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**Polyaluminium chloride for treatment of drinking
water**

生活饮用水用聚氯化铝

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Polyaluminium chloride for treatment of drinking water

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

1 Scope

This Standard specifies the requirements, test methods, inspection rules, marking, packaging, transportation and storage of polyaluminium chloride for treatment of drinking water.

This Standard applies to polyaluminium chloride for treatment of drinking water. The product is mainly used for the purification of drinking water.

2 Normative references

The following documents are indispensable for the application of this document. For the dated references, only the editions with the dates indicated are applicable to this document. For the undated references, the latest edition (including all the amendments) are applicable to this document.

GB/T 191 Packaging - Pictorial marking for handling of goods

GB/T 320 Synthetic hydrochloric acid for industrial use

GB/T 601 Chemical reagent - Preparations of reference titration 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 4294 Aluminium hydroxide

GB/T 6678 General principles for sampling chemical products

the excess EDTA solution, to get alumina (Al_2O_3) content.

6.2.1.2 Reagents and materials

6.2.1.2.1 Carbon dioxide-free water.

6.2.1.2.2 Nitric acid solution: 1+12.

6.2.1.2.3 Aqueous ammonia solution: 1+1.

6.2.1.2.4 Disodium ethylenediaminetetraacetic acid (EDTA) solution: $c(\text{EDTA})$ is about 0.05 mol/L.

6.2.1.2.5 Acetic acid-sodium acetate buffer solution (pH=5.5): WEIGH 272 g of sodium acetate (trihydrate) and dissolve it in water; ADD 19 mL of glacial acetic acid; and dilute to 1000 mL.

6.2.1.2.6 Alumina standard solution: 1 mL contains 0.001 g of Al_2O_3 . WEIGH 0.5293 g of high-purity aluminium ($\geq 99.99\%$), accurate to 0.2 mg. PLACE it in a 200 mL polyethylene cup; ADD 20 mL of water; ADD about 3 g of sodium hydroxide, to make it all dissolved and transparent (if necessary, heat on a water bath). USE hydrochloric acid solution (1+1) to adjust to acidic and then add 10 mL, to make it transparent, cool; TRANSFER to a 1000 mL volumetric flask; DILUTE to the mark and shake well.

6.2.1.2.7 Zinc chloride reference titration solution: $c(\text{ZnCl}_2)$ is about 0.025 mol/L; prepare as follows:

- a) Preparation: WEIGH 3.5 g of zinc chloride (ZnCl_2); DISSOLVE it in hydrochloric acid solution [0.05% (volume fraction)]; DILUTE to 1 L and shake well.
- b) Calibration: PIPETTE 20 mL of EDTA solution and 40 mL of alumina standard solution; PUT them in a 250 mL conical flask; follow the steps in 6.2.1.3 "ADD 10 mL of nitric acid solution..." to start the operation. READ the consumption V (mL) of the zinc chloride reference titration solution. In addition, pipette 20 mL of EDTA solution and place it in a 250 mL conical flask. Follow the steps in 6.2.1.3 "ADD 10 mL of nitric acid solution..." to start the operation. READ the consumption V_0 (mL) of the zinc chloride reference titration solution.
- c) Result calculation: The concentration of zinc chloride reference titration solution, $c(\text{ZnCl}_2)$, value expressed in moles per liter (mol/L), is calculated according to formula (1):

Where:

V_0 - The value of the volume of sodium hydroxide reference titration solution consumed in the blank test, in milliliters (mL);

V - The value of the volume of sodium hydroxide reference titration solution consumed for determination of the sample, in milliliters (mL);

c - The exact value of the actual concentration of sodium hydroxide reference titration solution, in moles per liter (mol/L);

M - The value of the molar mass of hydroxide (OH^-), in grams per mole (g/mol) ($M=16.99$);

m - The value of the mass of the test portion, in grams (g);

w_1 - The mass fraction of alumina measured in 6.2;

V_1 - The value of the volume of test solution A pipetted, in milliliters (mL) ($V_1=25$);

V_A - The value of the total volume of test solution A, in milliliters (mL) ($V_A=250$);

M_1 - The value of the molar mass of aluminium, in grams per mole (g/mol) ($M_1=26.98$);

M_2 - The value of the molar mass of alumina, in grams per mole (g/mol) ($M_2=101.96$).

