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PLASTICS

METHODS FOR DETERMINING THE DENSITY AND RELATIVE DENSITY

(SPECIFIC GRAVITY) OF PLASTICS EXCLUDING CELLULAR PLASTICS

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BRIEF HISTORY

The ISO Recommendation R 1183, *Plastics – Methods for determining the density and relative density (specific gravity) of plastics excluding cellular plastics*, was drawn up by Technical Committee ISO/TC 61, *Plastics*, the Secretariat of which is held by the American National Standards Institute (ANSI).

Work on this question led to the adoption of Draft ISO Recommendation No. 823, which was circulated to all ISO Member Bodies for enquiry in July 1965. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Argentina	France	Romania
Australia	Germany	South Africa, Rep. of
Austria	Greece	Spain
Belgium	Hungary	Sweden
Brazil	India	Switzerland
Canada	Italy	U.A.R.
Chile	Japan	United Kingdom
Colombia	Netherlands	U.S.A.
Czechoslovakia	New Zealand	U.S.S.R.
Finland	Poland	Yugoslavia

No Member Body opposed the approval of the Draft.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in February 1970, to accept it as an ISO RECOMMENDATION.

ISO Recommendation   R 1183   February 1970

PLASTICS

METHODS FOR DETERMINING THE DENSITY AND RELATIVE DENSITY

(SPECIFIC GRAVITY) OF PLASTICS EXCLUDING CELLULAR PLASTICS

1. SCOPE

This ISO Recommendation describes four methods for the determination of the density and relative density (specific gravity) of non-cellular plastics in the forms of films, sheet, tube, moulded objects, powders and moulding powders :

Method A

Immersion method for plastics in a finished condition, whether machined or otherwise formed, such as sheet, rod, tube and moulded articles.

Method B

Pycnometer method for plastics in preformed condition such as moulding powder or pellets, flake and the like.

Method C

Sink-float method for plastics in forms similar to those required for Method A, and pellets.

Method D

Density gradient column method for plastics in forms similar to those required for Method A, and pellets.

Density gradient columns are columns of liquid the densities of which increase uniformly from top to bottom. They are particularly suited to measurements of small specimens, materials with density less than 1 g/cm³ and density differences.

Density and relative density are frequently used to follow the variations in the physical structure of specimens and in calculations of the amount of material necessary to fill a given volume. Density is the preferred property relating the mass and volume of an object, specimen or material. These properties may also be useful in determining uniformity among samples or specimens. These methods are designed to yield results accurate to at least 0.2 % without applying corrections for weighings in air and to 0.05 % with such corrections.

The density of plastics often depends on the methods employed in preparing the test specimens. When this is the case, precise details of the methods of preparation must be given; these are ordinarily stipulated in the specifications for the material.

2. DEFINITIONS

2.1 *Density*. The mass per unit volume of material at t °C; it is expressed as

density, t °C (symbol : ρ_t) in kg/m^3, g/cm^3 or g/ml

Ordinarily t will be one of the standard laboratory temperatures, 20, 23 or 27 °C.

2.2 *Relative density*. The ratio of the mass of a given volume of material to that of an equal volume of a reference material at the same temperature; it is expressed as

relative density, t/t °C (symbol : d_t^t)

where t is the temperature in degrees Celsius. Ordinarily, t will be one of the standard laboratory temperatures, 20, 23 or 27 °C.

The term "specific gravity" is often used for "relative density" when the reference substance is water.

Relative density may also be defined as the ratio of the density of a substance to the density of a reference substance under conditions that should be specified for both substances.

Density in grammes per cubic centimetre at t °C may be converted to specific gravity at these temperatures as follows :

$$S = \frac{\rho_t}{k}$$

where

S is the specific gravity;

ρ_t is the density of the specimen;

k is the density of water, at temperature t, values at the standard laboratory temperatures being as follows :

t	k
°C	g/cm^3
20*	0.9982
23	0.9976
27	0.9965

Consult the International Bureau of Weights and Measures for exact relationships between density and specific gravity at other temperatures.

NOTE. – The following equivalent terms based upon ISO Recommendation R 31, Part III, *Quantities and units of mechanics*, are given here for clarification.

English term	French term	Symbol	Formulation	Units
1. density (mass density)	masse volumique	ϱ, ρ	$\dfrac{m}{V}$	kg/m^3 g/cm^3 g/ml
2. relative density	densité relative	d	$\dfrac{\rho_1}{\rho_2}$	dimensionless
3. specific volume	volume massique	v	$\dfrac{V}{m} = \dfrac{1}{\rho}$	m^3/kg cm^3/g ml/g

* The reference temperature : see ISO Recommendation R 291, *Standard atmospheres for conditioning and testing.*

3. APPARATUS

3.1 Methods A and B

3.1.1 *Balance* with a sensitivity of 0.1 mg.

