### International Standard



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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION MEЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ ORGANISATION INTERNATIONALE DE NORMALISATION

# General methods of test for pigments and extenders — Part 23: Determination of density (using a centrifuge to remove entrained air)

Méthodes générales d'essai des pigments et matières de charge — Partie 23 : Détermination de la masse volumique (en utilisant une centrifugeuse pour chasser l'air entraîné)

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#### **Foreword**

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and nongovernmental, 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.

International Standard ISO 787/23 was developed by Technical Committee ISO/TC 35, *Paints and varnishes*, and was circulated to the member bodies in March 1978.

It has been approved by the member bodies of the following countries:

Poland Ireland Austria Romania Israel Bulgaria South Africa, Rep. of Italy Canada Kenya Sweden Egypt, Arab Rep. of Korea, Rep. of Switzerland France Turkey Germany, F. R. Netherlands United Kingdom New Zealand India Norway Yugoslavia Iran

No member body expressed disapproval of the document.

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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 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
- 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 6 : Determination of residue on sieve Oil method
- Part 7 : Determination of residue on sieve Water method
- 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 12: Visual comparison of hue of powdered white pigment (Hollow cone method)<sup>1)</sup>
- 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 in linseed stand oil using the automatic muller
- Part 17: Comparison of lightening power of white pigments
- Part 18: Determination of residue on sieve by a 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)

<sup>1)</sup> This part will be withdrawn as the specified method is no longer in use.

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## General methods of test for pigments and extenders — Part 23: Determination of density (using a centrifuge to remove entrained air)

#### 0 Introduction

This document is a part of ISO 787, General methods of test for pigments and extenders.

#### 1 Scope and field of application

**1.1** Part 23 of this International Standard specifies a general method of test for determining the density of a sample of pigment or extender, using a centrifuge to remove entrained air.

NOTE — When this general method is applicable to a given pigment or extender, only a cross-reference to it 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 this general method is not applicable to a particular material should a special method for determination of density using a centrifuge be specified.

**1.2** Part 10 of this International Standard specifies a general method of test for determining the density of a sample of pigment or extender, using a pyknometer.

#### 2 References

ISO 787, General methods for pigments and extenders — Part 10: Determination of density — Pyknometer method. 1)

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

#### 3 Preliminary considerations

#### 3.1 Displacement liquids of known density

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 vacuum. High-boiling aromatic or aliphatic hydrocarbon solvents with a boiling point over 170 °C are suitable.

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. When using this method, therefore, it is most important that each weighing should be made at a constant temperature. It is desirable that a constant temperature room or enclosure should be used but if these are not available the temperature at which each weighing is made should be noted and corrections made to the density of the displacement liquid.

#### 4 Apparatus

- **4.1** Centrifuge tube of glass or other suitable material such as polypropylene or stainless steel.
- **4.2** Cradle and loop of fine platinum or nickel-chromium wire not more than 0,12 mm diameter to suspend the tube from the balance.
- **4.3** Glass stirring rod, slightly longer than the tube (4.1).
- 4.4 Centrifuge, laboratory type.
- **4.5** Sieve, with nominal mesh aperture of 500 μm.
- 4.6 Balance, accurate to 1 mg or better.

#### 5 Sampling

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

<sup>1)</sup> At present at the stage of draft. (Revision of ISO/R 787/10-1970.)

#### 6.1 Test portion

Thoroughly mix a sufficient quantity of the test sample to occupy about half the volume of the centrifuge tube (4.1), and pass it through the sieve (4.5). Dry by heating it at 105  $\pm$  2 °C for 2 h and allow to cool to room temperature in a desiccator.

#### 6,2 Determination

Clean and thoroughly dry the tube and stirring rod (4.3). Suspend them from the balance (4.6) by means of the cradle and loop (4.2) and weigh them in air.

Completely immerse the tube and stirring rod in the displacement liquid contained in a 250 ml beaker, and allow to stand for a sufficient time in the constant-temperature room or enclosure for the whole to reach the temperature of the room or enclosure (the time necessary for this may be 1 h or longer). Suspend the tube and stirring rod from the balance and weigh.

Remove the tube and stirring rod from the displacement liquid and wipe them clean and dry. Weigh into the tube a quantity of the dried sample sufficient to occupy about half the volume (the quantity required for this is from 1 to 10 g depending on the density). Add displacement liquid to the material in the tube, gradually and with careful stirring, until the material is wetted and completely covered and has a layer of clear liquid above it. Add further displacement liquid to bring the level to about 13 mm from the top of the tube.

Place the tube, complete with stirring rod, into the centrifuge (4.4), suitably balanced, and spin it until the occluded air is removed and the solid material is reduced to a closely packed mass (for a centrifuge operating at 4 500 r/min a period of 15 min should be sufficient).

Remove the tube from the centrifuge and fill it with displacement liquid. Lower the tube and stirring rod carefully into a 250 ml beaker containing displacement liquid, and allow the whole to stand in the constant-temperature room or enclosure until the temperature of the whole is at the temperature of the room or enclosure. Suspend the tube and stirring rod by means of the cradle and loop and weigh.

#### 7 Expression of results

Calculate the density of the material tested, expressed in grams per millilitre, by the formula

$$\frac{\varrho \times m_2}{m_2 - (m_3 - m_1)}$$

#### where

 $m_1$  is the mass, in grams, of the cradle (4.2), tube (4.1) and stirring rod (4.3) in the displacement liquid;

 $m_2$  is the mass, in grams, of the material in air;

 $m_3$  is the mass, in grams, of the cradle, tube, stirring rod and material in the displacement liquid;

 $\varrho$  is the density, in grams per millilitre, of the displacement liquid at the temperature t.

#### 8 Precision

#### 8.1 Repeatability (r)

The value below which the absolute difference between two single test results on identical material, obtained by one operator in one laboratory using the same equipment within a short interval of time using the standard test method, may be expected to lie with a 95 % probability, is 0,03 g/ml.

#### 8.2 Reproducibility (R)

The value below which the absolute difference between two test results, each the mean of duplicate measurements, on identical material, obtained by operators in different laboratories, using the standardized test method, may be expected to lie with a 95 % probability, is 0,05 g/ml.

#### 9 Test report

The test report shall include at least the following information:

- a) the type and identification of the product tested;
- b) a reference to this International Standard or to a corresponding national standard;
- c) details of the displacement liquid used and the temperature of the determination;
- d) any deviation, by agreement or otherwise, from the test procedure specified;
- e) the result of the test;
- f) the date of test.