GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

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National Food Safety Standard - Food Additives - Silicon dioxide

食品安全国家标准

食品添加剂 二氧化硅

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National Food Safety Standard - Food Additives - Silicon dioxide

1 Scope

This Standard is applicable to food additive - silicon dioxide that is obtained by using gas phase method (chlorosilane is hydrolyzed in oxygen hydrogen flame) and precipitation method (including gel method, produced by reacting sodium silicate solution with acid).

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.

3 Molecular formula and relative molecular mass

3.1 Molecular formula

SiO_2

3.2 Relative molecular mass

60.08 (according to 2007 international relative atomic mass)

4 Classification

Food additive - silicon dioxide, according to production process and product form, is classified into the following three categories:

Class I: Fumed silica.

Class II: Hydrated silica gel.

Class III: Precipitated silica, silica gel.

Annex A

(normative)

Inspection methods

A.1 Warning

Some reagents used in inspection methods in this Standard are toxic, corrosive. Operators must be cautious! If splashed on skin, immediately use water to rinse. In severe cases, treat immediately.

A.2 General rules

Reagents and water used in inspection methods in this Standard, if no other requirements indicated, shall be analytically-pure reagents and grade-three water specified in GB/T 6682-2008. Impurity standard solutions, preparations and products used in tests, if no other requirements indicated, shall be prepared according to the provisions of HG/T 3696.2, HG/T 3696.3.

A.3 Identification test

A.3.1 Reagents and materials

Ammonium molybdate solution: Dissolve 6.5 g of ammonium molybdate powder into 14 mL of water and 14.5 mL of ammonia mixture. Cool. Under stirring, slowly add into pre-cooled mixed solution of 32 mL of nitric acid and 40 mL of water. Place for 48 h. Perform suction-filtration. Filtrate is stored at a dark place. This solution shall deteriorate and become invalid after long-time placement. When 2 mL of sodium phosphate is added into 5 mL of above solution and a large amount of yellow precipitation is not immediately generated, this solution shall be invalid.

A.3.2 Silicon identification method

A.3.2.1 Weigh about 5 mg of sample into platinum crucible. Add 200 mg of anhydrous potassium carbonate to mix. In red heat, burn about 10 min. Cool. Add 2 mL of water to dissolve. If necessary, it may heat up. Then slowly add 2 mL of ammonium molybdate solution. There shall be dark yellow.

A.3.2.2 Drop 1 drop of sample that is dissolved in A.3.2.1 on filter paper. Evaporate this solution. Add into 1 drop of O-benzidine's glacial acetic acid saturated solution. Then put test paper on concentrated ammonia. There shall be green spot.

A.4 Determination of silica

The absolute difference between two parallel determination results is not more than 0.2%.

A.5 Determination of dry reduction

A.5.1 Apparatus and equipment

A.5.1.1 Electric constant-temperature drying oven: temperature can be controlled at 105°C ± CD.

A.5.1.2 Weighing bottle: Φ 40mm×25mm.

A.5.2 Analysis steps

Use weighing bottle that has been pre-dried at 105°C ± 2°C to constant mass to weigh 2g~3g of sample (weigh 8g~10g for Class II), to the nearest of 0.0002 g. Move into electric constant-temperature drying oven. Dry at 105°C ± 2°C to constant mass. Keep this dried test material as test material A for determination of burning reduction in A.6.

A.5.3 Result calculation

Dried reduction is calculated in mass fraction w_2. Value is represented in %. It is calculated according to formula (A.2):

$$w_2 = \frac{m_1 - m_2}{m_1 - m_0} \times 100\% \quad \cdots\cdots (A.2)$$

Where,

m_1 - value of mass of test material and weighing bottle before drying, in grams (g);

m_2 - value of mass of test material and weighing bottle after drying, in grams (g);

m_0 - value of mass of weighing bottle, in grams (g).

Take arithmetic mean of parallel measurement results as measurement result. The absolute difference between two parallel determination results is not more than 0.2%.

