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Water treatment chemicals - Aluminum sulfate

水处理剂 硫酸铝

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Water treatment chemicals - Aluminum sulfate

WARNING -- The strong acids and bases used in this Standard are corrosive. It shall avoid inhalation or contact with the skin during use. If splashed on the body, rinse immediately with plenty of water. In severe cases, seek medical attention immediately.

1 Scope

This Standard specifies the requirements, classification, test methods, inspection rules, marks, labels and packaging of aluminum sulfate.

This Standard applies to aluminum sulfate used as a water treatment agent. The product is mainly used for drinking water and industrial water, waste water and sewage treatment. Among them, the raw sulfuric acid used for drinking water shall be industrial sulfuric acid; aluminum-containing raw materials shall be industrial aluminum hydroxide.

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.

GB/T 191, *Packaging and storage marks*

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

GB/T 602, *Chemical reagent -- Preparations of standard solutions for impurity*

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

GB/T 610-2008, *Chemical reagent -- General method for the determination of arsenic*

GB/T 6678, *General principles for sampling chemical products*

GB/T 6680, *General rules for sampling liquid chemical products*

GB/T 6682, *Water for analytical laboratory use -- Specification and test methods*

GB/T 8170, *Rules of rounding off for numerical values and expression and*

6 Test methods

6.1 General

Reagents used in this Standard, unless otherwise specified, only use analytically pure reagents.

The standard solutions, impurity standard solutions, preparations and products required in the test shall be prepared according to the provisions of GB/T 601, GB/T 602 and GB/T 603 unless other requirements are specified.

6.2 Determination of alumina content

6.2.1 Method summary

The aluminum in the specimen reacts with a known excess of disodium edetate solution to form a complex. When the pH value is about 6, xylenol orange is used as the indicator. Use zinc chloride standard titration solution to titrate excess disodium edetate solution.

6.2.2 Reagents and materials

6.2.2.1 Water: meet the grade three water specification in GB/T 6682.

6.2.2.2 Hydrochloric acid solution: 1+1.

6.2.2.3 Sodium acetate solution: 272 g/L.

6.2.2.4 Zinc chloride standard stock solution: $c(ZnCl_2) = 0.1$ mol/L.

6.2.2.5 Zinc chloride standard titration solution: $c(ZnCl_2) = 0.025$ mol/L. Prepare according to GB/T 601 and dilute 4 times.

6.2.2.6 Disodium ethylenediaminetetraacetic acid (EDTA) standard solution: c(EDTA) = 0.05 mol/L.

6.2.2.7 Xylenol orange indicator solution: 2 g/L.

6.2.3 Analysis steps

6.2.3.1 Preparation of test solution

Weigh about 5 g of solid specimen or 10 g of liquid specimen, accurate to 0.2 mg. Place in a 250 mL beaker. Add 100 mL of water and 2 mL of hydrochloric acid solution. Heat to dissolve and boil for 5 min (filter if necessary). After cooling, transfer all to a 500 mL volumetric flask. Use water to dilute to the scale. Shake well. This solution shall be the test solution A, for the determination of alumina and iron content.

6.2.3.2 Preparation of blank test solution

Weigh (1.00±0.01)g of specimen. Place it in a 100 mL beaker. Add about 50 mL of carbon dioxide-free water to dissolve. Transfer all to a 100 mL volumetric flask. Use carbon dioxide-free water to dilute to the scale. Shake well. Pour the specimen solution into the beaker. Measure its pH value on a pH meter that has been positioned.

6.6 Determination of arsenic content

6.6.1 Atomic fluorescence spectroscopy

6.6.1.1 Method principle

After the specimen is treated with acid, add thiourea to pre-reduce pentavalent arsenic to trivalent arsenic. Then add sodium borohydride or potassium borohydride to reduce and generate arsine, which is decomposed into atomic arsenic by loading argon gas into a quartz atomizer. Atomic fluorescence is excited by the emitted light from an arsenic hollow cathode lamp. Its fluorescence intensity is directly proportional to the concentration of arsenic in the tested solution under fixed conditions. Conduct quantitative comparison with standard series.

