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Rubber compounding ingredients — Magnesium oxide — Methods of test

Ingrédients de mélange du caoutchouc — Oxyde de magnésium — Méthodes d'essai

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Foreword

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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).

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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 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This second edition cancels and replaces the first edition (ISO 21869:2006), which has been technically revised.

The main changes are as follows:

— a new Clause 6, Loss on ignition, and a new Clause 7, Magnesium oxide content, have been added;

— information on the determination of copper and manganese content have been moved to Annex B;

— a 75 µm sieve opening has been added as an alternative in 9.2;

— a new Clause 12, Ash of hydrochloric acid-insoluble matter, Clause 13, Water–soluble matter content and Clause 14, Bulk density have been added;

— a new Annex A, Determination of calcium oxide content, has been added;

— high, medium and low activity for α, β, and γ, respectively, have been specified in Annex B.

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.

Introduction

Magnesium oxide is used in the rubber industry as a stabilizer, as an agent for modifying the vulcanization process and to enhance the heat resistance of rubber articles. The performance of magnesium oxide in these roles is dependent on its particle size, surface properties and purity. This document specifies the methods used to determine these properties.

Rubber compounding ingredients — Magnesium oxide — Methods of test

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices.

1 Scope

This document specifies the test methods to be used for magnesium oxide intended for use in the rubber industry as a stabilizer and vulcanizing agent.

The choice of the properties to be determined and the values required are subject to agreement between the interested parties.

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 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

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

ISO 3819, *Laboratory glassware — Beakers*

ISO 4652, *Rubber compounding ingredients — Carbon black — Determination of specific surface area by nitrogen adsorption methods — Single-point procedures*

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

ISO 18852, *Rubber compounding ingredients — Determination of multipoint nitrogen surface area (NSA) and statistical thickness surface area (STSA)*

3 Terms and definitions

No terms and definitions are listed in this document.

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 https://www.electropedia.org/

4 Sampling

Sampling shall be carried out in accordance with ISO 15528.

5 Moisture, magnesium hydroxide and magnesium carbonate content

Two methods are included: thermogravimetry and oven heating.

5.1 Thermogravimetry

5.1.1 Procedure

The tests are performed on a thermogravimetric analyser capable of controlling temperature at 105 °C ± 10 °C, 390 °C ± 20 °C and 750 °C ± 50 °C.

The tests are performed in either an air or a nitrogen flow of 100 cm^3/min ± 20 cm^3/min. The temperature increase rate should be between 20 °C/min and 40 °C/min while the temperature sweep shall go from ambient to 800 °C.

5.1.2 Expression of the results

5.1.2.1 Moisture content (mass loss from ambient to 105 °C)

The moisture content, M, is given by Formula (1):

$$M = \frac{(m_1 - m_2)}{m_1} \times 100 \quad (1)$$

where

M is the moisture content, in mass fraction %;

m_1 is the mass of the test portion, in grams;

m_2 is the mass after heating to within the 95 °C to 115 °C temperature range, in grams.

5.1.2.2 Magnesium hydroxide content (mass loss from 105 °C to 390 °C)

The magnesium hydroxide content, w_h, is given by Formula (2):

$$w_h = 3{,}2 \times \frac{(m_2 - m_3)}{m_1} \times 100 \quad (2)$$

where

w_h is the magnesium hydroxide content, in mass fraction %;

m_1 as defined in 5.1.2.1;

m_2 as defined in 5.1.2.1;

m_3 is the mass after heating to within the 370 °C to 410 °C temperature range, in grams;

3,2 is the ratio between 58, the molecular mass of magnesium hydroxide, and 18, the molecular mass of water, calculated on the basis of the following reaction:

$Mg(OH)_2 \Rightarrow MgO + H_2O$

5.1.2.3 Magnesium carbonate content (mass loss from 390 °C to 750 °C)

The magnesium carbonate content, w_c, is given by Formula (3):

$$w_c = 1{,}9 \times \frac{(m_3 - m_4)}{m_1} \times 100 \quad (3)$$

where

w_c is the magnesium carbonate content, in mass fraction %;

m_1 as defined in 5.1.2.1;

m_3 as defined in 5.1.2.2;

m_4 is the mass after heating to above 700 °C, in grams;

