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**Inorganic chemical products for industrial use -
General method for determination of chloride content
- Potentiometric method**

无机化工产品中氯化物含量测定的通用方法 电位滴定法

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Inorganic chemical products for industrial use - General method for determination of chloride content - Potentiometric method

1 Scope

This standard specifies the general method for the determination of chloride content in inorganic chemical products - Potentiometric titration.

This standard applies to test solutions, which have a chloride ion content of 1 mg/L ~ 1500 mg/L. The chloride (calculated as Cl) content in the test solution, which is used for titration, is 0.01 mg ~ 75 mg. When the concentration of the silver nitrate standard solution used is less than 0.02 mol/L, the titration shall be carried out in an ethanol-water solution.

K^+ 、 Na^+ 、 Ca^{2+} 、 Mg^{2+} 、 Mn^{2+} 、 Pb^{2+} 、 Cu^{2+} 、 Ba^{2+} 、 NO_3^- 、 SO_4^{2-} 、 BO_3^{3-} 、 CO_3^{2-} 、 PO_4^{3-} does not interfere with the determination. The ions that form insoluble precipitates or complexes with Ag^+ , as well as MnO_4^- , etc. interfere with the determination. For the limit and elimination method, please refer to Appendix D (informative) and Appendix E (informative).

2 Normative reference

The provisions contained in the following standards constitute the provisions of this standard through quotation in this standard. When this standard is published, the versions shown are valid. All standards will be revised. All parties using this standard shall explore the possibility of using the latest version of the following standards.

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

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

3 Method summary

In acidic water or ethanol-water solution, use silver (silver-silver sulfide) electrode as the measuring electrode [see Appendix B (normative) for the

selection of electrode types]; use calomel electrode as the reference electrode; use silver nitrate standard titration solution for titration. The end point of the reaction is determined by means of a potential jump.

4 Reagents and materials

The reagents and water, which are used in this standard, refer to analytical reagents and the grade 3 water, which are specified in GB/T 6682, unless otherwise specified.

The preparations and products, which are used in this standard, shall be prepared in accordance with the provisions of GB/T 603, unless otherwise specified.

4.1 95% ethanol.

4.2 Saturated potassium nitrate solution.

4.3 Sodium hydroxide solution: 200 g/L.

4.4 Nitric acid solution 2 + 3.

4.5 Potassium chloride standard solution: $c(\text{KCl}) = 0.1 \text{ mol/L}$. Accurately weigh 3.728 g of the reference potassium chloride (accurate to 0.001 g), which was previously baked at 130 °C to a constant weight. Place it in a beaker. After adding water to dissolve, transfer it into a 500 mL volumetric flask. Use water to dilute it to the mark. Shake well.

Potassium chloride standard titration solution of 0.01 mol/L, 0.005 mol/L, 0.001 mol/L or other concentration: Accurately dilute the 0.1 mol/L potassium chloride standard titration solution to the required multiple.

4.6 Silver nitrate standard titration solution: $c(\text{AgNO}_3) = 0.1 \text{ mol/L}$.

Silver nitrate standard titration solution of 0.01 mol/L, 0.005 mol/L, 0.001 mol/L or other concentrations: Accurately dilute the 0.1 mol/L silver nitrate standard titration solution to the required multiple.

Calibration:

Use a pipette to take 5 mL or 10 mL of the potassium chloride standard solution of the selected concentration. Place it in a 50 mL beaker. Add 1 drop of bromophenol blue indicator solution. Add 1 ~ 2 drops of nitric acid solution, to make the solution just yellow. Then add 15 mL or 30 mL of ethanol. Put in the electromagnetic stirrer. Place the beaker on the electromagnetic stirrer. Start the stirrer. Insert the measuring electrode and the reference electrode into the

$$c = \frac{c_0 V_2}{V} \dots\dots\dots (2)$$

Where:

c_0 - The concentration of the potassium chloride standard solution taken, mol/L;

V_2 - The volume of the potassium chloride standard solution taken, mL.

4.7 Bromophenol blue indicator solution: 0.1% ethanol solution.

5 Instruments and equipment

5.1 Potentiometer: The accuracy is 2 mV/div. The range is -500 mV ~ +500 mV.

5.2 Reference electrode: Double-liquid junction type saturated calomel electrode, filled with saturated potassium chloride solution. During titration, the outer tube is filled with saturated potassium nitrate solution AND is connected to the calomel electrode.

5.3 Measuring electrode: Silver electrode or 0.5 mm silver wire (with 99.9% silver content; use shielded wire when connecting with potentiometer)

When the concentration of the silver nitrate standard solution used is lower than 0.005 mol/L, it shall use a silver electrode with a silver sulfide coating. The preparation method is detailed in Appendix A (normative)

5.4 Micro burette: The graduation value is 0.02 mL or 0.01 mL.

6 Analytical procedures

6.1 Preparation of test solution

Weigh an appropriate amount of the specimen. Treat it with an appropriate method. OR otherwise pipette an appropriate amount of the test solution after chemical treatment [to make the interference ion not greater than the specified limit, see Appendix D (informative)]. Place it in a beaker. Add 1 drop of Bromophenol blue indicator solution. Use sodium hydroxide solution or nitric acid solution to adjust the color of the solution just yellow. Transfer it to a volumetric flask of appropriate size. Add water to the mark. Shake well. This test solution is solution A. The concentration of chloride ions is 1 mg/L ~ 1.5 x 10³ mg/L.

6.2 Titration

Appendix E

(Informative)

Elimination method of ions interfering with determination

This Appendix cannot include the elimination methods of all interfering substances. It shall check the possible interference frequently. The processing methods described in E1 to E5 cannot cover all situations.

E1 Iodine and bromine

In addition to fluorine, iodine and bromine will react with silver nitrate, while titrating chlorine, which will interfere with the determination. The elimination method is to first use oxidant (such as hydrogen peroxide) to make oxidization, in a dilute nitric acid medium; then use an appropriate solvent to extract it.

E2 Oxidant

In an acidic solution, $\text{Cr}_2\text{O}_7^{2-}$ and ClO_3^- etc., can oxidize Cl^- to Cl_2 , which interferes with the determination. The elimination method is to control the pH of the tested solution, to make titration at pH 5 ~ 6.

MnO_4^- reacts with chlorine AND interferes with the determination. The elimination method is to slowly add an appropriate amount of 30% H_2O_2 -1 mol/L HNO_3 mixed solution (1 + 10) in the test solution, to make MnO_4^- fade.

The oxidant can oxidize the silver electrode. Use sodium sulfite, ascorbic acid solution or other reducing agents, that do not affect the measurement, to destroy the oxidant.

E3 Nitrite

NO_2^- and Ag^+ generate AgNO_2 precipitation. The elimination method of interference is to drop 10% sulfamic acid solution into the test solution; then heat and boil it, after there is no air bubble.

E4 Cyanide, thiocyanate, sulfide, thiosulfate, sulfite, etc.

The anions in these salts form insoluble precipitates with Ag^+ , which interfere with the determination. The elimination method is to slowly add an appropriate amount of 30% hydrogen peroxide solution in an alkaline medium; slowly heat it; finally boil until it, until there is no small bubble.

E5 Ammonium salt and iron (III) salt

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