6.6.1.2 Reagents and materials

6.6.1.2.1 Water: meet the grade two water specification in GB/T 6682.

6.6.1.2.2 Hydrochloric acid: guaranteed reagent.

6.6.1.2.3 Nitric acid: guaranteed reagent.

6.6.1.2.4 Thiourea solution: 100 g/L.

6.6.1.2.5 Hydrochloric acid solution: 1+49.

6.6.1.2.6 Nitric acid solution: 1+1.

6.6.1.2.7 Potassium borohydride-sodium hydroxide solution: Weigh 2.0 g of sodium hydroxide and 10.0 g of potassium borohydride into a polyethylene beaker. Dissolve with water and dilute to 1000 mL. Store in polyethylene bottles.

6.6.1.2.8 Arsenic standard stock solution: 0.1 mg/mL.

6.6.1.2.9 Arsenic standard solution: pipette 10.00 mL of arsenic standard stock solution into a 100 mL volumetric flask. Use water to dilute to the scale. Mix well. Just before use, pipette 10.00 mL of this solution into a 100 mL volumetric flask. Use water to dilute to the scale. Mix well. 1.00 mL of this solution contains 1 μg of As.

6.6.1.3 Instruments and equipment

6.6.1.3.1 Atomic fluorescence spectrometer: equipped with an arsenic hollow cathode lamp.

before use, pipette 10.00 mL of this solution into a 100 mL volumetric flask. Use water to dilute to the scale. Mix well. 1.00 mL contains 0.001mg of As.

6.6.2.2.12 Lead acetate cotton.

6.6.2.3 Instruments and equipment

6.6.2.3.1 Spectrophotometer.

6.6.2.3.2 Arsenic fixer: meet the requirements in 4.2.2.3 of GB/T 610-2008.

6.6.2.4 Analysis steps

6.6.2.4.1 Preparation of specimen solution

Weigh about 25 g of liquid specimen or 12.5 g of solid specimen, accurate to 0.2 mg. Place in a 100 mL beaker. Add 30 mL of water and 5 mL of nitric acid solution. Cover and boil in a watch glass for about 1 min. Cool to room temperature and transfer to a 250 mL volumetric flask. Dilute to the scale. Shake well. This shall be test solution B, used for testing As, Pb, Cd, Hg, Cr.

6.6.2.4.2 Drawing of calibration curve

6.6.2.4.2.1 Add 0.00 mL, 1.00 mL, 2.00 mL, 3.00 mL, 4.00 mL and 5.00 mL of arsenic standard solution to six dry arsenic determination bottles in sequence. Then add 30 mL, 29 mL, 28 mL, 27 mL, 26 mL, 25 mL of water in sequence to make the total volume of the solution 30 mL.

6.6.2.4.2.2 Add 20 mL of stannous chloride hydrochloric acid solution, 5 mL of potassium iodide solution and 1 mL of copper sulfate solution to each arsenic bottle. Shake well. At this time, the acidity c (calculated as H^+) in the solution shall be between 1.8 mol/L ~ 2.6 mol/L. Place in the dark for 30 min ~ 40 min. Add 5 g of arsenic-free zinc particles to the arsenic-fixing device. Immediately put the absorption tube plugged with lead acetate cotton and filled with 5.0 mL of silver diethyldithiocarbamate-triethylamine chloroform solution (absorption solution) on the arsenic bottle. React for 25 min ~ 35 min (avoid direct sunlight. If the absorption solution evaporates too quickly, attention shall be paid to replenishing chloroform). Remove the absorption tube (do not suck back the absorption liquid). Supplement the absorption solution to 5.0 mL with chloroform. Mix well.

6.6.2.4.2.3 At a wavelength of 510 nm, use a 1 cm absorption cell and use the reagent blank as a reference to measure the absorbance.

6.6.2.4.2.4 Take the measured absorbance as the ordinate and the corresponding arsenic content (mg) as the abscissa, draw a calibration curve. Compute the regression equation.

