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**Rubber compounding ingredients—
Magnesium oxide—Methods of test**

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Foreword

This Japanese Industrial Standard has been established by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee according to the proposal for establishment of Japanese Industrial Standard submitted by The Japan Rubber Manufacturers Association (JRMA)/Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law.

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Rubber compounding ingredients— Magnesium oxide—Methods of test

Introduction

This Japanese Industrial Standard has been prepared based on **ISO 21869:2006**, Edition 1, with some additions and modifications of the technical contents to appropriately evaluate the quality of magnesium oxide used as a rubber compounding ingredient.

The vertical lines on both sides and dotted underlines indicate changes from the corresponding International Standard. A list of modifications with the explanations is given in Annex JD.

1 Scope

This Standard specifies the test methods for physical and chemical properties of magnesium oxide used as a rubber compounding ingredient.

NOTE : The International Standard corresponding to this Standard and the symbol of degree of correspondence are as follows.

ISO 21869:2006 *Rubber compounding ingredients—Magnesium oxide—Methods of test* (MOD)

In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standard and **JIS** are IDT (identical), MOD (modified), and NEQ (not equivalent) according to **ISO/IEC Guide 21-1**.

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

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS K 0050 *General rules for chemical analysis*

JIS K 0116 *General rules for atomic emission spectrometry*

JIS K 0121 *General rules for atomic absorption spectrometry*

JIS K 0211 *Technical terms for analytical chemistry (General part)*

JIS K 5600-1-2 *Testing methods for paints—Part 1: General rules—Section 2: Sampling*

NOTE : Corresponding International Standard: ISO 15528 *Paints varnishes and raw materials for paints and varnishes—Sampling* (IDT)

JIS K 8005 Reference materials for volumetric analysis

JIS K 8034 Acetone (Reagent)

JIS K 8085 Ammonia solution (Reagent)

JIS K 8107 Disodium dihydrogen ethylenediamine tetraacetic acid dihydrate (Reagent)

JIS K 8116 Ammonium chloride (Reagent)

JIS K 8180 Hydrochloric acid (Reagent)

JIS K 8201 Hydroxylammonium chloride (Reagent)

JIS K 8432 Magnesium oxide (Reagent)

JIS K 8576 Sodium hydroxide (Reagent)

JIS K 8617 Calcium carbonate (Reagent)

JIS K 8736 Eriochrome black T (Reagent)

JIS K 8891 Methanol (Reagent)

JIS P 3801 Filter paper (for chemical analysis)

JIS R 1301 Porcelain crucibles for chemical analysis

JIS R 3505 Volumetric glassware

JIS Z 8401 Guide to the rounding of numbers

JIS Z 8801-1 Test sieves—Part 1: Test sieves of metal wire cloth

3 Terms and definitions

For the purpose of this Standard, the terms and definitions given in **JIS K 0211** apply.

4 Common requirements

4.1 General requirements

The general requirements common to the chemical analysis shall be in accordance with **JIS K 0050**.

Since magnesium oxide is hygroscopic, the sample shall be handled with care not to allow it to absorb moisture.

4.2 Sampling

Sampling shall be carried out in accordance with **JIS K 5600-1-2**.

4.3 Properties and test methods

The properties of magnesium oxide for rubber shall be measured by test methods indicated in Table 1. Unless otherwise specified, the number (n) of tests conducted by each test method shall be one ($n = 1$). Tests may be carried out multiple times, if necessary.

Table 1 Properties of magnesium oxide for rubber and test methods

Property	Test method	Clause
Ignition loss [mass fraction (%)]	Ignition loss method at 900 °C to 980 °C	5
Magnesium oxide content [mass fraction (%)]	EDTA titrimetry	6
Loss on heating [mass fraction (%)]	Loss-on-heating method at 105 °C	7
Specific surface area (m ² /g)	Single point nitrogen adsorption method	8
Residue on sieve [mass fraction (%)]	Wet method	9
Hydrochloric acid-insoluble matter [mass fraction (%)]	Acid dissolution method	10
Water-soluble matter [mass fraction (%)]	Water dissolution method	11
Bulk density (g/ml)	Constant mass method utilizing bulk density measurement apparatus	12

5 Ignition loss

5.1 Outline

Ignite the sample at 900 °C to 980 °C and measure the amount of loss.

