BS EN ISO 3262-9:1998

Extenders for paints — Specifications and methods of test —

Part 9: Calcined clay

The European Standard EN ISO 3262-9:1998 has the status of a British Standard

ICS 87.060.10

National foreword

This British Standard is the English language version of EN ISO 3262-9:1998. It is identical with ISO 3262-9:1997. Together with the other parts of BS EN ISO 3262, it will supersede BS 1795:1976 which is declared obsolescent.

The UK participation in its preparation was entrusted to Technical Committee STI/1, Pigments, which has the responsibility to:

- — aid enquirers to understand the text;
- — present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- — monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

Cross-references

Attention is drawn to the fact that CEN and CENELEC standards normally include an annex which lists normative references to international publications with their corresponding European publications. The British Standards which implement international or European publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled “International Standards Correspondence Index”, or by using the “Find” facility of the BSI Standards Electronic Catalogue.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the EN ISO title page, page 2, the ISO title page, page ii, pages 1 to 6 and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

This British Standard, having been prepared under the direction of the Sector Board for Materials and Chemicals, was published under the authority of the Standards Board and comes into effect on
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June 1998

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Descriptors: See ISO document

English version

Extenders for paints — Specifications and methods of test — Part 9: Calcined clay

(ISO 3262-9:1997)

Matières de charge pour peintures —
Spécifications et méthodes d'essai —
Partie 9: Kaolin calciné
(ISO 3262-9:1997)

Füllstoffe für Beschichtungsstoffe —
Anforderungen und Prüfverfahren —
Teil 9: Calcinierter Kaolin
(ISO 3262-9:1997)

This European Standard was approved by CEN on 14 May 1998.

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European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Ref. No. EN ISO 3262-9:1998 E

Foreword

The text of the International Standard from Technical Committee ISO/TC 35 "Paints and varnishes" of the International Organization for Standardization (ISO) has been taken over as an European Standard by Technical Committee CEN/TC 298 "Pigments and extenders", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 1998, and conflicting national standards shall be withdrawn at the latest by December 1998.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 3262-9:1997 has been approved by CEN as a European Standard without any modification.

NOTE Normative references to International Standards are listed in Annex ZA (normative).

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INTERNATIONAL STANDARD

ISO 3262-9

First edition
1997-06-15

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);*
- *— 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).*

Descriptors: Paints, extenders, clays, kaolin, specifications, materials specifications, tests.

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[a]
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

[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]
Resistivity of aqueous extract	Ω · m		ISO 787-14

[a] 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.*

[b] 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 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.

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

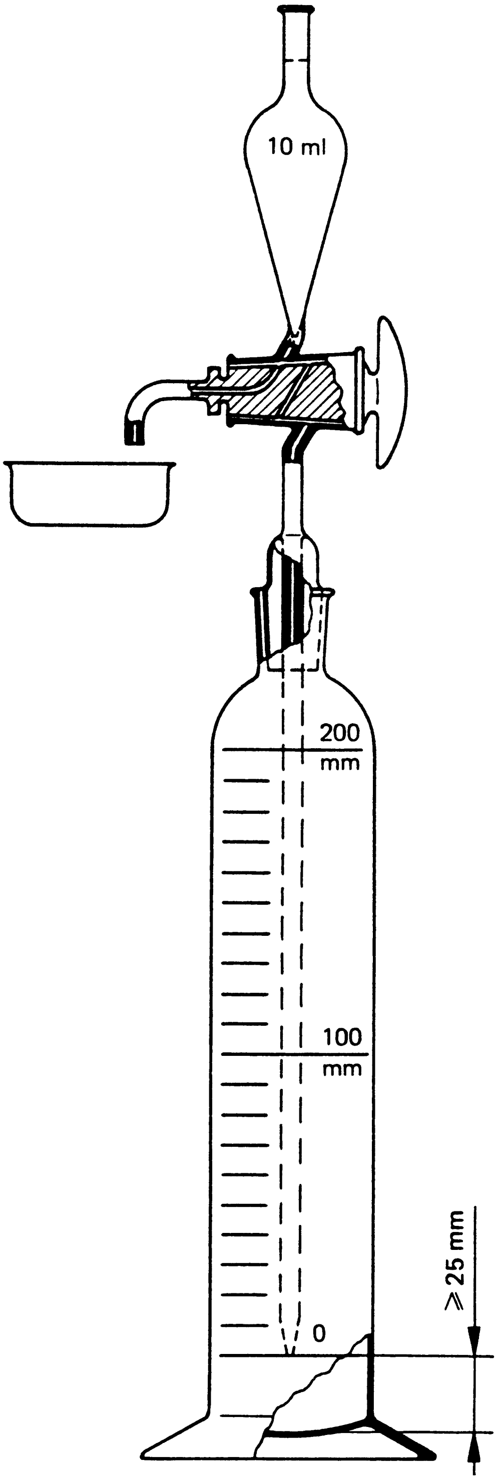

Figure 1 — Sedimentation vessel and pipette

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

6.3.6 *Analytical balance,* capable of weighing to 0,1 mg.

