INTERNATIONAL STANDARD

ISO 3262-8

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Extenders for paints — Specifications and methods of test —

Part 8:
Natural clay

Matières de charge pour peintures — Spécifications et méthodes d'essai —

Partie 8: Kaolin naturel

Reference number
ISO 3262-8:1999(E)

Contents

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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.

International Standard ISO 3262-8 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.

At present, the following parts of ISO 3262 are published or in preparation, 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: Kieselguhr*

Extenders for paints — Specifications and methods of test —

Part 8:
Natural clay

1 Scope

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

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 787-3:—[1], *General methods of test for pigments and extenders — Part 3: Determination of matter soluble in water — Hot extraction method.*

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

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 15528:—[2], *Paints and varnishes — Sampling.*

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[1] To be published. (Revision of ISO 787-3:1979)

[2] To be published. (Revision of ISO 842:1984 and ISO 1512:1991)

3 Term and definition

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

3.1

natural clay

naturally occurring hydrated aluminium silicates with a lamellar crystal structure, predominantly consisting of kaolinite of chemical composition $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O \mid Al_4[(OH)_8/Si_4O_{10}]$

4 Requirements and test methods

For natural 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**				**Test method**
		Grade A	**Grade B**	**Grade C**	**Grade D**	
Kaolinite content, min.	% (*m*/*m*)	90	85	70	50	X-ray diffraction or to be agreed between the interested parties
Residue on 45 µm sieve, max.	% (*m*/*m*)	0,02	0,05	0,1	0,5	ISO 787-7
Particle-size distribution (Andreasen method), $<$ 2 µm, min.	% (*m*/*m*)	90	70	40	20	See clause 6
Matter volatile at 105 °C, max.	% (*m*/*m*)	1				ISO 787-2[a]
Loss on ignition	% (*m*/*m*)	12 to 14	11 to 14	10 to 14	6 to 9	ISO 3262-1
Matter soluble in water (hot extraction method), max.	% (*m*/*m*)	0,3			0,5	ISO 787-3
pH value of aqueous suspension		4 to 9				ISO 787-9

[a] 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[a]
Colour			ISO 3262-1
Lightness			To be agreed between the interested parties[b]
Reistivity of aqueous extract	Ω·m		ISO 787-14

[a] A general description of a sedimentation method using X-ray absorption is given in EN 725-5:1996, *Advanced technical ceramics — Methods of test for ceramic powders — Part 5: Determination of the particle size distribution.*

[b] Test method in preparation.

5 Sampling

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

6 Determination of particle-size distribution

6.1 General

Because of its simple procedure and good reproducibility, the Andreasen method[3] 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.

6.2 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, 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.

6.3 Reagents

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

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

6.4 Apparatus (see Figure 1)

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

6.4.1 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 of the base of the vessel, and the capacity of the vessel up to the 200 mm line shall be between 550 ml and 620 ml.

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

NOTE 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.

6.4.3 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.

6.4.4 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 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.

6.4.5 Dispersion vessel, of appropriate dimensions, such as a 1 000 ml gas jar.

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[3] Andreasen, A.H.M., Lundberg, I; "Berichte aus der deutschen Keramischen Gesellschaft" **11** (1930), 5, pp. 312-323.

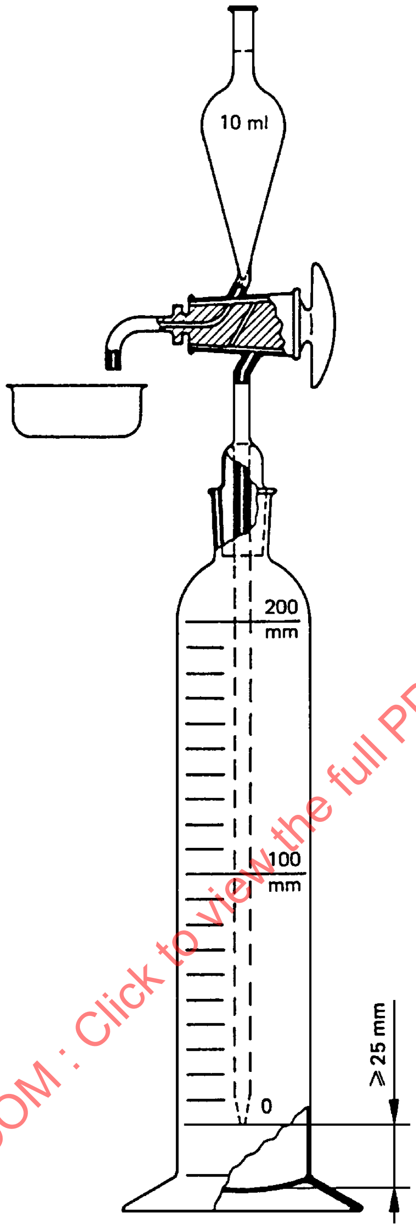

Figure 1 — Sedimentation vessel and pipette

6.4.6 Analytical balance, capable of weighing to 0,1 mg.

6.4.7 Drying oven, capable of being maintained within a temperature range suitable for evaporation of the suspending liquid, for example (105 ± 2) °C for water.

