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Polnila - Specifikacije in preskusne metode - 19. del: Oborjeni silicijev dioksid (ISO/DIS 3262-19:2020)

Extenders - Specifications and methods of test - Part 19: Precipitated silica (ISO/DIS 3262-19:2020)

Füllstoffe - Anforderungen und Prüfverfahren - Teil 19: Gefällte Kieselsäure (ISO/DIS 3262-19:2020)

Matières de charge - Spécifications et méthodes d'essai - Partie 19: Silice précipitée (ISO/DIS 3262-19:2020)

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Part 19: Precipitated silica

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

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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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*.

This second edition cancels and replaces the first edition (ISO 3262-19:2000), 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 6.3.8 magnesium perchlorate has been changed to an example for a desiccant;

— in 7.2.3 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 19: Precipitated silica

1 Scope

This document specifies requirements and corresponding methods of test for precipitated 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 degrees C*

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

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

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

ISO 5794-1:1994, *Rubber compounding ingredients — Silica, precipitated, hydrated — Part 1: Non-rubber tests*

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:

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

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

3.1
precipitated silica
amorphous silica precipitated by reaction of sodium silicate solution with a mineral acid and/or carbon dioxide

4 Requirements and test methods

For precipitated silica complying with this document, 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.	% (mass fraction)	95	95	See Clause 6
Carbon content[a]		< 0,3	≥ 0,3[b]	See Clause 7
Organic surface treatment and surface coating	—	No	Yes	See Clause 7
Matter volatile at 105 °C	% (mass fraction)	max. 8		ISO 787-2
Loss on ignition	% (mass fraction)	3 to 8	3 to 15	ISO 3262-1
Oil absorption value[c]	g/100g	120		ISO 787-5
pH value of aqueous suspension[d]	—	3,5 to 9		ISO 787-9

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

[b] Usually does not exceed 15 %.

[c] A test method with higher reproducibility and repeatability is described in ASTM D2414. However, the results cannot be compared directly with oil absorption values determined in accordance with ISO 787-5.

[d] For hydrophobic silica, use a 1:1 (mass fraction) mixture by mass of water and methanol.

Table 2 — Conditional requirements

Characteristic	Unit	Requirement		Test method
		Grade A	Grade B	
Residue on 45 μm sieve, max.	% (mass fraction)	To be agreed between the interested parties	Not applicable	Spray method (Clause 8)[a]
Particle size distribution (instrumental method)	% (mass fraction)	To be agreed between the interested parties		
Apparent density after tamping	g/ml	To be agreed between the interested parties		ISO 787-11
Specific surface area	m^2/g			ISO 5794-1:1994, Annex D

[a] Only for hydrophilic materials.

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 repeatedly treated with hydrochloric acid and evaporated to dryness. To render the dehydrated silicic acid thus formed as insoluble as possible, it is then heated for 2 h at (140 ± 5) °C. Any chlorides present are removed by extracting the precipitate with hot dilute hydrochloric acid.

The precipitate is ignited at 1 000 °C, giving impure silicon dioxide, which is treated with sulphuric and hydrofluoric acid. The silicon tetrafluoride formed is evaporated off and the silica content is calculated from the resulting loss in mass.

6.2 Reagents

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

6.2.1 Hydrochloric acid, concentrated, approximately 32 % (mass fraction), $\rho \approx 1{,}16$ g/ml.

6.2.2 Hydrochloric acid, diluted 1 + 1.

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

6.2.3 Sulfuric acid, diluted 1 + 1.

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

6.2.4 Hydrofluoric acid, 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 Dish.

6.3.2 Platinum crucible.

6.3.3 Water bath, capable of being maintained at 100 °C.

6.3.4 Infrared evaporator.

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

6.3.6 Drying oven, capable of being maintained at (140 ± 5) °C.

6.3.7 Filter paper.

The filter paper used for filtration of the silica shall be of such texture as to retain the smallest particles of precipitate and nevertheless permit rapid filtration[1)].

6.3.8 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 a dish (6.3.1).

1) For example Whatman No. 40 or 41 or Schleicher und Schüll No. 589/2 "Weißband". This information is given for the convenience of users of this International Standard and does not constitute and endorsement by ISO of these products.

6.4.3 Determination

Add slowly 20 ml of concentrated hydrochloric acid (6.2.1) and evaporate to dryness under the infrared evaporator (6.3.4). Add again 20 ml of concentrated hydrochloric acid and evaporate to dryness. Repeat this step once more. After the third evaporation, place the dish in the drying oven (6.3.6), maintained at (140 ± 5) °C, and leave for 2 h.

Remove the dish from the oven and allow to cool. Add 50 ml of 1 + 1 hydrochloric acid (6.2.2) to the residue in the dish and warm it for approximately 20 min on the water bath (6.3.3) at 100 °C. Filter through a suitable filter paper (6.3.7) and wash the residue on the filter with hot water until the washings are neutral.

Pour the filtrate and washings into the original dish and evaporate to dryness. Repeat this evaporation step another two times, adding each time 10 ml of concentrated hydrochloric acid to the residue. After the third evaporation, heat at (140 ± 5) °C for 2 h in the drying oven.

Add 20 ml of 1 + 1 hydrochloric acid to the residue in the dish and warm it for approximately 10 min on the water bath at 100 °C. Filter through a fresh filter paper and wash the residue on the filter with hot water until the washings are neutral.

If it is felt necessary, check the filtrate for any silicon which may have passed through the filter.

Place the two filter papers with the washed precipitates in the platinum crucible (6.3.2). Dry, char at low temperature, ignite in the muffle furnace (6.3.5) at (1 000 ± 20) °C to constant mass (this should take approximately 1 h) and allow to cool in the desiccator (6.3.8). Weigh the ignited precipitate to the nearest 0,2 mg (m_1).

Wet the ignited precipitate in the platinum crucible with 2 ml to 3 ml of water, add 1 ml of 1 + 1 sulfuric acid (6.2.3) and 15 ml of hydrofluoric acid (6.2.4) and evaporate to a syrup, 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).

6.4.4 Determination of the total loss on ignition

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

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

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

Calculate the total loss on ignition w (TLI), expressed as a percentage mass fraction , using Formula (1):

$$w(\text{TLI}) = \frac{m_3 - m_4}{m_3} \times 100 \qquad (1)$$

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 0,1 %.