6.6.2.4.3 Determination

V_1 - the value of the volume of test solution B pipetted, in milliliters (mL);

V_B - the value of the total volume of test solution B, in milliliters (mL) (V_B=250).

6.7.1.6 Tolerance

The arithmetic mean of the parallel determination results is taken as the measurement result. The absolute difference of parallel determination results is not more than 0.00005%.

6.7.2 Flame atomic absorption spectrometry

6.7.2.1 Method summary

Add sodium diethyldithiocarbamate solution to the specimen to chelate the lead. Extract with 4-methyl-2-pentanone. Measure the absorbance at a wavelength of 283.3 nm by atomic absorption spectrometry. Find the lead content.

6.7.2.2 Reagents and materials

6.7.2.2.1 Water: meet the grade two water specification in GB/T 6682.

6.7.2.2.2 Nitric acid solution: 1+1.

6.7.2.2.3 4-Methyl-2-pentanone.

6.7.2.2.4 Ammonia solution: 1+5.

6.7.2.2.5 Hydrochloric acid solution: 1+3.

6.7.2.2.6 Ammonium citrate solution: 500 g/L.

6.7.2.2.7 Ammonium sulfate solution: 400 g/L.

6.7.2.2.8 Sodium diethyldithiocarbamate solution: 100 g/L.

6.7.2.2.9 Lead standard stock solution: 0.1 mg/mL.

6.7.2.2.10 Lead standard solution: Pipette 10.00 mL of lead standard stock solution into a 100 mL volumetric flask. Add 2 mL of nitric acid solution. Use water to dilute to the scale. Shake well. 1 mL of this solution contains 0.01 mg of Pb.

6.7.2.3 Instruments and equipment

6.7.2.3.1 Atomic absorption spectrometer.

6.7.2.3.2 Lead hollow cathode lamps.

6.7.2.4 Analysis steps

Use electric heating atomic absorption spectrometry to measure the absorbance at a wavelength of 228.8 nm. Find the cadmium content.

6.8.1.2 Reagents and materials

6.8.1.2.1 Water: meet the grade two water specification in GB/T 6682.

6.8.1.2.2 Nitric acid solution: 1+1.

6.8.1.2.3 Cadmium standard stock solution: 0.1mg/1mL.

6.8.1.2.4 Cadmium standard solution: Pipette 10.00 mL of cadmium standard stock solution into a 1000 mL volumetric flask. Add 20 mL of nitric acid solution. Use water to dilute to the scale. Shake well. Then take 10.00 mL of the solution in a 100 mL volumetric flask. Add 2 mL of nitric acid solution. Use water to dilute to the scale. Shake well. 1 mL of this solution contains 0.1 μg of Cd. Prepare the solution when needed.

6.8.1.3 Instruments and equipment

6.8.1.3.1 Trace liquid inlet device: equipped with a push-button 5 μL ~ 500 μL trace liquid flow meter or an automatic sampler.

6.8.1.3.2 Electric heating atomic absorption analysis device: electrified heating method; reverse grounding compensation can be performed.

6.8.1.3.3 Heating furnace: made of graphite or high temperature resistant metal.

6.8.1.3.4 Cadmium hollow cathode lamps.

6.8.1.4 Analysis steps

6.8.1.4.1 Pipette 0.00 mL, 0.50 mL, 1.00 mL and 1.50 mL of chromium standard solution into four 50 mL volumetric flasks respectively. Add 1 mL of nitric acid solution. Use water to dilute to the scale. Shake well. Inject the prepared specimen into the heating furnace with a micro-injection device. After drying, ashing and atomization, measure its absorbance at 228.8 nm. With the mass concentration of cadmium standard solution (μg/L) as the abscissa and the corresponding absorbance as the ordinate, draw the calibration curve. Compute the regression equation.

6.8.1.4.2 Pipette an appropriate volume of test solution B. Follow 6.8.1.4.1 to operate. Determine. The mass concentration of cadmium is obtained from the calibration curve or regression equation.