The ignited sample shall be used for the measurement in Clause 6.

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

5.2 Instrument and apparatus

The instrument and apparatus shall be as follows.

- Balance**, capable of weighing to the nearest 0.1 mg.
- Crucible**, as specified in **JIS R 1301** and of Type B with a nominal capacity of 15 ml.
- Desiccator**, with desiccating agents (silica gel) inside.
- Electric furnace**, adjustable to a temperature within the range of 900 °C to 980 °C with a precision of ± 25 °C.

5.3 Test procedure

The test procedure shall be as follows.

- Ignite a crucible for 30 min at the ignition temperature for testing.
- Allow the crucible to cool to room temperature in the desiccator and weigh the mass of the crucible to the nearest 0.1 mg. Take the mass as m_1 .
- Put the sample into the crucible up to about 2/3 of its capacity and weigh the mass to the nearest 0.1 mg. Take the mass as m_2 .

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

- d) Put the crucible in an electric furnace and ignite it at a temperature selected within the range of 900 °C to 980 °C. The standard ignition time at 900 °C is 3 h, and that at 980 °C is 2 h.
- e) Allow the crucible ignited to cool to room temperature in the desiccator and weigh the mass to the nearest 0.1 mg. Take the mass as m_3 .
- f) Preserve the sample ignited in the desiccator to prevent moisture absorption and use it in the measurement specified in Clause 6.

5.4 Calculation

Calculate the ignition loss by Formula (1). Round off the result to one decimal place in accordance with **JIS Z 8401**.

$$I = \frac{m_2 - m_3}{m_2 - m_1} \times 100 \dots\dots\dots (1)$$

where, I : ignition loss [mass fraction (%)]

m_1 : mass of crucible (g)

m_2 : mass of sample and crucible before ignition (g)

m_3 : mass of sample and crucible after ignition (g)

5.5 Test report

The test report shall include the following items:

- a) historical records of the sample;
- b) title and number of this Standard;
- c) test conditions (ignition temperature and ignition time);
- d) test results;
- e) details of tests including operations not specified in this Standard;
- f) test date.

6 Magnesium oxide content

6.1 Outline

Dissolve the sample, of which the ignition loss has been measured according to Clause 5, with hydrochloric acid. Titrate the solution with disodium dihydrogen ethylenediamine tetraacetate dihydrate (EDTA) aqueous solution and determine the titration volume of EDTA based on the total sum of magnesium oxide and calcium oxide content. Then, determine the calcium oxide content by atomic absorption spectrometry (AAS) or inductively coupled plasma atomic emission spectrometry (ICP-AES), and convert it into the titration volume of EDTA corresponding to the content. Further, subtract this volume from the titration volume of EDTA corresponding to the total sum of magnesium oxide and calcium oxide contents, and calculate the magnesium oxide content after ignition.

NOTE : As MgO content is nearly 100 %, the solution needs to be diluted to a significant extent when measured by AAS or ICP-AES. This lowers analytical precision. Moreover, the measurement result by AAS or ICP-AES is expressed to about two significant figures; and therefore, while the sensitivity is high, the precision is low compared to the titration method (about three significant figures). Due to the reasons above, the calcium content to be simultaneously detected by the titration method is determined using the highly sensitive measurement method.

6.2 Total sum of magnesium oxide and calcium oxide contents

6.2.1 Reagents

The reagents shall be as follows.

