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Standard

ISO 3262-17

Extenders — Specifications and methods of test —

Part 17:
Precipitated calcium silicate

Matières de charge — Spécifications et méthodes d'essai —
Partie 17: Silicate de calcium précipité

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

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Contents

Foreword

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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 document 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).

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This document was prepared by Technical Committee ISO/TC 256, *Pigments, dyestuffs 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 3262-17:2000), which has been technically revised.

The main changes are as follows:

- the first part of the title has been changed to "Extenders";
- ISO 19246 has been added as an alternative test method for oil absorption value in Table 1;
- ISO 13320 has been added as test method for particle size distribution in Table 2;
- DIN 53163 has been added as test method for lightness (tristimulus value *Y*) in Table 2;
- CAS-numbers have been added to all reagents used for the test methods specified;
- 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 17:
Precipitated calcium silicate

1 Scope

This document specifies requirements and corresponding methods of test for precipitated calcium silicate.

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 787-5, *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 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 5794-1:2022, *Rubber compounding ingredients — Silica, precipitated, hydrated — Part 1: Non-rubber tests*

ISO 13320, *Particle size analysis — Laser diffraction methods*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

ISO 18451-1, *Pigments, dyestuffs and extenders — Terminology — Part 1: General terms*

ISO 19246, *Rubber compounding ingredients — Silica — Oil absorption of precipitated silica*

DIN 53163, *Pigments and extenders — Determination of lightness of extenders and white pigments in powder form*

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 terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

3.1

precipitated calcium silicate

amorphous silicate precipitated by reaction of sodium silicate solution with a mineral acid and/or a calcium salt

4 Requirements and test methods

For precipitated calcium silicate complying with this document, the essential requirements are specified in Table 1 and the conditional requirements are listed in Table 2. The test methods in Tables 1 and 2 shall apply.

Table 1 — Essential requirements

Characteristic	Unit	Requirements	Test method
Silica content, min.	% mass fraction	75	See Clause 6 or Clause 7
Oxide content			
— Na_2O		1 to 5	See Clause 7
— CaO		2 to 24	
Residue on 45 µm sieve, max.	% mass fraction	2	See Clause 8
Matter volatile at 105 °C, max.	% mass fraction	10	ISO 787-2
Loss on ignition	% mass fraction	3 to 10	ISO 3262-1
Oil absorption value, min.	g/100 g	80	ISO 787-5 or ISO 19246
pH value of aqueous suspension	—	8 to 12	ISO 787-9

Table 2 — Conditional requirements

Characteristic	Unit	Requirements	Test method
Particle size distribution (laser diffraction method)	µm	To be agreed between the interested parties	ISO 13320
Lightness (tristimulus value Y)	—		DIN 53163
Specific surface area	m^2/g		ISO 5794-1:2022, Annex E

5 Sampling

Take a representative sample of the product to be tested, as specified 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 which has been 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 specified in ISO 3696.

6.2.1 Hydrochloric acid, CAS Registry Number®[1] 7647-01-0, concentrated, approximately 32 % mass fraction, $\rho \approx 1{,}16$ g/ml.

6.2.2 Hydrochloric acid, CAS-No 7647-01-0, 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, 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.

6.2.4 Hydrofluoric acid, 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 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. Examples of filter paper include Whatman No 40 or No 41, or Schleicher und Schüll No 589/2 "Weißband".[2]

6.3.8 Desiccator, containing magnesium perchlorate as desiccant.

6.3.9 Balance, with an accuracy of 0,000 1 g.

6.4 Procedure

6.4.1 Number of determinations

Carry out the determination in duplicate.

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[1] Chemical Abstracts Service (CAS) Registry Number® is a trademark of the American Chemical Society (ACS). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

[2] Whatman No 40 or No 41, or Schleicher und Schüll No 589/2 "Weißband" 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 these products.

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 has possibly passed through the filter.

Place the two filter papers with the washed precipitates in the platinum crucible (6.3.2). Dry, reduce to ashes at low temperature, ignite in the muffle furnace (6.3.5) at (1 000 ± 20) °C to constant mass (this is expected to 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.

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 is expected to 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 by mass, 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 %.

6.5 Expression of results

Calculate the silica content $w(\text{SiO}_2)$, expressed as a percentage by mass, using Formula (2):

$$w(\text{SiO}_2) = \frac{(m_1 - m_2)}{m_0 \left(1 - \frac{w_{\text{TLI}}}{100}\right)} \times 100 \qquad (2)$$

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_{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 document, *r* is 0,6 %, with a 95 % probability.

6.6.2 Reproducibility, *R*

No reproducibility data are currently available.

7 Determination of the composition by spectrometry

7.1 Principle

This spectrometric method gives the calcium oxide and sodium oxide contents as well as the calcium silicate content although the precision of the determination of the calcium silicate content may be lower than that of the method specified in Clause 6.

The elements calcium, sodium and silicon are analysed using either flame atomic absorption spectrometry (FAAS) or inductively coupled plasma atomic emission spectrometry (ICP-AES), depending on which type of instrument is available in the laboratory.

