HG

CHEMICAL INDUSTRY STANDARD

OF THE PEOPLE'S REPUBLIC OF CHINA

Filing No.: 7474-2000

HG 2934-2000

Replacing HG 2934-1987

Feed grade zinc sulphate

饲料级硫酸锌

Issued on: June 05, 2000 **Implemented on: March 01, 2001**

Issued by: State Bureau of Petroleum and Chemical Industry

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Feed grade zinc sulphate

1 Scope

This standard specifies the requirements, test methods, inspection rules, signs, labels, packaging, transportation, storage of feed grade zinc sulfate.

This standard applies to feed grade zinc sulfate, which is produced by reacting zinc-containing raw materials with sulfuric acid. This product is used as a zinc supplement in the feed, after pretreatment.

Molecular formula: $ZnSO_4 \cdot H_2O$

Relative molecular mass: 179.47 (according to the international relative atomic mass in 1997)

Molecular formula: $ZnSO_4 \cdot 7H_2O$

Relative molecular mass: 287.56 (according to the international relative atomic mass in 1997)

2 Normative references

The provisions in following documents become the provisions of this Standard through reference in this Standard. The versions indicated at the time of publication of this standard are valid. All standards are subjected to revision, AND parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable.

GB/T 601-1988 Chemical reagent - Preparations of standard volumetric solutions

GB/T 602-1988 Chemical reagent - Preparations of stock standard solutions

GB/T 603-1988 Chemical reagent - Preparations of reagent solutions for use in test methods

GB/T 6678-1986 General principles for sampling chemical products

GB/T 6682-1992 Water for laboratory use - Specifications (neq ISO 3696:1987)

GB/T 8450-1987 Determination method of arsenic in food additives (arsenic spot method)

5.1.1 Reagents and materials

5.1.1.1 Chloroform.

5.1.1.2 Acetic acid solution: 1 + 10.

5.1.1.3 Sodium sulfate solution: 250 g/L.

5.1.1.4 Dithizone carbon tetrachloride solution: 1 + 100.

5.1.1.5 Barium chloride solution: 50 g/L.

5.1.2 Identification method

5.1.2.1 Identification of zinc ions

Weigh 0.2 g of specimen. Dissolve it in 5 mL water. Pipette 1 mL of the test solution. Use acetic acid solution to adjust the pH of the solution to 4 ~ 5. Add 2 drops of sodium sulfate solution. Add few drops of disulfiram carbon tetrachloride solution and 1 mL of chloroform. After shaking, the organic layer shows purple red.

5.1.2.2 Identification of sulfate ion

Dissolve the specimen in water. Add barium chloride solution, to produce a white precipitate, which is insoluble in hydrochloric acid solution.

5.2 Determination of zinc sulfate content and zinc content

5.2.1 Method summary

Dissolve zinc sulfate in sulfuric acid solution. Add appropriate amount of water. Add ammonium fluoride solution, thiourea, ascorbic acid, as masking agent. Use acetic acid-sodium acetate solution, to adjust pH value to 5 ~ 6. Use xylenol orange as indicator. Titrate with disodium ethylenediaminetetraacetic acid standard titration solution, until the solution turns from purple red to bright yellow, which is the end point.

5.2.2 Reagents and materials

5.2.2.1 Potassium iodide.

5.2.2.2 Ascorbic acid.

5.2.2.3 Thiourea solution: 200 g/L.

5.2.2.4 Ammonium fluoride solution: 200 g/L.

5.2.2.5 Sulfuric acid solution: 1 + 1.

5.2.2.6 Acetic acid-sodium acetate buffer solution: pH = 5.5.

5.2.5 Tolerance

Take the arithmetic mean of the parallel determination results, as the measurement result. The absolute difference of parallel determination results: zinc sulfate monohydrate and zinc sulfate heptahydrate is not more than 0.2%, zinc (Zn) is not more than 0.15%.

5.3 Determination of arsenic content

5.3.1 Method summary

In acidic medium, potassium iodide and stannous chloride will reduce As (V) to As (III); generate arsenic spot with nascent hydrogen on the mercury bromide. It is compared with the standard arsenic spot.

