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

**Part 2:
Baryte (natural barium sulfate)**

*Matières de charge — Specifications et méthodes d'essai —
Partie 2: Partie 2: Barytine (sulfate de baryum naturel)*

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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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).

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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 3262-2:1998), 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 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 2: Baryte (natural barium sulfate)

1 Scope

This document specifies requirements and corresponding methods of test for baryte (natural barium sulfate).

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 787-7, *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 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*

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

barytenaturally occurring barium sulfate, BaSO₄

4 Requirements and test methods

For baryte 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
		Grade A	Grade B	
BaSO ₄ content, min.	% mass fraction	90	80	See Clause 6 or Clause 7 ^a
Residue on 45 μm sieve, max.	% mass fraction	0,5	See Table 2	ISO 787-7
Matter volatile at 105 °C, max.	% mass fraction	0,5		ISO 787-2 ^b
Loss on ignition, max.	% mass fraction	1	See Table 2	ISO 3262-1
Matter soluble in water, max.				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 By agreement between the interested parties, test portions other than 10 g may be used.				

Table 2 — Conditional requirements

Characteristic	Unit	Requirement		Test method
		Grade A	Grade B	
Residue on 45 µm sieve	% mass fraction	See Table 1	To be agreed between the interested parties	ISO 787-7
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		To be agreed between the interested parties
Loss on ignition	% mass fraction	See Table 1	To be agreed between the interested parties	ISO 3262-1
Matter soluble in water	% mass fraction	See Table 1	To be agreed between the interested parties	ISO 787-3
Resistivity of aqueous extract	Ω m	To be agreed between the interested parties		ISO 787-14
Matter soluble in hydrochloric acid	% mass fraction			See Clause 7
^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, in accordance with ISO 15528.

6 Determination of BaSO₄ content

6.1 General

For barytes, the acid-insoluble barium, calculated as barium sulfate, is 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 Hydrochloric acid, CAS Registry Number[®] 7647-01-0, diluted 1 + 100. Add 1 part by volume of concentrated hydrochloric acid ([6.2.1](#)) to 100 parts by volume of water.

6.2.4 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.

6.2.5 Ammonia solution, CAS Registry Number[®] 1336-21-6, diluted 1 + 3.

Add 1 part by volume of concentrated ammonia solution (approximately 32 % mass fraction, $\rho \approx 0,880$ g/ml) to 3 parts by volume of water.

6.2.6 Bromine water, CAS Registry Number[®] 7726-95-6, saturated solution.

6.2.7 Sodium carbonate solution, CAS Registry Number[®] 497-91-8, 10 g/l. Dissolve 1 g of anhydrous sodium carbonate in 100 ml of water.

6.2.8 Hydroxylammonium chloride solution, CAS Registry Number[®] 5470-11-1, 50 g/l.

6.2.9 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.

6.3 Apparatus

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

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.

6.3.1 Platinum crucible, with lid.

6.3.2 Sintered-silica filter crucible, pore size index P 16 (pore diameter of 10 µm to 16 µm) as specified in ISO 4793.

6.3.3 Air oven, capable of being maintained at $(105 \pm 2) ^\circ\text{C}$.

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.

6.4 Procedure

6.4.1 Weigh, to the nearest 1 mg, approximately 1 g (m_0) of the test sample, previously dried in accordance with ISO 787-2, into a 400 ml beaker. Add 100 ml of 1 + 100 hydrochloric acid (6.2.3) and allow to stand for 1 h at room temperature, with occasional stirring. Filter through a 125 mm fine-textured ashless filter paper and wash once with 10 ml of hot water.

6.4.2 Transfer the filter paper to the platinum crucible (6.3.1), dry and ignite. Add 5 g of the fusion mixture (6.2.9), mix well, and then place a further 1 g of the fusion mixture on top of the mixture in the crucible.

6.4.3 Heat the crucible, fitted with its lid, at bright-red heat ($800 ^\circ\text{C}$ to $900 ^\circ\text{C}$) for 15 min. Then allow the crucible and lid to cool.

6.4.4 Measure 100 ml of hot water into a 600 ml beaker (A) and place into it the crucible and lid. Heat gently for 15 min to extract the melt, then, using clean nickel or other suitable tongs, remove the crucible and lid. Rinse the crucible and lid and tongs with water into the beaker. Remove any white residue from the crucible with a rubber-tipped glass stirring rod and rinse again with water.

6.4.5 Measure 95 ml of water and 5 ml of concentrated hydrochloric acid (6.2.1) into a second 600 ml beaker (B) and put the crucible and lid into it.

6.4.6 Allow the aqueous fusion extract in beaker A to settle on a warm surface for 30 min. Decant the hot solution through a 125 mm fine-textured ashless filter paper. Wash the precipitate three times by decantation, using 10 ml portions of hot sodium carbonate solution (6.2.7) and transfer to the filter paper. Repeat the washing three times with 5 ml portions of sodium carbonate solution and discard the washings.

6.4.7 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 then filter through a 125 mm filter paper into beaker B, which originally contained the acid solution. Wash the filter papers thoroughly six times with 10 ml portions of hot water.