6.3.5 Allowable difference

USE the arithmetic mean of the parallel determination results as the determination result. The absolute difference of the parallel determination results is not more than 2.0%.

6.4 Determination of density

6.4.1 Method summary

From the depth of immersion when the densitometer reaches the equilibrium state in the measured liquid, read the density value.

6.4.2 Apparatus

6.4.2.1 Densitometer: The division value is 0.001 g/cm^3 .

6.4.2.2 Constant-temperature water bath: Controllable temperature $(20 \pm 0.1)^\circ\text{C}$.

6.4.2.3 Thermometer: The division value is 0.1°C .

standard solution respectively in five 100 mL volumetric flasks. Respectively add 5.0 mL of hydrochloric acid and 20 mL of thiourea solution; USE water to dilute to the mark and shake well; PLACE for more than 30 min. The mass concentrations of arsenic in this series of solutions are 0 µg/L, 4 µg/L, 8 µg/L, 12 µg/L, 16 µg/L, respectively.

6.8.1.4.2 After the instrument is stable, use potassium borohydride-sodium hydroxide solution as the reducing agent; USE hydrochloric acid solution as the carrier solution; under the best working conditions of the instrument, measure its fluorescence value. USE the measured fluorescence value as the ordinate; USE the corresponding mass concentration of arsenic (µg/L) as the abscissa, to draw a calibration curve and calculate the regression equation.

Note: When using an atomic fluorescence spectrometer for determination, the required potassium borohydride solution concentration, carrier solution concentration, linear range of calibration curves of various elements, pH value of sample solution, etc. will vary depending on the model of the instrument. Users can, according to the instrument model, choose the best test conditions.

6.8.1.5 Analytical procedure

6.8.1.5.1 Pre-cleaning of glassware

The glassware used in the test shall, before use, be soaked in nitric acid solution (1+4) for 24 h; then rinsed with water for later use.

6.8.1.5.2 Sample determination

WEIGH about 1.5 g of liquid sample or 0.5 g of solid sample, accurate to 0.2 mg; PLACE it in a 100 mL beaker. ADD 30 mL of water, 1 mL of nitric acid solution (1+1); COVER with a watch glass and boil for about 1 min. After cooling to room temperature, transfer to a 100 mL volumetric flask; respectively add 5.0 mL of hydrochloric acid, 20 mL of thiourea solution; USE water to dilute to the mark and shake well. Perform the determination according to the steps of 6.8.1.4.2 (if there is turbidity, use a medium-speed quantitative filter paper to dry-filter before the determination). The arsenic content is obtained from the calibration curve or regression equation.

6.8.1.6 Result calculation

The arsenic content is calculated as the mass fraction w_4 . The value is expressed in %, calculated according to formula (7):

$$w_4 = \frac{\rho V \times 10^{-9}}{m} \times 100 \quad \dots\dots\dots (7)$$

arsenic standard stock solution in a 100 mL volumetric flask; USE water to dilute to the mark and mix well. Before use, pipette 10.00 mL of this solution into a 100 mL volumetric flask; USE water to dilute to the mark and mix well.

6.8.2.2.9 Lead acetate cotton.

6.8.2.3 Apparatus

6.8.2.3.1 Spectrophotometer: With a 1 cm absorption cell.

6.8.2.3.2 Arsenic detector: Comply with the provisions of 4.2.2.3 in GB/T 610-2008.

6.8.2.4 Analytical procedure

6.8.2.4.1 Drawing of calibration curve

6.8.2.4.1.1 In 6 dry arsenic determination bottles, add 0.00 mL, 1.00 mL, 2.00 mL, 3.00 mL, 4.00 mL, 5.00 mL of arsenic standard solution in sequence; and 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 be 30 mL.

6.8.2.4.1.2 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 determination bottle; SHAKE well. At this time, the acidity c (in terms of H^+) in the solution shall be between 1.8 mol/L and 2.6 mol/L. PLACE in a dark place for 30 min~40 min; ADD 5 g of arsenic-free zinc granules to the arsenic determination bottle. Immediately install the absorption tube, filled with lead acetate cotton and containing 5.0 mL of silver diethyldithiocarbamate-triethylamine trichloromethane solution (absorption liquid), on the arsenic determination bottle; react for 25 min~35 min (Avoid direct sunlight. If the absorption liquid volatilizes too fast, it shall pay attention to add trichloromethane). TAKE off the absorption tube (do not suck the absorption liquid backwards); USE trichloromethane to replenish the absorption liquid to 5.0 mL and mix well.