5.3.2 Reagents and materials

5.3.2.1 Hydrochloric acid.

5.3.2.2 Potassium carbide.

5.3.2.3 Arsenic-free metal zinc.

5.3.2.4 Stannous chloride solution: 400 g/L.

5.3.2.5 Arsenic standard solution: 1 mL solution contains 0.001 mg of As.

Preparation: Use a pipette to pipette 1.00 mL of arsenic standard solution, which is prepared according to GB/T 602. Place it in a 100 mL volumetric flask. Use water to dilute it to the mark. Shake well. This solution is prepared before use.

5.3.2.6 Mercury bromide test paper.

5.3.2.7 Lead acetate cotton.

5.3.3 Instruments and equipment

Arsenic fixer.

5.3.4 Analytical procedures

Weigh (1.0 ± 0.01) g of specimen. Place it in the jar of the arsenic fixer. Add a small amount of water to moisten it. Add 6 mL of hydrochloric acid, to dissolve the specimen. Use water to dilute it to 70 mL. Add 1 g of potassium iodide and 0.2 mL of stannous chloride solution. Shake well. Let it stand for 10 min. Add 2.5 g of arsenic-free metal zinc. Immediately plug the arsenic measuring tube, which was pre-installed with lead acetate cotton and mercury bromide test paper. Place it in a dark place, at 25 ~ 30 °C for 1 ~ 1.5 h. The brown-yellow color of the mercury bromide test paper shall not be

deeper than the color spots, which are produced by the standard.

The standard is to use a pipette to pipette 5 mL of arsenic standard solution. Place it in the jar of the arsenic fixer. Use water to dilute it to 70 mL. Add 6 mL of hydrochloric acid. The following operations are performed at the same time and method as the specimen.

5.4 Determination of lead content

5.4.1 Atomic absorption spectrophotometry (arbitration method)

5.4.1.1 Method summary

In the dilute nitric acid medium, at the wavelength of 283.3 nm of the atomic absorption spectrophotometer, use the air-acetylene flame, to make measurement, through the standard addition method.

5.4.1.2 Reagents and materials

a) Nitric acid solution: 1 + 1.

b) Lead standard solution: 1 mL of the solution contains 0.01 mg of Pb, which is prepared before use.

Preparation: Use a pipette to pipette 10 mL of lead standard solution, which was prepared according to GB/T 602. Place it in a 100 mL volumetric flask. Use water to dilute it to the mark. Shake well.

5.4.1.3 Instruments and equipment

a) Atomic absorption spectrophotometer.

b) Lead hollow cathode lamps.

5.4.1.4 Instrument working conditions

a) Wavelength: 283.3 nm.

b) Flame: Air - Acetylene.

5.4.1.5 Analytical procedures

Weigh about 30 g of the specimen (accurate to 0.01 g). Dissolve it in 50 mL of water. Add 5 mL of nitric acid solution. Transfer it into a 250 mL volumetric flask, after dissolving. Use water to dilute it to the mark. Shake well.

Use a pipette to pipette 25 mL of the test solution into four 100 mL volumetric flasks. Then use pipette to add 0, 10 mL, 20 mL, 30 mL of lead standard solution. Use water

b) Ammonium citrate solution: 400 g/L.

c) Ammonia-ammonium chloride buffer solution (A): pH = 10.

d) Potassium cyanide solution: 150 g/L.

e) Dithizone-chloroform solution: 0.02 g/L.

Preparation: Dissolve 0.02 g of dithizone in 1 L of chloroform. Store at 10 °C in the dark.

f) Lead standard solution: 1 mL of solution contains 0.010 mg Pb.

Preparation: Use a pipette to pipette 10.00 mL of lead standard solution, which was prepared according to GB/T 602. Place it in a 100 mL volumetric flask. Use water to dilute it to the mark. Shake well. Prepare the diluent before use.

5.4.2.3 Analytical procedures

Weigh (0.5 ± 0.01) g of specimen. Put it in a 50 mL colorimetric tube. Add 25 mL of water to dissolve it. Add 5 mL of ammonium citrate solution, 5 mL of ammonia-ammonium chloride buffer solution (A), 10 mL of potassium cyanide solution. Shake well. Add 0.5 g of hydroxylamine hydrochloride. Shake well. Add 5 mL of dithizone-chloroform solution. Shake for 1 min. After the solution is layered, the color of the dithizone-chloroform extraction layer shall not be redder than that of the standard colorimetric solution.

For the standard colorimetric solution, use a pipette to pipette 1.00 mL (for determination of zinc sulfate heptahydrate) or 0.50 mL (for determination of zinc sulfate monohydrate) of lead standard solution, which is treated using the same time and method as the specimen.