3.1.2 *Pan straddle* or other stationary support.

3.1.3 *Pycnometer* of, for example, 50 ml capacity, with side-arm overflow capillary, for determining the density of the immersion liquid when this liquid is not water. The pycnometer should be equipped with a thermometer graduated in 0.1 °C from 0 to 30 °C.

3.1.4 *Liquid bath* capable of thermostatic control to within 0.1 °C.

3.2 Method C

3.2.1 *Stoppered glass vessels*, 200 ml, approximately 100 mm high.

3.2.2 *Thermostatically controlled bath* as for Methods A and B.

3.2.3 *Means for determining the density of liquids* to an accuracy of ± 0.001 g/ml.

3.3 Method D

3.3.1 *Density gradient column* consisting of a suitable tube, which may be graduated, not less than 40 mm diameter, with cover.

3.3.2 *Thermostatically controlled bath* as for Methods A and B.

3.3.3 *A number of calibrated glass floats* covering the density range to be studied and approximately evenly distributed throughout this range.

3.3.4 *A set of suitable hydrometers* covering the range of densities to be studied and having density graduations of 0.001 g/cm³, or other suitable means for measuring densities of liquids.

3.3.5 *Balance* with a sensitivity of 0.1 mg.

3.3.6 *Siphon* or *pipette assembly* for filling the gradient tube such as shown in the Figure (page 10) or any other which will yield an equivalent result.

3.3.7 *Cathetometer* (optional).

4. IMMERSION LIQUIDS

The liquid or solution with which the specimen comes into contact during the measurement should have no effect on the specimen and should not be absorbed by the specimen in any significant quantity.

4.1 Methods A and B

Freshly distilled water or other suitable liquid containing not more than 0.1 % of a wetting agent to help in removing air bubbles.

4.2 Method C

Mixtures of liquids in various proportions or solutions of various concentrations having densities evenly distributed throughout the range of interest, containing, if desired, not more than 0,1 % of a wetting agent.

4.3 Method D

Mixtures of two liquids selected from those in Table 2, or other suitable combinations known to meet the requirements of this ISO Recommendation, prepared according to the procedure described in clause 7.4.2.

5. SPECIMENS

5.1 Method A

Specimens may consist of films, sheets, tubes, moulded objects, granules other than powder, or specimens taken from such forms with surfaces made smooth in an appropriate way to minimize the entrapment of air bubbles upon immersion in the liquid. The specimen should be of any convenient size to give adequate clearance between the specimen and beaker (a mass of approximately 1 to 5 g is often convenient).

5.2 Method B

Specimens of powder or flake should be measured in the form in which they are received. Specimen size may be approximately 1 to 5 g or more.

5.3 Method C

Specimens should be in any convenient solid form.

5.4 Method D

Specimens should consist of pieces of the material cut to any convenient shape for ease of identification. Dimensions should be chosen to permit accurate measurement of the position of the centre of the volume. Surfaces should be smooth and free from cavities to preclude the entrapment of air bubbles upon immersion.

6. CONDITIONING

In general, conditioning specimens to constant temperature will not be required because the determination itself brings the specimen to the constant temperature of the test. Conditioning specimens to constant moisture content, on the other hand, may be important.

Specimens whose change in density upon conditioning may be greater than the accuracy required of the density determination should be conditioned before measurement in accordance with the applicable material specifications.

Specimens whose change in density with time or conditioning is the primary purpose of the measurements should be conditioned in accordance with any agreed plan.

7. PROCEDURES

7.1 Method A

Weigh the specimen suspended with a wire of diameter 0.125 mm (0.005 in) or less*.

Immerse the specimen, still suspended by the wire, in the immersion liquid contained in a beaker on the pan straddle or other stationary support. The temperature of the immersion liquid should be $t \pm 0.1\ ^\circ C$ where t is 20, 23 or 27 $^\circ C$. Remove adhering air bubbles with a fine wire. It is convenient to mark the level of immersion, for example by a shallow notch filed in the wire. Weigh the immersed specimen.

Determine the density of immersion liquids other than water by weighing the pycnometer empty and then containing freshly distilled water at temperature t and weighing the same pycnometer, after cleaning and drying, filled with the immersion liquid (also at temperature t). Calculate the density, ρ_x, of the immersion liquid as follows :

$$\rho_x = \frac{m_x}{m_w} \rho_w$$

where

m_x is the mass of liquid;

m_w is the mass of water;

$\rho_w = k$ as defined in clause 2.2.

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* Such a fine wire renders correction for the apparent loss in mass of the wire unnecessary.

