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Extenders — Specifications and methods of test —

Part 22: Flux-calcined kieselguhr

Matières de charge — Spécifications et méthodes d'essai —

Partie 22: Kieselguhr flux-calciné

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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 3262-22:2001), 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.

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 22: Flux-calcined kieselguhr

1 Scope

This document specifies requirements and corresponding methods of test for flux-calcined kieselguhr.

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 °C*

ISO 787-3, *General methods of test for pigments and extenders — Part 3: Determination of matter soluble in water — Hot extraction method*

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

ISO 787-7, *General methods of test for pigments and extenders — Part 7: Determination of residue on sieve — Water method — Manual procedure*

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, *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
flux-calcined kieselguhr
siliceous material of diatomaceous origin

Note 1 to entry: Flux-calcined kieselguhr is produced by adding soda ash or salt to the siliceous material and heating to 1 148 °C. The loss on ignition is then up to 3 % Na_2O and K_2O. Flux calcining converts the iron oxide to a colourless glassy phase and produces a white rather than pink diatomaceous earth.

4 Requirements and test methods

For flux-calcined kieselguhr 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	Requirement		Test method
		Grade A	Grade B	
Silica content, SiO_2, min.	% mass fraction	85		See Clause 6
Loss on ignition, max.	% mass fraction	0,5		ISO 3262-1
pH-value of aqueous suspension	—	7,0 to 10,5		ISO 787-9
Matter soluble in water, max.	% mass fraction	1,0		ISO 787-3
Matter volatile at 105 °C, max.	% mass fraction	0,5		ISO 787-2
Residue on sieve, max.	% mass fraction			ISO 787-7
63 µm		1	0	
45 µm		10	0,1	
Specific surface area	m^2/g	3 max.	3 min.	ISO 5794-1
Oil absorption value	g/100 g	120 to 200	90 to 140	ISO 787-5

Table 2 — Conditional requirements

Characteristic	Unit	Requirement	Test method
Lightness	—	To be agreed between the interested parties	To be agreed between the interested parties

5 Sampling

Take a representative sample of the product to be tested, as specified in ISO 15528.

6 Determination of silica content

6.1 Reagents

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

6.1.1 Sulfuric acid, CAS Registry Number®[1] 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.

6.1.2 Hydrofluoric acid, CAS Registry Number® 7664-39-3, concentrated, approximately 40 % mass fraction, $\rho \approx$ 1,13 g/ml.

6.2 Apparatus

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

6.2.1 Platinum dish

6.2.2 Muffle furnace, capable of being maintained at up to (1 000 ± 25) °C.

6.2.3 Desiccator containing phosphorus pentoxide as desiccant.

6.2.4 Balance, with a maximum permissible error of 0,000 1 g.

6.3 Procedure

6.3.1 Number of determinations

Carry out the determination in duplicate.

6.3.2 Test portion

Weigh, to the nearest 1 mg, approximately 2 g of the sample (see Clause 5), previously dried at 105 °C in accordance with ISO 787-2, into the tared platinum dish (6.2.1).

6.3.3 Determination

Ignite the test portion in the platinum dish in the muffle furnace (6.2.2) at (1 000 ± 5) °C to constant mass (m_1) and allow to cool in the desiccator (6.2.3) containing phosphorus pentoxide.

Add approximately 1 ml of sulfuric acid (6.1.1). Heat the platinum dish gently until fuming ceases and then continue the heating at 900 °C for 15 min in the muffle furnace. Remove from the furnace, cool in the desiccator and weigh (m_2).

Add to the residue in the platinum dish (6.2.1) 5 ml of hydrofluoric acid (6.1.2) and evaporate to a syrup, taking care to avoid loss by spitting. Cool the platinum dish and wash the sides down with small quantities of water. Then add a further 2 ml of hydrofluoric acid and evaporate to dryness. Heat the residue on a hot-plate until white fumes are no longer evolved, then ignite in the muffle furnace at 900 °C for 15 min. Remove the dish from the furnace, allow to cool in the desiccator and weigh (m_3).

6.4 Expression of results

Calculate the silica content, $w(SiO_2)$, expressed as a percentage by mass, using Formula (1):

$$w(SiO_2) = \frac{(m_2 - m_3)}{m_1} \times 100 \quad (1)$$

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.

where

m_1 is the mass, expressed in grams, of the test portion after ignition;

m_2 is the mass, expressed in grams, after treatment with sulfuric acid and igniting;

m_3 is the mass, expressed in grams, after treatment with hydrofluoric acid and igniting.

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

7 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the product tested;

b) a reference to this document, i.e. ISO 3262-22:2023;

c) the results of the tests, the test method used, and whether or not the product complies with the relevant specification limits;

d) any deviation from the test methods specified;

e) any unusual features (anomalies) observed during the test;

f) the dates of the tests.