
**Extenders — Specifications and
methods of test —**

**Part 3:
Blanc fixe**

*Matières de charge — Spécifications et méthodes d'essai —
Partie 3: Blanc fixe*

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 256, *Pigments, dyestuffs and extenders*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 298, *Pigments and extenders*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 3262-3:1998), which has been technically revised.

The main changes are as follows:

- the title has been changed to “Extenders”;
- an additional method for BaSO₄ content (X-ray fluorescent analysis) has been added;
- the gravimetric method for BaSO₄ content has been simplified;
- the sodium carbonate solution in the gravimetric method has been replaced by ammonium carbamate solution;
- the normative references have been updated.

A list of all parts in the ISO 3262 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Extenders — Specifications and methods of test —

Part 3: Blanc fixe

1 Scope

This document specifies requirements and corresponding methods of test for blanc fixe.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 787-2, *General methods of test for pigments and extenders — Part 2: Determination of matter volatile at 105 °C*

ISO 787-3, *General methods of test for pigments and extenders — Part 3: Determination of matter soluble in water — Hot extraction method*

ISO 787-5, *General methods of test for pigments and extenders — Part 5: Determination of oil absorption value*

ISO 787-7, *General methods of test for pigments and extenders — Part 7: Determination of residue on sieve — Water method — Manual procedure*

ISO 787-9, *General methods of test for pigments and extenders — Part 9: Determination of pH value of an aqueous suspension*

ISO 787-14, *General methods of test for pigments and extenders — Part 14: Determination of resistivity of aqueous extract*

ISO 3262-1, *Extenders — Specifications and methods of test — Part 1: Introduction and general test methods*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 12677, *Chemical analysis of refractory products by X-ray fluorescence (XRF) — Fused cast-bead method*

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

ISO 18451-1, *Pigments, dyestuffs and extenders — Terminology — Part 1: General terms*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18451-1 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

blanc fixe

synthetic (as opposed to naturally occurring) barium sulfate, produced by a precipitation process

4 Requirements and test methods

For blanc fixe complying with this document, the essential requirements are specified in [Table 1](#) and the conditional requirements are listed in [Table 2](#). The test methods listed in [Tables 1](#) and [2](#) shall apply.

Table 1 — Essential requirements

Characteristic	Unit	Requirement	Test method
BaSO ₄ content, min.	% mass fraction	95	See Clause 6 or Clause 7 ^a
Residue on 45 µm sieve, max.	% mass fraction	0,2	ISO 787-7 ^b
Matter volatile at 105 °C, max.	% mass fraction	0,5 ^c	ISO 787-2 ^d
Loss on ignition, max.	% mass fraction	3 ^b	ISO 3262-1
Matter soluble in water, max.	% mass fraction	0,5	ISO 787-3
pH value of aqueous suspension		6 to 10	ISO 787-9
^a Both methods (gravimetric or X-ray fluorescent analysis) can be used. ^b Use a brush to obtain reproducible results. ^c For ultra-fine and surface-treated types, higher values are possible. The requirements for these types shall therefore be agreed between the interested parties. ^d By agreement between the interested parties, test portions other than 10 g may be used.			

Table 2 — Conditional requirements

Characteristic	Unit	Requirement	Test method
Particle size distribution (instrumental method)	% mass fraction	To be agreed between the interested parties ^a	
Colour		To be agreed between the interested parties	ISO 3262-1
Lightness			To be agreed between the interested parties
Oil absorption value	g/100 g		ISO 787-5
Resistivity of aqueous extract	Ω m		ISO 787-14
^a A general description of a sedimentation method using X-ray absorption is given in EN 725-5.			

5 Sampling

Take a representative sample of the product to be tested, as specified in ISO 15528.

6 Determination of BaSO₄ content (gravimetric)

6.1 General

For precipitated barium sulfate, the acid-insoluble barium compounds, calculated as barium sulfate, are taken as the BaSO₄ content. Strontium sulfate, which is precipitated in a similar way and can also be present, can be subtracted using additional analysis e.g. inductively coupled plasma optical emission spectrometry (ICP-OES). Alternatively, X-ray fluorescent analysis can be used to determine the BaSO₄ content (see [Clause 7](#)) instead of the gravimetric determination.

NOTE This determination includes strontium sulfate.

