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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Plastics — Polymer dispersions — Determination of density

Plastiques — Dispersions de polymères — Détermination de la masse volumique

Reference number
ISO 8962 : 1987 (E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 8962 was prepared by Technical Committee ISO/TC 61, *Plastics*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Plastics — Polymer dispersions — Determination of density

1 Scope and field of application

This International Standard specifies two methods of test for determining the density of polymer dispersions (aqueous and non-aqueous) :

— Method I : Pyknometer method for high-precision measurements at all viscosity ranges of polymer dispersions

— Method II : Hydrometer method for polymer dispersions of low to moderately high viscosity ranges (approximately < 0,5 Pa·s)

NOTES

1 The presence of foam in the dispersion exerts a pronounced influence on results. The only dispersions that can be expected to be free, for the most part, from microfoam have a very low viscosity. Many dispersions of medium to high viscosity contain finely divided foam bubbles that are extremely difficult to remove. In such cases, a wide margin of error can be expected.

2 A dilution of polymer dispersions of very high viscosity does give approximately linear density changes within the precision of the method (to three decimal places)

2 References

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

ISO 2811, *Paints and varnishes — Determination of density.*

3 Definition

density : Mass divided by volume.

4 Method I : Pyknometer method for high-precision measurements at all viscosity ranges of polymer dispersions

NOTE — This method is based on ISO 2811.

4.1 Materials

4.1.1 Chromic acid, cleaning solution.

4.1.2 Distilled water or **water of equivalent purity.**

4.1.3 Solvent, leaving no residue on evaporation.

4.2 Apparatus

4.2.1 Pyknometers, of capacity 20 to 100 ml. Glass pyknometers are shown in figures 1 and 2; a metal pyknometer is shown in figure 3.

4.2.2 Thermometer, graduated in divisions of 0,1 °C and accurate to 0,2 °C.

4.2.3 Water-bath or **constant-temperature room,** capable of being maintained within ± 0,5 °C of the test temperature (usually 23 ± 0,5 °C) when high accuracy is required, or within ± 2 °C for production control purposes.

