International Standard ISO 787/10

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

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

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The purpose of this International Standard is to establish a series of general test methods for pigments and extenders which are suitable for all or many of the individual pigments and extenders for which specifications might be required. In such cases, a cross-reference to the general method should be included in the International Standard relating to that pigment or extender, with a note of any detailed modifications which might be needed in view of the special properties of the product in question.

Technical Committee ISO/TC 35, *Paints and varnishes*, decided that all the general methods should be published as they become available, as parts of a single International Standard, in order to emphasize the relationship of each to the whole series.

The Technical Committee also decided that, where two or more procedures were widely used for determining the same or a similar characteristic of a pigment or extender, there would be no objection to including more than one of them in the ISO series. In such cases it will, however, be essential to state clearly in a specification which method is to be used and, in the test report, which method has been used.

Parts of the series already published are as follows :

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
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 of coloured pigments of similar types to light from a specified light source
Part 16 : Comparison of relative tinting strength (or equivalent colouring value) and colour on reduction — 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 method.

General methods of test for pigments and extenders — Part 10 : Determination of density — Pyknometer method

0 Introduction

This document is a part of ISO 787, *General methods of test for pigments and extenders.* This revision now includes three methods which use the same general principle but differ in that somewhat different apparatus is used. Method B is more convenient for less dense pigments.

1 Scope and field of application

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

NOTE — When these general methods are applicable to a given pigment or extender, only a cross-reference to them should be included in the International Standard relating to that pigment or extender, with a note of any detailed modification which may be needed in view of the special properties of the material in question. Only when these general methods are not applicable to a particular material should a special method for the determination of density be specified.

1.2 Part 23 of ISO 787 specifies a method using a centrifuge.

2 References

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

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

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

3 Preliminary considerations

3.1 Displacement liquid

A liquid should 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. High-boiling aromatic or aliphatic hydrocarbon solvents with final boiling point over 170 °C are suitable. In addition to organic liquids, water with a wetting agent may also be selected.

Particular care, however, may be necessary in the selection of the liquid if carbon black or organic dyestuffs are to be examined.

3.2 Temperature of the determination

The temperature t at which the determination is carried out will significantly affect 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 should be at least 5 °C above room temperature.

4 Sampling

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

5 Method A

5.1 Apparatus

5.1.1 Pyknometer, Gay-Lussac type, of capacity 25 or 50 ml with loose-fitting cap (see figure 1).

Alternatively, other types of pyknometer fitted with a capillary stopper may be used.

5.1.2 Vacuum apparatus, of suitable design, for example comprising the following items.

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

5.1.2.2 Vacuum pump, or other apparatus which is capable of reducing the pressure to not greater than 2 kPa.[1)]

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

1) 1 kPa = 10 mbar

5.1.4 Sieve, with 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 Procedure

Carry out the procedure in duplicate.

5.2.1 Volume of pyknometer

5.2.1.1 Clean and dry the pyknometer (5.1.1), stopper and cap. Fill the pyknometer with the displacement liquid (see 3.1) and, after allowing it to attain the temperature of the bath as described in 5.2.3.2, insert the stopper, wipe off the excess liquid, attach the cap and wipe dry the pyknometer as in the previous operation. Transfer the pyknometer and the cap to the balance case (5.1.5), allow to stand for 15 min and weigh to the nearest 1 mg.

NOTE — 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 again empty, clean and dry the pyknometer, stopper and cap and fill with distilled water. Carry out the procedure described in 5.2.1.1.

5.2.2 Test portion

Thoroughly mix the sample and pass a sufficient quantity of it through the sieve (5.1.4). Dry by heating it at 105 ± 2 °C[1] for 2 h and allow to cool to room temperature in a desiccator.

5.2.3 Determination

5.2.3.1 Wash and dry the pyknometer (5.1.1), stopper and cap and weigh to an accuracy of 1 mg or better. Introduce into the pyknometer, by means of a dry funnel, a suitable quantity (1 to 10 g when the 25 ml pyknometer is used, or 2 to 20 g when the 50 ml pyknometer is used, depending on the density) of the dried sample so that the bottle 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 arrange 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 not greater than 2 kPa, close the three-way stopcock and gradually open the stopcock of the funnel to admit sufficient displacement liquid to cover the test portion completely to a depth of about 15 mm. 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 entrained air. Stop the pump and gradually open the three-way stopcock to admit air into the desiccator until room pressure is restored.

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 t ± 0,1 °C (see 3.2).

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

If the difference between the results of two determinations is greater than 0,03 g/ml, repeat the determinations.

6 Method B

6.1 Apparatus

6.1.1 Vacuum apparatus as shown in figure 2 is required. This consists of a glass-tube into which the stem of a dropping funnel is sealed, the seal being strong enough to withstand the manipulation of the funnel and the applied vacuum. The glass tube has the same interior diameter as the neck of the pyknometer. The stem of the dropping funnel is approximately 10 mm longer than the part of the glass tube leading to the pyknometer. The pyknometer is connected by means of a rubber tube so that the stem of the dropping funnel projects some way into the neck of the pyknometer and a gap of approximately 4 mm remains between the neck of the pyknometer and the end of the glass tube, allowing the pyknometer to be shaken.

6.1.2 The apparatus specified in 5.1, except for item 5.1.2.1, is also required.

6.2 Procedure

Carry out the procedure in duplicate.

6.2.1 Volume of pyknometer

Carry out the procedure described in 5.2.1.

1) For materials which decompose when dried under the conditions stated, the temperature and time should be adjusted to avoid decomposition.

6.2.2 Test portion

Carry out the procedure described in 5.2.2.

