INTERNATIONAL STANDARD

ISO 3262-9

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Extenders for paints — Specifications and methods of test —

Part 9:
Calcined clay

Matières de charge pour peintures — Spécifications et méthodes d'essai —
Partie 9: Kaolin calciné

Reference number
ISO 3262-9:1997(E)

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.

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.

International Standard ISO 3262-9 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes,* Subcommittee SC 2, *Pigments and extenders.*

Together with the subsequent parts, this International Standard cancels and replaces ISO 3262: 1975 which has been technically revised and divided into parts. Part 1 comprises the definition for the term extender and a number of test methods that are applicable to most extenders, whilst parts 2 and the following specify requirements and, where appropriate, particular test methods for individual extenders.

At present, the following parts of ISO 3262 are in preparation, under the general title

Extenders for paints - Specification and methods of test

- Part 1: Introduction and general test methods

- Part 2: Baryte (natural barium sulfate)

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- *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: Diatomaceous earth (kieselguhr)*

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Extenders for paints — Specifications and methods of test —

Part 9:
Calcined clay

1 Scope

This part of ISO 3262 specifies the requirements and the corresponding methods of test for calcined clay.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 3262. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 3262 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO 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 787-3: 1979, *General methods of test for pigments and extenders - Part 3: Determination of matter soluble in water - Hot extraction method.*

ISO 787-9: 1981, *General methods of test for pigments and extenders - Part 9: Determination of pH value of an aqueous suspension.*

ISO 787-14: 1973, *General methods of test for pigments - Part 14: Determination of resistivity of aqueous extract.*

ISO 787-18: 1983, *General methods of test for pigments and extenders - Part 18: Determination of residue on sieve - Mechanical flushing procedure.*

ISO 842: 1984, *Raw materials for paints and varnishes - Sampling.*

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.*

3 Definition

For the purposes of this part of ISO 3262, the following definition applies:

3.1 calcined clay: Aluminium silicate ($Al_2O_3 \cdot 2SiO_2$), lamellar, mainly amorphous in structure as determined by X-ray diffraction, produced from natural clay by thermal dehydration, consisting partly of crystalline mullite ($3Al_2O_3 \cdot 2SiO_2$).

4 Requirements and test methods

For calcined clay 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			Test method
		A	B	C	
Content of $Al_2O_3 \cdot 2SiO_2$	% (*m/m*) min.	90			X-ray fluoresence
Residue on sieve, 45 μm	% (*m/m*) max.	0,02	0,05	0,1	ISO 787-18
Particle size distribution (Andreasen method) < 2 μm	% (*m/m*) min.	90	70	40	See clause 6
Matter volatile at 105 °C	% (*m/m*) max.	0,5			ISO 787-2[1])
Loss on ignition	% (*m/m*) max.	1			ISO 3262-1
Matter soluble in water (hot extraction method)	% (*m/m*) max.	0,2			ISO 787-3
pH value of aqueous suspension		5 to 9			ISO 787-9

[1]) By agreement between the interested parties, test portions other than 10 g may be used.

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[1])
Colour			ISO 3262-1
Lightness			To be agreed between the interested parties[2])
Resistivity of aqueous extract	Ω · m		ISO 787-14

[1]) A general description of the sedimentation method, with the detection of X-ray absorption, is given in EN 725-5, *Advanced technical ceramics - Methods of test for ceramic powders - Part 5: Determination of particle size distribution.*

[2]) Test method in preparation.

5 Sampling

Take a representative sample of the product to be tested, as described in ISO 842.

6 Determination of the particle size distribution

Because of its simple procedure and good reproducibility the Andreasen method[1]) is given as the referee method. Other methods may, however, be used by agreement between the interested parties, but in such cases it will be necessary to agree on appropriate limits.

6.1 Principle

The rate of fall of spherical particles through a medium in which they are dispersed is proportional to the square of the particle diameter (Stokes' law). The Andreasen method for determination of particle size distribution makes use of this relationship

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[1]) Andreasen, A.H.M., Lundberg, I.; "Berichte aus der deutschen Keramischen Gesellschaft" 11 (1930), 5, pages 312 to 323

expressing the particle size distribution in terms of the distribution of spherical particles having the same settlement rate. A suspension is prepared and the concentration of solids at a fixed distance below the surface is determined at a series of time intervals selected to correspond to a series of given sphere diameters.

