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Extenders — Specifications and methods of test —

Part 20:
Fumed silica

Matières de charge — Spécifications et méthodes d'essai —

Partie 20: Silice pyrogénée

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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, dyestuff 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 3262-20:2000), which has been technically revised.

The main changes compared to the previous edition are as follows:

— the main title has been changed from "Extenders for paints" to "Extenders";

— in Table 1, carbon content has been revised and organic surface has been refined;

— in 6.3.4, magnesium perchlorate has been changed to an example for a desiccant;

— in 8.2.3, suitable examples for carbon steel have been added;

— the text has been editorially revised and 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 20:
Fumed silica

1 Scope

This document specifies requirements and corresponding methods of test for fumed 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 648, *Laboratory glassware — Single-volume pipettes*

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

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

ISO 787-11, *General methods of test for pigments and extenders — Part 11: Determination of tamped volume and apparent density after tamping*

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

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

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 3819, *Laboratory glassware — Beakers*

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 terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
fumed silica
amorphous silica produced from silicon halides by high-temperature flame hydrolysis

4 Requirements and test methods

For fumed silica complying with this document, the essential requirements are specified in Table 1 and the conditional requirements are listed in Table 2.

In order to determine the pH value in an aqueous suspension for Grade B, a 1 + 1 mass fraction mixture of water and methanol is used.

Table 1 — Essential requirements and test methods

Characteristic	Unit	Requirement		Test method according to
		Grade A	Grade B	
Silica content, min.	% mass fraction	99,8		Clause 6
Oxide content — Al_2O_3 — TiO_2 — Fe_2O_3		max. 0,05 max. 0,03 max. 0,003		Clause 7
Carbon content[a]		<0,3	≥0,3[b]	Clause 8
Chloride content		max. 0,025		Clause 9
Organic surface treatment or surface coating	—	No	Yes	Clause 8
Residue on 45 µm sieve	% mass fraction	max. 0,05	Not applicable	ISO 787-18
Matter volatile at 105 °C	% mass fraction	max. 3	max. 1	ISO 787-2
Loss on ignition	% mass fraction	max. 2,5	max. 10	ISO 3262-1
pH value of aqueous suspension	—	3,6 to 4,5	3,4 to 8	ISO 787-9

[a] The carbon content is also part of the loss on ignition.

[b] Usually does not exceed 15 %.

Table 2 — Conditional requirements and test methods

Characteristic	Unit	Requirement		Test method according to
		Grade A	Grade B	
Apparent density after tamping	g/ml	To be agreed between the interested parties		ISO 787-11
Specific surface area	m^2/g			To be agreed between the interested parties

5 Sampling

Take a representative sample of the product to be tested according to ISO 15528.

6 Determination of silica content

6.1 Principle

A test portion is ignited, weighed and treated with sulfuric acid and hydrofluoric acid. The silicon tetrafluoride thus formed is evaporated off and the silica content is calculated from the impurities is not required.

6.2 Reagents

WARNING — Hydrofluoric acid is corrosive and toxic. The related operations shall be performed in fume hood. This document does not point out all possible safety problems. It is the responsibility of the user to take proper safety and health measures and to determine the applicability of regulatory limitations prior to use.

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

6.2.1 Sulfuric acid (H_2SO_4), CAS-No 7664-93-9[1)], 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.

6.2.2 Hydrofluoric acid (HF), CAS 7664-39-3, concentrated, approximately 40 % mass fraction $\rho \approx 1{,}13$ g/ml.

6.3 Apparatus

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

6.3.1 Platinum dish.

6.3.2 Muffle furnace, capable of being maintained at (1 000 ± 20) °C.

6.3.3 Infrared evaporator.

6.3.4 Desiccator, containing for example magnesium perchlorate as desiccant.

6.4 Procedure

6.4.1 Number of determinations

Carry out the determination in duplicate.

6.4.2 Test portion

Weigh, to the nearest 0,2 mg, approximately 1 g (m_0) of the sample (see Clause 5) into the tared platinum dish (6.3.1).

6.4.3 Determination

Ignite the test portion in the muffle furnace (6.3.2) at (1 000 ± 20) °C to constant mass (this should take approximately 2 h) and allow to cool in the desiccator (6.3.4). Weigh the test portion again (m_1).

Wet the ignited test portion in the platinum dish with 2 ml to 3 ml of water, add 1 ml of sulfuric acid (6.2.1) and 15 ml of hydrofluoric acid (6.2.2) and evaporate to a syrup on the infrared evaporator (6.3.3), taking care to avoid loss by spitting. Allow to cool and wash the sides down with small quantities of water. Then add a further 10 ml of hydrofluoric acid and evaporate to dryness. If the evaporation of the silicon tetrafluoride is not complete, add a further 10 ml of hydrofluoric acid and evaporate to dryness again.