6.4.8 Transfer the filter papers to the platinum crucible and dry, then ignite at dull-red heat ($650 ^\circ\text{C}$ to $700 ^\circ\text{C}$) and add 2 g of the fusion mixture (6.2.9) to the crucible. Heat the crucible and lid at bright-red heat for 15 min. Allow the crucible and lid to cool.

6.4.9 Measure 50 ml of hot water into beaker A and place into it the crucible and lid. Heat gently for 15 min to extract the melt. Filter through a 125 mm filter paper. Wash twice with 5 ml portions of sodium carbonate solution (6.2.7) and discard the washings.

6.4.10 Add to beaker A (which still contains the crucible and lid), 20 ml of 1 + 3 hydrochloric acid (6.2.2) and 50 ml of hot water. Filter this solution through the filter paper, and collect the filtrate in beaker B, which contained the original acid extract. Wash six times with 5 ml portions of hot water. Dilute the filtrate with water to approximately 300 ml.

6.4.11 Boil the solution in beaker B to remove carbon dioxide and then add bromine water (6.2.6) until a yellow colour appears. Add ammonia solution (6.2.5) in slight excess and boil to precipitate the hydroxides of iron, aluminium and manganese. Filter the precipitate on a fast filter paper and wash three times with hot ammonia solution (6.2.5), collecting the filtrate and washings in a 400 ml beaker.

6.4.12 Dissolve the precipitate in another beaker with approximately 8 ml of 1 + 3 hydrochloric acid (6.2.2) and 12 ml of water to which a few drops of hydroxylammonium chloride solution (6.2.8) have been added. Dilute this solution to 50 ml, add bromine water (6.2.6) until a yellow colour appears and then add ammonia solution (6.2.5) in slight excess. Filter and wash the precipitate as described above. Add the first and second filtrates to beaker B and acidify the content of beaker B with 1 + 3 hydrochloric acid (6.2.2) against methyl orange, adding approximately 4 ml of acid in excess.

6.4.13 Heat the acid extract to boiling. Stir and add slowly 20 ml of hot dilute sulfuric acid (6.2.4). Allow the covered beaker to stand on a warm surface for 3 h.

6.4.14 Filter through a sintered-silica filter crucible (6.3.2) which has been previously dried in the air oven (6.3.3) at $(105 \pm 2) ^\circ\text{C}$, cooled in a desiccator (6.3.6) and weighed. Wash four times with 10 ml portions of hot water. Dry in the air oven at $(105 \pm 2) ^\circ\text{C}$ for 15 min and heat in the muffle furnace (6.3.4) at a temperature of $500 ^\circ\text{C}$ to $600 ^\circ\text{C}$ to constant mass. Allow to cool in a desiccator and weigh the precipitate to the nearest 0,1 mg (m_1). Alternatively, the precipitate may be filtered on a filter paper, placed in a platinum crucible, and dried and ignited before cooling in the desiccator.

6.5 Expression of results

Calculate the barium compound content, $w(\text{BaSO}_4)$, expressed as a percentage by mass of barium sulfate, using the 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 BaSO_4 content (X-ray fluorescent analysis)

7.1 General

For baryte, the barium content in a borax melt (di-sodium tetra borate), calculated as barium sulfate, are taken as the BaSO_4 content. Alternatively, to X-ray fluorescent analysis, the gravimetric method can be used to determine the BaSO_4 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, $\text{Na}_2\text{B}_4\text{O}_7$.

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

7.3.3 Muffle furnace

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, weighed to the nearest 0,1 mg, of the test sample. Mix the blend thoroughly and heat the blend 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, BaO.

8 Determination of matter soluble in hydrochloric acid

8.1 Reagents

During the analysis, use only reagents of recognized analytical grade and only water of at least grade 3 purity as specified in ISO 3696.

8.1.1 Hydrochloric acid, CAS Registry Number® 7647-01-0, approximately 25 % mass fraction, $\rho \approx 1,125$ g/ml.

8.2 Apparatus

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

8.2.1 Membrane filter, pore size 0,8 µm.

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

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

8.3 Procedure

Weigh, to the nearest 0,1 mg, approximately 10 g (m_0) of the test sample into a 600 ml beaker. Add 50 ml of water and, carefully, approximately 50 ml of hydrochloric acid (8.1.1). Cover the beaker with a watch glass and boil the solution for 15 min.

Dry the membrane filter (8.2.1) in the air oven (8.2.2) at (105 ± 2) °C to constant mass, cool in a desiccator to room temperature and weigh it to the nearest 0,1 mg (m_1). Then filter the solution through it. Wash the residue on the filter eight times with hot distilled water. Dry the residue on the filter in the

air oven at $(105 \pm 2) ^\circ\text{C}$ for about 1 h. Allow to cool in a desiccator to room temperature and weigh to the nearest 0,1 mg (m_2).

8.4 Expression of results

Calculate the matter soluble in hydrochloric acid, expressed as a percentage by mass, using the [Formula \(2\)](#):

$$100 - \left(\frac{m_2 - m_1}{m_0} \cdot 100 \right) \quad (2)$$

where

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

m_1 is the mass, in grams, of the dried membrane filter;

m_2 is the mass, in grams, of the dried membrane filter plus the residue.

9 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 3262-2:2023;
- 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.