6.8.2.4.1.3 At a wavelength of 510 nm, use a 1 cm absorption cell; USE the reagent blank as a reference; determine the absorbance.

6.8.2.4.1.4 USE the mass (mg) of arsenic as the abscissa; USE the corresponding absorbance as the ordinate; DRAW a calibration curve, or calculate a regression equation.

6.8.2.4.2 Determination

WEIGH about 10 g of liquid sample or 3.3 g of solid sample, accurate to 0.2 mg;

6.9.1.2 Reagents and materials

6.9.1.2.1 Nitric acid solution: 1+1.

6.9.1.2.2 Lead standard stock solution: 0.1 mg/mL.

6.9.1.2.3 Lead standard solution: PIPETTE 10.00 mL of lead standard stock solution into a 1000 mL volumetric flask; ADD 20 mL of nitric acid solution; USE water to dilute to the mark and shake well. 1 mL of this solution contains 1 μ g of Pb.

6.9.1.3 Apparatus

6.9.1.3.1 Micro-liquid feeding device: Equipped with a button type 5 μ L~500 μ L micro-liquid flowmeter or automatic sampler.

6.9.1.3.2 Electric heating atomic absorption analysis device: Electric heating method. Reverse grounding compensation can be carried out.

6.9.1.3.3 Heating furnace: Made of graphite or high-temperature-resistant metal.

6.9.1.3.4 Lead hollow cathode lamp.

6.9.1.4 Analytical procedure

6.9.1.4.1 WEIGH about 10 g of liquid sample or 3.3 g of solid sample, accurate to 0.2 mg. PLACE it in a 250 mL beaker; ADD 30 mL of water and 10 mL of nitric acid solution. COVER with a watch glass and boil for 1 min. After cooling to room temperature, transfer to a 250 mL volumetric flask; USE water to dilute to the mark and shake well. This solution is test solution C.

6.9.1.4.2 Respectively pipette 0.00 mL (blank), 1.00 mL, 2.00 mL, 3.00 mL of lead standard solution into four 50 mL volumetric flasks. ADD 1 mL of nitric acid solution; USE water to dilute to the mark and shake well. USE a micro-liquid feeding device to inject the prepared sample into the heating furnace; after drying, ashing and atomization, measure the absorbance at 283.3 nm. USE the mass concentration (μ g/L) of lead standard solution as the abscissa; USE the corresponding absorbance as the ordinate; DRAW the calibration curve and calculate the regression equation.

6.9.1.4.3 PIPETTE an appropriate volume of test solution C; operate according to 6.9.1.4.2, and determine. The mass concentration of lead is obtained from the calibration curve or regression equation.

6.9.1.5 Result calculation

The lead content is calculated as the mass fraction w_5 . The value is expressed

6.9.2.2.7 Sodium diethyldithiocarbamate solution: 100 g/L.

6.9.2.2.8 Lead standard stock solution: 1 mL contains 0.1 mg of Pb.

6.9.2.2.9 Lead standard solution: PIPETTE 10.00 mL of lead standard stock solution into a 100 mL volumetric flask; ADD 15 mL of nitric acid solution; USE water to dilute to the mark and shake well. 1 mL of this solution contains 0.01 mg of Pb.

6.9.2.3 Apparatus

6.9.2.3.1 Atomic absorption spectrometer.

6.9.2.3.2 Lead hollow cathode lamp.

6.9.2.4 Analytical procedure

6.9.2.4.1 WEIGH about 5 g of liquid sample or 1.5 g of solid sample, accurate to 0.2 mg. PLACE it in a 250 mL beaker; ADD 30 mL of water, 2 mL of nitric acid solution; COVER with a watch glass and boil for about 1 min; and cool to room temperature.

