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Extenders — Specifications and methods of test —

Part 15:
Vitreous silica

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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 documents 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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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*.

This second edition cancels and replaces the first edition (ISO 3262-15:2000), which has been technically revised.

The main changes are as follows:

— the first part of the title has been changed to "Extenders";

— the test method for particle size distribution in Table 2 has been changed to ISO 8130-13;

— the normative references have been updated and the text has been editorially revised.

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 15: Vitreous silica

1 Scope

This document specifies requirements and corresponding methods of test for vitreous silica.

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 787-9, *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 787-18, *General methods of test for pigments and extenders — Part 18: Determination of residue on sieve — Mechanical flushing procedure*

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 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
vitreous silica
non-crystalline modification of silica

Note 1 to entry: For use as an extender it is supplied ground to a powder. There are two different grades of vitreous silica in use, the grade depending on the raw materials used in its manufacture:

Grade A (quartz glass): A transparent form of vitreous silica that is usually made from molten rock crystal.

Grade B (fused silica): An opaque form of vitreous silica that is made from molten silica sand. The opacity of the product is due to very small gas bubbles incorporated in it.

4 Requirements and test methods

For vitreous silica complying with this document, the essential requirements are specified in Table 1 and the conditional requirements are listed in Table 2. The test method in Tables 1 and 2 shall be in accordance with the standards listed.

Table 1 — Essential requirements

Characteristic	Unit	Requirement		Test method
		Grade A	Grade B	
Silica content, min.	% mass fraction	99,5	99	See clause 6
Quartz content, max.	% mass fraction	0,5	1,0	X-ray diffraction
Cristobalite content, max.				
Matter volatile at 105 °C, max.	% mass fraction	0,2		ISO 787-2[a]
Loss on ignition, max.	% mass fraction	0,1 0,5[b]	0,2 0,5[b]	ISO 3262-1
Matter soluble in water, max.	% mass fraction	0,1		ISO 787-3
pH value of aqueous suspension	—	5 to 7 5 to 9[b]		ISO 787-9

[a] Another method may be agreed between the interested parties it gives the same results.

[b] These values take into account the effect on the result if the product has undergone surface treatment.

Table 2 — Conditional requirements

Characteristic	Unit	Requirement	Test method
Residue on 45 µm sieve	% mass fraction	To be agreed between the interested parties	ISO 787-7
Particle size distribution (instrumental method)	% mass fraction	To be agreed between the interested parties	ISO 8130-13
Colour	—	To be agreed between the interested parties	ISO 3262-1
Lightness	—		To be agreed between the interested parties
Resistivity of aqueous extract	Ω · m		ISO 787-14
Oil absorption value	g/100 g		ISO 787-5

5 Sampling

Take a representative sample of the product to be tested, in accordance with ISO 15528.

6 Determination of silica content

6.1 Reagents

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

6.1.1 Sulfuric acid, CAS[1)] No. 7664-93-9, diluted 1 + 1.

Add 1 part by volume of concentrated sulfuric acid [approximately 96 % mass fraction, $\rho \approx 1{,}84$ g/ml] slowly to 1 part by volume of water.

6.1.2 Hydrofluoric acid, CAS No. 7664-39-3, [approximately 40 % mass fraction, $\rho \approx 1{,}13$ g/ml].

6.2 Apparatus

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

6.2.1 Platinum dish.

6.2.2 Muffle furnace.

6.2.3 Balance, with an accuracy of 0,0001 g.

6.2.4 Desiccator, containing a suitable desiccant.

6.3 Procedure

Weigh, to the nearest 1 mg, approximately 2 g of the test sample, previously dried at 105 °C in accordance with ISO 787-2, into the tared platinum dish (6.2.1), ignite in the muffle furnace (6.2.2) at 1 000 °C ± 25 °C to constant mass (m_1) and allow to cool in a desiccator (6.2.4) containing phosphorus pentoxide.

Add approximately 1 ml of the sulphuric acid (6.1.1). Heat the platinum dish gently until fuming ceases and then continue the heating at 900 °C for 15 min in the muffle furnace. Remove from the furnace, allow to cool in the desiccator and weigh to the nearest 0,1 mg (m_2).

Add to the residue in the platinum dish 15 ml of the hydrofluoric acid (6.1.2) and 1 ml of the sulphuric acid (6.1.1) and evaporate to a syrup, taking care to avoid loss by spitting. Cool the dish and wash the sides down with small quantities of water. Then add a further 10 ml of hydrofluoric acid and evaporate to dryness, Heat the residue on a hot-plate until white fumes are no longer evolved, then ignite in the muffle furnace at 900 °C for 15 min. Remove the dish from the furnace, cool in the desiccator and weigh to the nearest 0,1 mg (m_3).

6.4 Expression of results

Calculate the silica content $w(SiO_2)$, expressed as a percentage by mass of barium sulfate, using the Formula (1)

$$w(SiO_2) = \frac{m_2 - m_3}{m_1} \times 100 \qquad (1)$$

1) Chemistry Abstracts Service Registry Number.