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Analytical methods of anhydrous potassium fluoride

无水氟化钾分析方法

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Analytical methods of anhydrous potassium fluoride

WARNING -- Personnel using this Standard should have practical experience in formal laboratory work. This standard does not address all possible safety issues. Users are responsible for taking appropriate safety and health measures and ensuring compliance with the conditions stipulated in relevant national laws and regulations.

1 Scope

This Standard specifies the analytical methods for potassium fluoride content and various impurities in anhydrous potassium fluoride.

This Standard applies to anhydrous potassium fluoride products.

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 6682-2008, *Water for analytical laboratory use -- Specification and test methods*

HG/T 3696.1, *Inorganic chemicals for industrial use -- Preparations of standard and reagent solutions for chemical analysis -- Part 1: Preparations of standard volumetric solutions*

HG/T 3696.2, *Inorganic chemicals for industrial use. Preparations of standard and reagent solutions for chemical analysis. Part 2: Preparations of standard solutions for impurity*

HG/T 3696.3, *Inorganic chemicals for industrial use. Preparations of standard and reagent solutions for chemical analysis. Part 3: Preparations of reagent solutions*

3 Safety tips

Some of the reagents used in this analytical method are toxic or corrosive. Exercise caution when handling. If splashed on skin, rinse immediately with water. In severe cases, seek medical attention immediately. Potassium fluoride is inherently toxic and highly corrosive. Exercise caution when handling and wear protective equipment.

4 General

Unless otherwise specified, the reagents and water used in this Standard refer to analytically pure reagents and Grade three water as specified in GB/T 6682-2008. The standard titration solutions, impurity standard solutions, preparations, and products used in the tests, unless otherwise specified, are prepared in accordance with HG/T 3696.1, HG/T 3696.2, and HG/T 3696.3.

5 Determination of potassium fluoride content

5.1 Method summary

Dissolve the test material in water. Exchange the solution with a strong acid cation exchange resin to generate hydrofluoric acid. Titrate with a standard sodium hydroxide solution using phenolphthalein as an indicator to obtain the result.

5.2 Reagents and materials

5.2.1 Sodium hydroxide standard titration solution: $c(\text{NaOH}) \approx 0.5 \text{ mol/L}$.

5.2.2 Phenolphthalein indicator solution: 10 g/L.

5.2.3 Asbestos.

5.2.4 H-732 ion exchange resin.

5.2.5 Carbon dioxide-free water.

5.3 Instruments and equipment

5.3.1 Ion exchange column: polyethylene plastic tube; inner diameter is 25 mm; length is 500 mm; capable of controlling flow rate.

5.3.2 Plastic beaker.

5.4 Analysis procedure

5.4.1 Preparation of ion exchange column

Secure the ion exchange column to the stand. Close the stopcock. Fill the bottom of the column with 1 cm thick asbestos. Pour approximately 10 mL of water into the column to moisten it. Pour pretreated H-732 ion exchange resin into the column to a resin bed height of 400 mm. Before use, treat the column according to the instructions in Annex A.

5.4.2 Determination

Weigh approximately 1 g of specimen to the nearest 0.0002 g. Place in a 100 mL plastic beaker. Dissolve in 50 mL of CO₂-free water and inject into the ion exchange column. Use CO₂-free water as the eluent. Control the column flow rate to 5.0 mL/min~6.0 mL/min. Wash until the effluent is neutral (check with pH paper). Collect the effluent in a 500 mL plastic beaker. Add 2 drops of phenolphthalein indicator solution. Titrate with sodium hydroxide standard titrant until the solution turns pink and remains so for 30 s.

5.5 Result calculation

The potassium fluoride content is calculated as the mass fraction w_1 of potassium fluoride (KF) according to formula (1):

$$w_1 = \frac{(V/1\,000) cM}{m} \times 100\% - 1.639w_2 - 2.904w_4 - 1.210w_6 \quad \dots\dots\dots (1)$$

Where,

V - The volume of the sodium hydroxide standard titrant consumed during the titration, in milliliters (mL);

c - The exact concentration of the sodium hydroxide standard titrant, in moles per liter (mol/L);

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

w_2 - The mass fraction of chloride measured in Chapter 6;

1.639 - The conversion factor for chlorine to potassium fluoride;

w_4 - The mass fraction of free acid measured in Chapter 8;

2.904 - The conversion factor for hydrofluoric acid to potassium fluoride;

w_6 - The mass fraction of sulfate measured in Chapter 9;

1.210 - The conversion factor for sulfate to potassium fluoride;

M - The molar mass of potassium fluoride (KF), in grams per mole (g/mol) (M = 58.10).

The arithmetic mean of the parallel determination results is taken as the determination result. The absolute difference between the two parallel determination results shall not exceed 0.2%.