6.2.3 Determination

6.2.3.1 Carry out the procedure described in 5.2.3.1.

6.2.3.2 Attach the pyknometer to the apparatus described in 6.1.1, start the vacuum pump (5.1.2.2) and slowly close the air inlet stopcock and reduce the pressure to not greater 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 pressure until no further air bubbles escape from the wetted test portion. Carefully shake the pyknometer to assist in removing entrained air.

Gradually open the stopcock of the air inlet to admit air into the pyknometer until room 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 $t \pm 0{,}1$ °C (see 3.2).

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

If the difference between the results of two determinations is greater than 0,03 g/ml, repeat the determinations.

7 Method C

7.1 Apparatus

7.1.1 Centrifuge, laboratory type.

7.1.2 Apparatus specified in 5.1, except for item 5.1.2.1.

7.2 Procedure

Carry out the procedure in duplicate.

7.2.1 Volume of pyknometer

Carry out the procedure described in 5.2.1.

7.2.2 Test portion

Carry out the procedure described in 5.2.2.

7.2.3 Determination

7.2.3.1 Carry out the procedure described in 5.2.3.1.

7.2.3.2 Slowly add the displacement liquid to the contents of the pyknometer until the surface of the liquid is 15 mm above the surface of the test portion. Wrap the pyknometer well in cotton wool and place it, while open, into one of the holders for the centrifuge tubes. Centrifuge until the entrained air is removed and the test portion is reduced to a closely packed mass.

NOTE — With a centrifuge operating at a relative centrifugal acceleration of 100 km/s^2 a period of 15 min should be sufficient.

Remove the pyknometer from the centrifuge, fill it completely with the displacement liquid and place it in the water bath (5.1.3) maintained at $t \pm 0{,}1$ °C (see 3.2).

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

If the difference between the results of two determinations is greater than 0,03 g/ml, repeat the determinations.

8 Expression of results

Calculate the density of the displacement liquid at the temperature t by the equation

$$\varrho_l = \frac{m_4 - m_1}{m_5 - m_1} \times \varrho_o$$

Calculate the density of the material tested by the equation :

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

where

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;

ϱ_o is the density, in grams per millilitre, of the water at the temperature t (see the table for values at different temperatures);

ϱ_l is the density, in grams per millilitre, of the displacement liquid at the temperature t;

ϱ_m is the density, in grams per millilitre, of the material tested.

Table

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

Take the mean of two determinations and report the result to two decimal places as the density of the material at the temperature of the determination.

9 Test report

The test report shall contain at least the following information :

a) the type and identification of the product tested;

b) a reference to this International Standard (ISO 787/10) and to the method used (method A, B or C);

c) the result of the test as indicated in clause 8;

d) details of the displacement liquid used and the temperature of the determination;

e) any deviation, by agreement or otherwise, from the test procedures specified;

f) the date of the test.

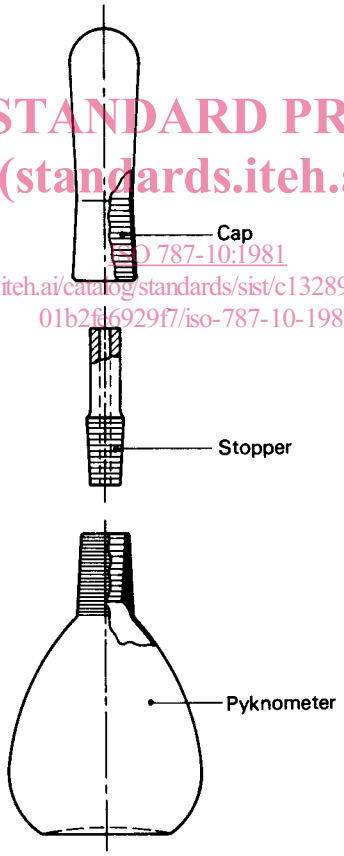

Figure 1 — Pyknometer, Gay-Lussac type

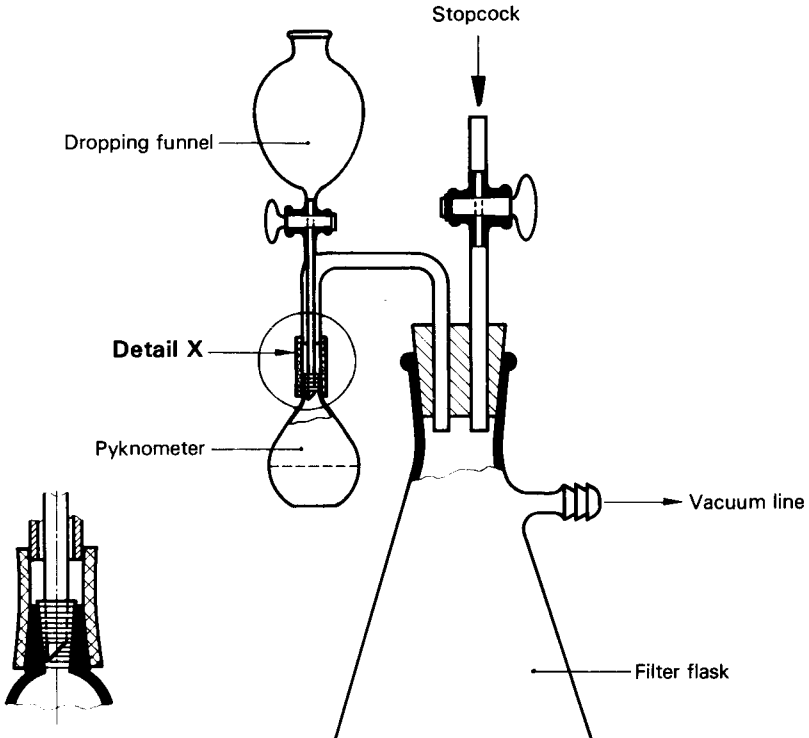

Figure 2 — Vacuum apparatus