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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

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

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

Reference number
ISO 1183 : 1987 (E)

Foreword

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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 1183 was prepared by Technical Committee ISO/TC 61, *Plastics*.

It cancels and replaces ISO Recommendation R 1183 : 1970, of which it constitutes a technical revision.

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 — Methods for determining the density and relative density of non-cellular plastics

1 Scope and field of application

1.1 This International Standard specifies four methods for the determination of the density and relative density of non-cellular plastics in the form of sheet, film, tube, moulded objects, and moulding powders, granules and pellets.

- Method A

Immersion method for plastics in a finished condition, whether machined or otherwise formed (see 5.1.3), but not powders.

- Method B

Pyknometer method for plastics in the form of powder, granules, pellets, flake or moulded articles reduced to small particles.

- Method C

Titration method for plastics in forms similar to those required for method A, including pellets.

- Method D

Density gradient column method for plastics in forms similar to those required for method A, and including pellets. Density gradient columns are columns of liquid, the densities of which increase uniformly from top to bottom. They are particularly suited to measurement of small samples of products and to comparison of densities.

1.2 Density and relative density are used frequently, both to follow the variations in the physical structures of specimens and in calculation 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 assessing 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.

1.3 Often the density of plastics will depend upon the methods employed in the preparation of test specimens. When this is the case, precise details of the methods of preparation shall be given; these are ordinarily included in the specifications for the material.

2 References

ISO 31-3, *Quantities and units of mechanics.*

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

3 Definitions

3.1 density, ϱ_t**:** The ratio of the mass of the sample to its volume V_t (at the temperature t), expressed in kilograms per cubic metre, grams per cubic centimetre or grams per millilitre.

Ordinarily, t will be one of the standard laboratory temperatures specified in ISO 291 (23 or 27 °C).

3.2 relative density: The ratio of the mass of a given volume of material at the temperature t_1 to that of an equal volume of a reference material at the temperature t_2; it is expressed as relative density, at t_1 and t_2 (symbol: $d^{t_1}_{t_2}$) where t is the temperature, in degrees Celsius.

Ordinarily, t will be one of the standard laboratory temperatures specified in ISO 291 (23 or 27 °C).

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 are specified for both substances.

NOTES

1 When the reference substance is water, the English term "specific gravity" is often used instead of "relative density", and the French term "densité" instead of "densité relative".

Density at t_1 °C may be converted to specific gravity, using the equation

$$d^{t_1}_{t_2} = \frac{\varrho_{\mathrm{S},t_1}}{\varrho_{\mathrm{W},t_2}}$$

where

$d^{t_1}_{t_2}$ is the specific gravity of the sample;

ϱ_{S,t_1} is the density, in grams per cubic centimetre, of the specimen, at temperature t_1;

ϱ_{W,t_2} is the density, in grams per cubic centimetre, of water, at temperature t_2; values at some laboratory temperatures are as follows:

t	$\varrho_{W,t}$
°C	g/cm^3
20	0,998 2
23[1]	0,997 6
27	0,996 5

The International Bureau of Weights and Measures should be consulted for exact relationships between density and specific gravity at other temperatures.

2 The following equivalent terms, based upon ISO 31-3, are given here for clarification.

English term	French term	Symbol	Formulation	Units
density (mass density)	masse volumique	ϱ	$\frac{m}{V}$	kg/m^3 g/cm^3 g/ml
relative density	densité relative	d	$\frac{\varrho_1}{\varrho_2}$	dimensionless
specific volume	volume massique	v	$\frac{V}{m} = \frac{1}{\varrho}$	m^3/kg cm^3/g ml/g

4 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 required.

Specimens which change in density during the test to such an extent that it may exceed the accuracy required of the density determination shall be conditioned before measurement in accordance with the applicable material specifications. When changes in density with time or atmospheric conditions are the primary purpose of the measurements, the speciments shall be conditioned as agreed upon by the interested parties.

5 Methods

5.1 Method A

5.1.1 Apparatus

5.1.1.1 Balance, accurate to 0,1 mg.

5.1.1.2 Pan straddle or other stationary support.

5.1.1.3 Pyknometer, 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 pyknometer shall be equipped with a thermometer graduated in 0,1 °C from 0 to 30 °C.

5.1.1.4 Liquid bath, capable of being thermostatically controlled to within 0,1 °C.

5.1.2 Immersion liquids: Freshly distilled water or other 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 and shall not be absorbed by the specimen in any significant quantity.