6.9.2.4.2 ADD 3 mL of ammonium citrate solution and 15 mL of ammonium sulfate solution; USE aqueous ammonia solution or hydrochloric acid solution, to adjust the pH value to 5.0~5.2 (use a pH meter). Then, respectively add 3 mL of sodium diethyldithiocarbamate solution and mix well.

6.9.2.4.3 After standing for 3 min, transfer to the separatory funnel. Successively add 25.00 mL of 4-methyl-2-pentanone; MIX for 2 min. After standing for another 10 min, discard the aqueous layer; COLLECT the extract in a dry volumetric flask.

6.9.2.4.4 Under the best working conditions of the instrument, at a wavelength of 283.3 nm, use the reagent blank for zero setting; determine the absorbance.

6.9.2.4.5 Respectively pipette 0.00 mL (blank), 2.5 mL, 5.00 mL, 7.5 mL of lead standard solution into 100 mL beakers; ADD water to about 30 mL. Follow the steps of 6.9.2.4.2~6.9.2.4.4. USE the measured absorbance as the ordinate; USE the corresponding lead mass (mg) as the abscissa; DRAW the calibration curve and calculate the regression equation.

6.9.2.5 Result calculation

The lead content is calculated as the mass fraction w_5 . The value is expressed in %, calculated according to formula (10):

mercury in the sample is reduced by potassium borohydride (KBH_4) to atomic mercury, which is brought by the carrier gas (argon) into the atomizer. Under the irradiation of a special mercury hollow cathode lamp, the ground state mercury atoms are excited to the upper state. When deactivated to the ground state, they emit fluorescence with a characteristic wavelength. Its fluorescence intensity is proportional to the mercury content. Quantitatively compare with the standard series.

6.11.1.2 Reagents and materials

6.11.1.2.1 Nitric acid: Guaranteed reagent.

6.11.1.2.2 Hydrochloric acid: Guaranteed reagent.

6.11.1.2.3 Nitric acid solution: 1+1.

6.11.1.2.4 Nitric acid solution: (1+4)

6.11.1.2.5 Hydrochloric acid solution: 1+19.

6.11.1.2.6 Potassium borohydride-sodium hydroxide solution: WEIGH 2.5 g of sodium hydroxide and 10.0 g of potassium borohydride in a polyethylene beaker; USE water to dissolve and dilute to 1000 mL. Prepare this solution at the time of use.

6.11.1.2.7 Mercury standard stock solution: 0.1 mg/mL.

6.11.1.2.8 Mercury standard solution (I): 5 $\mu\text{g/mL}$. PIPETTE 5 mL of mercury standard stock solution in a 100 mL volumetric flask; ADD 0.05 g of potassium dichromate, 5 mL of nitric acid; USE water to dilute to the mark. Prepare this solution at the time of use.

6.11.1.2.9 Mercury standard solution (II): 0.05 $\mu\text{g/mL}$. PIPETTE 1 mL of mercury standard solution (I) into a 100 mL volumetric flask; ADD 0.05 g of potassium dichromate, 5 mL of hydrochloric acid; USE water to dilute to the mark. Prepare this solution at the time of use.

6.11.1.3 Apparatus

6.11.1.3.1 Atomic fluorescence spectrophotometer.

6.11.1.3.2 Mercury hollow cathode lamp.

6.11.1.4 Drawing of calibration curve

6.11.1.4.1 Respectively take 0.00 mL (blank), 2.00 mL, 4.00 mL, 6.00 mL, 8.00 mL, 10.00 mL of mercury standard solution (II) into six 100 mL volumetric flasks.

(V=100);

m - The value of the mass of the test portion, in grams (g).

6.11.1.7 Allowable difference

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

6.11.2 Cold atomic absorption method

6.11.2.1 Method summary

In an acid medium, oxidize the mercury in the sample to divalent mercury ions; USE stannous chloride to reduce the mercury ions to mercury atoms; USE cold atomic absorption method to determine mercury.

6.11.2.2 Reagents and materials

6.11.2.2.1 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 and mix well.

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

6.11.2.2.3 Hydrochloric acid (guaranteed reagent) solution: 1+11.

6.11.2.2.4 Potassium permanganate (guaranteed reagent) solution: 10 g/L.

6.11.2.2.5 Hydroxylamine hydrochloride solution: 100 g/L.

6.11.2.2.6 Stannous chloride solution: 50 g/L. WEIGH 5.0 g of stannous chloride; PLACE it in a 200 mL beaker. ADD 10 mL of hydrochloric acid solution and an appropriate amount of water to dissolve it; DILUTE to 100 mL and mix well.

