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Extenders for paints — Specifications and methods of test —

Part 19: Precipitated silica

Matières de charge pour peintures — Spécifications et méthodes d'essai — Partie 19: Silice précipitée

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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 3262-19 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 19:
Precipitated silica

1 Scope

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

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 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 787-11:1981, *General methods of test for pigments and extenders — Part 11: Determination of tamped volume and apparent density after tamping.*

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.*

3 Term and definition

For the purposes of this part of ISO 3262, the following term and definition apply:

3.1
precipitated silica
amorphous silica precipitated by reaction of sodium silicate solution with a mineral acid and/or carbon dioxide

1) To be published. (Revision of ISO 842:1984 and ISO 1512:1991)

4 Requirements and test methods

For precipitated silica 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
		Grade A	Grade B	
Silica content, min.	% (*m*/*m*)	95	95	See clause 6
Carbon content[a]		max. 0,2	min. 0,3	See clause 7
Organic surface coating?	—	No	Yes	See clause 7
Matter volatile at 105 °C, max.	% (*m*/*m*)	8		ISO 787-2
Loss on ignition	% (*m*/*m*)	3 to 8	3 to 15	ISO 3262-1
Oil absorption value[b], min.	g/100 g	120		ISO 787-5
pH value of aqueous suspension[c]	—	3,5 to 9		ISO 787-9

[a] The carbon content is also part of the loss on ignition.

[b] 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.

[c] For hydrophobic silicas, use a 1:1 (*m*/*m*) mixture by mass of water and methanol.

Table 2 — Conditional requirements

Characteristic	Unit	Requirement		Test method
		Grade A	Grade B	
Residue on 45 µm sieve, max.	% (*m*/*m*)	To be agreed between the interested parties	Not applicable	Spray method (see clause 8)[a]
Particle size distribution (instrumental method)	% (*m*/*m*)	To be agreed between the interested parties		
Apparent density after tamping	g/ml	To be agreed between the interested parties		ISO 787-11
Specific surface area	m^2/g			ISO 5794-1:1994, annex D

[a] Only for hydrophylic materials.

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.

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2) For example Whatman No. 40 or 41 or Schleicher und Schüll No. 589/2 "Weißband".