5.1.3 Specimens

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 shall be of any convenient size to give adequate clearance between the speciment and beaker (a mass of approximately 1 to 5 g is often convenient).

5.1.4 Procedure

5.1.4.1 Weigh the specimen suspended with a wire of diameter 0,125 mm or less[2].

5.1.4.2 Immerse the specimen, still suspended by the wire, in the immersion liquid (5.1.2), contained in a beaker on the pan straddle or other stationary support (5.1.1.2). The temperature of the immersion liquid shall be $t \pm 0{,}1$ °C, where t is 23 or 27 °C. Remove adhering air bubbles with a fine wire. It is convenient to mark the level of immersion, for example, by a shallow notch in the wire. Weigh the immersed specimen.

5.1.4.3 Determine the density of immersion liquids other than water. Weigh the pyknometer (5.1.1.3) empty and then containing freshly distilled water at temperature t. Weigh the same pyknometer, after cleaning and drying, filled with the immersion liquid (also at temperature t). Calculate the density ϱ_{IL} of the immersion liquid, using the equation

$$\varrho_{IL} = \frac{m_{IL}}{m_W} \times \varrho_{W,t}$$

where

m_{IL} is the mass of the immersion liquid;

m_W is the mass of water;

$\varrho_{W,t}$ is the density of water at temperature t.

5.1.4.4 Calculate the density $\varrho_{S,t}$ of the specimen, using the equation

$$\varrho_{S,t} = \frac{m_{S,A}\,\varrho_{IL}}{m_{S,A} - m_{S,IL}}$$

where

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

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[1] Reference temperature: see ISO 291.

[2] Such a fine wire renders corrections for the apparent loss in mass of the wire unnecessary.

$m_{\mathrm{S,IL}}$ is the uncorrected mass, in grams, of the specimens in the immersion liquids;

ϱ_{IL} is the density of the immersion liquid determined as in 5.1.4.3, expressed in grams per cubic centimetre or grams 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 Δm of the sinker on immersion may be considered as a part of the suspending wire; it should be subtracted from $m_{\mathrm{S,IL}}$ in the above equation.

Thus,

$$\varrho_{\mathrm{S},t} = \frac{m_{\mathrm{S,A}}\,\varrho_{\mathrm{IL}}}{m_{\mathrm{S,A}} - (m_{\mathrm{S,IL}} - \Delta m)}$$

(if no sinker is used, $\Delta m = 0$)

where

$m_{\mathrm{S,A}}$ and $m_{\mathrm{S,IL}}$ have the same meaning as in the preceding equation;

Δm is the apparent loss in mass of the sinker on immersion in the liquid.

For correction of buoyancy, see clause 6.

5.2 Method B

5.2.1 Apparatus

5.2.1.1 Balance, accurate to 0,1 mg.

5.2.1.2 Pan straddle or other stationary support.

5.2.1.3 Pyknometer (see 5.1.1.3).

5.2.1.4 Liquid bath (see 5.1.1.4).

5.2.2 Immersion liquids

See 5.1.2.

5.2.3 Specimens

Specimens of powders, granules, pellets or flake shall be measured in the form in which they are received. Specimen mass shall be in the range of 1 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 air from the specimen by placing the pyknometer in a desiccator and applying a vacuum. Break the vacuum and fill the pyknometer with the immersion liquid. Bring it to constant temperature in the liquid bath (5.2.1.4) and then complete filling exactly to the limits of the capacity of the pyknometer with its contents. Wipe dry and weigh the pyknometer with specimen and immersion liquid.

5.2.4.2 Empty and clean the pyknometer. Fill it with boiled distilled water, removing air as above, and determine the mass of the pyknometer and 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 calculate its density as specified in 5.1.4.3.

5.2.4.4 Calculate the density of the specimen, $\varrho_{\mathrm{S},t}$, using the equation

$$\varrho_{\mathrm{S},t} = \frac{m_{\mathrm{S}}\,\varrho_{\mathrm{IL}}}{m_1 - m_2}$$

where

m_{S} is the mass, in grams, of the specimen;

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

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

ϱ_{IL} is the density of the immersion liquid determined as specified in 5.1.4.3, in grams per cubic centimetre.

For correction of buoyancy, see clause 6.

5.3 Method C

5.3.1 Apparatus

5.3.1.1 Thermostatically controlled bath (see 5.1.1.4).

5.3.1.2 Glass cylinder, of capacity 250 ml.

5.3.1.3 Thermometer, graduated in 0,1 °C divisions, with a range suitable for the test temperature used.

5.3.1.4 Measuring flask, of capacity 100 ml.

5.3.1.5 Draw glass stirrers.

5.3.1.6 Automatic burette, of capacity 25 ml, graduated in 0,1 ml and kept in the thermostatically controlled bath (5.3.1.1).

