

ICS 67.220.20
CCS X 40



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB 29211-2012

Food additives ferrous sulfate

食品添加剂 硫酸亚铁

(English Translation)

Issued on: December 25, 2012

Implemented on: January 25, 2013

Issued by: Ministry of Health of the People's Republic of China

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1 Scope

This standard applies to iron and sulfuric acid reaction, the refined production of food additives seven hydrated ferrous sulfate and ferrous sulfate dry goods.

2.1 Chemical name

Heptahydrate ferrous sulfate Ferrous sulfate dry product

2.2 Molecular formula

Hexahydrate ferrous sulfate. $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ Ferrous sulfate dry product. $\text{FeSO}_4 \cdot n\text{H}_2\text{O}$

2.3 Relative molecular mass

Heptahydrate ferrous sulfate. 278.01 (according to the 2007 international relative atomic mass)

3 Terms and definitions

The following terms and definitions apply to this document.

3.1 Ferrous sulfate dry product

Hexahydrate ferrous sulfate produced by heating drying process of ferrous sulfate. The degree of heating and drying is different from ferrous sulfate There are different proportions of ferrous sulfate hydrate and ferrous sulfate tetrahydrate.

4.1 Sensory requirements

Shall comply with the provisions of Table 1.

Table 1 sensory requirements

project

Claim

Heptahydrate ferrous sulfate ferrous sulfate ferrous sulfate Testing method Color gray or blue-green gray or light green State granular crystal powder Take the appropriate amount of sample in a 50 mL beaker and observe the color under natural light status

4.2 Physical and chemical indicators

Shall comply with the provisions of Table 2.

Table 2 Physical and chemical indicators

project

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Heptahydrate ferrous sulfate ferrous sulfate ferrous sulfate Testing method Ferrous sulfate content, w /% ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) 99.5 ~ 104.5 (FeSO_4) 86.0 ~ 89.0 Appendix A A.4 Lead (Pb)/(mg/kg) ≤ 2 Appendix A A.5 Mercury (Hg)/(mg/kg) ≤ 1 Appendix A A.6 Arsenic (As)/(mg/kg) ≤ 3 GB/T 5009.76 Acid insoluble matter, w /% ≤ 0.05 Appendix A A.7 Appendix A Testing method A.1 Warning the samples of this standard are highly corrosive and some of the reagents used in the test methods are toxic or corrosive and should be taken in proper operation Safety and protective measures.

A.2 General provisions the reagents and water used in this standard, when not specified, refer to the analytical reagent and the tertiary water specified in GB/T 6682-2008.

Standard titration solution used in the test, the standard solution for the determination of impurities, preparations and products, in the absence of other requirements, according to GB/T 601, GB/T 602, GB/T 603. The solution used refers to an aqueous solution when it is not specified with any solvent formulation.

A.3 Identification test A.3.1 Reagents and materials A.3.1.1 Potassium ferricyanide solution. 100 g/L.

A.3.1.2 barium chloride solution. 100 g/L.

A.3.1.3 Hydrochloric acid solution. 1 1.

A.3.1.4 Lead acetate solution. 80 g/L.

A.3.1.5 Ammonium acetate solution. 100 g/L.

A.3.2 Instruments and equipment Acidity meter. scale value of 0.02.

A.3.3 Identification method A.3.3.1 Identification of ferrous ions Weigh about 0.2g sample, dissolved in 10mL water, dropping potassium ferricyanide solution to produce dark blue precipitate.

A.3.3.2 Identification of Sulfate A.3.3.2.1 Take about 1g sample, dissolve in 20mL water, add barium chloride solution, which produces a white precipitate, this white precipitate insoluble in hydrochloric acid Solution.

A.3.3.2.2 Take about 1g sample, dissolve in 20mL water, add lead acetate solution, which produces white precipitate, this white precipitate dissolved in ammonium acetate Solution.

A.3.3.2.3 Take about 1g sample, dissolve in 20mL water, add hydrochloric acid does not produce precipitation.

A.3.3.3 Identification of acidity Weigh $10\text{g} \pm 0.01\text{g}$ sample, dissolved in 100mL water, stir well. Use the calibrated pH meter to measure the pH of the solution 3.7 ± 0.5 .

