INTERNATIONAL STANDARD

ISO 21869

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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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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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.

ISO 21869 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*.

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 International Standard specifies the methods used to determine these properties. It is based on NF T 45-006 (France).

Rubber compounding ingredients — Magnesium oxide — Methods of test

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard 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 and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard 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 shall be agreed between the interested parties.

2 Normative references

The following referenced documents are indispensable for the application 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 3819, *Laboratory glassware — Beakers*

ISO 4652-1, *Rubber compounding ingredients — Carbon black — Determination of specific surface area by nitrogen adsorption methods — Part 1: 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 Sampling

Sampling shall be carried out in accordance with ISO 15528.

4 Moisture, magnesium hydroxide and magnesium carbonate content

Two methods are included: thermogravimetry and oven heating.

4.1 Thermogravimetry

4.1.1 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.

4.1.2 Expression of results

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

$$\text{Moisture content (\%)} = 100 \times (m_1 - m_2)/m_1$$

where

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.

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

$$Mg(OH)_2 \text{ content (\%)} = 100 \times 3{,}2(m_2 - m_3)/m_1$$

where

m_1 and m_2 are as defined in 4.1.2.1;

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:

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

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

$$MgCO_3 \text{ content (\%)} = 100 \times 1{,}9(m_3 - m_4)/m_1$$

where

m_1 is as defined in 4.1.2.1;

m_3 is as defined in 4.1.2.2;

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:

$$MgCO_3 \Rightarrow MgO + CO_2$$

4.1.2.4 Precision

The repeatability of the mass measurements is ± 2 %.

4.2 Loss in mass on heating

4.2.1 Moisture content

4.2.1.1 Apparatus

4.2.1.1.1 Weighing dish, low form, approx. 70 mm diameter and 30 mm high (tared).

4.2.1.1.2 Oven, controlled at 115 °C ± 10 °C.

4.2.1.1.3 Analytical balance, accurate to 0,1 mg.

4.2.1.2 Procedure

Weigh into the tared weighing dish 5 g of magnesium oxide 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. Weigh.

The mass loss represents the moisture content.

4.2.1.3 Expression of results

The moisture content is given by the equation:

$$\text{Moisture content (\%)} = \omega_m = 100(\Delta m_1/m_{01})$$

where

Δm_1 is the mass loss after heating, in grams;

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

4.2.2 Magnesium hydroxide content

4.2.2.1 Apparatus

4.2.2.1.1 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.)

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

4.2.2.1.3 Analytical balance, accurate to 0,1 mg.

4.2.2.2 Procedure

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

Place the crucible containing the magnesium oxide in the furnace 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 was 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.

4.2.2.3 Expression of results

The magnesium hydroxide content is given by the equation:

$$Mg(OH)_2\ (\%) = 3{,}2 \times [100(\Delta m_2/m_{02}) - \omega_m] = \omega_h$$

where

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 4.2.1), 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:

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

4.2.3 Magnesium carbonate content

4.2.3.1 Apparatus

4.2.3.1.1 Crucible (tared), platinum or porcelain. (If a porcelain crucible is used, it shall be heated to over 700 °C and cooled in a desiccator before the test.)

4.2.3.1.2 Furnace, capable of reaching over 700 °C.

4.2.3.1.3 Analytical balance, accurate to 0,1 mg.

4.2.3.2 Procedure

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

Place the crucible containing the magnesium oxide in a furnace 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 was 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 plus magnesium carbonate content.

4.2.3.3 Expression of results

The magnesium carbonate content is given by the equation:

$$MgCO_3\ (\%) = 1{,}9 \times [100(\Delta m_3/m_{03}) - \omega_m - \omega_h]$$

where

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 4.2.1), in grams;

ω_h is the magnesium hydroxide content (determined as specified in 4.2.2), 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:

$$MgCO_3 \Rightarrow MgO + CO_2$$

4.2.4 Precision

With an analytical balance accurate to 0,1 mg for 5 g, the repeatability of the mass measurements can be considered as ± 1 %.

5 Determination of the specific surface area

The determination of the specific surface area shall be by the method described in ISO 4652-1 or ISO 18852.

6 Determination of the copper and manganese content

6.1 Principle

A test portion is dissolved in hydrochloric acid and the resulting solution is analysed using atomic absorption or atomic emission spectrometry. Any silicates that may be present are removed using hydrofluoric and sulfuric acids.

6.2 Reagents

During the analysis, unless stated otherwise, use only analytical-quality reagents and distilled water or water of equivalent purity.

6.2.1 Hydrochloric acid, ρ_{20} = 1,19 Mg/m^3.

6.2.2 Hydrochloric acid, diluted 1+2.

Dilute 1 volume of hydrochloric acid (6.2.1) with 2 volumes of water.

6.2.3 Sulfuric acid, ρ_{20} = 1,84 Mg/m^3.

6.2.4 Sulfuric acid, diluted 1+3.

Pour carefully 1 volume of concentrated sulfuric acid (6.2.3) into 3 volumes of water.