5.3.2 Immersion liquids: Two miscible freshly distilled liquids of different densities. The densities given for various liquids in the table in the annex may serve as an appropriate guide.

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

5.3.3 Specimens

Specimens shall be in any suitable solid form.

5.3.4 Procedure

5.3.4.1 Choose a liquid whose density is next below the one of the material to be tested. If necessary, make a preliminary rapid test in a few millilitres of the liquid.

5.3.4.2 By means of the measuring flask (5.3.1.4), accurately measure 100 ml of one of the immersion liquids and transfer it to the clean, dry 250 ml test glass cylinder (5.3.1.2). Secure the cylinder in the thermostatically controlled bath (5.3.1.1) at the temperature *t*. Ordinarily, *t* will be one of the standard laboratory temperatures (23 or 27 °C).

5.3.4.3 Place the pieces of the test specimen in the cylinder. They shall fall to the bottom and be free of air bubbles. Allow about 5 min for the cylinder and its contents to stabilize at the bath temperature, stirring several times.

5.3.4.4 When the temperature of the liquid is *t* °C, add the second immersion liquid (5.3.2) millilitre by millilitre supplied from the automatic burette (5.3.1.6). Stir the liquid vertically after each addition by means of a flat-tipped glass rod (5.3.1.5). and avoid producing air bubbles.

After each addition of the second liquid and mixing, observe the behavior of the test pieces. At first, they fall rapidly to the bottom, then their rate of fall becomes slower. At this point, add the second liquid in 0,1 ml fractions. Note the total amount of second liquid added when the first pieces keep afloat within the liquid, at the level to which they were brought by stirring, without moving up or down for at least 1 min.

Add more of the second liquid until the heaviest pieces reach a state of neutral equilibrium. For each sample, note the amount of the second liquid required. For each pair of liquids, the functional relationship between the amount of the second liquid added and the density shall be established and plotted in graphical form.

NOTES

1 It is recommended that the thermometer (5.3.1.3) be kept permanently in the solution. 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.

2 The density of the liquid mixture at each point of the graph of functional relationship can be evaluated by the pyknometer method.

5.4 Method D

5.4.1 Apparatus

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

5.4.1.2 Thermostatically controlled bath (see 5.1.1.4).

5.4.1.3 Calibrated glass floats, covering the density range to be studied and approximately evenly distributed throughout this range.

5.4.1.4 Set of suitable hydrometers, covering the range of densities to be studied, having density graduations of 0,001 g/cm^3, or other suitable means for measuring densities of liquids.

5.4.1.5 Balance (see 5.1.1.1).

5.4.1.6 Siphon or **pipette assembly,** for filling the gradient tube (5.4.1.1), such as shown in the figure or any other which will yield an equivalent result.

5.4.1.7 Cathetometer (optional).

5.4.2 Immersion liquids

See 5.3.2.

The mixtures of the two liquids selected shall be prepared as specified in 5.4.4.2.

5.4.3 Specimens

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

5.4.4 Procedure

5.4.4.1 Preparation of glass floats

5.4.4.1.1 Glass floats (see 5.4.1.3), prepared by any convenient method, shall be fully annealed, approximately spherical and of diameter not greater than 5 mm.

5.4.4.1.2 Prepare a solution of about 500 ml of the immersion liquids (5.4.2) to be used in the gradient tube (5.4.1.1), such that the density of the solution as measured with hydrometers (5.4.1.4) is approximately equal to the lowest desired density. When the floats are at ambient temperature, drop them gently into the solution. Retain the floats that sink very slowly and discard those that sink rapidly or retain them for another tube. If necessary, in order to obtain a suitable range of floats,

a) adjust 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 silicon carbide with less than 38 µm (400 mesh) particle size, or other appropriate abrasive, or

b) etch floats with hydrofluoric acid.

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

5.4.4.1.3 Determine the density of each standard glass float prepared as above by placing the float in a solution of two suitable liquids (5.4.2), 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. Allow the solution to rest and add no further liquid until the float shows signs of moving. If it does move, repeat the above procedure until the float remains stationary for at least 30 min. It is convenient to make these measurements in the bath (5.4.1.2) at the same temperature as is used for the density gradient tube. In any case, the solution for calibration of the floats shall be maintained at $t \pm 0{,}1$ °C where t is 23 or 27 °C.

