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Extenders — Specifications and methods of test —

Part 1:
Introduction and general test methods

Matières de charge — Spécifications et méthodes d'essai —

Partie 1: Introduction et méthodes d'essai générales

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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*, 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 3262-1:1997), which has been technically revised.

The main changes compared to the previous edition are as follows:

- the main title has been changed from “Extenders for paints” to “Extenders”;
- clarification and focus on extenders for all/undefined applications;
- CAS numbers have been added to all reagents used.

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 1:
Introduction and general test methods

1 Scope

This document gives the definition for the term extender and specifies test methods that are required for most of the subsequent parts of ISO 3262.

NOTE The subsequent parts of ISO 3262 specify requirements and the corresponding methods of test for extenders for use in paints, related coating materials and other applications.

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 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — Single-volume pipettes*

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

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

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

ISO 3819, *Laboratory glassware — Beakers*

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

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

3.1
extender
material in granular or powder form, practically insoluble to somewhat soluble in the application medium and used to modify or influence certain physical properties

Note 1 to entry: The German terms “Extender”, “Extenderpigment”, Pigmentextender” or “Verschnittmittel” should be avoided.

Note 2 to entry: Whether a given substance is to be considered as pigment or extender depends on its application.

Note 3 to entry: In some industries, such as rubber industries, “extender” is also called “filler” (see ISO 1382). In coatings industries, the term “extender” is preferred, because in ISO 4618, “filler” is defined as “coating material with a high proportion of extender, intended primarily to even out irregularities in substrates to be painted and to improve surface appearance”.

[SOURCE: ISO 18451-1:2019, 3.34]

4 Sampling

Take a representative sample of the product to be tested according to ISO 15528.

5 Test methods

NOTE For particular test methods for individual extenders, see the appropriate parts of ISO 3262.

5.1 Comparison of colour

NOTE There is no objection to the use of a particular instrumental method, agreed between the interested parties, for comparison of colour. ISO 11664-4 defines procedures for calculating the respective coordinates in the CIE 1976 L*a*b* colour space. However, in view of differences between the results obtained with various instruments, the simple visual method is given as the referee method. A method for the assessment of hue of near-white specimens is described in ISO 18314-1.

Place approximately equal masses of the test sample and the agreed reference sample on to a ground glass plate placed on a piece of white paper.

Place a thin glass plate vertically between the two samples so as to form a partition. Push the samples towards each other until they come into contact with the thin glass plate. Remove the glass plate and flatten the surface of the two heaps with another glass plate, removing the latter before viewing.

Examine the colour of the samples in diffuse daylight or, if good daylight is not available, make the comparison in artificial daylight.

Carefully add a few drops of either white spirit or water (as agreed between the interested parties) in such a way that the two samples are just wetted without being disturbed, then repeat the colour comparison by the same method as before.

5.2 Determination of loss on ignition

NOTE For the determination of loss on ignition of aluminium hydroxides, see ISO 3262-16.

5.2.1 Apparatus

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

5.2.1.1 **Platinum or porcelain dish, shallow**.

5.2.1.2 **Muffle furnace**.

5.2.2 Procedure

Weigh, to the nearest 1 mg, about 0,5 g to 2 g of the test sample, previously dried in accordance with ISO 787-2, into the tared platinum or porcelain dish (5.2.1.1), ignite in the muffle furnace (5.2.1.2) at (1 000 ± 25) °C to constant mass together with a cover lid, and cool immediately in a desiccator containing a suitable desiccant such as phosphorus pentoxide or orange gel. To avoid uptake of moisture during handling, cover the dish with the lid.

NOTE The mass depends on the specific density of the extender.

5.2.3 Expression of results

Calculate the loss on ignition, *LI*, expressed as mass portion in per cent, using Formula (1):

$$LI = \frac{100(m_0 - m_1)}{m_0} \tag{1}$$

where

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

m_1 is the mass, in grams, of the test portion after ignition.

5.3 Determination of calcium and magnesium contents

5.3.1 Reagents

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

5.3.1.1 Hydrochloric acid, CAS-No 7647-01-0, c(HCl) = 6 mol/l.

5.3.1.2 Ammonia solution, CAS-No 1336-21-6, approximately 32 % (mass portion), ρ approximately 0,880 g/ml.

5.3.1.3 Potassium hydroxide solution, CAS-No 1310-58-3, c(KOH) = 4 mol/l.

5.3.1.4 Ammonium chloride, CAS-No 12125-02-9.

5.3.1.5 Sodium chloride, CAS-No 7647-14-5.

5.3.1.6 EDTA, standard volumetric solution, CAS-No 60-00-4, c(EDTA) = 0,05 mol/l.

Dissolve 18,612 6 g of disodium ehylenediamine tetraacetate dihydrate (EDTA disodium salt) in water in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix well. Standardize the solution against a standard magnesium solution, using the procedure described in 5.3.3.3.