6.2 Reagents

Use only reagents of recognized analytical grade and only water of at least grade 3 purity as specified in ISO 3696.

6.2.1 Hydrochloric acid, CAS Registry Number^{®1)} 7647-01-0, concentrated, approximately 37 % mass fraction, $\rho \approx 1,19$ g/ml.

6.2.2 Hydrochloric acid, CAS Registry Number[®] 7647-01-0, diluted 1 + 3.

Add 1 part by volume of concentrated hydrochloric acid (6.2.1) to 3 parts by volume of water.

6.2.3 Sulfuric acid, CAS Registry Number[®] 7664-93-9, diluted 1 + 9.

Add 1 part by volume of concentrated sulfuric acid (approximately 96 % mass fraction, $\rho \approx 1,84$ g/ml) slowly to 9 parts by volume of water.

6.2.4 Ammonium carbamate solution, CAS Registry Number[®] 1111-78-0, 10 %.

Dissolve 10 g of ammonium carbamate in 90 ml of water.

6.2.5 Fusion mixture

Mix equal parts by mass of sodium carbonate, CAS Registry Number[®] 497-91-8, and potassium carbonate, CAS Registry Number[®] 584-08-7.

6.2.6 Ammonia solution, CAS Registry Number[®] 1336-21-6, approximately 25 % mass fraction, $\rho \approx 0,903$ g/ml.

6.2.7 Ammonium sulfate solution, CAS Registry Number[®] 7783-20-2, 10 %.

Dissolve 10 g of ammonium sulfate in 90 ml of water.

6.3 Apparatus

Use ordinary laboratory apparatus and glassware, together with the following.

6.3.1 Platinum crucible, with lid, 40 ml.

6.3.2 Sintered-silica crucible

6.3.3 Air oven, capable of being maintained at (105 ± 2) °C.

6.3.4 Muffle furnace

6.3.5 Balance, with an accuracy of 0,000 1 g.

6.3.6 Desiccator, containing a suitable desiccant, for example dried silica gel.

1) Chemical Abstracts Service (CAS) Registry Number[®] is a trademark of the American Chemical Society (ACS). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

6.4 Procedure

6.4.1 Weigh 19 g of the fusion mixture (6.2.5) into the platinum crucible (6.3.1). Add approximately 1 g (m_0), weighed to the nearest 0,1 mg, of the test sample, previously dried in accordance with ISO 787-2. Mix well and place a further 1 g of the fusion mixture on top of the mixture in the crucible.

6.4.2 Heat the crucible, fitted with its lid, at bright-red heat (800 °C to 900 °C) for a minimum of 15 min. Then allow the crucible and lid to cool.

6.4.3 Measure 250 ml of hot water into a 500 ml beaker (beaker A) and place into it the crucible and lid. Heat gently for at least 15 min to extract the melt until it is disintegrated, then, using a clean nickel or other suitable tongs, remove the crucible and lid. Rinse the crucible, lid and tongs with a small amount of hot water into the beaker. Remove any white residue from the crucible with a rubber-tipped glass stirring rod and rinse again with a small amount of hot water. Let the solution in the beaker completely cool down for at least 4 h or overnight.

6.4.4 Measure 95 ml of water and 5 ml of concentrated hydrochloric acid (6.2.1) into a second 500 ml beaker (beaker B) and put the crucible and lid into it.

6.4.5 Allow the aqueous fusion extract in beaker A to settle and decant the solution through a 125 mm fine-textured ashless filter paper. Wash the precipitate three times by decantation, using 10 ml portions of ammonium carbamate solution (6.2.4) and transfer to the filter paper. Repeat the washing three times with 5 ml portions of ammonium carbamate solution and discard the washings.

6.4.6 Remove the crucible and lid from the acid solution in beaker B and wash down with water into the beaker. Transfer this solution to beaker A, in which the melt was extracted, and add to it the filter paper and residue. Cover the beaker with a watch glass, boil for 5 min and after that allow the solution to cool down.

