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Extenders for paints — Specifications and methods of test —

Part 17:
Precipitated calcium silicate

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

Partie 17: Silicate de calcium précipité

Reference number
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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 734 10 79
E-mail copyright@iso.ch
Web www.iso.ch

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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 3262-17 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 17:
Precipitated calcium silicate

1 Scope

This part of ISO 3262 specifies requirements and corresponding methods of test for precipitated calcium silicate.

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 787-5:1980, *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 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*.

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[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
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 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
Silica content, min.	% (*m*/*m*)	75	See clause 6 or 7
Oxide content — Na_2O		1 to 5	See clause 7
— CaO		2 to 24	
Residue on 45 µm sieve, max.	% (*m*/*m*)	2	See clause 8
Matter volatile at 105 °C, max.	% (*m*/*m*)	10	ISO 787-2
Loss on ignition	% (*m*/*m*)	3 to 10	ISO 3262-1
Oil absorption value[a], min.	g/100 g	80	ISO 787-5
pH value of aqueous suspension	—	8 to 12	ISO 787-9

[a] 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.

Table 2 — Conditional requirements

Characteristic	Unit	Requirement	Test method
Particle size distribution (instrumental method)	% (*m*/*m*)	To be agreed between the interested parties	To be agreed between the interested parties
Lightness	%		To be agreed between the interested parties[a]
Specific surface area	m^2/g		ISO 5794-1:1994, annex D

[a] Test method in preparation.

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.

[2)] For example Whatman No. 40 or No. 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 $w(\text{SiO}_2)$, expressed as a percentage by mass, using the equation

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

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);

$w(\text{TLI})$ 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 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 described 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.

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

7.2.1 Lithium metaborate.

7.2.2 Acid mixture.

Add 50 ml of nitric acid [approximately 65 % (m/m), $\rho \approx$ 1,40 g/ml] and then 17 g of tartaric acid 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 in approximately 500 ml of water and add 50 ml of 30 % (m/m) hydrochloric acid ($\rho \approx$ 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 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), commercial grade, in a steel cylinder.

7.2.7 Compressed air.

7.2.8 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:

- 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

- an 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 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 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 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.

7.4 Procedure

7.4.1 Preparation of standard matching solutions

Introduce, with pipettes (7.3.8), suitable volumes of the standard stock solutions (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.

NOTE The standard matching solutions for FAAS and ICP-AES analysis must 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 determinations 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).