1,9 is the ratio between 84, the molecular mass of magnesium carbonate, and 44, the molecular mass of carbon dioxide, calculated on the basis of the following reaction:

$MgCO_3 \Rightarrow MgO + CO_2$

5.2 Loss in mass on oven heating

5.2.1 Moisture content

5.2.1.1 Apparatus

5.2.1.1.1 Weighing dish, low form, approximately 70 mm diameter and 30 mm high (tared).

5.2.1.1.2 Oven, capable of controlling temperature at 115 °C ± 10 °C.

5.2.1.1.3 Analytical balance, accurate to 0,1 mg.

5.2.1.2 Procedure

Weigh into the tared weighing dish 5 g of magnesium oxide sample to the nearest 1 mg.

Spread the test portion to form an even layer in the bottom of the weighing dish. Place the dish, without its cover, in the oven with the temperature previously set at 115 °C ± 10 °C and dry to constant mass (to the nearest 1 mg).

On removal from the oven, always place the cover on the weighing dish. Allow to cool in a desiccator and weigh, the mass loss represents the moisture content.

5.2.1.3 Expression of the results

The moisture content, ω_m, is given by Formula (4):

$$\omega_m = \left(\frac{\Delta m_1}{m_{01}} \right) \times 100 \quad (4)$$

where

ω_m is the moisture content, in mass fraction %;

Δm_1 is the mass loss after heating, in grams;

m_{01} is the original mass of the test portion, in grams.

5.2.2 Magnesium hydroxide content

5.2.2.1 Apparatus

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

5.2.2.1.2 Furnace, capable of reaching 450 °C ± 20 °C.

5.2.2.1.3 Analytical balance, accurate to 0,1 mg.

5.2.2.1.4 Desiccator, with desiccating agents (silica gel) inside.

5.2.2.2 Procedure

Weigh into the tared crucible 2 g of magnesium oxide sample to the nearest 1 mg.

Place the crucible containing the magnesium oxide sample in the furnace and set at 390 °C ± 20 °C.

If a porcelain crucible is used, raise the temperature gradually. When 390 °C is reached, maintain it for 2 h in an oxidative atmosphere. Remove the crucible from the furnace, allow to cool in a desiccator and weigh.

Repeat the calcination to verify that a constant mass has been reached.

It is preferable to allow a porcelain crucible to cool slowly in the furnace before placing it in the desiccator.

The mass loss represents the moisture plus magnesium hydroxide content.

5.2.2.3 Expression of the results

The magnesium hydroxide content, ω_h, is given by Formula (5):

$$\omega_h = 3,2 \times \left[\frac{\Delta m_2}{m_{02}} \times 100 - \omega_m \right] \quad (5)$$

where

ω_h is the magnesium hydroxide content, in mass fraction %;

Δm_2 is the loss in mass after heating to 390 °C, in grams;

m_{02} is the original mass of the test portion, in grams;

ω_m is the moisture content (determined as specified in 5.2.1.3), in mass fraction %;

3,2 is the ratio between 58, the molecular mass of magnesium hydroxide, and 18, the molecular mass of water, calculated on the basis of the following reaction:

$Mg(OH)_2 \Rightarrow MgO + H_2O$

5.2.3 Magnesium carbonate content

5.2.3.1 Apparatus

5.2.3.1.1 Crucible (tared), platinum or porcelain.

5.2.3.1.2 **Furnace**, capable of reaching over 700 °C.

5.2.3.1.3 **Analytical balance**, accurate to 0,1 mg.

5.2.3.1.4 **Desiccator**, with desiccating agents (silica gel) inside.

5.2.3.2 Procedure

Weigh into the tared crucible 2 g of magnesium oxide sample to the nearest 1 mg.

Place the crucible containing the magnesium oxide sample in a furnace and set at over 700 °C.

