INTERNATIONAL STANDARD

ISO 3262-1

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Extenders for paints — Specifications and methods of test —

Part 1:
Introduction and general test methods

Matières de charge pour peintures — Spécifications et méthodes d'essai —
Partie 1: Introduction et méthodes d'essai générales

Reference number
ISO 3262-1:1997(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 3262-1 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 part 2 and the following parts 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 - Specifications and methods of test

- Part 1: Introduction and general test methods

- Part 2: Baryte (natural barium sulfate)

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- *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)*

Extenders for paints — Specifications and methods of test —

Part 1:
Introduction and general test methods

1 Scope

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

The subsequent parts of ISO 3262 specify requirements and the corresponding methods of test for extenders for use in paints and related coating materials. These parts may also be suitable for the various materials covered by the standard when they are used for other applications.

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 385-1: 1984, *Laboratory glassware - Burettes - Part 1: General requirements.*

ISO 648: 1977, *Laboratory glassware - One-mark pipettes.*

ISO 787-2: 1981, *General methods of test for pigments and extenders - Part 2: Determination of matter volatile at 105 °C.*

ISO 842: 1984, *Raw materials for paints and varnishes - Sampling.*

ISO 1042: 1983, *Laboratory glassware - One-mark volumetric flasks.*

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 extender: A material in granular or powder form, practically insoluble in the application medium and used as a constituent of paints to modify or influence certain physical properties.

4 Sampling

Take a representative sample of the product to be tested, as described in ISO 842.

5 Test methods¹)

5.1 Comparison of colour

NOTE 1 There is no objection to the use of a particular instrumental method, agreed between the interested parties, for comparison of colour. 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 7724-5, *Paints and varnishes - Colorimetry - Part 5: Determination of chroma and hue of near-white paint films.*²)

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.

Add carefully 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, and repeat the colour comparison by the same method as before.

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¹) For particular test methods for individual extenders, see the appropriate parts of ISO 3262.

²) To be published. (At present ISO/DIS 11958).

5.2 Determination of loss on ignition[3]

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 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 (1000 ± 25) °C to constant mass, and cool immediately in a desiccator containing a desiccant such as phosphorus pentoxide.

5.2.3 Expression of results

Calculate the loss on ignition, *LI*, expressed as a percentage by mass, using the equation

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

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.

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[3] Determination of loss on ignition of aluminium hydroxides in accordance with ISO 3262-16.

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, $c(\mathrm{HCl}) = 6$ mol/l.

5.3.1.2 Ammonia solution, approximately 32 % (m/m), $\rho \approx 0{,}880$ g/ml.

5.3.1.3 Potassium hydroxide solution, $c(\mathrm{KOH}) = 4$ mol/l.

5.3.1.4 Ammonium chloride.

5.3.1.5 Sodium chloride.

5.3.1.6 EDTA, standard volumetric solution, $c(\mathrm{EDTA}) = 0{,}05$ mol/l.

Dissolve 18,6126 g of disodium ethylenediamine tetraacetate dihydrate (EDTA disodium salt) in water in a 1000 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[4]**) indicator** (or other appropriate indicator).

Grind 1 part of Calcon with 99 parts of anhydrous sodium sulfate to a fine powder.

5.3.1.8 Mordant black II (Eriochrome Black T)[5]) (or other appropriate indicator).

Grind 1 part of Mordant black II with 99 parts of sodium chloride to a fine powder.

5.3.1.9 Indicator paper, pH 9,5 to 13.

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[4]) Calcon is a registered trade name for sodium-1-(2-hydoxy-1-naphthylazo)-2-naphthyl-4-sulfonate.

[5]) Mordant black II and Eriochrome black T are registered trade names for sodium-1-(1-hydroxy-2-naphthylazo)-6-nitronaphthalene-4-sulfonate.

5.3.2 Apparatus

Use ordinary laboratory apparatus and glassware complying with the requirements of ISO 385-1, ISO 648 and ISO 1042.

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 a 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 percentage of mass of calcium carbonate, $w(\mathrm{CaCO_3})$, using the equation

$$w(CaCO_3) = \frac{50{,}04 \times V_1 \times T}{m}$$

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 mol/l, of the EDTA solution (5.3.1.6).

5.3.3.3 Determination of magnesium content

NOTE 2 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 may 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 3 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 percentage by mass of magnesium carbonate, $w(\mathrm{MgCO_3})$, using the equation

$$w(MgCO_3) = \frac{42,16\,(V_2 - V_1)\,T}{m}$$

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 mol/l, of the EDTA solution (5.3.1.6).

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