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Replacing HG/T 2573-2006

Light Magnesium Oxide for Industrial Use

工业轻质氧化镁

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Table of Contents

Foreword

This Standard was drafted in accordance with the rules in GB/T 1.1-2009.

This Standard serves as a replacement of HG/T 2573-2006 *Light Magnesium Oxide for Industrial Use*. In comparison with HG/T 2573-2006, apart from editorial modifications, there are several main technical changes as follows:

---Type-I Grade-1 sulfate content index is added to the requirements (see 5.2; 4.2 in Version 2006);

---Testing method for magnesium oxide content is modified (see 6.4; 5.3 in Version 2006);

---Testing method for chlorine content determination is modified: the determination of chlorine content is modified into precipitation titration method (see 6.11; 5.10 in Version 2006);

---Testing method for bulk density determination is modified (see 6.13; 5.12 in Version 2006).

The formulation of this Standard adopts the redrafting law and takes Russian standard ГОСТ844-1979 (4th modification since October 1, 1990) *Technical Conditions for Industrial Calcination of Magnesium Oxide* as a reference. The degree of consistency with ГОСТ844-1979 is non-equivalent.

This Standard was proposed by China Petroleum and Chemical Industry Federation.

This Standard shall be under the jurisdiction of Inorganic Chemical Industry Subcommittee of National Technical Committee 63 on Chemical of Standardization Administration of China (SAC/TC63/SC1).

The drafting organizations of this Standard: CenerTech Tianjin Chemical Research and Design Institute Co., Ltd.; Yuncheng Environmental Protection Technology Co., Ltd. Asia-Hing; Shanghai Dunhuang Chemical Factory; Xingtai Magnesium Chemical Co., Ltd.; Wuxi Zehui Chemical Co., Ltd.; Shouguang Huihuang Chemical Co., Ltd.

The main drafters of this Standard: Lixia, Wei Guanya, Shi Zuyu, Sunwen, Mo Yunze, Liwei, Han Jianqiu, Xu Shunjuan, Jiang Chunning, Guo Fengxin.

The issuing of the previous versions of the standard replaced by this Standard is as follows:

---GB 9004-1988.

---HG/T 2573-1994; HG/T 2573-2006.

Light Magnesium Oxide for Industrial Use

1 Scope

This Standard stipulates the classification, requirements, testing methods, inspection rules, marking, labels, packaging, transportation and storage of light magnesium oxide for industrial use.

This Standard is applicable to light magnesium oxide calcinated through basic magnesium carbonate and magnesium hydroxide for industrial use. The product is mainly applied to industries like plastics, rubber, wires, cables, dye, grease, glass ceramics, rubber tires, adhesives, tanning and fuel suppressant, etc.

2 Normative References

The following documents are indispensable to the application of this document. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

GB/T 191-2008 *Packaging - Pictorial Marking for Handling of Goods;*

GB/T 3049-2006 *Chemical Products for Industrial Use - General Method for Determination of Iron Content - 1,10-Phenanthroline Spectrophotometric Method* (idt ISO 6685:1982);

GB/T 6003.1-1997 *Test Sieves of Metal Wire Cloth;*

GB/T 6678 *General Principles for Sampling Chemical Products;*

GB/T 6682-2008 *Water for Analytical Laboratory Use - Specification and Test Methods;*

GB/T 8170 *Rules of Rounding off for Numerical Values & Expression and Judgement of Limiting Values;*

HG/T 3696.1 *Inorganic Chemicals for Industrial Use - Preparations of Standard and Reagent Solutions for Chemical Analysis - Part 1: Preparations of Standard Volumetric Solutions;*

HG/T 3696.2 *Inorganic Chemicals for Industrial Use - Preparations of Standard and Reagent Solutions for Chemical Analysis - Part 2: Preparations of Standard Solutions for Impurity;*

HG/T 3696.3 *Inorganic Chemicals for Industrial Use - Preparations of Standard*

6.4.1 Method summary

Use triethanolamine to conceal a small amount of trivalent iron, trivalent aluminum and divalent manganese plasma. When pH is 10, use chrome black T as the indicator; use ethylene diamine tetra-acetic acid standard volumetric solution to titrate calcium and magnesium content. Subtract calcium content from it; calculate the content of magnesium oxide.