6.11.2.2.7 Mercury standard stock solution: 1 mL of the solution contains 0.1 mg of Hg.

6.11.2.2.8 Mercury standard solution: 1 mL contains 0.001 mg of Hg.

6.11.2.3 Apparatus

6.11.2.3.1 Atomic absorption spectrophotometer or mercury vapor meter.

6.11.2.3.2 Mercury hollow cathode lamp.

6.11.2.6 Allowable difference

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

6.12 Determination of chromium content

6.12.1 Method summary

At a wavelength of 429.0 nm, use electrically heated atomic absorption spectrometry to measure the absorbance of chromium atoms, to obtain the chromium content.

6.12.2 Reagents and materials

6.12.2.1 Nitric acid solution: 1+1.

6.12.2.2 Chromium standard stock solution: 0.1 mg/mL.

6.12.2.3 Chromium standard solution: PIPETTE 10.00 mL of chromium standard stock solution in a 1000 mL volumetric flask; ADD 20 mL of nitric acid solution; USE water to dilute to the mark and shake well. 1 mL of this solution contains 1 µg of Cr.

6.12.3 Apparatus

6.12.3.1 Micro-liquid feeding device: Equipped with a button type 5 µL~500 µL micro-liquid flowmeter or automatic sampler.

6.12.3.2 Electric heating atomic absorption analysis device: Electric heating method. Reverse grounding compensation can be carried out.

6.12.3.3 Heating furnace: Made of graphite or high-temperature-resistant metal. Atomic absorption spectrophotometer.

6.12.3.4 Chromium hollow cathode lamp.

6.12.4 Analytical procedure

6.12.4.1 Respectively pipette 0.00 mL (blank), 1.00 mL, 2.00 mL, 3.00 mL of chromium standard solution into four 50 mL volumetric flasks; ADD 1 mL of nitric acid solution; USE water to dilute to the mark and shake well. USE a micro-liquid feeding device to inject the prepared sample into the heating furnace; after drying, ashing and atomization, measure the absorbance at 429.0 nm. USE the mass concentration (µg/L) of chromium standard solution as the abscissa; USE the corresponding absorbance as the ordinate; DRAW the

polyaluminium chloride, see Appendix A.

7.3 Each lot of products shall not exceed 200 t for liquid; and shall not exceed 60 t for solid.

7.4 According to GB/T 6678, determine the number of sampling units.

7.5 When sampling liquid products, the sampler shall be deep into the barrel. The sampling volume from the upper, middle and lower parts shall not be less than 100 mL. MIX the sampled product well; TAKE out about 800 mL from it; DIVIDE it into two clean and dry glass bottles, and seal.

7.6 When sampling liquid products shipped in storage tanks, samplers shall be used to sample from the upper, middle, and lower parts of the tank. The sampling volume of each part is not less than 250 mL. MIX the sampled product well; TAKE out about 800 mL from it; DIVIDE it into two clean and dry glass bottles, and seal.

7.7 When sampling solid products, the sampler shall be inserted vertically into three-quarters of the depth of the bag for sampling. The sample taken from each bag shall not be less than 100 g. MIX the sampled product evenly; reduce it to about 500 g using the quartering method; DIVIDE it into two clean and dry glass bottles, and seal.

7.8 STICK a label on the sealed sample bottle, indicating the name of the manufacturer, product name, lot number, sampling date, and the name of the sampler. One bottle is for inspection; the other bottle is kept for 3 months for future reference.

7.9 Determine according to the comparison method of rounded value in GB/T 8170.

7.10 If there is one index in the inspection results that does not meet the requirements of this Standard, it shall re-sample from twice the number of packaging units for verification. If there is still one item in the verification results that does not meet the requirements of this Standard, the entire lot of products is nonconformity.

8 Marking, packaging, transportation and storage

8.1 The outer packaging of polyaluminium chloride shall have a firm and clear mark; which indicates the name of the manufacturer, product name, trademark, net mass, lot number, production date, standard number, and the "avoid rain" marking specified in GB/T 191.

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