Heat the residue until white fumes are no longer evolved, then ignite for 30 min in the muffle furnace at (1 000 ± 20) °C. Remove from the furnace, allow to cool in the desiccator and weigh to the nearest 0,2 mg (m_2).

1) CAS-No – Chemical Abstracts Service Registry Number.

6.5 Expression of results

Calculate the silica content $w(SiO_2)$, expressed as a mass fraction in percent, using Formula (1):

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

where

m_1 is the mass, in grams, of the test portion after ignition;

m_2 is the mass, in grams, after treatment with hydrofluoric acid and ignition.

Calculate the mean of the two determinations and report the result to the nearest 0,1 %.

6.6 Precision

No precision data are available at the time of publication.

7 Determination of aluminium oxide, titanium (IV) oxide and iron (III) oxide contents by spectrometry

7.1 Principle

A test portion is treated with sulfuric acid and hydrofluoric acid in a platinum dish. The resulting silicon tetrafluoride is volatized and the residue is dissolved in hydrochloric acid. After diluting with water to a constant, known volume, the Al, Ti and Fe impurities are determined either by flame atomic absorption spectrometry (FAAS) or by inductively coupled plasma atomic emission spectrometry (ICP-AES), depending on which instrument is available in the laboratory.

The advantages of the ICP-AES method include its wide dynamic range and multi-element capabilities. Both methods (FAAS and ICP-AES) are relative analytical techniques. For quantitative analytical results, both measurement techniques shall be calibrated using standard matching solutions.

7.2 Reagents and materials

Use only reagents of recognized analytical grade except for acids, which shall be ultrapure, and use only water of at least grade 3 purity according to ISO 3696.

7.2.1 Sulfuric acid (H_2SO_4), CAS-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.

7.2.2 Hydrofluoric acid (HF), CAS-No 7664-39-3, concentrated, approximately 40 % mass fraction, $\rho \approx 1{,}13$ g/ml.

7.2.3 Hydrochloric acid (HCl), CAS-No 7647-01-0, concentrated, approximately 30 % mass fraction, $\rho \approx 1{,}15$ g/ml.

7.2.4 Hydrochloric acid (HCl), CAS-No 7647-01-0, diluted, approximately 3 % mass fraction, $\rho \approx 1{,}01$ g/ml.

7.2.5 Caesium chloride buffer solution

Dissolve 50 g of caesium chloride in approximately 500 ml of water and add 50 ml of concentrated hydrochloric acid (7.2.3). Make up to 1 000 ml with water and mix well.

7.2.6 Standard stock solutions, containing 1,000 g/l of aluminium, titanium and iron, respectively.

Store each solution in a fluorinated-polyethylene/polypropylene (FEP) bottle.

7.2.7 Standard solutions, containing 10 mg of the element per litre.

Prepare these solutions on the day of use.

Pipette 1 ml of the appropriate standard stock solution (see 7.2.6) into a 100 ml one-mark volumetric flask, add 10 ml of concentrated hydrochloric acid (7.2.3), make up to the mark with water and mix well.

1 ml of the standard solution contains 10 µg of the element concerned.

Prepare a more dilute or more concentrated standard solution, if necessary, depending on the concentration of Al, Ti or Fe in the product under test.

7.2.8 Ethanol, 96 % volume fraction.

7.2.9 Acetylene (C_2H_2), commercial grade, in a steel cylinder.

7.2.10 Compressed air.

7.2.11 Dinitrogen oxide (N_2O), commercial grade, in a steel cylinder.

7.3 Apparatus

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

7.3.1 Flame atomic absorption spectrometer (FAAS), suitable for measurements at the following wavelengths:

— 309,3 nm for aluminium (Al),

— 364,3 nm for titanium (Ti),

— 248,3 nm for iron (Fe),

fitted with a suitable burner fed with

— an N_2O/C_2H_2 mixture for Ai and Ti determinations,

— a C_2H_2/air mixture for Fe determinations,

and also fitted with hollow-cathode lamps for the elements Al, Ti and Fe and a deuterium background corrector.

7.3.2 Inductively coupled plasma atomic emission spectrometer (ICP-AES), preferably with high resolution (≤ 0,01 nm), automatic control of all plasma operating functions and a computer-controlled signal compensation system.

7.3.3 Platinum dish.

7.3.4 100 ml one-mark volumetric flasks, conforming with the requirements of ISO 1042.