6.4.2 Reagents

6.4.2.1 Hydrochloric acid solution: 1+1.

6.4.2.2 Ammonia solution: 1+1.

6.4.2.3 Triethanolamine solution: 1+3.

6.4.2.4 Ammonia-ammonium chloride buffer solution A (pH ≈ 10).

6.4.2.5 Silver nitrate solution: 10 g/L.

6.4.2.6 Ethylene diamine tetra-acetic acid standard volumetric solution: c (EDTA) ≈ 0.02 mol/L.

6.4.2.7 Chrome black T indicator.

6.4.3 Analytical procedures

6.4.3.1 Preparation of test solution A

Weigh-take around 5 g of sample, accurate to 0.0002 g; place it into a 250 mL beaker. Use a small amount of water to moisten it. Add around 55 mL of hydrochloric acid solution, stir it, till the sample completely dissolves. Put on the watch glass; boil it for 3 min ~ 5 min. While it is still hot, use medium-speed quantitative filter paper to filter it. Use hot water to rinse it, till there is no chloride ion (use silver nitrate solution to examine it). After cooling it down, transfer all the filtrate and rinsing solution into a 500 mL volumetric flask. Use water to dilute to the scale, then, mix it up. This is test solution A; it shall be used for the determination of magnesium oxide content, calcium oxide content, iron content and sulfate content. Retain the filter paper and the residue for the determination of hydrochloric acid insoluble matter content.

6.4.3.2 Determination

Use a transfer pipette to transfer-take 25 mL of test solution A; place it in a 250 mL volumetric flask. Use water to dilute to the scale, then, mix it up. Use a transfer pipette to transfer-take 25 mL of the test solution, then, place it in a 250 mL conical flask. Add 50 mL of water; use ammonia solution to adjust the pH value of the solution into 7 ~ 8 (use pH test paper to examine it). Add 5 mL of triethanolamine solution, 10 mL of ammonia-ammonium chloride buffer solution A, 0.1 g of chrome black T indicator; use

6.7.2.5.1 Determination

Transfer-take a certain amount of test solution A (6.4.3.1) (Type-I: transfer-take 10.00 mL of superior product, 3.30 mL of first-class product; Type-II: transfer-take 4.00 mL of superior product, 2.50 mL of first-class product and 2.00 mL of qualified product). Place it into a 50 mL colorimetric tube; add water to around 20 mL. Use ammonia solution to adjust the solution to neutral (use pH test paper to examine it). Add 1 mL of hydrochloric acid solution and 2 mL of barium chloride solution; add water to the scale, then, mix it up. Place it in water bath at 40 °C ~ 50 °C. After 10 min, compare the turbidity. The turbidity manifested by the test solution shall not be more than the standard turbidity-comparing solution.

6.7.2.5.2 Preparation of standard turbidity-comparing solution

Transfer-take 2.00 mL of sulfate standard solution; place it into a 50 mL colorimetric tube. The following procedures shall comply with the operation described in 6.7.2.5.1: “add water to around 20 mL……after 10 min, compare the turbidity”.

6.8 Determination of Sieve Residue

6.8.1 Method summary

Pour the sample into the sieve. Use soft brush to softly brush it, till no powdered sample passes through the sieve. Then, weigh the sieve residue.

6.8.2 Instruments and equipment

6.8.2.1 Test sieve: R40/3 series, ϕ200 × 50–0.15/0.1 (GB/T 6003.1-1997).

6.8.2.2 Soft brush: brush length: around 3 cm; brush width: around 3 cm ~ 5 cm.

6.8.3 Analytical procedures

Weigh-take around 10 g of sample, accurate to 0.01 g. Transfer it into test sieve; use soft brush to slightly brush the sample, so that powder can pass through the sieve. In the end, place a black paper under the sieve; brush the sieve, till there is no trace of the sample on the black paper. Transfer the sieve residue into a watch glass, whose mass is already known; weigh the sieve residue, accurate to 0.0002 g.

6.8.4 Result calculation

Sieve residue, counted by the mass fraction w_4, expressed in (%), shall be calculated in accordance with Formula (4):

$$w_4 = \frac{m_1 - m_2}{m} \times 100 \quad \cdots\cdots (4)$$

Where,

Iron content, counted by the mass fraction w_5 of iron (Fe), expressed in (%), shall be calculated in accordance with Formula (5):

$$w_5 = \frac{(m_1 - m_0)/1\,000}{m \times (20/500)} \times 100 \cdots\cdots (5)$$

Where,

m_1---in accordance with the measured absorbance of the test solution, obtain the numerical value of the mass of iron through the working curve, expressed in (mg);

m_0---in accordance with the measured absorbance of the blank test solution, obtain the numerical value of the mass of iron through the working curve, expressed in (mg);

m---the numerical value of the mass of sample in 6.4.3.1, expressed in (g).