4.2.4 Analytical balance, accurate to within 0,1 mg.

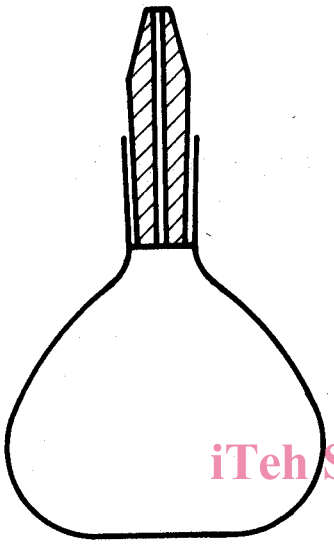

Figure 1 — Gay-Lussac pyknometer

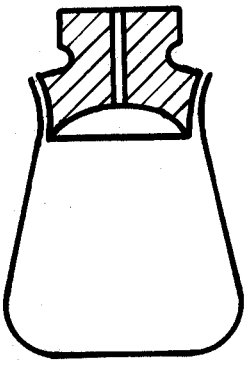

Figure 2 — Hubbard pyknometer

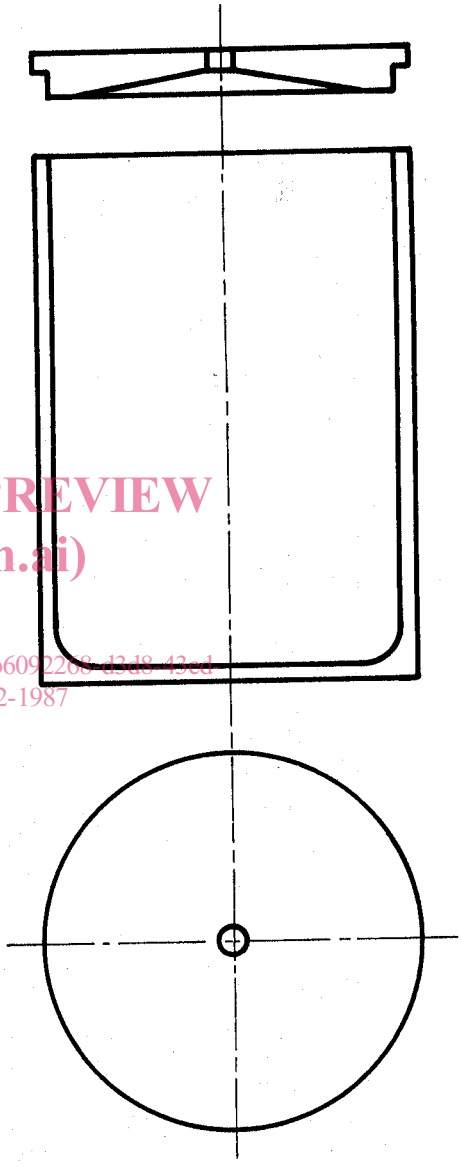

Figure 3 — Metal pyknometer

4.3 Test temperature

Unless otherwise specified or agreed by the interested parties, the measurement shall be made at 23 °C (see ISO 291) with the tolerance specified in the method used.

NOTE — Some countries specify a test temperature of 20 °C for density measurements.

4.4 Procedure

4.4.1 Calibration of pyknometer

4.4.1.1 It is essential that the pyknometer be calibrated and the density of the product be determined at the same temperature.

Clean a glass pyknometer (4.2.1) by using in turn the chromic acid solution (4.1.1) and the distilled water (4.1.2) and/or a solvent (4.1.3). Thoroughly dry the pyknometer. Clean a metal pyknometer by using the solvent and dry it. Allow the pyknometer to attain room temperature and weigh it (m_0). If maximum accuracy is required, the cleaning, drying and weighing of the pyknometer shall be continued until the difference between two successive weighings does not exceed 0,5 mg.

4.4.1.2 Fill the pyknometer with distilled water at a temperature not more than 1 °C below the test temperature (see 4.3). Stopper or cap the pyknometer, leaving the overflow orifice open. Every care shall be taken to prevent the formation of bubbles in the pyknometer.

Place the pyknometer in the water-bath or the constant-temperature room (4.2.3) until the temperature of the bottle and its contents is constant within the tolerance specified in 4.2.3. Remove the overflow by wiping with absorbent material (see note 1) and thoroughly dry the outside of the pyknometer by wiping with absorbent material. Do not remove any subsequent overflow (see note 2). Immediately weigh (see note 3) the filled apparatus to the nearest 0,001 % of its mass (m_1).

NOTES

1 A paper tissue is recommended for this purpose.

2 Handling the pyknometer with bare hands will increase the temperature and cause more overflow from the overflow orifice, and will also leave fingerprints; hence, handling only with tongs and with hands protected by clean, dry, absorbent material is recommended.

3 Immediate and rapid weighing of the filled pyknometer is recommended in order to minimize loss in mass due to evaporation of the water through orifices, and from overflow subsequent to the first wiping after attainment of temperature when this overflow is not retained within a capped enclosure.

4.4.1.3 Calculate the volume of the pyknometer V, in cubic centimetres, using the equation

$$V = \frac{m_1 - m_0}{\varrho}$$

where

m_0 is the mass, in grams, of the empty pyknometer;

m_1 is the mass, in grams, of the pyknometer and water;

ϱ is the density, in grams per cubic centimetre, of water at 23 °C or other agreed temperature (see the table).

Table — Density of water at various temperatures

Temperature °C	Density g/cm³
15	0,999 1
16	0,998 9
17	0,998 7
18	0,998 6
19	0,998 4
20	0,998 2
21	0,998 0
22	0,997 8
23	0,997 5
24	0,997 3
25	0,997 0
26	0,996 8
27	0,995 5
28	0,996 2
29	0,996 0
30	0,995 7

4.4.2 Determination of density of product

Repeat the procedure specified in 4.4.1.1 and 4.4.1.2, using the product in place of the distilled water. Remove any residues from the outside of the pyknometer by wiping with absorbent material moistened with the solvent (4.1.3) and thoroughly dry, wiping with clean absorbent material. Let the mass of the pyknometer and product be m_2.

NOTES

1 When using glass pyknometers with pigmented products, difficulties can be experienced in removing residual pigment, especially from ground glass surfaces. Such residues can be removed by ultrasonic vibration in a water- or solvent-bath.

