INTERNATIONAL STANDARD

ISO 787-10

Second edition
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General methods of test for pigments and extenders —

Part 10:
Determination of density — Pyknometer method

Méthodes générales d'essai des pigments et matières de charge —

Partie 10: Détermination de la masse volumique — Méthode utilisant un pycnomètre

Reference number
ISO 787-10:1993(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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 787-10 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Sub-Committee SC 2, *Pigments and extenders*.

This second edition cancels and replaces the first edition (ISO 787-10:1981), which has been technically revised. The second edition includes two methods, rather than three, that use the same general principle but differ somewhat in the apparatus used. Method B is more convenient for pigments of lower density. Method C, given in the first edition, has been omitted in this second edition.

ISO 787 consists of the following parts, under the general title *General methods of test for pigments and extenders*:

— *Part 1: Comparison of colour of pigments*

— *Part 2: Determination of matter volatile at 105 °C*

— *Part 3: Determination of matter soluble in water — Hot extraction method*

— *Part 4: Determination of acidity or alkalinity of the aqueous extract*

— *Part 5: Determination of oil absorption value*

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— *Part 7: Determination of residue on sieve — Water method — Manual procedure*

— *Part 8: Determination of matter soluble in water — Cold extraction method*

— *Part 9: Determination of pH value of an aqueous suspension*

— *Part 10: Determination of density — Pyknometer method*

— *Part 11: Determination of tamped volume and apparent density after tamping*

— *Part 13: Determination of water-soluble sulphates, chlorides and nitrates*

— *Part 14: Determination of resistivity of aqueous extract*

— *Part 15: Comparison of resistance to light of coloured pigments of similar types*

— *Part 16: Determination of relative tinting strength (or equivalent colouring value) and colour on reduction of coloured pigments — Visual comparison method*

— *Part 17: Comparison of lightening power of white pigments*

— *Part 18: Determination of residue on sieve — Mechanical flushing procedure*

— *Part 19: Determination of water-soluble nitrates (Salicylic acid method)*

— *Part 20: Comparison of ease of dispersion (Oscillatory shaking method)*

— *Part 21: Comparison of heat stability of pigments using a stoving medium*

— *Part 22: Comparison of resistance to bleeding of pigments*

— *Part 23: Determination of density (using a centrifuge to remove entrained air)*

— *Part 24: Determination of relative tinting strength of coloured pigments and relative scattering power of white pigments — Photometric methods*

— *Part 25: Comparison of the colour, in full-shade systems, of white, black and coloured pigments — Colorimetric method*

— *Part 26: Determination of relative tinting strength and remaining colour difference on reduction — Colorimetric method*

Further parts are planned. Parts 6 and 12 have been withdrawn. Parts 13, 14 and 17 are printed together in the same document.

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General methods of test for pigments and extenders —

Part 10:
Determination of density — Pyknometer method

1 Scope

This part of ISO 787 specifies general methods of test for determining the density of a sample of pigment or extender, using a pyknometer.

ISO 787-23:1979, *General methods of test for pigments and extenders — Part 23: Determination of density (using a centrifuge to remove entrained air)*, specifies a general method using a centrifuge to remove trapped air.

NOTE 1 The general methods given in the various parts of ISO 787 are usually applicable to any pigment or extender. Thus only a cross-reference to the appropriate part of ISO 787 needs to be included in the International Standard giving the specification for that pigment or extender, indicating any detailed modification that may be needed in view of the special properties of the material in question. Only when the general methods are not applicable to a particular material is a different method for determination of density to be specified.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 787. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 787 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings.*

ISO 842:1984, *Raw materials for paints and varnishes — Sampling.*

3 Preliminary considerations

3.1 Displacement liquid

3.1.1 A liquid shall be selected in which the material to be tested is insoluble and which has good wetting properties and a low evaporation rate under a vacuum. A high-boiling aliphatic hydrocarbon solvent with a final boiling point over 170 °C is normally suitable.

NOTE 2 In addition to organic liquids, water with added wetting agent is also suitable.

3.1.2 Particular care is necessary in the selection of the liquid if carbon black is to be examined. The liquid selected shall have particularly good wetting properties for carbon black.

NOTE 3 Carbon tetrachloride has been found suitable.

3.2 Temperature of the determination

The temperature at which the determination is carried out significantly affects the density of the displacement liquid used, but not that of the material tested. In order that the determination may be carried out conveniently in the laboratory, the temperature of the determination shall be at least 5 °C above laboratory temperature.