5.4.4.1.4 Determine the density of the solution, to the nearest 0,000 1 g/ml, in which the float remains in equilibrium, using the pyknometer method (see 5.1.4.3) or other convenient method, for example, with hydrometers. Apply the buoyancy correction (see clause 6) if necessary. Record this density as the density of the float and repeat the procedure for each float. If 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.

5.4.4.2 Preparation of density gradient column

5.4.4.2.1 Place the graduated tube in the thermostatically controlled bath (5.4.1.2), maintained at $t \pm 0{,}1$ °C. Select a suitable combination of liquids (5.4.2) from the table in the annex such that the resulting sensitivity of the column will preferably 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 shall not be used, and readings shall not be taken outside the calibrated part.

5.4.4.2.2 Any of several methods for preparing the gradient may be used, including the following:

Assemble the apparatus as shown in the figure, using two vessels of the same size. Then select an appropriate amount of two suitable liquids which previously have been carefully deaerated by gentle heating or application of a vacuum. The volume of liquid used in the mixer (vessel 2 in the figure) shall be equal to at least one-half of the total volume desired in the gradient tube (see note 1).

Place an appropriate mass of the less dense liquid into vessel 2 of suitable size and begin to stir, using a magnetic stirrer. Adjust the speed of stirring so that the surface of the liquid does not fluctuate greatly. Place an equal mass of the denser liquid into vessel 1. Take care that no air is dispersed in the liquid.

Use the less dense liquid (starting liquid in vessel 2) to prime the siphon (5.4.1.6), which should be equipped with a capillary tip at the delivery end for flow control; then start the delivery of the liquid to the gradient tube, Fill the tube to the top graduation desired. (See note 2.)

Allow the density gradient column so prepared to settle for at least 24 h.

NOTES

1 Calculate the density ϱ_2, of the liquid in vessel 2 (see the figure), used to prepare a desired gradient, using the equation

$$\varrho_2 = \varrho_{\text{max}} - \frac{2(\varrho_{\text{max}} - \varrho_{\text{min}})V_2}{V}$$

where

ϱ_{min} 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 indivisual gradient tube;

ϱ_{max} is the upper limit of required density and starting density for the liquid in vessel 1, chosen to be 0,005 g/ml heavier than the density of the densest glass float calibrated for the individual gradient tube;

V is the total volume required in the gradient tube;

V_2 is the volume of the starting liquid in vessel 2.

2 Preparation of a suitable gradient column may require 1 to 1½ h or longer, depending upon the volume required in the gradient tube.

5.4.4.2.3 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 procedures.

Alternatively, place the floats in the tube immediately upon preparation of the column. If the floats appear to group together and do not spread 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 h. At the end of this time, measure the distances 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 distances. The curve so produced shall

a) be monotonic;

b) have no discontinuity;

c) have not more than one point of inflection.

The solution shall 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.

5.4.4.3 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 min or more. Films of thickness less than 0,05 mm require at least 1½ h 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) in order not to disturb the density gradient.

5.4.4.4 Calculations

The densities of the specimens may be determined graphically or by calculation from the levels to which they settle, as follows:

a) Make a plot of float density *versus* float position on a chart large enough to be read accurately to ± 0,000 1 g/ml and ± 1 mm. Plot the positions of the specimens on the chart and read their corresponding densities, or

b) Calculate the density $\varrho_{\mathrm{S},x}$ of the specimen by interpolation, using the equation

$$\varrho_{\mathrm{S},x} = \varrho_{\mathrm{F}_1} + \frac{(x - y)(\varrho_{\mathrm{F}_2} - \varrho_{\mathrm{F}_1})}{z - y}$$

where

ϱ_{F_1} and ϱ_{F_2} are the respective densities of the two standard floats bracketing the specimen;

x is the distance of the specimen above an arbitrary level;

y and z are the distances (measured from the same arbitrary level) of the two standard floats.

NOTE — This method does not reveal calibration errors, which can be detected with the graphical method, and may be applied only when the calibration is known to be linear within the range under test.

6 Correction for buoyancy of air

Because the weighings are made in air, the values of the "apparent masses" obtained shall be corrected, if necessary to compensate for the different effect of the air buoyancy on the specimen and on the weights used, if required. (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 is calculated using the equation

$$m_\mathrm{T} = m_\mathrm{APP} \left(1 + \frac{0{,}001\,2}{\varrho_{\mathrm{S},t}} - \frac{0{,}001\,2}{\varrho_\mathrm{L}} \right)$$

where

m_APP is the apparent mass;

$\varrho_{\mathrm{S},t}$ is the density of the specimen;

ϱ_L is the density of the weight used.

