
**General methods of test for pigments and
extenders —**

Part 3:

**Determination of matter soluble in water —
Hot extraction method**

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

*Partie 3: Détermination des matières solubles dans l'eau — Méthode par
extraction à chaud*



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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

Attention is drawn to the possibility that some of the elements of this part of ISO 787 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 787-3 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 2, *Pigments and extenders*.

This second edition cancels and replaces the first edition (ISO 787-3:1979), which has been technically revised.

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*
- *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 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 residual colour difference of colorants — Weighted K/S value method

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General methods of test for pigments and extenders —

Part 3:

Determination of matter soluble in water — Hot extraction method

1 Scope

This part of ISO 787 specifies a general method of test for determining the percentage by mass of matter soluble in boiling water, in a sample of pigment or extender.

ISO 787-8 specifies a method for determining the percentage by mass of matter soluble in water by cold extraction. For most pigments and extenders, these two test methods will give different results, and it is therefore essential to state clearly in a specification which method is to be used, and in the test report which method has been used.

NOTE 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 water-soluble matter to be specified.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 787. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 787 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 787-4:1981, *General methods of test for pigments and extenders — Part 4: Determination of acidity or alkalinity of the aqueous extract.*

ISO 787-8:2000, *General methods of test for pigments and extenders — Part 8: Determination of matter soluble in water — Cold extraction method.*

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks.*

ISO 15528:2000, *Paints, varnishes and raw materials for paints and varnishes — Sampling.*

3 Reagent

3.1 Water, freshly double-distilled or de-ionized, of pH 6 to 7.

Other types of water may be used but only by agreement between the interested parties.

4 Apparatus

4.1 One-mark volumetric flask, of capacity 250 ml, complying with the requirements of ISO 1042.

4.2 Membrane filter.

Other types of filter may be used but only by agreement between the interested parties.

4.3 **Evaporating dish**, flat-bottomed, of glass, platinum, glazed porcelain or silica, dried to constant mass.

4.4 **Water bath**.

4.5 **Oven**, capable of being maintained at (105 ± 2) °C.

4.6 **Balance**, accurate to 1 mg or better.

4.7 **Desiccator**, containing an efficient desiccant.

5 Sampling

Take a representative sample of the material to be tested, as described in ISO 15528.

6 Procedure

6.1 General

Carry out the determination in duplicate.

6.2 Test portion

Weigh, to the nearest 0,01 g, 2 g to 20 g of the sample (m_0) into a beaker.

The mass of the test portion used shall be chosen according to the type of material and the amount of water-soluble matter in the material. This is particularly important for materials that contain large amounts of matter soluble in water. In any case, the same test portion mass shall be taken for repeat tests or for tests carried out in several different laboratories.

6.3 Determination

Moisten the test portion in the beaker with a few millilitres of water (3.1).

If the material does not disperse easily in water, use a wetting agent. In the case of materials which are not soluble in ethanol, an appropriate volume of ethanol may be added; in the case of pigments soluble in ethanol, use a non-ionic wetting agent such as 10 ml of a 0,01 % solution of an ethylene oxide condensate. If the wetting agent is non-volatile under the conditions of test, an appropriate correction derived from a blank test shall be made.

Add 200 ml of boiling water (3.1) and stir. Unless a different period is specified for the material under test, boil the suspension for 5 min. A coagulating agent can be used if the semi-colloidal nature of the material makes this desirable, provided that the agent selected is not such as to affect a subsequent determination of the acidity or alkalinity of the aqueous extract (see ISO 787-4), and provided that only the minimum quantity is used.

Cool rapidly to room temperature, transfer the solid and the liquid to the volumetric flask (4.1) and dilute to the mark with water (3.1). Mix thoroughly by shaking and inverting, and filter immediately through the membrane filter (4.2), returning the filtrate to the filter until the filtrate is clear.

If the time between the end of the boiling and the beginning of the filtration is specified, it shall be indicated in the test report.

Suspensions which are difficult to filter shall be centrifuged.

Pipette 100 ml of the perfectly clear filtrate or centrifugate into a previously dried and weighed evaporating dish (4.3) and then evaporate on the water bath (4.4).

Dry the residue in the evaporating dish in the oven (4.5) at (105 ± 2) °C, cool in the desiccator (4.7) and weigh to the nearest 1 mg. Repeat the heating and cooling until the results of the two last weighings, at an interval including at least 30 min heating, do not differ by more than 10 % of the final weighing. From the lower of the last two weighings, calculate and record the mass of the residue (m_1).

7 Expression of results

7.1 Calculation

Calculate the water-soluble matter (hot extraction method), $M_{ws,h}$, expressed as a percentage by mass, using the equation

$$M_{ws,h} = \frac{m_1 \times 2,5}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the residue.

If the two results do not differ by more than 10 % of the lower value, calculate and report the arithmetic mean. If the difference exceeds 10 %, carry out a third determination and report the arithmetic mean of all three results.

If the difference between the result of the third determination and the mean of the other determinations is also greater than 10 %, state this and the individual results in the test report.

Express the results to one decimal place.

7.2 Reproducibility/repeatability

Precision data are currently not available.

Following the publication of ISO/R 787:1968, work was carried out in an attempt to improve reproducibility. As a result of this work, it was found that three factors were of prime importance. The first was the type of water used, the second the type of filter used and the third the mass of the test portion. Unfortunately, insufficient testing was carried out to be able to fix reliable reproducibility limits, but it is considered inappropriate to proceed further with this work.

8 Test report

The test report shall contain at least the following information:

- a) a reference to this part of ISO 787 (ISO 787-3);
- b) all details necessary to identify the product tested;
- c) the result of the test, as indicated in clause 7;
- d) the mass of the test portion used;
- e) the type of water used;
- f) the type and volume of any wetting agent added;
- g) the type of filter used, or whether the suspension was centrifuged;
- h) any deviation from the test method specified;
- i) the date of the test.

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