6 Determination of chloride content

6.1 Method summary

milliliters (mL);

V_0 - The volume of the silver nitrate standard titrant consumed during the blank test, in milliliters (mL);

c - The exact concentration of the silver nitrate standard titrant, in moles per liter (mol/L);

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

M - The molar mass of chlorine (Cl), in grams per mole (g/mol) ($M = 35.45$).

The arithmetic mean of the parallel determination results is taken as the determination result. The absolute difference between the two parallel determination results shall not exceed 0.01%.

7 Determination of loss on ignition

7.1 Apparatus and equipment

7.1.1 Platinum crucible.

7.1.2 High-temperature furnace: the temperature can be controlled within $650^{\circ}\text{C} \pm 20^{\circ}\text{C}$.

7.2 Analysis steps

Weigh approximately 5 g of specimen to the nearest 0.0002 g in a platinum crucible that has been calcined at $650^{\circ}\text{C} \pm 20^{\circ}\text{C}$ to a constant mass. Place the crucible in a high-temperature furnace at $650^{\circ}\text{C} \pm 20^{\circ}\text{C}$ for 2 h, remove it, and weigh it after cooling.

7.3 Result calculation

The loss on ignition is calculated as mass fraction w_3 according to formula (3):

$$w_3 = \frac{m_1 - m_2}{m} \times 100\% \quad \dots\dots\dots (3)$$

Where,

m_1 - The mass of the test material and crucible before ignition, in grams (g);

m_2 - The mass of the test material and crucible after ignition, in grams (g);

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

The arithmetic mean of the parallel determination results is taken as the determination result. The absolute difference between the two parallel determination results shall not

exceed 0.05%.

8 Determination of free acid or free base content

8.1 Reagents and materials

8.1.1 Sodium hydroxide standard titration solution: $c(\text{NaOH}) \approx 0.05 \text{ mol/L}$.

8.1.2 Hydrochloric acid standard titration solution: $c(\text{HCl}) \approx 0.05 \text{ mol/L}$.

8.1.3 Phenolphthalein indicator solution: 10 g/L.

8.1.4 Carbon dioxide-free water.

8.2 Apparatus

8.2.1 Plastic beaker.

8.2.2 Microburette: the graduation is 0.02 mL or 0.05 mL.

8.3 Analysis steps

Weigh approximately 5 g of specimen to the nearest 0.0002 g. Place in a 250 mL plastic beaker. Dissolve in 50 mL of carbon dioxide-free water. Add 2 drops of phenolphthalein indicator solution.

If the test solution is colorless, titrate with sodium hydroxide standard solution until the solution turns pink and remains pink for 30 s.

If the test solution is red, titrate with standard hydrochloric acid solution until the solution becomes colorless.

8.4 Result calculation

The free acid content is calculated as the mass fraction w_4 of hydrofluoric acid (HF) according to formula (4):

$$w_4 = \frac{(V/1\,000) cM}{m} \times 100\% \quad \dots\dots\dots (4)$$

Where,

V - The volume of the sodium hydroxide standard titrant consumed during the titration, in milliliters (mL);

c - The exact concentration of the sodium hydroxide standard titrant, in moles per liter (mol/L);

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

M - The molar mass of hydrofluoric acid (HF), in grams per mole (g/mol) (M = 20.01).

The free alkali content is calculated as the mass fraction w_5 of potassium hydroxide (KOH) according to formula (5):

$$w_5 = \frac{(V/1\,000) cM}{m} \times 100\% \quad \dots\dots\dots (5)$$

Where,

V - The volume of the standard hydrochloric acid solution consumed during the titration, in milliliters (mL);

c - The exact concentration of the standard hydrochloric acid solution, in moles per liter (mol/L);

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

M - The molar mass of potassium hydroxide (KOH), in grams per mole (g/mol) (M = 56.11).

The arithmetic mean of the parallel determination results is taken as the determination result. The absolute difference between the two parallel determination results shall not exceed 0.02%.

9 Determination of sulfate content

9.1 Method summary

The specimen is placed in a hydrochloric acid medium. Boric acid is added to complex the fluoride ions. Then, an excess of barium chloride solution is added to precipitate the sulfate ions into barium sulfate. Calculate the sulfate content based on the weight of the barium sulfate.

9.2 Reagents

9.2.1 Boric acid.

9.2.2 Hydrochloric acid solution: 1 + 3.

9.2.3 Barium chloride solution: 100 g/L.

9.2.4 Methyl orange indicator solution: 1 g/L.

9.3 Instruments and equipment

0.4116 - The coefficient for converting barium sulfate to sulfate ions;

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

The arithmetic mean of the parallel determination results is taken as the determination result. The absolute difference between the two parallel determination results shall not exceed 0.05%.