- a) **Water**, as specified in Annex D of **JIS K 0050**.
- b) **Hydrochloric acid (reagent)**, as specified in **JIS K 8180**.
- c) **Hydrochloric acid (1+1)** Add 1 volume of water to 1 volume of hydrochloric acid to dilute.
- d) **Sodium hydroxide (reagent)**, as specified in **JIS K 8576**.
- e) **40 g/L sodium hydroxide aqueous solution** Weigh out about 4 g of sodium hydroxide, add about 50 ml of water to dissolve and further add water to make total 100 ml.
- f) **0.8 g/L sodium hydroxide aqueous solution** Dilute 40 g/L sodium hydroxide aqueous solution 50-fold with water.
- g) **28 % mass fraction aqueous ammonia (reagent)**, as specified in **JIS K 8085**.
- h) **Ammonium chloride (reagent)**, as specified in **JIS K 8116**.
- i) **pH 10.7 ammonia-ammonium chloride buffer solution** Add water to 67.5 g of ammonium chloride to dissolve, add 570 ml of 28 % mass fraction aqueous ammonia and further add water to make total 1 000 ml.
- j) **Eriochrome black T (EBT) (reagent)**, as specified in **JIS K 8736**.
- k) **Hydroxylammonium chloride (reagent)**, as specified in **JIS K 8201**.
- l) **Methanol (reagent)**, as specified in **JIS K 8891**.
- m) **EBT indicator** Dissolve 0.6 g of EBT and 4.0 g of hydroxylammonium chloride in 100 ml of methanol. Store in dark place.
- n) **Zinc (reference material for volumetric analysis)**, as specified in **JIS K 8005**.
- o) **Acetone (reagent)**, as specified in **JIS K 8034**.
- p) **0.01 mol/L zinc aqueous solution**
 - 1) **Preparation** Wash zinc with diluted hydrochloric acid and then with water and acetone, and dry. Weigh out about 0.65 g of dry zinc to the nearest 0.1 mg and take the zinc mass as m_1 . Dissolve completely with 5 ml of hydrochloric acid (1+1) and add water to make total 1 000 ml.

- 2) **Calculation** Calculate the factor (f_1) of 0.01 mol/L zinc aqueous solution by Formula (2). Round off the result to three decimal places in accordance with **JIS Z 8401**. Take the factor of 0.01 mol/L zinc aqueous solution calculated above as f_1 in Formula (3).

$$f_1 = \frac{m_4}{0.6538} \times \frac{Z}{100} \dots\dots\dots (2)$$

where, f_1 : factor of 0.01 mol/L zinc aqueous solution
 m_4 : mass of zinc (g)
 Z : purity of zinc [mass fraction (%)]
0.6538 : mass of zinc contained in 1 000 ml of 0.01 mol/L zinc aqueous solution (g)

- q) **Disodium dihydrogen ethylenediamine tetraacetate dihydrate (EDTA) (reagent)**, as specified in **JIS K 8107**.

r) **0.01 mol/L EDTA aqueous solution**

- 1) **Preparation** Weigh out about 3.72 g of EDTA, dissolve in water and add water to make total 1 000 ml. Put it into an airtight container made of resin such as polyethylene to store.
- 2) **Standardization** Take 20 ml of 0.01 mol/L zinc aqueous solution in p) with a volumetric pipette, add 80 ml of water and then adjust the pH to about 7 with 0.8 g/L sodium hydroxide aqueous solution. Add 2 ml of ammonia-ammonium chloride buffer solution of pH 10.7 to this adjusted aqueous solution, titrate with 0.01 mol/L EDTA aqueous solution prepared in 1) using EBT indicator, and determine the equivalence point (when reddish violet turns blue not tinged with red).
- 3) **Calculation** Calculate the factor (f_2) of 0.01 mol/L EDTA aqueous solution by Formula (3). Round off the result to three decimal places in accordance with **JIS Z 8401**. Take the factor of 0.01 mol/L EDTA aqueous solution calculated above as f_2 in Formula (8).

$$f_2 = \frac{f_1 \times 20}{V} \dots\dots\dots (3)$$

where, f_2 : factor of 0.01 mol/L EDTA aqueous solution
 f_1 : factor of 0.01 mol/L zinc aqueous solution
 V : titration volume of 0.01 mol/L EDTA aqueous solution required for standardization (ml)

6.2.2 Instrument and apparatus

The instrument and apparatus shall be as follows.

- a) **Balance**, capable of weighing to the nearest 0.1 mg.
- b) **Volumetric flask**, complying with Class A requirements specified in **JIS R 3505**.
- c) **Volumetric pipette**, complying with Class A requirements specified in **JIS R 3505**.

- d) **Burette**, complying with Class A requirements specified in **JIS R 3505**.

6.2.3 Test procedure

The test procedure shall be as follows.