The test solution is prepared by fusing a test portion of precipitated calcium silicate with lithium metaborate and dissolving the melt in a mixture of nitric acid and tartaric acid.

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 and only water of at least grade 3 purity as specified in ISO 3696.

7.2.1 Lithium metaborate: CAS-No 13453-69-5.

7.2.2 Acid mixture

Add 50 ml of nitric acid (CAS-No 7697-37-2) approximately 65 % mass fraction, $\rho \approx$ 1,40 g/ml and then 17 g of tartaric acid (CAS-No 133-37-9) to 500 ml of water. After dissolution, make up to 1 000 ml with water and mix well.

7.2.3 Caesium chloride buffer solution.

Dissolve 50 g of caesium chloride (CAS-No 7647-17-8) in approximately 500 ml of water and add 50 ml of 30 % mass fraction hydrochloric acid (CAS-No 7647-01-0), $\rho \approx$ 1,15 g/ml. Make up to 1 000 ml with water and mix well.

7.2.4 Standard stock solutions, containing 1,000 g/l of calcium, sodium and silicon, respectively.

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

7.2.5 Matrix solution, free from calcium, sodium and silicon.

Dissolve 2 g of lithium metaborate (7.2.1) in 60 ml of the acid mixture (7.2.2) in a 200 ml polypropylene one-mark volumetric flask (7.3.7), make up to 200 ml with water and mix well.

7.2.6 Acetylene (C_2H_2), CAS-No 74-86-2, commercial grade, in a steel cylinder.

7.2.7 Compressed air

7.2.8 Dinitrogen oxide (N_2O), CAS-No 10024-97-2, 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:

- 422,7 nm for calcium (Ca),
- 589,0 nm for sodium (Na),
- 251,6 nm for silicon (Si),

fitted with a suitable burner fed with:

- a N_2O/C_2H_2 mixture for Ca and Si determinations,
- a C_2H_2/air mixture for Na determinations,

and also fitted with hollow-cathode lamps for the elements Ca, Na and Si and a deuterium background corrector.

7.3.2 Inductively coupled plasma 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 crucible

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

7.3.5 Magnetic stirrer, with a stirrer bar coated with polytetrafluoroethylene (PTFE).

7.3.6 PTFE beaker with cover, volume approximately 250 ml and a diameter approximately 6 cm.

7.3.7 200 ml one-mark volumetric flasks, made of polypropylene.

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

7.3.9 Balance, with an accuracy of 0,000 1 g.

7.4 Procedure

7.4.1 Preparation of standard matching solutions

Introduce, with pipettes (7.3.8), suitable volumes of the standard stock solution (7.2.4) into a series of several 50 ml one-mark volumetric flasks, add the required volume of matrix solution (7.2.5), make up to the mark with water and mix well. Prepare the blank matching solution by dilution of the required volume of matrix solution with water in a 50 ml one-mark volumetric flask.

The standard matching solutions for FAAS and ICP-AES analysis shall have the same matrix solution concentration as the test solution (see 7.4.4).

Each diluted standard matching solution shall be stored in FEP bottles and shall not be used for more than a few days.

In cases of samples with high concentrations of sodium or calcium which require a larger dilution, add e.g. caesium chloride buffer solution (7.2.3) in accordance with the instrument manufacturer's instruction manual when using the FAAS method.

7.4.2 Spectrometric measurement

7.4.2.1 Spectrometric measurement using FAAS

Install the required burner. Adjust the flow of C_2H_2 (7.2.6) and N_2O (7.2.8) or compressed air (7.2.7) (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 (Ca, Na or Si). 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
Ca	422,7	C_2H_2/N_2O	0,1 to 2
Na	589,0	C_2H_2/air	0,05 to 0,8
Si	251,6	C_2H_2/N_2O	10 to 200

Set the scale expansion, 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
Ca	317,93	1 to 100
Na	589,59	
Si	212,41	

7.4.3 Calibration graph

Correct each absorbance by subtracting the reading obtained from the blank 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 determination in duplicate.

Weigh, to the nearest 0,1 mg, approximately 200 mg (m_0) of the sample (see Clause 5) into a platinum crucible (7.3.3). Add 2 g of lithium metaborate (7.2.1) and fuse the mixture by heating for approximately 30 min at (1 000 ± 20) °C in the muffle furnace (7.3.4) until a clear melt is obtained. Allow the crucible to cool to room temperature and immerse it in 60 ml of the acid mixture (7.2.2) in a PTFE beaker (7.3.6). Place the stirrer bar of the magnetic stirrer (7.3.5) in the beaker and stir until complete dissolution is obtained (probably overnight). Remove the platinum crucible and the stirrer bar, rinse with water, transfer the solution to a 200 ml polypropylene volumetric flask (7.3.7), make up to the mark with water and measure using the FAAS method (7.4.2.1) or the ICP-AES method (7.4.2.2).

7.4.5 Determination

First, adjust the spectrometer as specified in 7.4.2.1 or 7.4.2.2 and measure the absorbance of the blank matching solution (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 blank 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 the required matrix solution (see 7.4.1). Note the dilution factor, D.