4 Sampling

Take a representative sample of the material to be tested, as described in ISO 842.

5 Method A

5.1 Apparatus

Ordinary laboratory apparatus and glassware, together with the following.

5.1.1 Pyknometer, Gay-Lussac type, of capacity 25 ml or 50 ml, with a stopper and loose-fitting cap (see figure 1) or other suitable type of pyknometer.

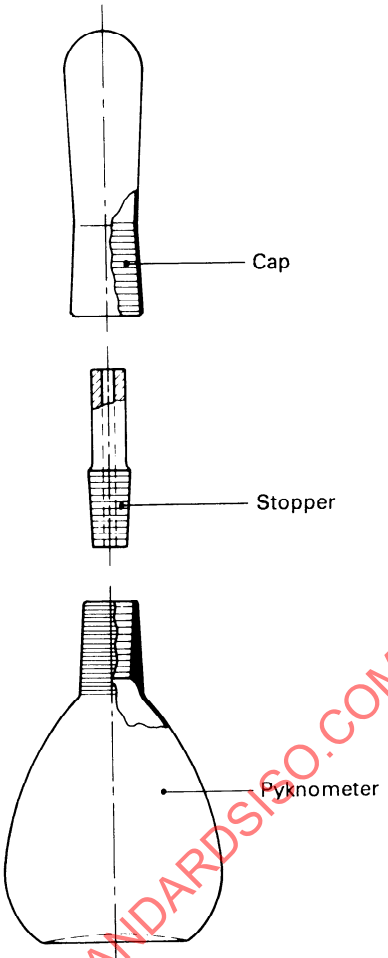

Figure 1 — Pyknometer, Gay-Lussac type

5.1.2 Vacuum apparatus, comprising the items described in 5.1.2.1 and 5.1.2.2.

NOTE 4 Other types of vacuum apparatus of suitable design may be used, in which case the procedure described in 5.2.3 may need to be modified.

1) 1 kPa = 10 mbar

5.1.2.1 Vacuum desiccator, fitted with a two-hole stopper. A glass tube with a three-way stopcock shall pass through one hole of the stopper and connect the desiccator to the vacuum pump (5.1.2.2), and the stem of a dropping funnel shall pass through the other hole of the stopper.

5.1.2.2 Vacuum pump, or other apparatus, capable of reducing the pressure to less than 2 kPa[1].

5.1.3 Water bath, thermostatically controlled, capable of being maintained to within ± 0,1 °C at a temperature within the range 25 °C to 30 °C (or at an agreed test temperature outside this range).

5.1.4 Sieve, with a nominal mesh aperture of 500 µm, complying with the requirements of ISO 565.

5.1.5 Balance, accurate to 1 mg or better.

5.2 Determination

Carry out the determination in duplicate.

5.2.1 Volume of pyknometer

5.2.1.1 Clean and dry the pyknometer (5.1.1), and its stopper and cap. Fill the pyknometer with the displacement liquid (see 3.1) and, after allowing it to attain the temperature of the water bath (5.1.3) as described in 5.2.3.3, insert the stopper, wipe the excess liquid from the outside of the stopper, attach the cap and wipe the pyknometer dry. Transfer the pyknometer and the cap to the case of the balance (5.1.5), allow to stand for 15 min and weigh to the nearest 1 mg.

NOTE 5 If the density of the displacement liquid is already known (for example from previous determinations), it is unnecessary to weigh the pyknometer filled with the displacement liquid.

5.2.1.2 Finally empty, clean and dry the pyknometer, stopper and cap, and fill with distilled water. Repeat the procedure described in 5.2.1.1.

NOTE 6 If the quantity of water that fills a pyknometer has already been determined several times, it is unnecessary to repeat the determination every time the pyknometer is used.

5.2.2 Preparation of sample

Thoroughly mix the sample and pass a sufficient quantity (see 5.2.3.1) of it through the sieve (5.1.4). Dry by heating it at (105 ± 2) °C for 2 h and allow to cool to room temperature in a desiccator.

For materials which decompose when dried under the conditions stated, a temperature and time shall be selected that avoid decomposition.

5.2.3 Procedure

5.2.3.1 Wash and dry the pyknometer, stopper and cap, and weigh to the nearest 1 mg. Introduce into the pyknometer, by means of a dry funnel, a suitable quantity of the dried sample (1 g to 10 g when a 25 ml pyknometer is used, or 2 g to 20 g when a 50 ml pyknometer is used, depending on the density) so that the pyknometer is not more than half-filled. Reweigh the stoppered pyknometer and cap.