- a) Weigh out about 500 mg of the sample, of which the ignition loss has been measured according to clause 5, into a beaker to the nearest 0.1 mg. Take the sample mass as m_5 .
- b) Gradually add 10 ml of hydrochloric acid (1+1) to the beaker in a) and heat to dissolve.
- c) After cooling, transfer to a 500 ml volumetric flask, wash the original beaker with a small amount of water, add the washings to the volumetric flask, further add water to make total 500 ml and take this as test solution.
- d) Take 10 ml of the test solution into a beaker with a volumetric pipette, add 80 ml of water and adjust the pH to about 7 with 0.8 g/L sodium hydroxide aqueous solution. Add 2 ml of ammonia-ammonium chloride buffer solution of pH 10.7 to this adjusted aqueous solution, titrate with 0.01 mol/L EDTA aqueous solution with a burette using EBT indicator, and determine the equivalence point (when reddish violet turns blue not tinged with red).
- e) Take the titration volume at this equivalence point as the titration volume equivalent to the total sum of magnesium oxide and calcium oxide content and take this as A in Formula (8).

6.3 Calculation of calcium oxide content

6.3.1 Outline

The following two methods are available to test calcium oxide content.

- a) **Atomic absorption spectrometry (AAS)** With an atomic absorption spectrometer, the calcium concentration is determined by dissociating calcium contained in the sample to ground-state atoms with flame and by measuring the absorbance of the atomic vapour layer.
- b) **Inductively coupled plasma atomic emission spectrometry (ICP-AES)** With an ICP atomic emission spectrometer, the calcium concentration is determined by gasifying and exciting calcium contained in the sample with radio-frequency inductively coupled plasma and by measuring the emission intensity of the atomic spectral line obtained.

6.3.2 Atomic absorption spectrometry (AAS)

6.3.2.1 Reagents

The reagents shall be as follows.

- a) **Water**, as specified in Annex D of **JIS K 0050**.
- b) **Hydrochloric acid (reagent)**, as specified in **JIS K 8180**.

- c) **Hydrochloric acid (1+1)** Add 1 volume of water to 1 volume of hydrochloric acid to dilute.
- d) **Lanthanum oxide**, La_2O_3 (not less than 99.5 % in mass fraction).
- e) **0.2 mol/L lanthanum oxide solution** Dissolve 6.5 g of lanthanum oxide in hydrochloric acid (1+1) and further add hydrochloric acid (1+1) to make total 100 ml.
- f) **Calcium carbonate (reagent)**, as specified in **JIS K 8617**.
- g) **Calcium standard stock solution (1 000 mg Ca/L)** Weigh out about 1.249 g of calcium carbonate, which has been dried at 150 °C to 180 °C for 1 h, to the nearest 0.1 mg, add 110 ml of hydrochloric acid (1+1) to dissolve and add water to make total 500 ml.

When a commercial item is used, it should be traceable to the national measurement standards provided based on Japan Calibration Service System (JCSS) and supplied with a JCSS calibration certificate.

- h) **Calcium standard solution (100 mg Ca/L)** Dilute calcium standard stock solution in g) 10-fold.

When a commercial item is used, it should be traceable to the national measurement standards provided based on Japan Calibration Service System (JCSS) and supplied with a JCSS calibration certificate.

6.3.2.2 Instrument and apparatus

The instrument and apparatus shall be as follows.

- a) **Balance**, capable of weighing to the nearest 0.1 mg.
- b) **Volumetric flask**, complying with Class A requirements specified in **JIS R 3505**.
- c) **Volumetric pipette**, complying with Class A requirements specified in **JIS R 3505**.
- d) **Mohr pipette**, complying with Class A requirements specified in **JIS R 3505**.
- e) **Atomic absorption spectrometer**, as specified in **JIS K 0121**.

6.3.2.3 Test procedure

The test procedure shall be as follows.

- a) Weigh out about 1 g of the sample, of which the ignition loss has been measured according to Clause 5, into a beaker to the nearest 0.1 mg. Take the sample mass as m_6 .
- b) Gradually add 20 ml of hydrochloric acid (1+1) to the beaker in a) and heat to dissolve.
- c) After cooling, transfer to a 100 ml volumetric flask, wash the original beaker with a small amount of water, add the washings to the volumetric flask and further add water to make total 100 ml. Measure out 2.5 ml of this solution into a 25 ml volumetric flask using a Mohr pipette and add 1 ml of 0.2 mol/L lanthanum oxide solution using a volumetric pipette. Further, add water to make total 25 ml and take this as test solution.