A.4 Determination of ferrous sulfate content A.4.1 Methodological Summary in an acidic medium, the solution was titrated with standard titration with cerium sulfate and the endpoint was indicated with 1,10-phenanthroline-ferrous as an indicator.

A.4.2 Reagents and materials A.4.2.1 Sulfuric acid solution. $c(1/2\text{H}_2\text{SO}_4) = 2 \text{ mol/L}$.

A.4.2.2 1,10-phenanthroline-ferrous indicator solution. 0.700 g of ferrous sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) was dissolved in 100 mL of 15 g/L 1,10-phenanthroline solution, the solution is stored in a closed container.

A.4.2.3 standard titration solution of cerium sulfate. $c(\text{CeSO}_4) = 0.1\text{mol/L}$.

A.4.3 Analysis steps Weigh about 1g of ferrous sulfate heptahydrate or about 0.6g ferrous sulfate dry, accurate to 0.0002g, placed in 500mL cone Bottle, add 25mL just boiled and cooled water and 25mL sulfuric acid solution dissolved, add a few drops of 1,10-phenanthroline - ferrous indicator solution, with sulfur the cerium standard titration solution changed from red to light blue.

While the blank test. Blank test in addition to no sample, the other operations and the type and amount of reagents added (standard titration solution and the same).

A.4.4 Calculation of results the mass fraction w_1 of ferrous sulfate (in $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ or FeSO_4) is calculated according to formula (A.1) $w_1 = (V-V_0) c_M m \times 1000 \times 100\%$ (A.1) where.

V --- titration sample solution consumption of cerium sulfate standard titration solution volume of the value in milliliters (mL);

V_0 --- titration blank sample solution consumption of cerium sulfate standard titration solution volume value in milliliters (mL);

c - the exact value of the standard titration solution of cerium sulfate standard in moles per liter (mol/L);

m - the mass of the sample, in grams (g);

M - the value of the molar mass of ferrous sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$), ferrous sulfate (FeSO_4), in grams per mole (g/mol) [$M(\text{FeSO}_4 \cdot 7\text{H}_2\text{O}) = 278.01$, $M(\text{FeSO}_4) = 151.91$];

1000 --- conversion factor.

The experimental results are based on the arithmetic mean of the parallel measurement results. The absolute difference between the two independent determinations obtained under repeatability conditions the value is not greater than 0.3%.

A.5 Determination of lead (Pb) A.5.1 Reagents and materials A.5.1.1 Hydrochloric acid solution. 3 1.

A.5.1.2 Ascorbic acid-sodium iodide solution. 100 mg/mL acid-resistant acid solution and 192.5 mg/mL sodium iodide aqueous solution mixing.

A.5.1.3 Tri-n-octylphosphine oxide solution. Weigh 5.0 g of tri-n-octylphosphine oxide into a 100 mL volumetric flask and use methyl isobutyl ketone Dissolved and diluted to the mark, shake.

WARNING. Tri-n-octylphosphine oxide is irritating and avoid contact with eyes, skin and clothing.

A.5.1.4 Lead (Pb) standard solution. 0.1 mg/mL.

A.5.1.5 Water. in line with GB/T 6682-2008 in the provisions of secondary water.

A.5.2 Instruments and equipment Atomic Absorption Spectrophotometer. With lead hollow cathode lamp.

A.5.3 Analysis steps A.5.3.1 Preparation of standard alignment solutions Remove the 1.0 mL lead standard solution into a 100 mL volumetric flask, dilute to the mark with water and shake well. Remove 2.0 mL of this solution to 50 mL in a volumetric flask, 10 mL of hydrochloric acid solution, 10 mL of water, 20 mL of ascorbic acid-sodium iodide solution, 5.0 mL of tri-n-octylphosphine oxide solution, Shake for 30s, standing stratified. Add water to the volumetric flask so that the organic layer rises to the neck of the volumetric flask and shake and stand still.

A.5.3.2 Preparation of sample solution Weigh 1.0g sample, accurate to 0.01g, placed in 100mL volumetric flask, add 10mL hydrochloric acid solution, 10mL water, shaking Sample dissolved, then add 20mL ascorbic acid - sodium iodide solution, 5.0mL tri-n-octylphosphine oxide solution, shaking 30s, standing stratification. To the capacity Add the water in the vial so that the organic layer rises to the neck of the volumetric flask and shake and stand still.