Take the arithmetic mean value of the parallel determination result as the determination result. The absolute difference of two parallel determination results shall be not more than 0.005%.

6.10 Determination of Manganese Content

6.10.1 Method summary

In strongly acidic medium where phosphate exists, use periodate to oxidize divalent manganese ion into permanganate ion, which is fuchsia. Use spectrophotometer to measure its absorbance at 525 nm.

6.10.2 Reagents

6.10.2.1 Phosphoric acid.

6.10.2.2 Potassium periodate.

6.10.2.3 Nitric acid solution: 1+1.

6.10.2.4 Manganese standard solution: 1 mL of solution contains 0.050 mg of manganese (Mn);

Use transfer pipette to transfer-take 5 mL of manganese standard solution, which is prepared in accordance with HG/T 3696.2. Place it into a 100 mL volumetric flask. Use water to dilute to the scale, then, mix it up. This solution shall be prepared right before usage.

6.10.3 Instruments and equipment

Spectrophotometer: equipped with a cuvette with the optical path of 3 cm.

6.10.4 Analytical procedures

m---the numerical value of the mass of sample, expressed in (g).

Take the arithmetic mean value of the parallel determination result as the determination result. In terms of superior products, the absolute difference of two parallel determination results shall be not more than 0.0005%. In terms of first-class products, the absolute difference of two parallel determination results shall be not more than 0.002%.

6.11 Determination of Chloride Content

6.11.1 Method summary

In slight alkaline medium, use silver nitrate standard volumetric solution for titration. In the sample, chloride ion and silver ion generate white silver chloride precipitation. Excessive silver nitrate and potassium chromate generate brick-red silver chromate precipitation, which indicates the ending point.

6.11.2 Reagents

6.11.2.1 Magnesium sulfate ($MgSO_4 \bullet 7H_2O$).

6.11.2.2 Silver nitrate standard volumetric solution: c ($AgNO_3$) ≈ 0.02 mol/L.

6.11.2.3 Potassium chromate solution: 50 g/L.

6.11.3 Instruments and equipment

Micro-burette: division value: 0.02 mL or 0.05 mL.

6.11.4 Determination

Weigh-take around 2 g of sample, accurate to 0.0002 g; place it into a 200 mL beaker. Add 50 mL of water, 1.0 mL of potassium chromate solution and 0.2 g of magnesium sulfate. After boiling the solution, use silver nitrate standard volumetric solution to titrate it, till slight brick-red emerges.

Meanwhile, conduct blank test. Apart from adding sample, the other operation, and the type and the amount of the added reagents shall be identical to the test solution in the blank test. Moreover, they shall receive the same treatment as the test solution.

6.11.5 Result calculation

Chloride content, counted by the mass fraction w_7 of chlorine (Cl), expressed in (%), shall be calculated in accordance with Formula (7):

$$w_7 = \frac{[(V_1 - V_0)/1\,000]cM}{m} \times 100 \quad \cdots\cdots (7)$$

Where,

V_1---the numerical value of the volume of silver nitrate standard volumetric solution consumed by the titration test solution, expressed in (mL);

V_0---the numerical value of the volume of silver nitrate standard volumetric solution consumed by the titration blank test solution, expressed in (mL);

c---the accurate numerical value of the concentration of silver nitrate standard volumetric solution, expressed in (mol/L);

m---the numerical value of the mass of sample, expressed in (g);

M---the numerical value of the molar mass of chlorine (Cl), expressed in (g/moL) (M = 35.45).

Take the arithmetic mean value of the parallel determination result as the determination result. The absolute difference of two parallel determination results shall be not more than 0.01%.

6.12 Determination of Loss on Ignition

6.12.1 Method summary

At 875 °C ± 25 °C, hydrated basic magnesium carbonate or magnesium hydroxide in the sample transforms into magnesium oxide. Meanwhile, free water is lost. In accordance with the reduced mass of the sample, determine the loss on ignition.

