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

无机化工产品中氯化物含量测定的通用方法 汞量法

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Inorganic chemical products for industrial use - General

method for determination of chloride content -

Mercurimetric method

1 Scope

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

This standard is applicable to specimen, which has a chloride (in terms of Cl) content of 0.01 mg ~ 80 mg. When the concentration of mercury nitrate standard solution used is less than 0.02 mol/L, the titration shall be carried out in an ethanol-water solution.

The ions K^+ , Na^+ , Ca^{2+} , Mg^{2+} , Ba^{2+} , Zn^{2+} , Pb^{2+} , NO_3^- , CO_3^{2-} , BO_3^{3-} do not interfere with the determination. The ions S^{2-} , SO_3^{2-} , SO_4^{2-} , PO_4^{3-} , $[Fe(CN)_6]^{3-}$, $[Fe(CN)_6]^{4-}$, $S_2O_3^{2-}$, NO_2^- , CNS^- , CN^- interfere with the determination; the limits and elimination methods are as shown in Appendix B (Normative) and Appendix C (Informative).

2 Normative references

The following standards contain provisions which, through reference in this standard, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties using this standard shall explore the possibility of using the latest edition 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 slightly acidic water or ethanol-aqueous solution, use strong ionized mercury nitrate standard titration solution, to convert chloride ion into weakly ionized mercuric chloride. Use diphenylcarbazone indicator and excess Hg^{2+} to generate purple complex compound, so as to determine the end point.

4 Reagents and materials

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

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

Safety tips: Mercury nitrate solution used in this standard is toxic; strong acid and strong alkali are corrosive, so users shall operate with care, to avoid splashing on the skin! If splashed on the skin, it shall be washed with water immediately, the severe cases shall be treated immediately.

4.1 Nitric acid solution: 1 + 1.

4.2 Nitric acid solution: 1 mol/L.

Measure 63 mL of nitric acid. Use water to dilute it to 1000 mL.

4.3 Sodium hydroxide solution: 1 mol/L.

Measure 52 mL of saturated sodium hydroxide solution. Use water to dilute it to 1000 mL.

4.4 Sodium chloride standard solution: $c(\text{NaCl}) = 0.1000 \text{ mol/L}$ or $c(\text{NaCl}) = 0.0500 \text{ mol/L}$.

Accurately weigh 5.844 g (or 2.922 g) of the benchmark sodium chloride, which was calcined to constant weight at $500^\circ\text{C} \sim 600^\circ\text{C}$, accurate to 0.0002 g. Place it in a beaker. Add a small amount of aqueous solution to dissolve it. Transfer all the solution into a 1000 mL volumetric flask. Add water to the mark. Shake well.

4.5 Sodium chloride standard solution $c(\text{NaCl}) = 0.0200 \text{ mol/L}$.

Dilute the sodium chloride standard solution (4.4) to the desired multiple accurately.

4.6 Mercury nitrate standard titration solution $c(1/2\text{Hg}(\text{NO}_3)_2)$ of about 0.1 mol/L OR $c[1/2\text{Hg}(\text{NO}_3)_2]$ of about 0.05 mol/L.

4.6.1 Preparation: Weigh 17.13 g (or 8.57 g) of mercury nitrate $[\text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}]$. Put it in a 250 mL beaker. Add 7 mL (or 4 mL) of nitric acid solution (4.1). Add a small amount of aqueous solution. Filter if necessary. Transfer it into a 1000 mL volumetric flask. Add water to the mark. Shake well.

Or, weigh 10.85 g (or 5.43 g) of mercury oxide. Put it in a 250 mL beaker. Add 20 mL (or 10 mL) of nitric acid solution (4.1). Add a small amount of aqueous solution. Filter if necessary. Transfer it into a 1000 mL volumetric flask. Add water to the mark. Shake

4.7.3 The amount concentration c (mol/L) of the substance in the mercury nitrate standard titration solution is calculated, according to formula (1).

4.8 Mercury nitrate standard titration solution: $c[1/2\text{Hg}(\text{NO}_3)_2]$ is about 0.01 mol/L, $c[1/2\text{Hg}(\text{NO}_3)_2]$ is about 0.005 mol/L, $c[1/2\text{Hg}(\text{NO}_3)_2]$ is about 0.001 mol/L or other concentrations.

Dilute the mercuric nitrate standard titration solution (4.6) to the required multiple accurately. Add an appropriate amount of nitric acid solution (4.1) during dilution, to prevent the hydrolysis of mercuric nitrate.

4.9 Bromophenol blue indicator solution: 1 g/L ethanol solution.

4.10 Diphenylcarbazone indicator solution: 5 g/L ethanol solution.

It shall be prepared again, when discoloration is insensitive.

5 Instruments and equipment

Micro-burette: Graduated value is 0.01 mL or 0.02 mL.

6 Analytical procedures

6.1 Preparation of test solutions

Weigh an appropriate amount of specimen. Use a suitable method to treat it. OR pipette an appropriate amount of the test solution after chemical treatment [so that the interfering ions do not exceed the specified limit, see Appendix B (normative), the chlorine content is 0.01 mg ~ 80 mg]. Place it in a conical flask. Control the total volume to be 100 mL ~ 200 mL (if the titration is performed in an ethanol-water solution, the total volume shall not be greater than 40 mL; the volume ratio of ethanol to water is 3:1). Add 2 ~ 3 drops of bromophenol blue indicator solution. Adjust the pH value of the solution to 2.5 ~ 3.5, according to one of the following steps.