5.2.3.2 Place the pyknometer containing the test portion in the vacuum desiccator (5.1.2.1) and position the dropping funnel so that the stem of the funnel extends into the pyknometer. Close the stopcock of the dropping funnel and the three-way stopcock connecting the desiccator to the vacuum pump (5.1.2.2), start the pump and gradually open the three-way stopcock to the pump.

Fill the dropping funnel with the displacement liquid (see 3.1) and, 15 min after the pressure in the desiccator has been reduced to less than 2 kPa, close the three-way stopcock and gradually open the stopcock of the funnel to add slowly the displacement liquid until the surface of the liquid is about 15 mm above the surface of the test portion. Close the stopcock of the funnel and re-open the three-way stopcock to the pump, taking care to avoid losses by suction. Allow the pyknometer to remain in the desiccator under reduced pressure (not greater than 2 kPa) for about 4 h or until no air bubbles are visible in the liquid. Tap the desiccator occasionally to assist in removing trapped air. Stop the pump and gradually open the three-way stopcock to admit air into the desiccator until atmospheric pressure is restored.

5.2.3.3 Remove the pyknometer from the desiccator, fill it completely with the displacement liquid and place it in the water bath (5.1.3) maintained at the selected test temperature to within ± 0,1 °C (see 3.2). Allow the pyknometer to remain in the bath for at least 30 min in order to allow the pyknometer to reach the temperature of the bath and then carefully insert the stopper so that the liquid just fills the capillary. Wipe the excess liquid from the outside of the stopper. Remove the pyknometer from the bath, attach the cap and carefully wipe the pyknometer dry. Transfer the pyknometer and cap to the case of the balance (5.1.5), allow to stand for 15 min and weigh to the nearest 1 mg.

5.2.3.4 If the difference between the duplicate results is greater than 0,03 g/ml, repeat the determination.

6 Method B

6.1 Apparatus

Ordinary laboratory apparatus and glassware, together with the following.

6.1.1 The apparatus specified in 5.1, except for the vacuum desiccator (5.1.2.1).

6.1.2 Vacuum apparatus, as shown in figure 2, with a glass tube into which the stem of a dropping funnel shall be sealed, the seal being strong enough to withstand the manipulation of the funnel and the applied vacuum. The glass tube shall have the same interior diameter as the neck of the pyknometer. The stem of the dropping funnel shall be approximately 10 mm longer than the part of the glass tube leading to the pyknometer. The pyknometer shall be connected to the glass tube by a rubber tube in such a way that the stem of the dropping funnel projects into the neck of the pyknometer and a gap of approximately 4 mm remains between the neck of the pyknometer and the wall of the glass tube, allowing the pyknometer to be shaken.

6.2 Determination

Carry out the determination in duplicate.

6.2.1 Volume of pyknometer

Determine the volume of the pyknometer as described in 5.2.1.

6.2.2 Preparation of the sample

Prepare the sample as described in 5.2.2.