Calculate the density of the specimen from the following formula :

$$\text{density}, t\,^{\circ}\text{C} = \frac{a\,\rho_x}{a - b} \text{ in g/cm}^3 \text{ or g/ml}$$

where

    a is the mass, in grammes, of the specimen in air;

    b is the uncorrected mass, in grammes, of the specimen in the immersion liquid;

    ρ_x is the density of the immersion liquid determined as above, expressed in grammes per cubic centimetre or grammes per millilitre.

NOTE. — 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 fluid level as does the specimen during immersion. The apparent loss in mass, c, of the sinker on immersion may be thought of as a part of the suspending wire; it must be subtracted from b in the above equation. Thus :

$$\text{density}, t\,^{\circ}\text{C} = \frac{a\,\rho_x}{a - (b - c)} \text{ (if no sinker is used, } c = 0\text{)}$$

7.2   Method B

Weigh the pycnometer empty and dry. Weigh a suitable quantity of the plastics material in the pycnometer. Cover the test specimen with immersion liquid and remove all air from the specimen by applying a vacuum to the pycnometer standing in a desiccator. Break the vacuum and fill the pycnometer with the immersion liquid. Bring it to constant temperature in the bath (3.1.4) and then complete filling exactly to the limits of the capacity of the pycnometer. Wipe dry and weigh the pycnometer with its contents.

Empty, clean and fill with boiled distilled water, removing air as above, and determine the mass of the pycnometer and contents at the temperature of the test. Repeat the process with the immersion liquid if an immersion liquid other than water was used, and calculate its density as described in clause 7.1.

Calculate the density of the specimen from the following formula :

$$\text{density}, t\,^{\circ}\text{C} = \frac{m\,\rho_x}{m_1 - m_2}$$

where

    m is the mass, in grammes, of the specimen;

    m_1 is the mass, in grammes, of liquid required to fill the pycnometer;

    m_2 is the mass, in grammes, of liquid required to fill the pycnometer containing the specimen;

    ρ_x is the density of the immersion liquid determined as above, in grammes per cubic centimetre.

7.3   Method C

Maintain quantities of immersion liquids having densities near that of the specimen each in a separate glass vessel at $t \pm 0.5\,^{\circ}\text{C}$. Place a test specimen in each vessel, taking care that no air bubbles are attached to the specimens.

Note the densest liquid in which the specimen sinks and the least dense in which it floats. Without delay, determine the densities at $t \pm 0.5\,^{\circ}\text{C}$ of these two liquids to the nearest 0.001 g/ml.

The density of the material under test lies between these two values.

NOTE. — If a more precise determination is required, a further set of liquids may be prepared to cover the requisite narrower range of densities and the procedure repeated.

7.4 Method D

7.4.1 *Preparation of glass floats.* Glass floats, prepared by any convenient method, should be fully annealed, approximately spherical and of diameter not greater than 5 mm.

Prepare a solution of about 500 ml of the liquids to be used in the gradient tube such that the density of the solution as measured with hydrometers is approximately equal to the desired lowest density. When the floats are at room temperature, drop them gently into the solution. Save the floats that sink very slowly and discard those that sink rapidly or save them for another tube. If necessary to obtain a suitable range of floats,

(*a*) grind selected floats to the desired density by rubbing the bead part of the float on a glass plate on which is spread a thin slurry of 400 or 600 mesh silicon carbide or other appropriate abrasive, or

(*b*) etch floats with hydrofluoric acid.

Progress may be followed by dropping the float in the test solution at intervals and noting the change in its rate of sinking.

Determine the density of each standard glass float prepared as above by placing the float in a solution of two suitable liquids the density of which can be varied over the desired range by the addition of either liquid to the mixture. If the float sinks, add the denser liquid stirring it well. Let the solution rest and add no further liquid until the float shows signs of moving. If it shows signs of moving, repeat the above procedure until the float remains stationary for at least 30 minutes. It is convenient to make these measurements in the same temperature bath as is used for the density gradient tube. In any case, the solution for calibration of the floats should be maintained at $t \pm 0.1\ °C$, where t is 20, 23 or 27 °C.

Determine the density of liquid, to the nearest 0.0001 g/ml, in which the float remains in equilibrium, using the pycnometer method described above in Method A or other convenient method, for example, with hydrometers. Make "in vacuo" corrections for all weighings (see Table 1). Record this density as the density of the float and repeat the procedure for each float. It is convenient to place all floats in the liquid together, then calibrate them in turn, starting with the least dense.

Alternatively, calculate the density of the liquid mixture in which the float remains stationary from the volumes of liquids used, taking care to apply corrections for fluid contractions.