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

6.3.8 *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.

6.3.9 *Stop-watch or stop-clock.*

6.4 Preparation for the test

6.4.1 *Calibration of pipette*

Thoroughly clean the pipette (**6.3.2**). Partly fill the sedimentation vessel (**6.3.1**) 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 (**6.3.8**). 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 Vp of the pipette.

6.4.2 *Calibration of sedimentation vessel*

Thoroughly clean the sedimentation vessel (**6.3.1**), and weigh it, with the pipette (**6.3.2**) 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.

6.5 Procedure

Carry out the determination in duplicate.

6.5.1 *Test portion*

Weigh to the nearest 1 mg, about 12 g of the sample. Prepare a dispersion by placing this test portion in the dispersion vessel (**6.3.5**), adding dispersing agent (**6.2.1**) at (23 ± 0,5) °C to give a total volume of about 500 ml and stirring with the mechanical stirrer (**6.3.4**) 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 (**6.3.3**) maintained at (23 ± 0,5) °C.

6.5.2 *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 thorougly by placing a finger over the vent hole and inverting the vessel several times. Immediately after mixing, replace the vessel and start the stop-clock or stop-watch (**6.3.9**).

Either withdraw from the suspension, by means of the pipette, three 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 **6.5.3**) and thereafter in progression so that the limiting diameters stand in a $\sqrt{2} : 1$ progression, or take samples at convenient times approximately in a 2 : 1 progression and calculate the exact spherical particle diameters corresponding to each sample.

NOTE 3 The time to fill the pipette should be about 20 s.

Discharge the suspension from the pipette into a tared weighing bottle (**6.3.8**). 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, which shall be determined by carrying out a blank test.

NOTE 4 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 5 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, the depth after the first fraction has been withdrawn will be 196 mm, and the mean depth h_1 used for calculation of the inital 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.

6.5.3 *Calculation of withdrawal time*

Calculate the withdrawal time, t_n, in seconds, for the nth sample, using the equation

$$t_n = \frac{18\eta h_n}{(\rho_2 - \rho_1)\, g d_n^2} \times 10^6$$

where

η is the absolute viscosity, in Newton seconds per square metre, of the medium;

h_n is the mean depth, in millimetres, at which the nth sample was withdrawn;

ρ_1 is the density, in grams per cubic centimetre, of the medium;

ρ_2 is the density, in grams per cubic centimetre, of the particle;

g is the acceleration due to gravity, in metres per second squared;

d_n is the limiting Stokes diameter, in micrometres, corresponding to the nth sample withdrawal.

6.6 Calculation of cumulative percentage undersize

Calculate the cumulative percentage by mass, $p_{n'}$ of particles smaller than each of the limiting Stokes diameters d_n for each time interval $t_{n'}$ using the equation

$$p_n = \frac{m_n \times V_s}{m_s \times V_p} \times 100$$

where

m_n is the mass, in grams, of the sediment in the fraction, corrected, by subtraction, for the mass of the sodium salts in the dispersing agent;

m_s is the mass, in grams, of the test portion;

V_p is the volume, in millilitres, of the pipette (**6.3.2**);

V_s is the volume, in millilitres, of the sedimentation vessel (**6.3.1**).

6.7 Acceptance of test values

The two values (duplicates) for each of the percentages by mass, of particles smaller than the same limiting Stokes diameters shall be accepted only if they do not differ by more than 4 %.

6.8 Expression of results

Plot the accepted values (**6.7**) with the micrometric sizes as abscissae and the percentages of undersize particles as ordinates. From the smooth curve drawn through the points, select the cumulative percentages corresponding to the series required.

Report the results to the nearest 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 3262-9);

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 date of the test.

Annex ZA (normative) Normative references to international publications with their relevant European publications

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to appies.

Publication	Year	Title	EN	Year
ISO 787-2	1981	General methods of test for pigments and extenders — Part 2: Determination of matter volatile at 105 °C	EN ISO 787-2	1995
ISO 787-3	1979	General methods of test for pigments and extenders — Part 3: Determination of matter soluble in water — Hot extraction method	EN ISO 787-3	1995
ISO 787-9	1981	General methods of test for pigments and extenders — Part 9: Determination of pH value of an aqueous suspension	EN ISO 787-9	1995
ISO 787-18	1983	General methods of test for pigments and extenders — Part 18: Determination of residue on sieve — Mechanical flushing procedure	EN ISO 787-18	1995
ISO 3262-1	1997	Extenders for paints — Specifications and methods of test — Part 1: Introduction and general test methods	EN ISO 3262-1	1998
ISO 3696	1987	Water for analytical laboratory use — Specification and test methods	EN ISO 3696	1995

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