5.5 Determination of cadmium content

5.5.1 Method summary

In dilute nitric acid medium, at the atomic absorption spectrophotometer wavelength of 228.8 nm, use air-acetylene flame to make determination, through the standard curve method.

5.5.2 Reagents and materials

5.5.2.1 Nitric acid solution: 1 + 1.

5.5.2.2 Cadmium standard solution: 1 mL contains 0.01 mg of Cd.

Preparation: Use a pipette to pipette 10 mL of cadmium standard solution, which was prepared according to GB/T 602. Put it in a 100 mL volumetric flask. Use water to

dilute it to the mark. Shake well.

5.5.3 Instruments and equipment

5.5.3.1 Atomic absorption spectrophotometer.

5.5.3.2 Cadmium hollow cathode lamps.

5.5.4 Instrument working conditions

5.5.4.1 Wavelength: 228.8 nm.

5.5.4.2 Flame: Air-acetylene.

5.5.5 Analytical procedures

Weigh about 1 g of specimen (accurate to 0.01 g). Dissolve it in 30 mL of water. Add 5 mL of nitric acid solution. Transfer it into a 100 mL volumetric flask after dissolving. Use water to dilute it to the mark. Shake well.

In a series of 100 mL volumetric flasks, pipette 0, 1 mL, 2 mL, 3 mL, 4 mL of cadmium standard solutions, respectively. Add 5 mL nitric acid solution for each. Use water to dilute it to the mark. Shake well.

According to the provisions in 5.5.4, adjust the instrument to the best working condition. Use water to adjust to zero. Measure the absorbance of the test solution and standard series solutions.

At the same time, do a blank test.

Take the mass of cadmium in the standard series solution as the abscissa AND the corresponding absorbance as the ordinate, to draw the working curve.

5.5.6 Expression of analysis results

The cadmium (Cd) content (X_5), expressed in mass percentage, is calculated according to formula (5):

$$X_5=\frac{(m_1-m_0)}{m\times 1000}\times 100=\frac{0.1(m_1-m_0)}{m} \quad \cdots\cdots (5)$$

Where:

m_1 - The mass of cadmium in the specimen, as found from the working curve, according to the measured absorbance of the test solution, in mg;

m_0 - The mass of cadmium in the specimen, as found from the working curve, according to the measured absorbance of the blank solution, in mg;

6 Inspection rules

6.1 All items specified in this standard are exit-factory inspection items.

6.2 Each batch of products shall not exceed 20 t.

6.3 Determine the number of sampling units, according to the provisions of GB/T 6678. When sampling, insert the sampler obliquely, from the top of the packaging bag to 3/4 of the depth of the material layer, to take sample. Mix the collected sample thoroughly. Divide it into quarters to about 500 g. Immediately put it into two clean, dry, stoppered jars. Seal it. Paste a label on the jar, indicating the name of the manufacturer, product name, batch number, sampling date, the name of the sampler. One jar is used for inspection, whilst the other jar is kept for three months for future reference.

6.4 Feed grade zinc sulfate shall be inspected by the quality supervision and inspection department of the manufacturer, according to the provisions of this standard. The manufacturer shall ensure that all the feed grade zinc sulfate, leaving the factory, meet the requirements of this standard. Each batch of feed-grade zinc sulfate leaving the factory shall be accompanied by a quality certificate, the content of which is the same as that stipulated in GB 10648.

6.5 The user organization has the right to inspect and accept the received feed grade zinc sulfate, in accordance with the provisions of this standard. The acceptance time is within one month of the arrival of the goods.

6.6 If one of the indicators of the inspection results does not meet the requirements of this standard, it shall take sample from twice the amount of packaging, for re-inspection. Even if one of the indicators does not meet the requirements of this standard in the re-inspection results, the entire batch of products is unqualified.

7 Signs, labels

7.1 There shall be firm and clear signs on the packaging of feed grade zinc sulfate. The content includes manufacturer name, manufacturer address, product name, trademark, "feed grade" words, net weight, batch number or production date, certificate of product quality conforming to this standard, the number of this standard.

7.2 The packaging of each batch of feed grade zinc sulfate leaving the factory shall have firm and clear signs, which are printed in accordance with the requirements of GB 10648.

8 Packaging, transportation, storage

8.1 Feed-grade zinc sulfate is packed in plastic woven bags. It is lined with a layer of

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