7.4.2 *Preparation of density gradient column.* Place the graduated glass tube in the thermostatically controlled bath (3.3.2) maintained at $t \pm 0.1\ °C$. Select a suitable combination of liquids from Table 2 such that the resulting sensitivity of the column will be no poorer than 0.001 g/ml per centimetre of tube length. Satisfactory density ranges for a column are, for example, 0.001 to 0.1 g/ml. The extreme upper and lower portions of the tube should not be used, and readings outside the calibrated part should not be taken.

Any of several methods of preparing the gradient may be used, of which one is as follows :

Assemble the apparatus as shown in the Figure, using beakers of the same diameter. Then select an appropriate amount of two suitable liquids which previously have been carefully de-aerated by gentle heating or an applied vacuum. The volume of liquid used in the mixer (beaker B in the Figure) must be equal to at least one half of the total volume desired in the gradient tube. Typical liquid systems for density gradient columns are listed in Table 2.

NOTE. – Calculate the density of the liquids in beaker A (see Figure) used to prepare any desired gradient as follows :

$$\rho_A = \rho_0 - \left[\frac{2(\rho_0 - \rho)\,V_B}{V} \right]$$

where

ρ_A is the density of the starting liquid in beaker A;

ρ_0 is the upper limit of required density and starting liquid density for beaker B, chosen to be 0.005 g/ml heavier than the density of the densest glass float calibrated for the individual gradient tube;

ρ is the lower limit of required density, chosen to be 0.01 g/ml lighter than the density of the least dense glass float calibrated for the individual gradient tube;

V_B is the volume of starting liquid in beaker B;

V is the total volume required in the gradient tube.

Place an appropriate mass of the denser liquid into beaker B of suitable size and begin to stir using a high-speed propeller-type stirrer. Adjust the speed of stirring such that the surface of the liquid does not fluctuate greatly, and place an equal mass of the less dense liquid into beaker A. Take care that no air is dispersed in the liquid.

After priming the siphon, which should be equipped with a capillary tip at the delivery end for flow control with liquid A, start the delivery of the liquid to the gradient tube. Then fill the tube to the top graduation desired.

NOTE. – Preparation of a suitable gradient column may require 1 to $1\frac{1}{2}$ hours or longer, depending upon the volume required in the gradient tube.

Allow the density gradient column so prepared to rest for at least 24 hours.

For every 25 cm of tube length dip a minimum of five clean calibrated floats spanning the effective range of the column into the less dense liquid used in the preparation of the gradient column, and add them to the tube. If it is observed that the floats group together and do not spread out evenly in the tube, discard the solution and repeat the procedure.

Alternatively, place the floats in the tube immediately upon preparation of the column. If the floats appear to group together and do not spread out evenly in the tube, discard the solution and repeat the preparation. Cap the tube, and retain it in the constant temperature bath for 24 to 48 hours. At the end of this time measure the heights of floats from the bottom of the tube to the nearest millimetre and plot a curve of the density of the floats as a function of their heights. The curve so produced should

(a) be monotonic,

(b) have no discontinuity,

(c) have not more than one point of inflection.

The solution should otherwise be discarded.

NOTE. – Density gradient columns normally remain stable for several months. A daily check of the original calibration will reveal when instability has been reached.

7.4.3 *Measurement of density*. Wet three representative test specimens with the less dense of the two liquids used in the tube and gently place them in the tube. Allow the tube and specimens to reach equilibrium, which will require 10 minutes or more. Films of thickness less than 0.05 mm require at least $1\frac{1}{2}$ hours to settle. Rechecking specimens of thin films after several hours is advisable.

NOTES

1. A fine wire carefully manipulated is suitable for removing air bubbles from specimens.

2. Old samples can be removed without destroying the gradient by slowly withdrawing a wire screen basket attached to a long wire. This can be done conveniently by means of a clock motor. Withdraw the basket from the bottom of the tube and, after cleaning, return it to the bottom of the tube. It is essential that this procedure be performed at a slow enough rate (approximately 10 mm length of column per minute) so as not to disturb the density gradient.

7.4.4 *Calculations*. The densities of the specimens may be determined graphically or by calculation from the levels to which they settle, using either of the following methods :

(a) Make a plot of float density versus float position on a chart large enough to be read accurately to ± 0.0001 g/ml and ± 1 mm. Plot the positions of the specimens on the chart and read their corresponding densities.

(b) Calculate the density by interpolation as follows :

$$\text{density at } x = a + \frac{(x-y)(b-a)}{(z-y)}$$

where

y and z are distances (measured from an arbitrary level) of the two standard floats bracketing the specimen;

a and b are the respective densities of these two standard floats;

x is the distance of the specimen above the same arbitrary level.

NOTE. – This method does not reveal calibration errors which can be detected with the graphical method (a).