# INTERNATIONAL STANDARD

# ISO 3262-19

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# Extenders for paints — Specifications and methods of test —

## Part 19:
## Precipitated silica

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

*Partie 19: Silice précipitée*

Reference number
ISO 3262-19:2000(E)

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# Contents

Page

# 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 3262-19 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 19:
## Precipitated silica

## 1 Scope

This part of ISO 3262 specifies requirements and corresponding methods of test for precipitated 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 787-2:1981, *General methods of test for pigments and extenders — Part 2: Determination of matter volatile at 105 °C.*

ISO 787-5:1980, *General methods of test for pigments and extenders — Part 5: Determination of oil absorption value.*

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

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

## 3 Term and definition

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

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

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

## 4 Requirements and test methods

For precipitated 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. | % ($m/m$) | 95 | 95 | See clause 6 |
| Carbon content[a] | | max. 0,2 | min. 0,3 | See clause 7 |
| Organic surface coating? | — | No | Yes | See clause 7 |
| Matter volatile at 105 °C, max. | % ($m/m$) | 8 | | ISO 787-2 |
| Loss on ignition | % ($m/m$) | 3 to 8 | 3 to 15 | ISO 3262-1 |
| Oil absorption value[b], min. | g/100 g | 120 | | ISO 787-5 |
| pH value of aqueous suspension[c] | — | 3,5 to 9 | | ISO 787-9 |

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

[b] A test method with higher reproducibility and repeatability is described in ASTM D 2414-97, *Standard test method for carbon black — n-Dibutyl phthalate absorption number.* However, the results cannot be compared directly with oil absorption values determined in accordance with ISO 787-5.

[c] For hydrophobic silicas, use a 1:1 ($m/m$) 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. | % ($m/m$) | To be agreed between the interested parties | Not applicable | Spray method (see clause 8)[a] |
| Particle size distribution (instrumental method) | % ($m/m$) | 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 hydrophylic materials.

## 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 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 sulfuric 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 as defined in ISO 3696.

**6.2.1 Hydrochloric acid,** concentrated, approximately 32 % ($m/m$), $\rho \approx 1{,}16$ 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 sulfuric acid, approximately 96 % ($m/m$), $\rho \approx 1{,}84$ g/ml, slowly to 1 part by volume of water.

**6.2.4 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 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.[2)]

**6.3.8 Desiccator,** containing magnesium perchlorate as desiccant.

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[2)] For example Whatman No. 40 or 41 or Schleicher und Schüll No. 589/2 "Weißband".

## 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).

### 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 may 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 $w(\text{TLI})$, expressed as a percentage by mass, using the equation

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

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 %.

## 6.5 Expression of results

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

$$w(\mathrm{SiO_2}) = \frac{(m_1 - m_2)}{m_0 \times \left[1 - \frac{w(\mathrm{TLI})}{100}\right]} \times 100$$

where

$m_0$ is the mass, in grams, of the test portion (see 6.4.2);

$m_1$ is the mass, in grams, of the dehydrated impure silica after ignition at (1 000 ± 20) °C to constant mass (see 6.4.3);

$m_2$ is the mass, in grams, of the silica after treatment with hydrofluoric acid and ignition to constant mass (see 6.4.3);

$w(\mathrm{TLI})$ is the total loss on ignition determined in 6.4.4.

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

## 6.6 Precision

### 6.6.1 Repeatability $r$

The repeatability $r$ is the value below which the absolute difference between two single test results, each the mean of duplicates, can be expected to lie when this method is used under repeatability conditions. In this case, the test results are obtained on identical material by one operator in one laboratory within a short interval of time. For this part of ISO 3262, $r$ is 0,6 %, with a 95 % probability.

### 6.6.2 Reproducibility $R$

No reproducibility data are currently available.

# 7 Determination of carbon content

## 7.1 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 can 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.

## 7.2 Reagents and materials

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

**7.2.1 Oxygen,** purity 99,99 % min.

**7.2.2 Catalyst,** comprising iron chips plus a mixture consisting of 9 parts by mass of tungsten and 1 part by mass of tin powder.3)

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

**7.2.3 Carbon reference materials (carbon steels).**4)

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

## 7.3 Apparatus

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

**7.3.1 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.

**7.3.2 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.

## 7.4 Procedure

### 7.4.1 Preparation of apparatus

Follow the operating instructions for the specific equipment used. After setting the controls, carry out several blank runs with a crucible (7.3.2) containing the required amount of catalyst but not the test portion. Successive blank values shall approach a low, constant value.

### 7.4.2 Calibration

Weigh, to the nearest 0,1 mg, approximately 0,5 g of reference material (7.2.3) into a crucible, combust and record the result if using equipment which gives the result automatically as described in 7.4.3, or use the equation given in 7.5 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.

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3) For example Lecocel II®.

4) Suitable examples are:

NIST SRM 131b (containing 0,001 8 % C); BAM 238-1(containing 0,018 % C); NIST SRM 151 (containing 0,55 % C); BAM EURO-ZRM 476-2 (containing 3,43 % C).