6.8.1.5 Calculation of results

The cadmium content is expressed as mass fraction w_6. The value is expressed in %, calculated according to formula (8):

regression equation, in milligrams (mg);

m_1 - the numerical value of the mass of the test material, in grams (g).

6.8.2.6 Tolerance

The arithmetic mean of the parallel determination results is taken as the measurement result. The absolute difference of parallel determination results is not more than 0.00005%.

6.9 Determination of mercury content

6.9.1 Atomic fluorescence spectroscopy

6.9.1.1 Method summary

After the specimen is heated and digested by acid, the mercury in the specimen is reduced to atomic mercury by potassium borohydride (KBH_4) in acidic medium. It is brought into the atomizer by the carrier gas (argon). Under the irradiation of a mercury hollow cathode lamp, the ground state mercury atoms are excited to a higher energy state. When deactivated to the ground state, it emits fluorescence at a characteristic wavelength. Its fluorescence intensity is proportional to the mercury content. Conduct quantitative comparison with standard series.

6.9.1.2 Reagents and materials

6.9.1.2.1 Water: meet the grade two water specification in GB/T 6682.

6.9.1.2.2 Nitric acid: guaranteed reagent.

6.9.1.2.3 Hydrochloric acid: guaranteed reagent.

6.9.1.2.4 Nitric acid solution: 1+1.

6.9.1.2.5 Hydrochloric acid solution: 1+49.

6.9.1.2.6 Potassium borohydride-sodium hydroxide solution: Weigh 3.0 g of sodium hydroxide and 0.5 g of potassium borohydride into a polyethylene beaker. Dissolve with water and dilute to 1000 mL. Prepare the solution when needed.

6.9.1.2.7 Mercury standard stock solution (I): 0.1 mg/mL.

6.9.1.2.8 Mercury standard stock solution (II): Pipette 5 mL of mercury standard stock solution (I) into a 100 mL volumetric flask. Add 0.05 g of potassium dichromate, 5 mL of nitric acid. Use water to dilute to the scale. 1 mL of this solution contains 5 μg of Hg. Prepare the solution when needed.

6.9.1.2.9 Mercury standard solution: pipette 1 mL of mercury standard stock solution (II) into a 100 mL volumetric flask immediately before use. Add 0.05 g of potassium

V - the value of the total volume of the specimen solution during the measurement, in milliliters (mL);

m_0 - the numerical value of the mass of the sample, in grams (g).

6.9.1.7 Tolerance

The arithmetic mean of the parallel determination results is taken as the measurement result. The absolute difference of parallel determination results is not more than 0.000005%.

6.9.2 Cold atomic absorption method

6.9.2.1 Method summary

In an acidic medium, the mercury in the specimen is oxidized to divalent mercury ions. Mercury ions are reduced to mercury atoms with stannous chloride. Mercury is determined by cold atomic absorption.

6.9.2.2 Reagents and materials

6.9.2.2.1 Water: meet the grade two water specification in GB/T 6682.

6.9.2.2.2 Sulfuric acid-nitric acid mixture: slowly add 200 mL of sulfuric acid (guaranteed reagent) to 300 mL of water while stirring continuously. After cooling, add 100 mL of nitric acid (guaranteed reagent). Mix well.

6.9.2.2.3 Sulfuric acid (guaranteed reagent) solution: 1+71.

6.9.2.2.4 Hydrochloric acid (excellent grade) solution: 1+11.

6.9.2.2.5 Potassium permanganate (premium grade) solution: 10 g/L.

6.9.2.2.6 Hydroxylamine hydrochloride solution: 100 g/L.

6.9.2.2.7 Stannous chloride solution: 50 g/L. Weigh 5.0 g of stannous chloride. Place in a 200 mL beaker. Add 10 mL of hydrochloric acid solution and appropriate amount of water to dissolve it. Dilute to 100 mL. Mix well.