7 Test report

The test report shall include the following particulars:

a) reference to this International Standard;

b) complete identification of the material tested;

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

d) the individual values and the arithmetic mean of the density ($\varrho_{\mathrm{S},t}$ or $\varrho_{\mathrm{S},x}$), in kilograms per cubic metre, grams per cubic centimetre or grams per millilitre, where t is the temperature of the test, or the relative density ($d_{t_2}^{t_1}$) and the identity of the reference substance.

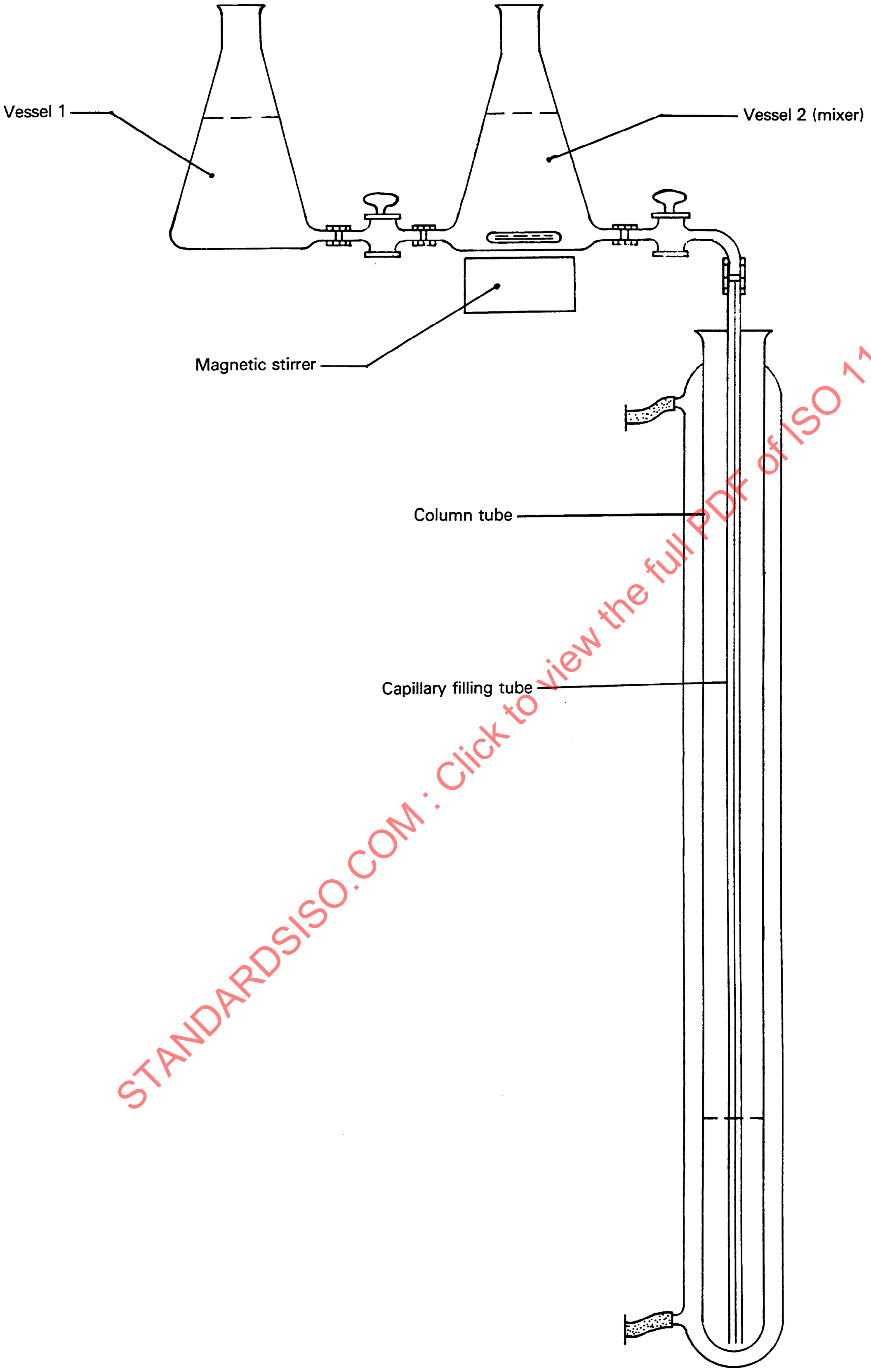

Figure — Density gradient column filling devices