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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, 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				Clause 7
— Al_2O_3		max. 0,05		
— TiO_2		max. 0,03		
— Fe_2O_3		max. 0,003		
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-No 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 takes 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).

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[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):

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

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 Al 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.

7.3.5 Pipettes, calibrated, of suitable volumes (see 7.4), conforming with the requirements of ISO 648.

7.4 Procedure

7.4.1 Preparation of standard matching solutions

Introduce, with pipettes (7.3.5), suitable volumes of the standard solutions (7.2.7) into a series of several 100 ml one-mark, volumetric flasks (7.3.4), add 10 ml of concentrated hydrochloric acid (7.2.3), make up to the mark with water and mix well. For the aluminium and titanium determination by FAAS, add 5 ml of caesium chloride solution (7.2.5) before making up to 100 ml with water.

7.4.2 Spectrometric measurement

7.4.2.1 Spectrometric measurement using FAAS

Install the required burner. Adjust the flow of C_2H_2 (7.2.9) and N_2O (7.2.11) or compressed air (7.2.10) (see Table 3) to suit the characteristics of the burner, and light the burner. Install the spectral source in the spectrometer (7.3.1) and optimize the conditions for the determination of the relevant element (Al, Ti or Fe). Adjust the instrument in accordance with the manufacturer's instructions in order to obtain the maximum absorbance. The test parameters are listed in Table 3.

Table 3 — FAAS test parameters

Element	Spectral resonance line nm	Flame	Measurement range µg of oxide/ml
Al	309,3	C_2H_2/N_2O	2 to 80
Ti	364,3	C_2H_2/N_2O	3 to 60
Fe	248,3	C_2H_2/air	0,2 to 6

Set the scale expansions, if fitted, so that the standard matching solution with the highest concentration gives an absorbance of about 0,55.

Aspirate into the flame each of the standard matching solutions in ascending order of concentration and repeat with the solution of lowest concentration to verify that the instrument is stable.

Aspirate water into the flame after each measurement.

7.4.2.2 Spectrometric measurement using ICP-AES

Introduce the standard matching solutions into the plasma using an air atomizer, having set the instrument to the manufacturer's recommended operating conditions and using spectral resonance lines as given in Table 4.

Table 4 — ICP-AES parameters

Element	Spectral resonance line nm	Measurement range µg of oxide/ml
Al	309,27	0,2 to 20
Ti	334,94	
Fe	238,20	0,1 to 10

7.4.3 Calibration graph

Correct each absorbance by subtracting the reading obtained from the standard matching solution and plot for each element a graph having the masses, in micrograms, of the element contained in 1 ml of standard matching solution as abscissae and the corresponding values of the absorbance as ordinates.

7.4.4 Preparation of the test solution

Carry out the determinations in duplicate.

Weigh, to the nearest 1 mg, approximately 5 g (m_0) of the sample (see Clause 5) into the platinum dish (7.3.3). Wet this test portion with 5 ml of water for grade A fumed silica and with 3 ml to 4 ml of ethanol (7.2.8) and 2 ml to 3 ml of water for grade B fumed silica.

Add 2 ml of sulfuric acid (7.2.1) and 30 ml of hydrofluoric acid (7.2.2) and evaporate carefully to a syrup. 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 again.

Heat the residue until white fumes are no longer evolved, then add 5 ml of hydrochloric acid (7.2.3) and 2 ml to 3 ml of water and dissolve the residue.

Transfer the solution obtained to a 50 ml one-mark volumetric flask. For aluminium and titanium determinations by FAAS, add 2,5 ml of caesium chloride buffer solution (7.2.5). Then make up to 50 ml with water.

7.4.5 Determination of aluminium, titanium and iron

First, adjust the spectrometer as described in 7.4.2.1 or 7.4.2.2 and measure the absorbance of the standard matching solution (see 7.4.1) in the spectrometer (7.3.1 or 7.3.2). Then measure the absorbance of the test solution (see 7.4.4) three times and, afterwards, that of the standard matching solution again. Finally, redetermine the absorbance of the standard matching solution with the highest concentration in order to verify that the response of the apparatus has not changed.

If the absorbance of the test solution is higher than the absorbance of the standard matching solution with the highest concentration, dilute the test solution with a known volume of dilute hydrochloric acid (7.2.4). Note the dilution factor D.

7.4.6 Determination of the total loss on ignition

Carry out the determination in duplicate.

Weigh, to the nearest 0,2 mg, approximately 1 g (m_3) of the sample (see Clause 5) into a platinum crucible (6.3.1).

Weighing out the test portions for the determination of the silica content (see 6.4.2) and the total loss on ignition may be carried out at the same time.

Ignite the test portion to constant mass in a muffle furnace (6.3.2) (this should take approximately 2 h) at (1 000 ± 20) °C and allow to cool in a desiccator (6.3.4). Weigh the ignited test portion to the nearest 0,2 mg (m_4).

Calculate the total loss on ignition w_{TLI}, expressed as a mass fraction in percent, using Formula (2):

$$w_{\mathrm{TLI}} = \frac{m_3 - m_4}{m_3} \times 100 \tag{2}$$

where

m_3 is the mass, in grams, of the test portion before ignition;

m_4 is the mass, in grams, of the ignited test portion.

Calculate the mean of the two determinations and report the result to the nearest 0,001 %.

7.5 Expression of results

7.5.1 Calculation of the concentration of each element

Calculate the concentration c_t, expressed in micrograms per millilitre, of each element (Al, Ti, Fe) in the test solution (see 7.4.4) using Formula (3):

$$c_t = \frac{c_c \times A_t}{A_c} \tag{3}$$

where

c_c is the concentration, in micrograms per millilitre, of the element in the standard matching solution;

A_t is the absorbance (or extinction) value for the test solution;

A_c is the absorbance (or extinction) value for the standard matching solution.