5.3.1.7 Calcon[1] indicator, (or other appropriate indicator).

Grind 1 part of Calcon, CAS-No 2538-85-4, with 99 parts of anhydrous sodium sulfate, CAS-No 7757-82-6, to a fine powder.

5.3.1.8 Mordant black II (Eriochrome black T)[2] (or other appropriate indicator).

Grind 1 part of Mordant black II, CAS-No 1787-61-7, with 99 parts of sodium chloride, CAS-No 7647-14-5, to a fine powder.

5.3.1.9 Indicator paper, pH 9,5 to pH 13.

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[1] Calcon is a registered trade name for sodium-1-(2-hydroxy-1-naphthylazo)-2-naphthyl-4-sulfonate. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named.

[2] Mordant black II and Eriochrome black T are registered trade names for sodium-1-(1-hydroxy-2-naphthylazo)-6-nitronaphthalene-4-sulfonate. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named.

5.3.2 Apparatus

Use ordinary laboratory apparatus and glassware complying with the requirements of ISO 385, ISO 648, ISO 1042 and ISO 3819.

5.3.3 Procedure

5.3.3.1 Preparation of filtrate

Weigh, to the nearest 0,1 mg, about 0,5 g of the test sample, previously dried in accordance with ISO 787-2, into a beaker. Moisten with a little water, add 7 ml of the hydrochloric acid (5.3.1.1), cover the beaker with a watch glass and boil for few minutes until dissolution is complete. Dilute to 100 ml.

Add 3 g of the ammonium chloride (5.3.1.4) and neutralize with ammonia solution (5.3.1.2). Then add 1 ml excess of the ammonia solution to precipitate iron and aluminium. Boil, filter rapidly, and wash the precipitate with hot water. Collect the filtrate and washings, acidify slightly, and dilute to 500 ml in a one-mark volumetric flask.

5.3.3.2 Determination of calcium content

5.3.3.2.1 Titration

By means of a pipette, transfer 100 ml of the filtrate obtained in 5.3.3.1 to a 400 ml beaker. Dilute to about 250 ml, and add approximately 10 ml of the potassium hydroxide solution (5.3.1.3) in order to obtain a pH value of between 12 and 13. Add 0,2 g to 0,4 g of the Calcon indicator (5.3.1.7). If the calcium carbonate content of pure calcium carbonate is being determined, add a small amount of a soluble magnesium salt at this stage to intensify the colour of the solution during the subsequent titration.

Titrate with the EDTA solution (5.3.1.6) with constant stirring until the colour changes from wine-red to clear blue. Record the volume of EDTA solution used (V_1).

5.3.3.2.2 Expression of results

Calculate the calcium content, expressed as a mass portion in per cent of calcium carbonate, $w(\text{CaCO}_3)$, using Formula (2):

$$w\left(\text{CaCO}_3\right)=\frac{50{,}04\times V_1\times T}{m} \qquad (2)$$

where

V_1 is the volume, in millilitres, of the EDTA solution (5.3.1.6) used;

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

T is the exact concentration, in moles per litre, of the EDTA solution (5.3.1.6).

5.3.3.3 Determination of magnesium content

NOTE By titration of the filtrate obtained in 5.3.3.1 against the same EDTA solution as used in 5.3.3.2.1 for calcium but using Mordant black II as indicator and a different pH value, the magnesium and calcium contents can be determined together. The magnesium content is then determined by subtraction of the calcium content.

5.3.3.3.1 Titration

By means of a pipette, transfer 100 ml of the filtrate obtained in 5.3.3.1 to a 400 ml beaker and dilute with water to about 250 ml. Add ammonia solution (5.3.1.2) to adjust the pH value to between 10 and 11 (up to 20 ml may be required).

Titrate with the EDTA solution (5.3.1.6). Add the Mordant black II indicator (5.3.1.8) towards the end of the titration. The end-point is reached when the colour changes from wine-red to blue. Record the volume of EDTA solution used (V_2).

NOTE The change in colour is not so marked as for the titration of calcium compounds using Calcon as indicator.

5.3.3.3.2 Expression of results

Calculate the magnesium content, expressed as a mass portion in per cent of magnesium carbonate, $w(\text{MgCO}_3)$, using Formula (3):

$$w(\text{MgCO}_3)=\frac{42{,}16(V_2-V_1)T}{m} \tag{3}$$

where

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

V_1 is the volume, in millilitres, of the EDTA solution (5.3.1.6) used in 5.3.3.2.1;

V_2 is the volume, in millilitres, of the EDTA solution (5.3.1.6) added in 5.3.3.3.1;

T is the exact concentration, in moles per litre, of the EDTA solution (5.3.1.6).