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 Dispersing agent. Dissolve 0,2 g of sodium carbonate and 0,1 g of sodium hexametaphosphate in 750 ml of water.

6.3 Apparatus (see figure 1)

Ordinary laboratory apparatus and glassware, together with the following:

6.3.1 Sedimentation vessel, glass, of 56 mm internal diameter and having a graduated scale from 0 mm to 200 mm marked on its side. The zero graduation line shall be not less than 25 mm from the inside base of the vessel, and the capacity of the vessel up to the 200 mm graduation line shall be between 550 ml and 620 ml.

6.3.2 Pipette, fitted with a two-way tap and a side discharge tube.

NOTE 1 The capacity of the pipette to the graduation line is conveniently 10 ml.

A bell-shaped dome with a ground glass joint to fit the neck of the sedimentation vessel shall be fused to the pipette. A small vent hole shall be made in this dome. The tip of the pipette stem shall be level with the zero line on the sedimentation vessel. The stem from the pipette bulb to the tip shall be made of capillary glass tubing with a bore not less than 1 mm and not more than 1,3 mm in diameter. The tube above the bulb shall have a bore of 4 mm to 4,5 mm in diameter.

6.3.3 Constant-temperature bath of at least 15 l capacity, having transparent walls, capable of being maintained at a temperature of (23 ± 0,5) °C, into which the sedimentation vessel can be immersed up to the 200 mm graduation line. The bath shall be positioned away from sources of vibration, and the circulating system shall not cause vibration.

6.3.4 Mechanical stirrer, capable of rotating at a suitable speed for complete dispersion [(1 000 ± 100) min^{-1} is generally suitable]. The stirrer shall be capable of lifting the dispersion and avoiding the creation of a vortex.

NOTE 2 A suitable stirrer may be made from an approximately 40 mm diameter brass disc with four equally spaced cuts, the cut sections being turned upwards at an angle of 30° to the horizontal.

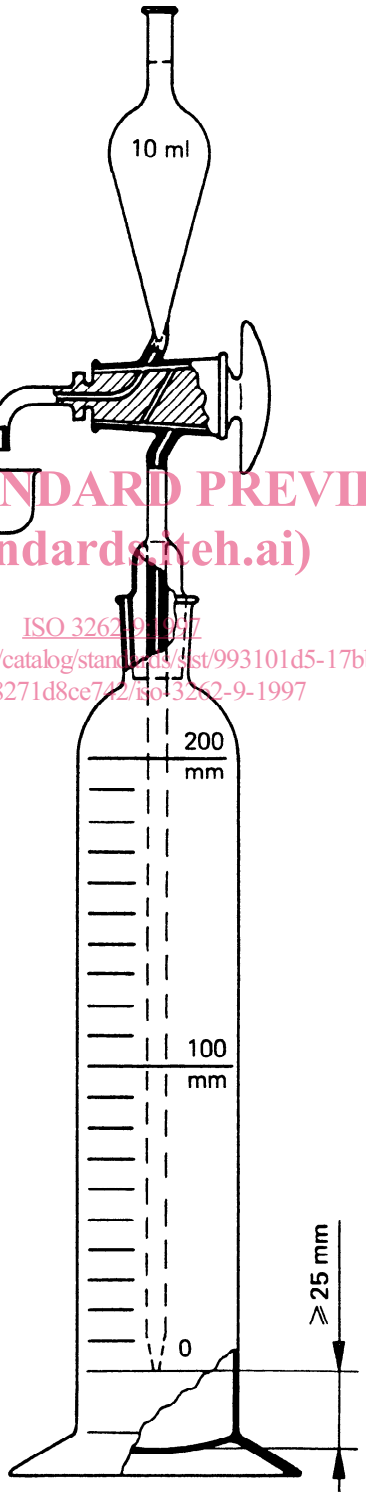

FIGURE 1 - Sedimentation vessel and pipette