7.5.2 Calculation of the oxide content for each element

Calculate the content of each oxide (Al_2O_3, TiO_2, Fe_2O_3), w_{ox}, expressed as a mass fraction in percent, using Formula (4):

$$w_{\mathrm{ox}} = \frac{c_t \times V_0 \times D \times f}{m_0 \times \left[1 - \frac{w_{\mathrm{TLI}}}{100}\right]} \times 100 \tag{4}$$

where

c_t is the concentration, in micrograms per millilitre, of the element in the test solution;

V_0 is the volume, in millilitres, of the test solution (50 ml);

D is the dilution factor (if applicable, see 7.4.5);

f is a conversion factor, for calculation of the oxide content rather than the content of the element, as given in Table 5;

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

w_{TLI} is the total loss on ignition as determined in 7.4.6.

Calculate the mean of the two determinations and report the result to the nearest 0,001 % (for Al_2O_3 and TiO_2) or 0,000 1 % (for Fe_2O_3).

Table 5 — Values of conversion factor *f*

Element	Oxide	Conversion factor
Al	Al_2O_3	1,889 5
Ti	TiO_2	1,668 1
Fe	Fe_2O_3	1,429 8

7.6 Precision

No precision data are available at the time of publication.

8 Determination of carbon content

8.1 Principle

A test portion in a crucible is covered, if necessary, with a suitable catalyst, and combusted in a stream of oxygen in an induction furnace.

Sulfur compounds, halogens and water vapour are removed from the combustion products, which are then passed over a platinum catalyst (to convert carbon monoxide to carbon dioxide), and the carbon dioxide concentration is measured using an infrared-cell detector.

Alternatively, the carbon may be determined by conductivity measurement. In this case, the specified combustion products are passed over a platinum catalyst and the carbon dioxide present is absorbed in a sodium hydroxide (NaOH) solution. The change in conductivity of the solution (caused by the conversion of some of the NaOH to disodium carbonate, Na_2CO_3) is measured.

8.2 Reagents and materials

Use only reagents of recognized analytical grade and only water of at least grade 3 purity according to ISO 3696.

8.2.1 Oxygen, CAS-No 7782-44-7, purity min. 99,99 %.

8.2.2 Catalyst, comprising iron chips plus a mixture consisting of 9 parts by mass of tungsten and 1 part by mass of tin powder[2].

NOTE The catalyst is used as required to obtain satisfactory results.

8.2.3 Carbon reference materials (carbon steels)[3].

8.2.4 Platinum catalyst pellets, suitable for use at 400 °C to 450 °C, to convert carbon monoxide to carbon dioxide.

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[2] Lecocel II® is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute and endorsement by ISO of this product.

[3] NIST SRM 131h (containing 0,000 78 % C); BAM CRM-No D 231-2 (containing 0,014 % C); NIST SRM 139b (containing 0,403 % C); BAM CRM 476-3 (containing 3,39 % C) are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

8.3 Apparatus

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

8.3.1 Low-carbon analyser, consisting of an induction-heated furnace suitable for operation at about 1 800 °C, a scavenging unit, a platinum catalyst system operating at about 450 °C, and an infrared-cell detection system. Alternatively, a carbon dioxide absorption unit including an NaOH solution and equipment for measuring the change in conductivity can be used.

The system shall include a carbon dioxide absorbent based on NaOH, a moisture absorbent (magnesium perchlorate) for purification purposes and a flowmeter for control of the oxygen stream.

8.3.2 Crucibles, expendable, made of alumina or similar refractory material. Both crucible and lid shall be ignited before use at a temperature of 1 000 °C or higher for a time (usually 20 min) sufficient to give a constant mass.

8.4 Procedure

8.4.1 Preparation of apparatus

Follow the operating instructions for the specific equipment used. After setting the controls, carry out several blank runs with a crucible (8.3.2) containing the required amount of catalyst but not the test portion. Successive blank values shall approach a low, constant value.

8.4.2 Calibration

Weigh, to the nearest 0,1 mg, approximately 0,5 g of reference material (8.2.3) into a crucible, combust and record the result if using equipment which gives the result automatically as described in 8.4.3, or use Formula (5) if using equipment which does not. Repeat at least twice. Adjust the calibration controls to produce the correct readings on the direct-reading meter. Combust additional samples of the reference material as required to produce the correct direct reading.

8.4.3 Determination

Carry out the determination in duplicate.

Weigh, to the nearest 0,1 mg, approximately 0,5 g of the sample (see Clause 5) (m_0) into a crucible, cover with 0,7 g of iron chips and 1,0 g of tungsten/tin powder (8.2.2) and place the crucible in the induction furnace of the low-carbon analyser (8.3.1). It is especially important to cover the test portion completely with the catalyst when the test portion is a powder as this prevents possible blow-out of test portion during ignition.

If the analyser has an integral balance, the test portion mass is automatically stored in the memory. In this case, press the "analyse" key and the analysis proceeds automatically. The result is displayed on the screen and is printed as percent by mass of carbon in the test portion.

8.5 Expression of results

If the equipment used does not print the result automatically, calculate the carbon content $w(\mathrm{C})$, expressed as a mass fraction in percent, using Formula (5):

$$w(\mathrm{C}) = \frac{m_\mathrm{t} - m_\mathrm{b}}{m_0 \times \left[1 - \frac{w_\mathrm{TLI}}{100}\right]} \times 100 \qquad (5)$$