INTERNATIONAL STANDARD

ISO 3262-9

Second edition
2023-07

Extenders — Specifications and methods of test —

Part 9:
Calcined clay

Matières de charge — Spécifications et méthodes d'essai —

Partie 9: Argile calcinée

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 3262-9:2023
https://standards.iteh.ai/catalog/standards/sist/b5cb60b8-b0db-4714-8011-318f61dd73a1/iso-3262-9-2023

Reference number
ISO 3262-9:2023(E)

© ISO 2023

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 3262-9:2023
https://standards.iteh.ai/catalog/standards/sist/b5cb60b8-b0db-4714-8011-318f61dd73a1/iso-3262-9-2023

COPYRIGHT PROTECTED DOCUMENT

© ISO 2023

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 3262-9:2023
https://standards.iteh.ai/catalog/standards/sist/b5cb60b8-b0db-4714-8011-318f61dd73a1/iso-3262-9-2023

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 document 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).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

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.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 3262-9:2023
https://standards.iteh.ai/catalog/standards/sist/b5cb60b8-b0db-4714-8011-318f61dd72a1/iso-3262-9-2023

This document was prepared by Technical Committee ISO/TC 256, *Pigments, dyestuffs and extenders*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 298, *Pigments and extenders*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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”;
- alkaline fusion has been added in Table 1 as a second method for determination of the aluminium silicate content;
- the test method for particle size distribution in Table 2 has been changed to ISO 8130-13;
- the normative references have been updated.

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.

© ISO 2023 – All rights reserved

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*

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 3262-9:2023
https://standards.iteh.ai/catalog/standards/sist/b5cb60b8-b0db-4714-8011-318f61dd73a1/iso-3262-9-2023

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/

© ISO 2023 – All rights reserved

3.1

calcined clay

aluminium silicate ($AI_2O_3 \cdot 2SiO_2$), lamellar, mainly amorphous in structure as determined by X-ray diffraction, produced from natural clay by thermal dehydration, consisting partly of crystalline mullite ($3AI_2O_3 \cdot 2SiO_2$)

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 methods listed in Tables 1 and 2 shall apply.

Table 1 — Essential requirements

Characteristic	Unit	Requirement grade			Test method
		A	B	C	
Content of $Al_2O_3 \cdot 2SiO_2$	% mass fraction min.	90			X-ray fluorescence or alkaline fusion
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.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 3262-9:2023
https://standards.iteh.ai/catalog/standards/sist/b5cb60b8-b0db-4714-8011-318f61dd73a1/iso-3262-9-2023

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	Ω·m		ISO 787-14

5 Sampling

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

© ISO 2023 – All rights reserved

6 Determination of the particle size distribution

6.1 General

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.

6.2 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 the 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 specified in ISO 3696.

6.3.1 Dispersing agent

iTeh STANDARD PREVIEW
(standards.iteh.ai)

Dissolve 0,2 g of sodium carbonate, CAS Registry Number®[1] 471-34-1, and 0,1 g of sodium hexametaphosphate, CAS Registry Number® 10124-56-8, in 750 ml of water.

6.4 Apparatus

ISO 3262-9:2023
https://standards.iteh.ai/catalog/standards/sist/b5cb00b8-b0db-4714-8011-318f61dd73a1/iso-3262-9-2023

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. 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 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 ± 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.

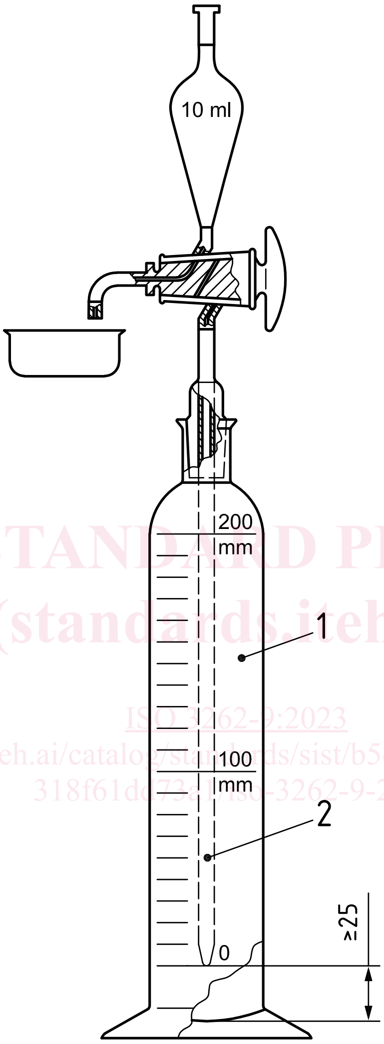
---

[1] Chemical Abstracts Service (CAS) Registry Number® is a trademark of the American Chemical Society (ACS). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

6.4.4 Mechanical stirrer, capable of rotation 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 can 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.

Dimensions in millimetres



iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 3262-9:2023
https://standards.iteh.ai/catalog/standards/sist/b5cb60b8-b0db-4714-8011-318f61dc73a1/iso-3262-9-2023

Key

1 sedimentation vessel

2 pipette

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 0,000 1 g.

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

© ISO 2023 – All rights reserved

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 **stop clock**

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 of the pipette, V_p.

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 of the vessel, V_S.

6.6 Procedure

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 3262-9:2023
https://standards.iteh.ai/catalog/standards/sist/b5cb60b8-b0db-4714-8011-318f61dd73a1/iso-3262-9-2023

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 sedimentation 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 stop clock or stopwatch (6.4.9).

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. Start the process at time, t_1, corresponding to a particle size 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. Alternatively, 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 been 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 can 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, if the decrease is 4 mm for each fraction withdrawn, and initially the depth of immersion of the pipette was 200 mm, then after the first fraction has been withdrawn, the depth will be 196 mm. The mean depth used for calculation of the initial diameter corresponding to the first fraction, $h_{1,}$ will be 198 mm. The mean depth for the second fraction, $h_{2,}$ will be 194 mm, and so on.

6.6.4 Calculation of withdrawal time

Calculate the withdrawal time t_n, in seconds, for the n^th sample, using Formula (1):

$$t_n = \frac{18 \eta h_n}{(\rho_2 - \rho_1) g d_n^2} \times 10^6 \qquad (1)$$

where

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

h_n is the mean depth, expressed in millimetres, at which the n^th sample was withdrawn;

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

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

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

d_n is the limiting Stokes diameter, expressed in micrometres, corresponding to the n^th sample withdrawal.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 3262-9:2023
https://standards.iteh.ai/catalog/standards/sist/b5cb60b8-b0db-4714-8011-

6.7 Calculation of cumulative percentage undersize

Calculate the cumulative percentage by mass, p_n, of particles smaller than each of the limiting Stokes diameter, d_n, for each time interval, t_n, using Formula (2):

$$p_n = \frac{m_n \times V_\mathrm{s}}{m_\mathrm{s} \times V_\mathrm{p}} \times 100 \qquad (2)$$

where

m_n is the mass, expressed 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, expressed in grams, of the test portion;

V_p is the volume, expressed in millilitres, of the pipette (6.4.2);

V_s is the volume, expressed in millilitres, of the sedimentation vessel (6.4.1).

6.8 Acceptance of test values

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

© ISO 2023 – All rights reserved