If the solution is yellow, add sodium hydroxide solution (4.3) dropwise to blue. Then add nitric acid solution (4.2) dropwise, until it is just yellow. Add another 2 ~ 6 drops (in ethanol-water solution, it shall add another 2 ~ 3 drops);

If the solution is blue, add nitric acid solution (4.2) dropwise, until it turns yellow. Then add another 2 ~ 6 drops (2 ~ 3 drops in ethanol-water solution).

6.2 Titration

Add 1 mL of diphenylcarbazone indicator solution, into the test solution (6.1). Use the appropriate concentration [refer to Appendix A (normative)] of mercury nitrate standard titration solution (4.6, 4.7, 4.8), to titrate, until the color of the test solution changes

Appendix C

(Informative)

Elimination method of interfering ions

C1 SO_4^{2-}

Sulfuric acid is a dibasic acid, that interferes with the determination, due to a slight buffering effect, which is caused by step ionization.

The elimination method is as follows: Add an excess of 1 mol/L nitric acid solution (generally 1 mL ~ 2 mL), depending on the SO_4^{2-} content. Strictly control the required pH value. It is recommended to use a pH meter to control it, during initial adjustment.

C2 SO_3^{2-}

In an acidic solution, SO_3^{2-} reacts with Hg^{2+} , which consumes the standard titration solution of mercury nitrate, therefore interfering with the determination.

SO_3^{2-} is oxidized to SO_4^{2-} by hydrogen peroxide, in alkaline medium.

The elimination method is as follows: When the content is below 10 g/L, adjust the tested solution to near neutral (use bromophenol blue as indicator, which changes from yellow to blue). Add 2 mL of 1 mol/L sodium hydroxide solution. Slowly add appropriate amount of 30% hydrogen peroxide solution dropwise. Heat it slightly. Finally heat to boil it, until there is no formation of small bubbles. After cooling, use nitric acid to adjust the pH value, to prepare for determination.

C3 S^{2-}

S^{2-} and Hg^{2+} generate HgS precipitation, which affects the determination.

In an alkaline medium, use hydrogen peroxide to oxidize S^{2-} to SO_4^{2-} . The elimination method is the same as C2.

C4 Fe^{2+}

When the content is above 0.3 g/L, precipitation occurs during neutralization.

When the content is below 1 g/L, the precipitate immediately dissolves after adding 4 drops of 1 mol/L nitric acid solution. The slight yellow color does not affect the observation of the end point.

C5 CrO_4^{2-} and $\text{Cr}_2\text{O}_7^{2-}$

CrO_4^{2-} first turns into $\text{Cr}_2\text{O}_7^{2-}$ (orange red) in acidic medium. $\text{Cr}_2\text{O}_7^{2-}$ has strong oxidizing property, which oxidizes diphenylazocarbohydrazide and affects the determination.

When the CrO_4^{2-} content is less than 50 mg/L, titrate it immediately, after adding diphenylazocarbohydrazide. At the end point, the color changes from orange-red to purple-red. When the content is greater than 50 mg/L, adding an appropriate amount of barium nitrate can form BaCrO_4 precipitation. Filter and remove CrO_4^{2-} ; however, there are many procedures. It is recommended to use the potentiometric titration method, for determination.

C6 CN^-

CN^- and Hg^{2+} generate $\text{Hg}(\text{CN})_2$ precipitation or $\text{Hg}(\text{CN})_4^{2-}$ complex.

The elimination method is: Add formaldehyde, which is 2 times the content of CN^- to the tested liquid. Place for 20 min. Use nitric acid to adjust the pH value, to prepare for determination.

C7 $[\text{Fe}(\text{CN})_6]^{4-}$ and $[\text{Fe}(\text{CN})_6]^{3-}$

$[\text{Fe}(\text{CN})_6]^{4-}$ and $[\text{Fe}(\text{CN})_6]^{3-}$ generate precipitates with Hg^{2+} , which affects the determination.

The elimination method is: Add zinc nitrate, which is 2 ~ 3 times of the specimen, to the tested liquid. Heat to boiling. Cool it. Transfer all into a volumetric flask. Add water to the mark. Shake well. Let it stand for layering. Use slow filter paper to make dry filtration. Discard the initial filtrate. The remaining filtrate is used for determination.

C8 NH_4^+

The interaction of NH_4^+ and Hg^{2+} consumes the standard titration solution of mercury nitrate, which affects the determination.

The elimination method is: Adjust the liquid to be tested to alkaline. Heat it. Boil it, to remove NH_3 .

C9 CNS^-

CNS^- and Hg^{2+} generate $\text{Hg}(\text{CNS})_2$ precipitation, which interferes with the determination.

The elimination method is: Slowly add an appropriate amount of 30% hydrogen peroxide solution to the test liquid. Heat and boil, until no small bubbles are generated. Operation shall be performed in a fume hood.

C10 NO_2^-

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