If a porcelain crucible is used, raise the temperature gradually. When 700 °C is reached maintain it for 2 h in an oxidative atmosphere. Remove the crucible from the furnace, allow to cool in a desiccator and weigh.

Repeat the calcination to verify that a constant mass has been reached.

The mass loss represents the moisture plus magnesium hydroxide plus magnesium carbonate content.

5.2.3.3 Expression of the results

The magnesium carbonate content, w_c, is given by Formula (6):

$$w_c = 1,9 \times \left[\left(\frac{\Delta m_3}{m_{03}} \right) \times 100 - \omega_m - \omega_h \right] \quad (6)$$

where

w_c is the magnesium carbonate content, in mass fraction %;

Δm_3 is the loss in mass after heating to over 700 °C, in grams;

m_{03} is the original mass of the test portion, in grams;

ω_m is the moisture content (determined as specified in 5.2.1.3), in mass fraction %;

ω_h is the magnesium hydroxide content (determined as specified in 5.2.2.3), in mass fraction %;

1,9 is the ratio between 84, the molecular mass of magnesium carbonate, and 44, the molecular mass of carbon dioxide, calculated on the basis of the following reaction:

$MgCO_3 \Rightarrow MgO + CO_2$

6 Loss on ignition

6.1 Principle

Ignite a test portion at 900 °C to 1 000 °C for more than 2 h and measure the amount of loss. The test portion after ignition is used for the measurement of magnesium oxide content in Clause 7.

NOTE Although loss on ignition measurement is one of the steps in the magnesium oxide content test procedure, it is described in an independent clause in consideration for its importance as an index for burning degree in a manufacturing calcination process.

6.2 Apparatus

6.2.1 **Analytical balance**, accurate to 0,1 mg.

6.2.2 Crucible, platinum or porcelain, nominal capacity of 15 cm^3.

6.2.3 Desiccator, with desiccating agents (silica gel) inside.

6.2.4 Electric furnace, capable of controlling the temperature accurately to within ±25 °C in the range of 900 °C to 1 000 °C.

6.3 Test procedure

The procedure is as follows:

a) Ignite the crucible (6.2.2) for 30 min at the ignition temperature for testing.

b) Allow the crucible to cool to room temperature in the desiccator (6.2.3) and weigh the mass of the crucible to the nearest 0,1 mg. Record the mass as m_5.

c) Take a test portion of the sample and put it into the crucible up to about 2/3 of its capacity and weigh the mass of the crucible including the test portion to the nearest 0,1 mg. Record the mass as m_6.

NOTE The mass of the test portion to be weighed in this procedure, which differs according to the bulk density, is approximately 2 g to 3 g.

d) Place the crucible in an electric furnace (6.2.4) and ignite at a selected temperature between 900 °C and 1 000 °C. Recommendation for the ignition time at the temperature between 900 °C and 1 000 °C is more than 2 h.

e) Remove the crucible from the electric furnace, allow to cool in a desiccator and weigh the mass to the nearest 0,1 mg. Record the mass as m_7.

f) Preserve the test portion in the desiccator to prevent moisture absorption and use it in the measurement specified Clause 7.

6.4 Calculation

Calculate loss on ignition, I, using Formula (7), with the result rounded to one decimal place.

$$I = \frac{m_6 - m_7}{m_6 - m_5} \times 100 \quad (7)$$

where

m_5 is the mass of the crucible, in grams;

m_6 is the mass of the crucible including the test portion before ignition, in grams;

m_7 is the mass of the crucible including the test portion after ignition, in grams.

7 Magnesium oxide content

7.1 Principle

The test portion prepared in 6.3 f) after the measurement of loss on ignition in Clause 6 is dissolved in hydrochloric acid. The solution is titrated with disodium dihydrogen ethylenediamine tetraacetate dihydrate (EDTA) aqueous solution and the total content of magnesium oxide and calcium oxide corresponding to the amount of EDTA titration is determined. Then, the calcium oxide content is determined by atomic absorption spectrometry (AAS) or inductively coupled plasma atomic emission spectrometry (ICP-AES), and converted into the titration volume of EDTA corresponding to the content.