INTERNATIONAL STANDARD

ISO 3262-6

Second edition
2022-11

Extenders — Specifications and methods of test —

Part 6: Precipitated calcium carbonate

Matières de charge — Spécifications et méthodes d'essai —

Partie 6: Carbonate de calcium précipité

Reference number
ISO 3262-6:2022(E)

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

Page

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*, 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 3262-6:1998), which has been technically revised.

The main changes are as follows:

— the first part of the title has been changed to “Extenders”;

— the values for $CaCO_3$ content and loss on ignition have been changed and the range of pH value of aqueous suspension has been expanded in Table 1;

— the test method for lightness has been specified in Table 2;

— a test method, furnace method, for the determination of matter insoluble in hydrochloric acid has been added in Clause 6;

— 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 6:
Precipitated calcium carbonate

1 Scope

This document specifies requirements and corresponding methods of test for precipitated calcium carbonate.

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 787-8, *General methods of test for pigments and extenders — Part 8: Determination of matter soluble in water — Cold 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 787-25, *General methods of test for pigments and extenders — Part 25: Comparison of the colour, in full-shade systems, of white, black and coloured pigments — Colorimetric method*

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 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/

3.1
precipitated calcium carbonate
synthetic calcium carbonate, consisting of trigonal crystals (like those of calcite) or rhombic bipyramidal crystals (like those of aragonite) with or without surface modification

4 Requirements

For precipitated calcium carbonate 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	Test method
$CaCO_3$ content, min.	% mass fraction	90	ISO 3262-1
Residue on 45 µm sieve, max.	% mass fraction	0,1	ISO 787-18
Matter volatile at 105 °C, max.	% mass fraction	1,0	ISO 787-2
Loss on ignition, max.	% mass fraction	50[a]	ISO 3262-1
Matter soluble in water, max.	% mass fraction	0,2	ISO 787-3 or ISO 787-8[b]
pH value of aqueous suspension		7 to 11[a]	ISO 787-9
Matter insoluble in hydrochloric acid, max.	% mass fraction	0,2	Clause 6

[a] These values take account of the effect on the result of any surface modification.

[b] Method to be agreed between the interested parties.

Table 2 — Conditional requirements

Characteristic	Unit	Requirement	Test method
Particle size distribution (D50 and D90) (instrumental method)	µm	To be agreed between the interested parties	
Colour		To be agreed between the interested parties	ISO 3262-1
Lightness			ISO 787-25
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.

6 Determination of matter insoluble in hydrochloric acid

6.1 General

An appropriate method to measure matter insoluble in hydrochloric acid shall be selected from the oven method and the furnace method (see 6.2 and 6.3). For surface-modified products, the furnace method shall be used (see 6.3).

6.2 Oven method

6.2.1 Reagents

During the analysis, use only reagents of recognized analytical grade and only water of at least grade 3 purity in accordance with ISO 3696.

6.2.1.1 Hydrochloric acid, approximately 25 % mass fraction, CAS-No 7647-01-0[1)], $\rho \approx 1{,}125$ g/ml.

6.2.2 Apparatus

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

6.2.2.1 Membrane filter, pore size 0,8 µm.

6.2.2.2 Air oven, capable of being maintained at (105 ± 2) °C.

6.2.3 Procedure

Weigh, to the nearest 0,1 mg, approximately 10 g (m_0) of the test sample into a 600 ml beaker. Add 50 ml of water and, carefully, approximately 50 ml of hydrochloric acid (6.2.1.1). Cover the beaker with a watch glass and boil the solution for 15 min.

Dry the membrane filter (6.2.2.1) in the air oven (6.2.2.2) at (105 ± 2) °C to constant mass, cool in a desiccator to room temperature and weigh it to the nearest 0,1 mg (m_1). Then filter the solution through it. Wash the residue on the filter eight times with hot distilled water. Dry the residue on the filter in the air oven at (105 ± 2) °C for about 1 h. Allow to cool in a desiccator to room temperature and weigh to the nearest 0,1 mg (m_2).

6.2.4 Expression of results

Calculate the matter insoluble in hydrochloric acid, expressed as a percentage by mass, using Formula (1):

$$\frac{m_2 - m_1}{m_0} \tag{1}$$

where

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

m_1 is the mass, in grams, of the dried membrane filter;

m_2 is the mass, in grams, of the dried membrane filter plus the residue.

6.3 Furnace Method

6.3.1 Reagents

During the analysis, use only reagents of recognized analytical grade and only water of at least grade 3 purity in accordance with ISO 3696.

6.3.1.1 Hydrochloric acid, approximately 17 % mass fraction, CAS-No 7647-01-0, $\rho \approx 1{,}085$ g/ml.

1) Chemical Abstracts Service Registry Number.

6.3.2 Apparatus

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

6.3.2.1 Quantitative filter paper, pore size 4 µm, ash content <0,01 % mass fraction.

6.3.2.2 Crucible, platinum or porcelain. If a porcelain crucible is used, it shall be heated to (390 ± 20) °C and cooled in a desiccator before the test.

6.3.2.3 Electric furnace, capable of being maintained at (900 ± 25) °C.

6.3.3 Procedure

Weigh, to the nearest 10 mg, approximately 2 g to 5 g (m_0) of the test sample into a beaker. Add a small amount of water and, carefully, approximately 10 ml to 12 ml of hydrochloric acid (6.3.1.1). Cover the beaker with a watch glass and boil the solution for 5 min.

Filter the matter insoluble using the filter paper (6.3.2.1), wash thoroughly with hot distilled water and dry the filter paper. Weigh the crucible (6.3.2.2) to the nearest 0,1 mg (m_1). Put the dried filter paper into the crucible and place the crucible in the electric furnace (6.3.2.3) set at (900 ± 25) °C for 30 min. Allow to cool in a desiccator to room temperature and weigh to the nearest 0,1 mg (m_2).

6.3.4 Expression of results

Calculate the matter insoluble in hydrochloric acid, expressed as a percentage by mass, using Formula (2):

$$\frac{m_2 - m_1}{m_0} \tag{2}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the dried crucible;

m_2 is the mass, in grams, of the dried crucible plus the residue.

7 Test report

The test report shall include at least the following information:

a) all details necessary to identify the product tested;

b) a reference to this document, i.e. ISO 3262-6:2022;

c) the results of the test, the method used, and whether or not the product complies with the relevant specification limits;

d) any deviation from the method of test specified;

e) any unusual features (anomalies) observed during the test;

f) the date of the test.

ICS 87.060.10

Price based on 4 pages