10 Determination of fluorosilicate content

10.1 Method summary

Fluoride ions in the specimen react with boric acid to form BF_4^- . Silicic acid reacts quantitatively with ammonium molybdate in a slightly acidic solution to form silicomolybdate heteropolyacid (silicomolybdate yellow). Adding a reducing agent reduces silicomolybdate yellow to silicomolybdate blue. Measure the color at 795 nm using a spectrophotometer or UV-visible spectrophotometer.

10.2 Reagents

10.2.1 Boric acid solution: 40 g/L.

10.2.2 Sulfuric acid solution: 1 + 33.

10.2.3 Ammonium molybdate solution: 100 g/L (refresh if precipitation occurs).

10.2.4 Oxalic acid solution: 50 g/L.

10.2.5 Stannous chloride hydrochloric acid solution: 20 g/L.

10.2.6 Silica standard solution: 1 mL of solution contains 0.01 mg of silicon dioxide (SiO_2).

Pipette 1.00 mL of the silica standard solution prepared according to HG/T 3696.2 into a 100 mL volumetric flask. Dilute to the mark with water and shake well. Prepare this solution immediately before use.

10.2.7 Saturated 2,4-dinitrophenol indicator solution.

10.3 Instrument

10.3.1 Spectrophotometer or UV-visible spectrophotometer: equipped with a 2 cm cuvette.

10.3.2 Plastic beaker.

10.4 Analysis steps

result. The absolute difference between the two parallel determination results shall not exceed 0.005%.

11 Determination of water-insoluble matter content

11.1 Method summary

Dissolve the specimen. Filter out the water-insoluble matter. Burn in a high-temperature furnace until the mass remains constant. Calculate the water-insoluble matter content.

11.2 Reagents

Barium chloride solution: 100 g/L.

11.3 Instruments and equipment

11.3.1 Plastic beaker (polytetrafluoroethylene).

11.3.2 Plastic funnel.

11.3.3 High-temperature furnace: the temperature can be controlled within $500^{\circ}\text{C} \pm 20^{\circ}\text{C}$.

11.4 Analysis steps

Weigh approximately 30 g of specimen to the nearest 0.01 g. Place in a 250mL plastic beaker. Add 100 mL of water and shake well. Heat the plastic beaker on a hot plate at low temperature to completely dissolve the specimen. Filter through a plastic funnel using slow-flow quantitative filter paper. Wash with water until fluoride ions are free (test with barium chloride solution). Place the filter paper in a porcelain crucible that has been calcined at $500^{\circ}\text{C} \pm 20^{\circ}\text{C}$ to a constant mass. Ash the crucible. Then calcine the crucible in a high-temperature furnace at $500^{\circ}\text{C} \pm 20^{\circ}\text{C}$ until the mass is constant. Remove the crucible. Cool and weigh.

11.5 Result calculation

The water-insoluble content is expressed as mass fraction w_8 and is calculated according to formula (8):

$$w_8 = \frac{m_1 - m_2}{m} \times 100\% \quad \dots\dots\dots (8)$$

Where,

m_1 - The mass of the porcelain crucible and water-insoluble substance, in grams (g);

m_2 - The mass of the porcelain crucible, in grams (g);

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

The arithmetic mean of the parallel determination results is taken as the determination result. The absolute difference between the two parallel determination results shall not exceed 0.05%.

12 Determination of arsenic, iron, lead, chromium, silicon, boron, calcium and nickel content

12.1 Method summary

Inductively coupled plasma atomic emission spectrometry (ICP-AES) is used for simultaneous determination of multiple elements.

12.2 Reagents

12.2.1 Nitric acid solution: 2+98 (guaranteed reagent).

12.2.2 Nitric acid solution: 1+10 (guaranteed reagent).

12.2.3 Mixed standard solution A: 1 mL of solution contains 0.02 mg each of arsenic (As), iron (Fe), lead (Pb), chromium (Cr), boron (B), calcium (Ca), and nickel (Ni).

Pipette 2.00 mL of arsenic, iron, lead, chromium, boron, calcium, and nickel standard solutions prepared according to HG/T 3696.2 into a 100 mL volumetric flask. Dilute to the mark with nitric acid solution (12.2.1). Shake well. It shall be valid for 6~12 months.

12.2.4 Standard solution B: 1 mL of solution contains 0.05 mg of silicon (Si).

Pipette 5.00 mL of the silicon standard solution prepared according to HG/T 3696.2 into a 100 mL volumetric flask. Dilute to the mark with nitric acid solution (12.2.1). Shake well. It shall be valid for 6~12 months.

12.2.5 Grade 1 water: comply with the requirements of GB/T 6682-2008.

12.3 Instruments and equipment

12.3.1 Inductively coupled plasma-atomic emission spectrometer (ICP-AES)

12.3.2 Fluorine-resistant torch

12.3.3 High-salt nebulizer

12.3.4 Plastic beaker (PTFE)

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