A.6 Determination of burning reduction

A.6.1 Apparatus and equipment

A.6.1.1 High-temperature furnace: temperature can be controlled at 950°C ± 50°C.

A.8.1 Reagents and materials

Same with Clause 3 of GB/T 5009.74-2003.

A.8.2 Analysis steps

Pipette 20.00 mL of test solution A and 3.00 mL of limited standard solution [1 mL of solution contains 10 μg of lead (Pb)]. Determine according to Clause 6 of GB/T 5009.74-2003.

A.9 Determination of arsenic

A.9.1 Atomic fluorescence spectrophotometry (arbitration method)

A.9.1.1 Reagents and materials

A.9.1.1.1 Hydrochloric acid solution: 1+20.

A.9.1.1.2 Hydrochloric acid solution: 1+1.

A.9.1.1.3 Potassium borohydride solution: Weigh 1.0 g of sodium hydroxide to dissolve in 250 mL of water. Mix well. Then weigh 5 g of potassium borohydride to dissolve in above sodium hydroxide solution. Mix well. This solution is prepared when it is needed.

A.9.1.1.4 Arsenic standard solution: 1 mL of solution contains 0.1 μg of arsenic (As).

Accurately pipette 10.00 mL of arsenic standard solution that is prepared according to HG/T 3696.2 into 1000 mL volumetric flask. Use water to dilute to scale. Shake well. Then accurately pipette 10.00 mL of this solution into 1000 mL volumetric flask. Add water to dilute to scale. Shake well.

A.9.1.1.5 Thiourea-ascorbic acid mixed solution:

Weigh 5 g of thiourea, 5 g of ascorbic acid into 100 mL of water. Mix well. Prepare it when it is needed.

A.9.1.1.6 Grade-two water: in accordance with GB/T 6682.

A.9.1.1.7 Argon: 99.99%.

A.9.1.2 Apparatus and equipment

A.9.1.2.1 Atomic fluorescence spectrophotometer.

A.9.1.2.2 Arsenic hollow cathode lamp.

A.9.1.3 Apparatus working conditions

represented in mg/kg. It is calculated according to formula (A.5):

$$w_5 = \frac{m_1 - m_0}{m \times 10^{-3} \times 2.5/100} \quad \text{(A.5)}$$

Where,

m_1 - value of arsenic mass that is found from working curve according to measured fluorescence strength of test solution, in milligrams (mg);

m_0 - value of arsenic mass that is found from working curve according to measured fluorescence strength of blank test solution, in milligrams (mg);

m - value of mass of test material (A.7.4.1), in grams (g).

Take arithmetic mean of parallel measurement results as measurement result. The absolute difference between two parallel determination results is not more than 0.5 mg/kg.

A.9.2 Arsenic speckle method

Move 20.00 mL of test solution A and 3.00 mL of arsenic standard solution [1 mL of solution contains 1.0 μg of arsenic (As)]. Operate according to Clause 11 of GB/T 5009.76-2003.

A.10 Determination of soluble dissociated salt

A.10.1 Reagents and materials

Sodium sulfate.

A.10.2 Apparatus and equipment

A.10.2.1 Vacuum suction-filtration pump: vacuum degree can reach 0.1MPa.

A.10.2.2 Glass core funnel: 100 mL; filter plate aperture is 5μm~15μm.

A.10.2.3 Conductivity meter.

A.10.2.4 Analysis steps

Weigh 5.00g ± 0.01g of sample that has been dried at 105°C ± 2°C for 2 h. Place in 250 mL beaker. Add 100 mL of water. Stir. Let insoluble substance settle down. Use glass core funnel for suction-filtration. Move filtrate to 250 mL volumetric flask. Use hot water to rinse insoluble substance 3 times, 20 mL of water for each time. Move filtrate to volumetric flask. At last, use 20 mL of hot water to rinse funnel and suction-filtration bottle. Cool filtrate to room temperature. Use water to dilute to scale. Shake well. Use conductivity meter

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