INTERNATIONAL STANDARD

ISO 3262-22

First edition
2001-10-15

Extenders for paints — Specifications and methods of test —

Part 22: Flux-calcined kieselguhr

Matières de charge pour peintures — Spécifications et méthodes d'essai — Partie 22: Kieselguhr, flux-calciné

Reference number
ISO 3262-22:2001(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

Printed in Switzerland

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 3262-22 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 22: Flux-calcined kieselguhr

1 Scope

This part of ISO 3262 specifies requirements and corresponding methods of test for flux-calcined kieselguhr.

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 150 °C*

ISO 787-3:2000, *General methods of test for pigments and extenders — Part 3: Determination of matter soluble in water — Hot extraction method*

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

ISO 787-7:1981, *General methods of test for pigments and extenders — Part 7: Determination of residue on sieve — Water method — Manual procedure*

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 15528:2000, *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
flux-calcined kieselguhr
siliceous material of diatomaceous origin

NOTE 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 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 Grade A	 B	Test method
Silica content, SiO_2, min.	% by mass	85		See clause 6
Loss on ignition, max.	% by mass	0,5		ISO 3262-1
pH-value of aqueous suspension		7,0 to 10,5		ISO 787-9
Matter soluble in water, max.	% by mass	1,0		ISO 787-3
Matter volatile at 105 °C, max.	% by mass	0,5		ISO 787-2
Residue on sieve, max.				
63 µm	% by mass	1	0	ISO 787-7
45 µm		10	0,1	
Specific surface area	m^2/g	max. 3	min. 3	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[a]
[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 Reagents

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

6.1.1 Sulfuric acid, diluted 1 + 1.

Add 1 part by volume of concentrated sulfuric acid, approximately 96 % by mass, $\rho \approx 1,84$ g/ml, slowly to 1 part by volume of water.

6.1.2 Hydrofluoric acid, concentrated, approximately 40 % by mass, $\rho \approx 1,13$ 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 $(1\,000 \pm 25)\,^{\circ}C$.

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 $(1\,000 \pm 25)\,^{\circ}C$ to constant mass (m_1) and allow to cool in a desiccator 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 the equation:

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

where

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

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

m_3 is the mass, 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 part of ISO 3262 (ISO 3262-22);

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

d) any deviation from the test methods specified;

e) the dates of the tests.