6.12.2 Instruments and equipment

High-temperature furnace: temperature may be controlled at 875 °C ± 25 °C.

6.12.3 Analytical procedures

Weigh-take around 1 g of sample, accurate to 0.0002 g. Place it a porcelain crucible, which is previously ignited at 875 °C ± 25 °C to constant mass. Place it into the high-temperature furnace, at 875 °C ± 25 °C, ignite it, till it reaches constant mass.

6.12.4 Result calculation

Loss on ignition, counted by the mass fraction w_8, expressed in (%), shall be calculated in accordance with Formula (8):

$$w_8 = \frac{m_1 - m_2}{m} \times 100 \quad \cdots\cdots (8)$$

Where,

m_1---the numerical value of the mass of the crucible and the sample before the ignition, expressed in (g);

6.13.2.3 Determination of tank volume

Clean and air-dry the tank. Put on a piece of glass. Weigh the mass of the tank and the piece of glass. Carefully pour water into the tank. When the tank is nearly full, use a burette to add water, till it is full. Then, put on the piece of glass. Use filter paper to suck water outside the tank and the piece of glass. There shall be no bubbles between the piece of glass and the water in the tank. Then, weigh the mass of the tank and the piece of glass.

6.13.2.4 Calculation of tank volume

Tank volume, counted by V, expressed in (mL), shall be calculated in accordance with Formula (9):

$$V=\frac{m_1-m_2}{\rho_{water}} \quad \cdots\cdots (9)$$

Where,

m_1---the numerical value of the mass of the tank, which is filled with water, and the piece of glass, expressed in (g);

m_2---the numerical value of the mass of the tank and the piece of glass, expressed in (g);

ρ_{water}---the numerical value of pure water density under the determination temperature, expressed in (g/mL), approximate to 1 g/mL (20 °C).

6.13.3 Analytical procedures

In accordance with Figure 1, install the bulk density determination device.

Weigh the mass of the tank, accurate to 0.1 g.

Close the lower bottom of the funnel. Naturally fill it up with the sample, which previously passes 150 μm test sieve. Use a straight ruler to scrape the prominent part. Properly place the tank, whose mass is already known. Open the lower bottom of the funnel, so that the sample can completely and automatically flow into the tank (or, use a glass rod to poke open it). Use a straight ruler to scrape the prominent part (before it is evenly scraped, do not move the tank). Weigh the mass of the sample and the tank, accurate to 0.1 g.

6.13.4 Result calculation

Bulk density, counted by the mass per unit volume ρ, expressed in (g/mL), shall be calculated in accordance with Formula (10):

$$\rho=\frac{m_1-m_2}{V} \quad (10)$$

Where,

m_1---the numerical value of the mass of the tank and the sample, expressed in (g);

m_2---the numerical value of the mass of the tank, expressed in (g);

V---the numerical value of the volume of the tank, expressed in (mL).

Take the arithmetic mean value of the parallel determination result as the determination result. The absolute difference of two parallel determination results shall be not more than 0.02 g/mL.

7 Inspection Rules

7.1 All the indexes stipulated in this Standard are exit-factory inspection items, which shall be inspected per batch.

7.2 The same grade of light magnesium oxide for industrial use, which is manufactured by manufacturers with the same materials, under basically the same manufacturing conditions in consecutive manufacture, or manufactured by the same manufacturing group, shall be considered as one batch. Each batch of products shall not exceed 10 t.

7.3 In accordance with the stipulations in GB/T 6678, determine the number of sampling units. During the sampling, vertically insert the sampler from above the packaging bag into 3/4 area of the depth of the sample for sampling. Mix up the collected sample; use quartering method to divide the sample to not less than 500 g. Sub-pack the sample into two clean and dry containers; seal them up; put on a label to indicate the name of the manufacturer, and product name, type, grade, batch No., sampling date and the name of the sampler. One shall be used in inspection; the other one shall be reserved for future reference. The retention time shall be determined by the manufacturer in accordance with the practical condition.

7.4 In the inspection result, if there are indicators that do not comply with the requirements of this Standard, re-take samples from a double size of package and conduct re-inspection. Even if one indicator in the re-inspection result does not comply with the requirements of this Standard, then, the whole batch of products shall be determined as disqualified.

7.5 Adopt the rounding-off comparison method stipulated in GB/T 8170 to determine whether the inspection result complies with the Standard.

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