are carried out before each calibration step.

In the first calibration step [see Figure A.1 a)], valves V_2 and V_3 are closed. By opening valve V_1, gas is allowed to flow into the measurement cell until the desired pressure p_1^{o} is reached. Then valve V_1 is closed, valve V_2 opened and the resulting equilibrium pressure p_2^{o} measured.

In the second calibration step [see Figure A.1 b)], the same procedure is carried out with a calibration specimen of known volume V_c in the measurement cell. The measurement cell is again filled with gas until the desired pressure p_1^{*} is reached and, after expansion, the resulting pressure p_2^{*} is measured.

The volume of the measurement cell and that of the expansion cell can be calculated using equations (A.1) and (A.2), respectively.

$$V_{meas} = \frac{V_c(p_1^{*} - p_2^{*})}{(p_1^{*} - p_2^{*}) - \frac{p_2^{*}}{p_2^{o}}(p_1^{o} - p_2^{o})} \tag{A.1}$$

$$V_{exp} = V_{meas}\frac{p_1^{o} - p_2^{o}}{p_2^{o}} \tag{A.2}$$

where

p_1^{o}, p_2^{o} are the pressures in the empty pyknometer respectively before and after expansion into the expansion cell;

p_1^{*}, p_2^{*} are the pressures in the pyknometer containing the calibration specimen respectively before and after expansion into the expansion cell;

V_c is the volume of the calibration specimen;

V_{meas} is the volume of the measurement cell;

V_{exp} is the volume of the expansion cell.

A.3 Procedure

After introducing the specimen into the measurement cell, the specimen volume is measured in the same way as in one of the calibration steps [see Figure A.1 c)]. Having brought the measurement cell to pressure p_1, the equilibrium pressure p_2 is obtained by allowing the gas to expand into the expansion cell. The volume of the test specimen at temperature T is given by equation (A.3):

$$V_s^T = V_{meas} - \frac{V_{exp}}{\frac{p_1}{p_2} - 1} \tag{A.3}$$

where

p_1, p_2 are the pressures in the pyknometer containing the test specimen respectively before and after expansion into the expansion cell;

V_sT is the volume of the test specimen at temperature T.

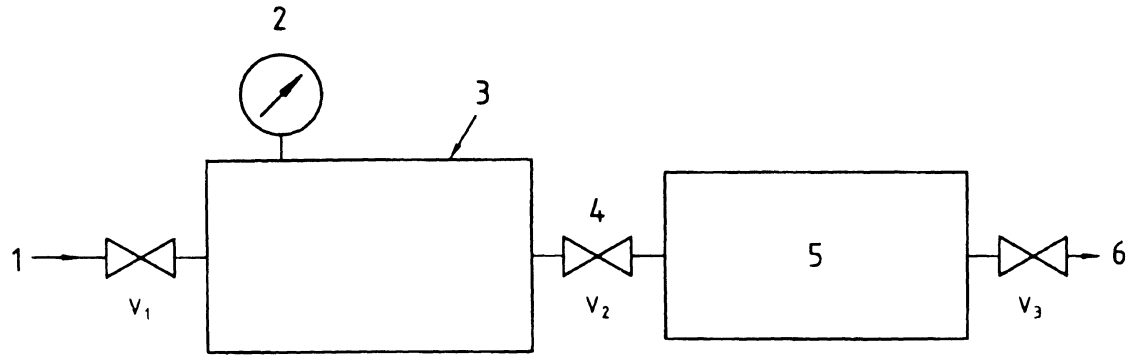
The density can be calculated by dividing the mass of the specimen by its volume:

$$\rho_s^T = \frac{m_s}{V_s^T} \qquad \text{(A.4)}$$

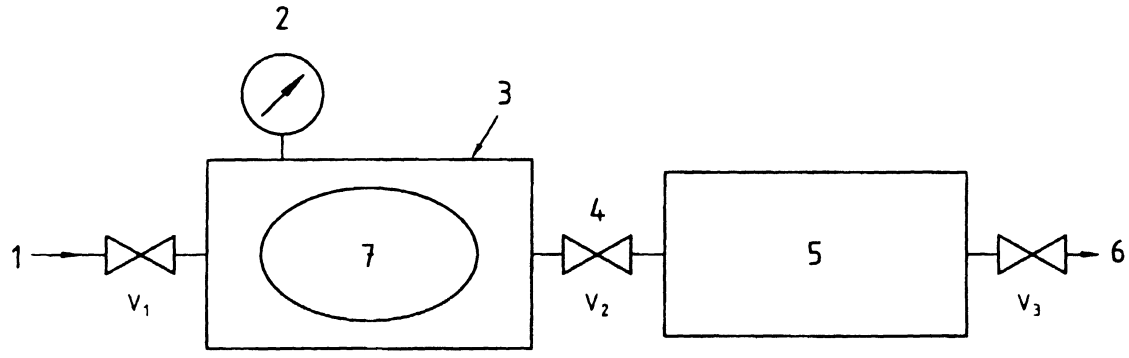
where

m_s is the mass of the test specimen;

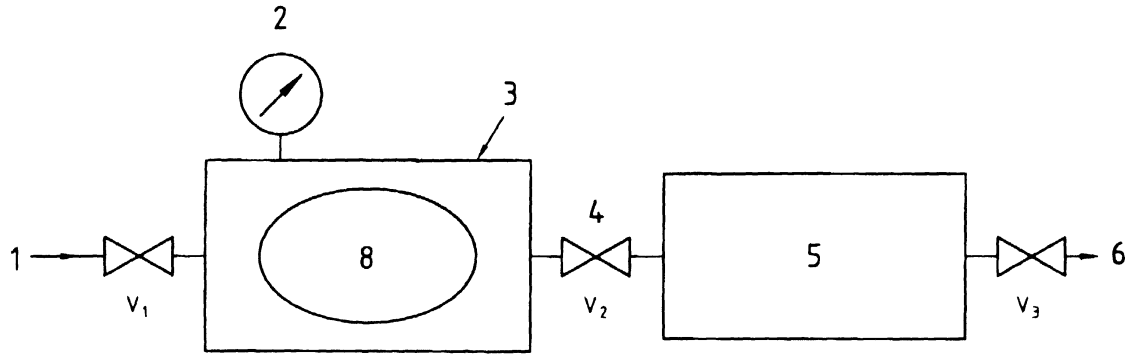
$\rho_s{}^T$ is the density of the test specimen at temperature T.



a) Schematic diagram of pyknometer



b) Calibration



c) Measurement

Key

1 Gas inlet
2 Pressure sensor
3 Measurement cell
4 Connecting valve
5 Expansion cell
6 Gas outlet
7 Calibration specimen
8 Test specimen

Figure A.1 — Two-chamber pressure-type gas pyknometer

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