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Extenders — Specifications and methods of test —

Part 18:
Precipitated sodium aluminium silicate

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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, dyestuffs and extenders*.

This second edition cancels and replaces the first edition (ISO 3262-18:2000), which has been technically revised.

The main changes are as follows:

- the first part of the title has been changed to "Extenders";
- the normative references have been updated and the text has been editorially revised.

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 18:
Precipitated sodium aluminium silicate

1 Scope

This document specifies requirements and corresponding methods of test for precipitated sodium aluminium 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 5794-1:1994, *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 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 sodium aluminium silicate
amorphous silica precipitated by reaction of sodium silicate solution with a mineral acid and/or an aluminium salt

4 Requirements and test methods

For precipitated sodium aluminium silicate complying with this document, the essential requirements are specified in Table 1 and the conditional requirements are listed in Table 2. The test method in Tables 1 and 2 shall be in accordance with the standards listed.

Table 1 — Essential requirements

Characteristic	Unit	Requirement	Test method
Silica content, min.	% mass fraction	75	See Clause 6 or 7
Oxide content			
— Na_2O		3 to 10	See Clause 7
— Al_2O_3		5 to 15	
Residue on 45 µm sieve, max.	% mass fraction	2	See Clause 8
Lightness, min.	%	95	Test method in preparation
Matter volatile at 105 °C, max.	% mass fraction	10	
Loss on ignition	% mass fraction	3 to 12	
Oil absorption value[a], min.	g/100 g	70	
pH value of aqueous suspension	—	7 to 12	

[a] A test method with higher reproducibility and repeatability is described in ASTM D 2412-97, *standard test method for carbon black — n-Dibutyl phthalate absorption number.* However, the results cannot be compared directly with oil absorption values determines in accordance with ISO 787-5.

Table 2 — Conditional requirements

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

5 Sampling

Take a representative sample of the product to be tested, in accordance with 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 a least grade 3 purity as specified in ISO 3696.

6.2.1 Hydrochloric acid, CAS1) No. 7664-93-9, concentrated, [approximately 32 % mass fraction, $\rho \approx 1,16$ g/ml].

6.2.2 Hydrochloric acid, CAS No. 7664-93-9, diluted 1 + 1.

Add 1 part by volume of concentrated hydrochloric acid (6.2.1) slowly 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 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, 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.2)

6.3.8 Desiccator, containing magnesium perchlorate as desiccant.

6.3.9 Balance, with an accuracy of 0,0001 g.

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).

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1) Chemistry Abstracts Service Registry Number.

2) For example Whatman No. 40 or 41 or Schleicher und Schüll No. 589/2 "Weißband". Trade names or trademarks of product 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 this 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 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 disiccator. 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 Formula (1):

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

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 the Formula (2):

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

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);
$w(\text{TLI})$	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 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 aluminium oxide and sodium oxide contents as well as the aluminium silicate content although the precision of the determination of the aluminium silicate content may be lower than that of the method described in Clause 6.

The elements aluminium, 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 sodium aluminium 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, 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. 526-83-0, 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 aluminium, sodium and silicon, respectively.

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

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

Dissolve 2 g of lithium metaborate, CAS No. 13453-69-5, 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:

- 309,3 nm for aluminium (Al),
- 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 Al and Si determinations,
- a C_2H_2/air mixture for Na determinations,

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

7.3.2 Inductively coupled plasma atomic 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.