6.9.2.2.8 Mercury standard stock solution: 0.1 mg/mL.

6.9.2.2.9 Mercury standard solution: pipette 10 mL of mercury standard stock solution into a 1000 mL volumetric flask. Dilute to the scale with sulfuric acid solution. Shake well. Then pipette 10 mL of the solution in a 1000 mL volumetric flask. Dilute to the scale with sulfuric acid solution. Shake well. 1 mL of this solution contains 0.001 mg of Hg.

6.9.2.3 Instruments and equipment

6.9.2.6 Tolerance

The arithmetic mean of the parallel determination results is taken as the measurement result. The absolute difference of parallel determination results is not more than 0.000002%.

6.10 Determination of chromium content

6.10.1 Method summary

Use electric heating atomic absorption spectrometry to measure the absorbance of chromium atoms at a wavelength of 429.0 nm. Find the chromium content.

6.10.2 Reagents and materials

6.10.2.1 Water: meet the grade two water specification in GB/T 6682.

6.10.2.2 Nitric acid solution: 1+1.

6.10.2.3 Chromium standard stock solution: 0.1 mg/mL.

6.10.2.4 Chromium standard solution: pipette 10.00 mL of chromium standard stock solution into a 1000 mL volumetric flask. Add 20 mL of nitric acid solution. Use water to dilute to the scale. Shake well. 1 mL of this solution contains 0.001 mg of Cr.

6.10.3 Instruments and equipment

6.10.3.1 Atomic absorption spectrometer.

6.10.3.2 Trace liquid inlet device: equipped with a push-button 5 μL ~ 500 μL trace liquid flowmeter or an automatic sampler.

6.10.3.3 Electric heating atomic absorption analysis device: electrified heating method; reverse grounding compensation can be performed.

6.10.3.4 Heating furnace: made of graphite or high temperature resistant metal.

6.10.3.5 Chromium hollow cathode lamps.

6.10.4 Analysis steps

6.10.4.1 Pipette 0.00 mL, 1.00 mL, 2.00 mL and 3.00 mL of chromium standard solutions into four 50 mL volumetric flasks respectively. Add 1 mL of nitric acid solution. Use water to dilute to the scale. Shake well. Inject the prepared specimen into the heating furnace with a micro-injection device. After drying, ashing and atomization, measure its absorbance at 429.0 nm. With the mass concentration of chromium standard solution (μg/L) as the abscissa and the corresponding absorbance as the ordinate, draw the calibration curve. Compute the regression equation.

6.10.4.2 Pipette an appropriate volume of test solution B. According to 6.10.4.1 from "add 1 mL of nitric acid solution, ..." to operate. Determine. The mass concentration of chromium is obtained from the calibration curve or regression equation.

6.10.5 Calculation of results

Chromium content is calculated as mass fraction w_8. The value is expressed in % and calculated according to formula (12):

$$w_8 = \frac{\rho V \times 10^{-9}}{m_0 V_1 / V_B} \times 100 \qquad \cdots\cdots (12)$$

Where,

ρ - the value of the mass concentration of chromium in the sample, in micrograms per liter (μg/L);

V - the value of the total volume of the specimen solution during the measurement, in milliliters (mL) (V=50);

m_0 - the value of the mass of the test material, in grams (g);

V_1 - the value of the volume of test solution B pipetted, in milliliters (mL);

V_B - the value of the total volume of test solution B, in milliliters (mL) (V_B=250).

6.10.6 Tolerance

The arithmetic mean of the parallel determination results is taken as the measurement result. The absolute difference of parallel determination results is not more than 0.00005%.

7 Inspection rules

7.1 All index items specified in this Standard are type inspection items. Class I products shall undergo type inspection at least once a month. Class II products shall undergo type inspection at least once every three months. Among them, alumina (Al_2O_3) content, iron (Fe) content, water-insoluble content and pH value shall be inspected batch by batch.

7.2 Each batch of products shall not exceed 150 t.

7.3 Determine the number of sampling units according to the provisions of GB/T 6678.

7.4 When sampling solid products, first remove the material layer about 30 cm thick on the upper layer of the packaging bag. Use a sampling tool to draw no less than 100 g of sample from the middle of each bag. The extracted samples are quickly broken down

------ The End ------