INTERNATIONAL STANDARD

ISO 3262-2

First edition
1998-07-01

Extenders for paints — Specifications and methods of test —

Part 2:
Barytes (natural barium sulfate)

Matières de charge pour peintures — Spécifications et méthodes d'essai —

Partie 2: Barytine (sulfate de baryum naturel)

Reference number
ISO 3262-2:1998(E)

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.

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.

International Standard ISO 3262-2 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 2, *Pigments and extenders.*

Together with the other parts (see below), this part of ISO 3262 cancels and replaces ISO 3262:1975, which has been technically revised. Part 1 comprises the definition of the term extender and a number of test methods that are applicable to most extenders, whilst part 2 and the following parts specify requirements and, where appropriate, particular test methods for individual extenders.

At present, the following parts of ISO 3262 are published or in preparation, under the general title *Extenders for paints — Specifications and methods of test:*

— *Part 1: Introduction and general test methods*

— *Part 2: Barytes (natural barium sulfate)*

— *Part 3: Blanc fixe*

— *Part 4: Whiting*

— *Part 5: Natural crystalline calcium carbonate*

— *Part 6: Precipitated calcium carbonate*

— *Part 7: Dolomite*

— *Part 8: Natural clay*

— *Part 9: Calcined clay*

— *Part 10: Natural talc/chlorite in lamellar form*

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet iso@iso.ch

Printed in Switzerland

— *Part 11: Natural talc, in lamellar form, containing carbonates*

— *Part 12: Muscovite-type mica*

— *Part 13: Natural quartz (ground*)

— *Part 14: Cristobalite*

— *Part 15: Vitreous silica*

— *Part 16: Aluminium hydroxides*

— *Part 17: Precipitated calcium silicate*

— *Part 18: Precipitated sodium aluminium silicate*

— *Part 19: Precipitated silica*

— *Part 20: Fumed silica*

— *Part 21: Silica sand (unground natural quartz)*

— *Part 22: Diatomaceous earth (kieselguhr)*

Extenders for paints — Specifications and methods of test —

Part 2:
Barytes (natural barium sulfate)

1 Scope

This part of ISO 3262 specifies requirements and corresponding methods of test for baryte (natural barium sulfate).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 3262. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 3262 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 787-2:1981, *General methods of test for pigments and extenders — Part 2: Determination of matter volatile at 105 °C.*

ISO 787-3:1979, *General methods of test for pigments and extenders — Part 3: Determination of matter soluble in water — Hot extraction method.*

ISO 787-7:1981, *General methods of test for pigments and extenders — Part 7: Determination of residue on sieve — Water method — Manual procedure.*

ISO 787-9:1981, *General methods of test for pigments and extenders — Part 9: Determination of pH value of an aqueous suspension.*

ISO 787-14:1973, *General methods of test for pigments — Part 14: Determination of resistivity of aqueous extract.*

ISO 842:1984, *Raw materials for paints and varnishes — Sampling.*

ISO 3262-1:1997, *Extenders for paints — Specifications and methods of test — Part 1: Introduction and general test methods.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 4793:1980, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation.*

3 Definition

For the purposes of this part of ISO 3262, the following definition applies:

3.1 barytes: Naturally occurring barium sulfate, $BaSO_4$.

4 Requirements and test methods

For barytes complying with this part of ISO 3262, the essential requirements are specified in table 1 and the conditional requirements are listed in table 2.

Table 1 — Essential requirements

Characteristic	Unit	Requirement		Test method
		Grade A	**Grade B**	
$BaSO_4$ content, min.	% (*m/m*)	90	80	See clause 6
Residue on 45 µm sieve, max.	% (*m/m*)	0,5	See table 2	ISO 787-7
Matter volatile at 105 °C, max.	% (*m/m*)	0,5		ISO 787-2 1)
Loss on ignition, max.	% (*m/m*)	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

1) By agreement between the interested parties, test portions other than 10 g may be used.

5 Sampling

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

6 Determination of $BaSO_4$ content 1)

6.1 General

For barytes, the acid-insoluble barium, calculated as barium sulfate, is taken as the $BaSO_4$ content.

6.2 Reagents

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

6.2.1 Hydrochloric acid, concentrated, approximately 37 % (*m/m*), $\rho \approx$ 1,19 g/ml.

6.2.2 Hydrochloric acid, 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, diluted 1 + 100.

Add 1 part by volume of concentrated hydrochloric acid (6.2.1) to 100 parts by volume of water.

---

1) This determination includes strontium sulfate.

Table 2 — Conditional requirements

Characteristic	Unit	Requirement		Test method
		Grade A	Grade B	
Residue on 45 µm sieve	% (m/m)	See table 1	To be agreed between the interested parties	ISO 787-7
Particle size distribution (instrumental method)	% (m/m)	To be agreed between the interested parties1)		
Colour		To be agreed between the interested parties		ISO 3262-1
Lightness				To be agreed between the interested parties2)
Loss on ignition	% (m/m)	See table 1	To be agreed between the interested parties	ISO 3262-1
Matter soluble in water				ISO 787-3
Resistivity of aqueous extract	Ω·m	To be agreed between the interested parties		ISO 787-14
Matter soluble in hydrochloric acid	% (m/m)			See clause 7

1) A general description of a sedimentation method using X-ray absorption is given in EN 725-5:1996, *Advanced technical ceramics — Methods of test for ceramic powders — Part 5: Determination of the particle size distribution.*

2) Test method in preparation.

6.2.4 Sulfuric acid, diluted 1 + 9.

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

6.2.5 Ammonia solution, diluted 1 + 3.

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

6.2.6 Bromine water, saturated solution.

6.2.7 Sodium carbonate solution, 10 g/l.

Dissolve 1 g of anhydrous sodium carbonate in 100 ml of water.

6.2.8 Hydroxylammonium chloride solution, 50 g/l.

6.2.9 Fusion mixture.

Mix equal parts by mass of sodium carbonate and potassium carbonate.

6.3 Apparatus

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

6.3.1 Platinum crucible, with lid.

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

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

6.3.4 Muffle furnace.

6.4 Procedure

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.

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.

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

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.

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.

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.

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.

Transfer the filter papers to the platinum crucible and dry, then ignite at dull-red heat (650 °C to 700 °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.

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.

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.

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.

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 whole with 1 + 3 hydrochloric acid (6.2.2) against methyl orange, adding approximately 4 ml of acid in excess.

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.

Filter through a sintered-silica filter crucible (6.3.2) which has been previously dried in the air oven (6.3.3) at (105 ± 2) °C, cooled in a desciccator and weighed. Wash four times with 10 ml portions of hot water. Dry in the air oven at (105 ± 2) °C for 15 min and heat in the muffle furnace (6.3.4) at a temperature of 500 °C to 600 °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, dried and ignited before cooling in the desiccator.

6.5 Expression of results

Calculate the barium compound content $w(\mathrm{BaSO_4})$, expressed as a percentage by mass of barium sulfate, using the equation

$$w(\mathrm{BaSO_4}) = \frac{m_1}{m_0} \times 100$$

where

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

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

7 Determination of matter soluble in hydrochloric acid

7.1 Reagents

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

7.1.1 Hydrochloric acid, approximately 25 % (*m/m*), $\rho \approx 1{,}125$ g/ml.

7.2 Apparatus

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

7.2.1 Membrane filter, pore size 0,8 µm.

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