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6.4.7 Transfer the solution into a 250 ml volumetric flask. 200 ml of this solution is transferred to a 1 000 ml Erlenmeyer flask and diluted with water to 600 ml. Use ammonia solution (6.2.6) and hydrochloric acid (6.2.1) to adjust the pH to slightly acidic conditions. Heat the solution and add 40 ml ammonium sulfate solution (6.2.7), then stir the solution boiling for 30 min. Let the solution and precipitate in the flask completely cool down for at least 4 h or overnight. Then filter through a 125 mm filter paper into a beaker, wash the filter paper thoroughly six times with 10 ml portions of hot water.

6.4.8 Transfer the filter paper to a sintered-silica crucible (6.3.2) which has been previously dried in the air oven (6.3.3) at (105 ± 2) °C, cooled in a desiccator (6.3.6) and weighed. Heat it in the muffle furnace (6.3.4) at a temperature of 800 °C to constant mass (approx. 4 h). Allow to cool in a desiccator. Add a few drops of sulfuric acid (6.2.3) to oxidize any BaS to BaSO₄ which has potentially developed. Dry and heat in the muffle furnace at a temperature of 800 °C to constant mass again (approx. 4 h). Allow to cool in a desiccator and weigh the precipitate to the nearest 0,1 mg (m_1).

6.4.9 If applicable, determine the strontium content of remaining solution in volumetric flask, for example, using inductively coupled plasma optical emission spectrometry (ICP-OES). Subtract calculated SrSO₄ content from the total result.

6.5 Expression of results

Calculate the BaSO₄ content, $w(\text{BaSO}_4)$, (which here includes potential SrSO₄), expressed as a percentage by mass of barium sulfate, using [Formula \(1\)](#):

$$w(\text{BaSO}_4) = \frac{m_1}{m_0} \times 100 \quad (1)$$

where

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

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

7 Determination of BaSO₄ content (X-ray fluorescent analysis)

7.1 General

For precipitated barium sulfate, the barium content in a borax melt (di-sodium tetra borate), calculated as barium sulfate, are taken as the BaSO₄ content. Unlike X-ray fluorescent analysis, the gravimetric method can be used to determine the BaSO₄ content (see [Clause 6](#)).

7.2 Reagents

Use only reagents of recognized analytical grade and only water of at least grade 3 purity as specified in ISO 3696.

7.2.1 Di-sodium tetra borate (borax), CAS Registry Number[®] 1330-43-4, Na₂B₄O₇.

7.3 Apparatus

Use ordinary laboratory apparatus and glassware, together with the following.

7.3.1 Wavelength dispersive X-ray fluorescence spectrometer

7.3.2 Platinum crucible

[ISO 3262-3:2023](https://standards.iteh.ai/catalog/standards/sist/85b029d1-7314-46af-ab5a-53aeac0d479b/iso-3262-3-2023)

[https://standards.iteh.ai/catalog/standards/sist/85b029d1-7314-46af-ab5a-](https://standards.iteh.ai/catalog/standards/sist/85b029d1-7314-46af-ab5a-53aeac0d479b/iso-3262-3-2023)

7.3.3 Muffle furnace

[53aeac0d479b/iso-3262-3-2023](https://standards.iteh.ai/catalog/standards/sist/85b029d1-7314-46af-ab5a-53aeac0d479b/iso-3262-3-2023)

7.3.4 Balance, with an accuracy of 0,000 1 g.

7.4 Procedure

Weigh 5 g of di-sodium tetra borate ([7.2.1](#)) into a platinum crucible ([7.3.2](#)). Add 0,5 g of the test sample, weighed to the nearest 0,1 mg. Mix the blend thoroughly and heat it in a muffle furnace ([7.3.3](#)) at a temperature of 1 150 °C for 1 h. Transfer the melt into a mould, allow it to cool down and measure the Ba content with a calibrated wavelength dispersive X-ray fluorescence spectrometer ([7.3.1](#)) in accordance with ISO 12677.

7.5 Expression of results

Calculate the BaSO₄ content, $w(\text{BaSO}_4)$, from the elemental Ba content given by the X-ray fluorescent analysis. The calculation shall take into consideration that the determination can include other compounds containing barium, such as BaCO₃, BaCl₂ and BaO.

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 document, i.e. ISO 3262-3:2023;

ISO 3262-3:2023(E)

- c) the results of the test, the method used, and whether or not the product complies with the relevant specification limits;
- d) any deviation from the method of test specified;
- e) any unusual features (anomalies) observed during the test;
- f) the date of the test.

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