6.2.3 Procedure

6.2.3.1 Carry out the operations described in 5.2.3.1.

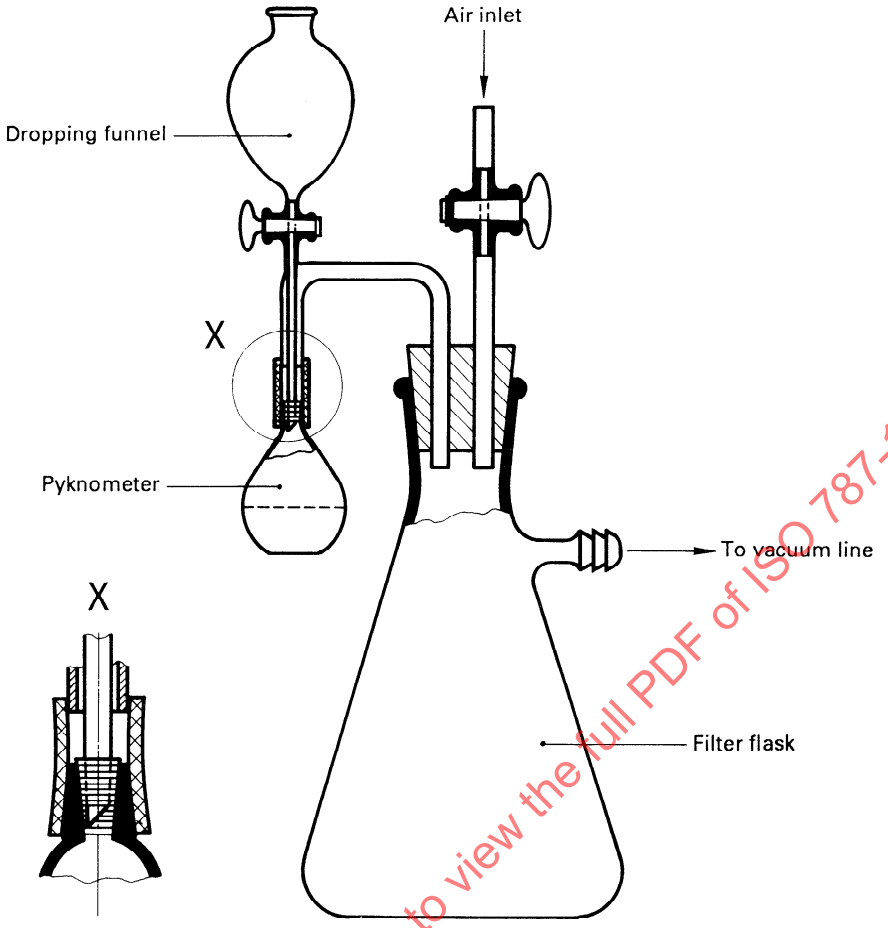

Figure 2 — Vacuum apparatus

6.2.3.2 Attach the pyknometer to the vacuum apparatus described in 6.1.2, start the vacuum pump (5.1.2.2), and slowly close the air inlet stopcock and reduce the pressure to less than 2 kPa. Maintain the pressure at this level for 15 min and then carefully open the stopcock of the dropping funnel previously filled with the displacement liquid.

Slowly add the displacement liquid until the surface of the liquid is about 15 mm above the surface of the test portion. Close the stopcock of the funnel and maintain the vacuum until no further air bubbles escape from the wetted test portion. Carefully shake the pyknometer to assist in removing trapped air.

6.2.3.3 Gradually open the air inlet stopcock to admit air into the pyknometer until atmospheric pressure is restored. Remove the pyknometer, fill it completely with the displacement liquid and place it in the water bath (5.1.3) maintained at the selected test temperature to within ± 0,1 °C (see 3.2).

Allow the pyknometer to remain in the bath for at least 30 min in order to allow the pyknometer to reach the temperature of the bath and then carefully insert the stopper so that the liquid just fills the capillary. Wipe the excess liquid from the outside of the stopper. Remove the pyknometer from the bath, attach the cap and carefully wipe the pyknometer dry. Transfer the pyknometer and cap to the case of the balance (5.1.5), allow to stand for 15 min and weigh to the nearest 1 mg.

6.2.3.4 If the difference between the duplicate results is greater than 0,03 g/ml, repeat the determination.

7 Expression of results

For both methods A and B, calculate the density ϱ_1 of the displacement liquid, expressed in grams per millilitre, at the temperature of the determination, using the following equation:

$$\varrho_1 = \frac{m_4 - m_1}{m_5 - m_1} \times \varrho_0$$

Calculate the density ϱ_m of the material tested, expressed in grams per millilitre, using the following equation:

$$\varrho_m = \frac{\varrho_1(m_2 - m_1)}{(m_4 - m_1) - (m_3 - m_2)}$$

where

ϱ_0 is the density, in grams per millilitre, of water at the temperature of the determination (see table 1);

m_1 is the mass, in grams, of the pyknometer, stopper and cap;

m_2 is the mass, in grams, of the pyknometer, stopper, cap and test portion;

m_3 is the mass, in grams, of the pyknometer, stopper, cap, test portion and displacement liquid;

m_4 is the mass, in grams, of the pyknometer, stopper, cap and displacement liquid;

m_5 is the mass, in grams, of the pyknometer, stopper, cap and distilled water.

Calculate the mean of two valid results (replicates) and report the test result to two decimal places as the density of the material at the temperature of the determination.

Table 1 — Density of water at different temperatures

Temperature of water °C	Density of water, ϱ_0 g/ml
15	0,999 1
20	0,998 2
25	0,997 0
30	0,995 6

8 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the product tested;

b) a reference to this part of ISO 787 (ISO 787-10) and to the method used (method A or B);

c) the result of the test, as indicated in clause 7;

d) details of the displacement liquid used (see 3.1) and the temperature of the determination (see 3.2);

e) any deviation from the test method specified;

f) the date of the test.