2 To minimize errors, joints should be firmly seated. For accurate determinations, glass pyknometers are preferred. Metal pyknometers (mass per volume cups) are normally used for density determinations required for production control purposes.

3 If the sample retains air bubbles that do not readily disperse on standing, the method is unsuitable.

4.5 Expression of results

Calculate the density of the product ϱ_t, in grams per cubic centimetre[1)], at the test temperature t, using the formula

$$\varrho_t = \frac{m_2 - m_0}{V}$$

where

m_0 is the mass, in grams, of the empty pyknometer;

1) The SI unit is the kilogram per cubic metre, but the gram per cubic centimetre is an authorized sub-multiple used in practice.

m_2 is the mass, in grams, of the pyknometer and product;

V is the volume, in cubic centimetres, of the pyknometer at the test temperature as determined in 4.4.1.3.

Express the result to three decimal places.

Details of the precision of this method are given for information in the annex.

5 Method II : Hydrometer method for polymer dispersions of low to moderately high viscosity ranges (approximately < 0,5 Pa·s)

5.1 Materials

See 4.1.

5.2 Apparatus

5.2.1 Hydrometers, with suitable graduations, dependent on the limits of the densities to be measured.

5.2.2 Graduated measuring cylinder, of capacity 250 ml, which has an inside diameter at least 25 mm greater than the maximum diameter of the hydrometer and a height 25 mm taller than the distance between the bottom end of the immersed hydrometer and the bottom of the measuring cylinder.

5.2.3 Thermometer, graduated in divisions of 0,1 °C, with a measuring range of 20 to 45 °C.

5.2.4 Water-bath, with thermostatic control and of sufficient depth to contain the graduated measuring cylinder (5.2.2), or **constant-temperature room,** for maintaining the temperature of the sample at 23 ± 0,5 °C (± 2 °C for production control) (or other agreed temperature within the same tolerances). See 4.3.

5.3 Test temperature

See 4.3.

5.4 Procedure

5.4.1 Carefully pour the polymer dispersion for testing into the clean dry measuring cylinder (5.2.2) so as to prevent the formation of bubbles. Place the measuring cylinder in the thermostat-controlled bath or in the constant-temperature room (5.2.4), stirring periodically until the temperature of the sample is 23 ± 0,5 °C (or other agreed temperature within the same tolerances).

NOTE — In order to reduce the time required for reaching thermal equilibrium, the apparatus and sample may first be heated to approximately the test temperature.

5.4.2 Carefully plunge the hydrometer (5.2.1) into the test portion; once equilibrium is obtained, push it two divisions into the liquid, then release it. Wait until the hydrometer is completely still without touching the internal wall of the measuring cylinder. Before taking any measurements, check that the temperature of the sample is 23 ± 0,5 °C (or other agreed temperature within the same tolerance).

5.4.3 Take the reading at the hydrometer scale where it is intersected by the surface of the non-transparent polymer dispersion. In order to read this height, adopt a position such that the eye is slightly below the level of the liquid, then rise again slowly until the surface appears in the form of a straight line intersecting the scale of the hydrometer.

5.5 Expression of results

Record the results as the value of the density obtained, in grams per cubic centimetre, to two decimal places and estimate the third.

6 Test report

The test report shall include the following particulars :

a) reference to this International Standard;

b) complete identification of the product tested;

c) reference to the method used (method I or method II);

d) test temperature;

e) the individual values and the arithmetic mean of the density measurements;

f) any operational details not specified in this International Standard or in the International Standards to which reference is made, or regarded as optional.

Annex

Precision of method I

(This annex does not form an integral part of the Standard.)

With accurate control and measurement of temperature, i.e. at the ± 0,5 °C level, it is possible to attain the following :

A.1 Repeatability

The difference between successive results obtained by the same operator within a short time-interval, with the same apparatus under constant operating conditions on identical test material, at the 95 % confidence level, should not exceed 0,000 6 g/cm^3.

A.2 Reproducibility

The difference between single and independent results obtained by different operators in different laboratories on identical test material, at the 95 % confidence level, should not exceed 0,001 2 g/cm^3.

NOTE — In the case of some products, especially those showing structural viscosity or thixotropy, the above precision may not be attained.

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Descriptors : plastics, polymers, suspensions, tests, determination, density (mass/volume), test equipment.

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