



SLOVENSKI STANDARD
oSIST prEN ISO 3262-9:2022
01-december-2022

Polnila za barve - Specifikacije in metode preskušanja - 9. del: Kalciniran kaolin (ISO/DIS 3262-9:2022)

Extenders - Specifications and methods of test - Part 9: Calcined clay (ISO/DIS 3262-9:2022)

Füllstoffe - Anforderungen und Prüfverfahren - Teil 9: Calciniertes Kaolin (ISO/DIS 3262-9:2022)

Matières de charge - Spécifications et méthodes d'essai - Partie 9: Argile calcinée (ISO/DIS 3262-9:2022)

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ICS:

87.060.10 Pigmenti in polnila Pigments and extenders

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Extenders — Specifications and methods of test —

Part 9: Calcined clay

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 256, *Pigments, dyestuffs and extenders*.

This second edition cancels and replaces the first edition (ISO 3262-9:1997), which has been technically revised.

The main changes are as follows:

- the first part of the title has been changed to "Extenders";
- the test method for particle size distribution in [Table 2](#) has been changed to ISO 8130-13;
- the normative references have been updated and the text has been editorially revised.

A list of all parts in the ISO 3262 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Extenders — Specifications and methods of test —

Part 9: Calcined clay

1 Scope

This document specifies requirements and corresponding methods of test for calcined clay.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 787-2, *General methods of test for pigments and extenders — Part 2: Determination of matter volatile at 105 °C*

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

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

ISO 787-14, *General methods of test for pigments and extenders — Part 14: Determination of resistivity of aqueous extract*

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

ISO 3262-1, *Extenders — Specifications and methods of test — Part 1: Introduction and general test methods*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 8130-13, *Coating powders — Part 13: Particle size analysis by laser diffraction*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

ISO 18451-1, *Pigments, dyestuffs and extenders — Terminology — Part 1: General terms*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18451-1 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

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3.1 calcined clay
aluminium silicate ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$), lamellar, mainly amorphous in structure as determined by X-ray diffraction, produced from natural clay by thermal dehydration, consisting partly of crystalline mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$)

4 Requirements and test methods

For calcined clay complying with this document, the essential requirements are specified in [Table 1](#) and the conditional requirements are listed in [Table 2](#). The test method in [Tables 1](#) and [2](#) shall be in accordance with the standards listed.

Table 1 — Essential requirements

Characteristic	Unit	Requirement grade			Test method
		A	B	C	
Content of $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$	% mass fraction min.	90			X-ray fluorescence
Residue on sieve, 45 μm	% mass fraction max.	0,02	0,05	0,1	ISO 787-18
Particle size distribution (Andreasen method) < 2 μm	% mass fraction min.	90	70	40	See clause 6
Matter volatile at 105 °C	% mass fraction max.	0,5			ISO 787-2 ^a
Loss on ignition	% mass fraction max.	1			ISO 3262-1
Matter soluble in water (hot extraction method)	% mass fraction 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)	% mass fraction	To be agreed between the interested parties	ISO 8130-13
Colour			ISO 3262-1
Lightness			To be agreed between the interested parties
Resistivity of aqueous extract	$\Omega \cdot \text{m}$		ISO 787-14

5 Sampling

Take a representative sample of the product to be tested, in accordance with ISO 15528.

6 Determination of the particle size distribution

6.1 General

Because of its simple procedure and good reproducibility the Andreasen method¹⁾ 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, CAS²⁾ No. 10124-56-8, 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 1 The capacity of the pipette to the graduate 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 $(23 \pm 0,5)$ °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.

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

2) Chemistry Abstracts Service Registry Number.

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6.4.4 Mechanical stirrer, capable of rotation at a suitable speed for complete dispersion $[(1\ 000 \pm 100) \text{ min}^{-1}]$ 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.

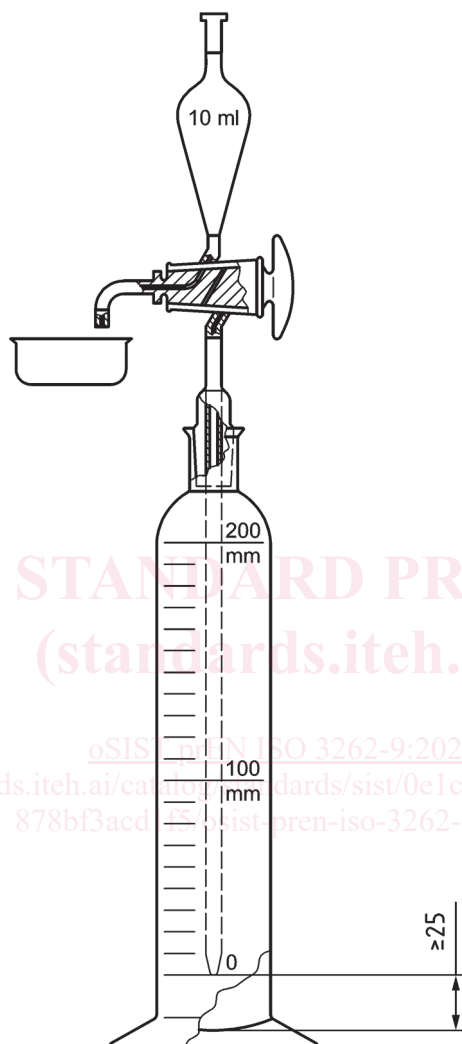


Figure 1 — Sedimentation vessel and pipette

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

6.4.6 Balance, with an accuracy of 0,0001 g.

6.4.7 Drying oven, capable of being maintained with a temperature range suitable for evaporation of the suspending liquid, for example $(105 \pm 2)^\circ\text{C}$ 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 not less than 10 ml capacity.

6.4.9 Stopwatch or stopclock.