6.4.8 Wide-mouthed weighing bottles, suitable for evaporation, of capacity not less than 20 ml, or a small laboratory centrifuge and centrifuge tubes, preferably of 20 ml capacity but of not less than 10 ml capacity.

6.4.9 Stopwatch or **stopclock.**

6.5 Preparation for the test

6.5.1 Calibration of pipette

Thoroughly clean the pipette (6.4.2). Partly fill the sedimentation vessel (6.4.1) with distilled water. Set the tap in the sampling position and, by means of a rubber tube, suck water into the bulb to the level of the graduation line. Reverse the tap to the discharge position and allow the water to drain into a tared weighing bottle (6.4.8). Apply pressure through the rubber tube to blow any water remaining in the bulb and the discharge tube into the weighing bottle. Weigh the bottle to the nearest 1 mg and calculate from this mass the internal volume V_p of the pipette.

6.5.2 Calibration of sedimentation vessel

Thoroughly clean the sedimentation vessel (6.4.1), and weigh it, with the pipette (6.4.2) in place, to the nearest 0,1 g. Then fill it to the 200 mm graduation line with water at (23 ± 0,5) °C and reweigh. Calculate from the mass of water the internal volume V_s of the vessel.

6.6 Procedure

6.6.1 Number of determinations

Carry out the determination in duplicate.

6.6.2 Test portion

Weigh, to the nearest 1 mg, about 12 g of the sample. Prepare a suspension by placing this test portion in the dispersion vessel (6.4.5), adding dispersing agent (6.3.1) at (23 ± 0,5) °C to give a total volume of about 500 ml and stirring with the mechanical stirrer (6.4.4) for 15 min. Immediately pour all the suspension into the sedimention vessel and make up to the 200 mm line with water. Transfer the sedimentation vessel to the constant-temperature bath (6.4.3), maintained at (23 ± 0,5) °C.

6.6.3 Sedimentation

Allow the sedimentation vessel to stand immersed to the 200 mm line until it has reached the temperature of the bath. When temperature equilibrium has been attained, mix the contents thoroughly by placing a finger over the vent hole and inverting the vessel several times. Immediately after mixing, replace the vessel and start the stopclock or stopwatch (6.4.9).

Either withdraw from the suspension, by means of the pipette, a series of samples of volume V_p at depths h_n at increasing time intervals after mixing, starting at time t_1 corresponding to a particle size of about 20 µm (calculated in accordance with 6.6.4) and thereafter in progression so that the limiting diameters are in a $\sqrt{2}$:1 progression, or take samples at convenient times approximately in a 2:1 progression and calculate the exact spherical-particle diameter corresponding to each sample.

The time to fill the pipette shall be about 20 s.

Discharge the suspension from the pipette into a tared weighing bottle (6.4.8). When the bulb has drained, remove the rubber suction tube and run 5 ml to 7 ml of water from an ordinary 10 ml pipette into the bulb of the sedimentation pipette to wash into the weighing bottle any particles adhering to the surface. The stem of the sedimentation pipette shall remain filled with suspension. Dry the contents of the weighing bottle at 105 °C until the difference between successive weighings is not greater than 0,1 mg. Calculate the mass of the fraction, m_n, allowing for the mass of dispersing agent (6.3.1), which shall be determined by carrying out a blank test.

NOTE 1 The determination of the different fractions may be carried out by means of chemical methods if this procedure is more suitable.

The depth of immersion of the pipette decreases as each fraction is withdrawn. Determine the exact decrease in depth by experiment and allow for it in the subsequent calculations.

NOTE 2 For example, suppose that the decrease is 4 mm for each fraction withdrawn, and that initially the depth of immersion of the pipette was 200 mm, then the depth after the first fraction has been withdrawn will be 196 mm, and the mean depth h_1 used for calculation of the initial diameter corresponding to the first fraction will be 198 mm. The mean depth h_2 for the second fraction will be 194 mm, and so on.