INTERNATIONAL STANDARD

ISO 3262-20

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Extenders for paints — Specifications and methods of test —

Part 20: Fumed silica

Matières de charge pour peintures — 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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

Attention is drawn to the possibility that some of the elements of this part of ISO 3262 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 3262-20 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.

ISO 3262 consists of the following parts, 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*

— *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: Flux-calcined kieselguhr*

Extenders for paints — Specifications and methods of test —

Part 20: Fumed silica

1 Scope

This part of ISO 3262 specifies requirements and corresponding methods of test for fumed silica.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 3262. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 3262 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*.

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

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

ISO 787-11:1981, *General methods of test for pigments and extenders — Part 11: Determination of tamped volume and apparent density after tamping.*

ISO 787-18:1983, *General methods of test for pigments and extenders — Part 18: Determination of residue on sieve — Mechanical flushing procedure.*

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks*.

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 3819:1985, *Laboratory glassware — Beakers.*

ISO 15528:—[1)], *Paints, varnishes and raw materials for paints and varnishes — Sampling.*

1) To be published. (Revision of ISO 842:1984 and ISO 1512:1991)

3 Term and definition

For the purposes of this part of ISO 3262, the following term and definition apply:

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 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	
Silica content, min.	% (*m*/*m*)	99,8		See clause 6
Oxide content — Al_2O_3, max.		0,05		See clause 7
— TiO_2, max.		0,03		
— Fe_2O_3, max.		0,003		
Carbon content[a]		max. 0,2	min. 0,3	See clause 8
Chloride content, max.		0,025		See clause 9
Organic surface coating?	—	No	Yes	See clause 8
Residue on 45 µm sieve, max.	% (*m*/*m*)	0,05	Not applicable	ISO 787-18
Matter volatile at 105 °C, max.	% (*m*/*m*)	3	1	ISO 787-2
Loss on ignition, max.	% (*m*/*m*)	2,5	10	ISO 3262-1
pH value of aqueous suspension	—	3,6 to 4,5	3,4 to 8[b]	ISO 787-9

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

[b] Use a 1:1 (*m*/*m*) mixture of water and methanol.

Table 2 — Conditional requirements

Characteristic	Unit	Requirement		Test method
		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, as described in 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 resulting loss of mass.

As the silica content is very high, a previous separation from the impurities is not required.

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 Sulfuric acid, diluted 1 + 1.

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

6.2.2 Hydrofluoric acid, concentrated, approximately 40 % (*m*/*m*), $\rho \approx 1{,}13$ 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 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).

6.5 Expression of results

Calculate the silica content $w(SiO_2)$, expressed as a percentage by mass, using the equation

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

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 currently available.

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 volatilized 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.

NOTE 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 must 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 as defined in ISO 3696.

7.2.1 Sulfuric acid, diluted 1 + 1.

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

7.2.2 Hydrofluoric acid, concentrated, approximately 40 % (*m*/*m*), $\rho \approx$ 1,13 g/ml.

7.2.3 Hydrochloric acid, concentrated, approximately 30 % (*m*/*m*), $\rho \approx$ 1,15 g/ml.

7.2.4 Hydrochloric acid, dilute, approximately 3 % (*m*/*m*), $\rho \approx 1{,}01$ 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 % (*V*/*V*).

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 ($\leqslant$ 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, complying with the requirements of ISO 1042.