GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

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Water Quality – Determination of Chloride – Silver Nitrate Titration Method

水质 氯化物的测定 硝酸银滴定法

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Water Quality – Determination of Chloride – Silver Nitrate Titration Method

1 Theme Content and Applicable Range

This Standard specifies using the silver nitrate titration method to determine the concentration of chloride in water.

This Standard is applicable to the determination of chloride in the natural water; it is also applicable to the highly mineralized water that has been diluted, such as salt water, seawater, etc.; as well as applicable to the domestic sewage and industrial wastewater that has removed the interference through the pretreatment.

This Standard is applicable to the chloride with concentration of 10~500mg/L. The water sample above such range can expand its determination range after dilution.

The bromide, iodide and cyanide can be titrated together with chloride. When the orthophosphate and polyphosphate separately exceed 250mg/L and 25mg/L, they have interference. When iron content exceeds 10 mg/L, the end-point is not obvious.

2 Principle

In the neutral or weak alkaline range (pH 6.5~10.5), take potassium chromate as an indicator; when using silver nitrate to titrate the chloride, since the solubility of silver chloride is less than that of the silver chromate, chloride ion is firstly precipitated fully; then the chromate was precipitated in the form of silver chromate; generate brick red, which indicates that the titration end-point is reached. The titration reaction of the precipitation is as follows:

$$Ag^+ + Cl^- \longrightarrow AgCl\downarrow$$

$$2Ag^+ + CrO_4 \longrightarrow Ag_2CrO_4\downarrow \text{ (Brick red)}$$

3 Reagents

Only use analytical reagent to prepare during the analyzing period; as well as distilled water or deionized water.

3.1 Potassium permanganate, *C* (1/5 $KMnO_4$) =0.01mol/L.

3.2 Hydrogen peroxide (H_2O_2), 30%.

3.3 Ethanol (C_6H_5OH), 95%.

3.4 Sulfuric acid solution, *C* (1/2 H_2SO_4) = 0.05mol/L.

3.5 Sodium hydroxide solution, *C* (NaOH) = 0.05mol/L.

3.6 Aluminum hydroxide suspension: dissolve 125g of potassium aluminum sulfate [$KAl(SO_4)_2 \cdot 12H_2O$] into 1L of distilled water; heating to 60°C; then slowly add 55mL of stronger ammonia water as stir, stand for about 1h; then move to larger bottle; use pouring method to repeatedly wash the precipitant till the eluate doesn't contain the chloride ions. Use water to dilute to about 300mL.

3.7 Sodium chloride standard solution, *C* (NaCl) = 0.0141mol/L, equivalent to 500mg/L chloride content: place the sodium chloride (NaCl) into a porcelain crucible to burn for 40~50min at 500~600°C. After cooling off in the dryer, take 8.2400g, dissolve into the distilled water; dilute to 1000mL in the volumetric flask. Pipette 10.0mL, accurately dilute to 100mL in the volumetric flask.

1.00ml of such standard solution contains about 0.50mg of chloride (Cl^-).

3.8 Silver nitrate standard solution, *C* ($AgNO_3$) = 0.0141 mol/L: take 2.3950g of silver nitrate ($AgNO_3$); dry for half an hour at 105°C; dissolve into the distilled water; dilute to 1000mL in the volumetric flask; store in the brown bottle.

Use sodium chloride standard solution (3.7) to calibrate its concentration:

Accurately pipette 25.00mL of sodium chloride standard solution (3.7) into 250mL conical flask; add 25mL of distilled water; take another conical flask to weigh 50mL of distilled water as blank. Separately add 1mL of potassium chromate solution (3.9); use the silver nitrate standard solution to titrate till the brick red precipitant just appears as continuously stir, which is the end-point. Calculate the chloride amount equivalent to the silver nitrate solution per milliliter; then calibrate its concentration; then do the final calibration.

1.00ml of such standard solution is equivalent to 0.50mg of chloride (Cl^-).

3.9 Potassium chromate solution, 50g/L: take 5g of potassium chromate (K_2CrO_4); dissolve into little amount of distilled water; titrate silver nitrate solution (3.8) till red precipitant is generated. Shake evenly, stand for 12h; then filter it, and use distilled water to dilute the filtrate to 100mL.

3.10 Phenolphthalein indicator solution: take 0.5g of phenolphthalein; dissolve into 50mL of 95% ethanol (3.3). Add 50mL of distilled water; then titrate 0.05mol/L sodium

hydroxide solution (3.5), so that the slight red appears.

4 Instruments

4.1 Conical flask, 250mL.

4.2 Burette, 25mL, brown.

4.3 Pipette, 50mL, 25mL.

5 Sample

Take representative water sample, place into the clean and chemically stable glass or polyethylene bottle. When storing, it is not necessary to add anti-corrosive agent.

6 Analytical Procedures

6.1 Interference exclusion

If there aren't various interferences below, such section can be omitted.

6.1.1 If the water sample is turbid and has a color, then take 150mL or appropriate amount of water sample to dilute to 150mL; then place into 250mL conical flask; add 2mL of aluminum hydroxide suspension (3.6); shake and filter; discard the 20mL of initial filtrate; then use clean conical flask to receive filtrate for spare-use.

6.1.2 If the organic substance content is high or the chroma is high, use muffle furnace ashing method to pre-treat the water sample. Take appropriate amount of waste water sample into porcelain evaporating dish; adjust pH value to be 8~9; place onto the water bath for dry; then place into muffle furnace to burn for 1h at 600°C; after taking out, cool off; add 10mL of distilled water; move into 250mL conical flask; use distilled water to wash for three times; then transfer them together into the conical flask; adjust pH value to be about 7; dilute to 50mL.

6.1.3 For the lighter chroma generated by organic substance, add 2mL of 0.01mol/L potassium permanganate (3.1), boil. Then titrate ethanol (3.3), so as to remove the excessive potassium permanganate till water sample fades; filter; the filtrate shall be stored in the conical flask for spare-use.

6.1.4 If the water sample contains sulfide, sulfite or thiosulfate, then add sodium hydroxide solution (3.5) to adjust the water sample into neutral or weakly alkaline; add 1mL of 30% hydrogen peroxide (3.2), shake evenly. After 1 minute, heat to 70~80°C; so as to remove the excessive hydrogen peroxide.

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