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Potassium Nitrate for Industrial Use

工业硝酸钾

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Table of Contents

Foreword 3
1 Scope 5
2 Normative References 5
3 Terms and Definitions 6
4 Molecular Formula and Relative Molecular Mass 6
5 Classification 6
6 Requirements 6
7 Test Methods 7
8 Inspection Rules 22
9 Marking and Labeling 23
10 Packaging, Transportation and Storage 24
Appendix A (informative) Commonly Used ICP-OES Spectral Lines and Detection Limits 25
Bibliography 26

Potassium Nitrate for Industrial Use

WARNING: in accordance with the provisions of Chapter 6 in GB 12268-2012, this product belongs to Category 5, Item 5.1 oxidizing substances, and caution shall be exercised when handling it. Personnel using this document shall have practical experience in formal laboratory work. This document does not point out all possible safety issues. Users are responsible for taking appropriate safety and health measures and ensuring the compliance with the conditions stipulated in relevant national regulations.

1 Scope

This document specifies the classification, requirements, test methods, inspection rules, marking, labeling, packaging, transportation and storage of potassium nitrate for industrial use.

This document is applicable to potassium nitrate for industrial use.

NOTE: this product is mainly used in molten salt, black powder, fuses, pharmaceutical intermediates, optical glass, ammonia catalyst, metal heat treatment, enamel and other industries.

2 Normative References

The contents of the following documents constitute indispensable clauses of this document through the normative references in the text. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

GB/T 191-2008 Packaging - Pictorial Marking for Handling of Goods

GB/T 3051-2000 Inorganic Chemical Products for Industrial Use - General Method for Determination of Chloride Content - Mercurimetric Method

GB/T 3600-2000 Fertilizers - Determination of Ammoniacal Nitrogen Content - Formaldehyde Method

GB/T 6678 General Principles for Sampling Chemical Products

GB/T 6682-2008 Water for Analytical Laboratory Use - Specification and Test Methods

GB/T 8170 Rules of Rounding off for Numerical Values & Expression and Judgement of Limiting Values

GB/T 23945-2009 Inorganic Chemicals for Industrial Use - General Method for the Determination of Chloride Content - Visible Turbidimetric Method

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

JT/T 617 (all parts) Regulations Concerning Road Transportation of Dangerous Goods

3 Terms and Definitions

This document does not have terms or definitions that need to be defined.

4 Molecular Formula and Relative Molecular Mass

Molecular formula: KNO_3

Relative molecular mass: 101.10 (in accordance with the international relative atomic mass in 2018)

5 Classification

Potassium nitrate for industrial use is classified into three categories. The main purposes are as follows:

---Category I products are mainly used for molten salt manufacturing;

---Category II products are divided into two types. Type I products are mainly used for the manufacturing of black powder, fuses, pharmaceutical intermediates and glass clarifiers, etc. Type II products are mainly used for metal heat treatment and the manufacturing of enamel paint, etc.;

---Category III products are products added inorganic salts as anti-caking agents and are mainly used as fluxes for glass and ceramics.

6 Requirements

6.1 Appearance: potassium nitrate for industrial use is white crystal or spherical particles.

6.2 Potassium nitrate for industrial use shall be tested in accordance with the test methods specified in this document and shall comply with the stipulations of Table 1.

3696.1, HG/T 3696.2 and HG/T 3696.3.

7.2 Appearance Inspection

Under natural light, adopt the method of visual inspection for determination.

7.3 Determination of Potassium Nitrate Content

7.3.1 Principle

In neutral medium, potassium ions react with sodium tetraphenylborate to generate potassium tetraphenylborate precipitate. If ammonium ions are present, formaldehyde solution can be added to eliminate the interference of ammonium ions. In accordance with the mass of the generated potassium tetraphenylborate, determine the potassium nitrate content. Its main reaction formula is as follows:

$$K^+ + [B(C_6H_5)_4]^- \rightarrow K[B(C_6H_5)_4]\downarrow$$

7.3.2 Reagents or materials

7.3.2.1 Absolute ethanol.

7.3.2.2 Formaldehyde solution: filter before use.

7.3.2.3 Acetic acid solution: 1 + 100.

7.3.2.4 Sodium hydroxide solution: 4 g/L.

7.3.2.5 Sodium tetraphenylborate ethanol solution.

7.3.2.6 Saturated ethanol solution of potassium tetraphenylborate.

7.3.2.7 Methyl orange indicator solution: 1 g/L.

7.3.2.8 Phenolphthalein indicator solution: 10 g/L.

7.3.3 Instruments and equipment

7.3.3.1 Electric thermostatic drying oven: the temperature can be controlled at 120 °C ± 2 °C.

7.3.3.2 Glass sand crucible: with a filter plate pore size of 5 μm ~ 15 μm.

7.3.4 Test steps

7.3.4.1 Preparation of test solution

Weigh-take 1.0 g ~ 1.2 g of sample, accurate to 0.0002 g, place it in a 100 mL beaker. Add water to dissolve it, transfer the solution to a 500 mL volumetric flask, use water to dilute to the scale and shake it well.

7.3.4.2 Test

Use a pipette to transfer 25 mL of the test solution, place it in a 150 mL beaker, add 20 mL of water and 2 drops of methyl orange indicator solution, and use acetic acid solution to adjust the solution to just turn red. If it contains ammonium salt, add 1 ~ 2 drops of phenolphthalein indicator solution and 2 mL of formaldehyde solution, and use sodium hydroxide solution to adjust it to turn reddish. Use a constant-temperature water bath to heat the solution to 45 °C (continue to keep the solution in reddish, and if there is any precipitate, filter and thoroughly wash it), while stirring, dropwise add 8 mL of sodium tetraphenylborate ethanol solution (the dropping time is about 5 minutes) and continue stirring for 1 minute. After letting it stand for 30 minutes, use a glass sand crucible that has been dried to a constant mass in an electric thermostatic drying oven at 120 °C ± 2 °C to suction filter it, use 20 mL of saturated ethanol solution of potassium tetraphenylborate to transfer the precipitate, and use 15 mL of saturated ethanol solution of potassium tetraphenylborate to wash the precipitate in 3 ~ 4 times (it shall be drained each time). Then, use 2 mL of absolute ethanol to wash once along the inner wall of the crucible, and drain it. In an electric thermostatic drying oven at 120 °C ± 2 °C, dry it, until reaching a constant mass.

7.3.4.3 Test data processing

The potassium nitrate content, which is calculated as the mass fraction w_1 of potassium nitrate (KNO_3), shall be calculated in accordance with Formula (1):

$$w_1 = \frac{0.282\ 2(m_2 - m_1)}{m(V_1/V)(1 - w_2)} \times 100\% - 3.37w_6 \qquad \cdots\cdots(1)$$

Where,

0.2822---the coefficient for converting potassium tetraphenylborate into potassium nitrate;

m_2---the mass of the glass sand crucible and the potassium tetraphenylborate precipitate after drying to a constant mass, expressed in (g);

m_1---the mass of the glass sand crucible after drying to a constant mass, expressed in (g);

m---the mass of the sample, expressed in (g);

V_1---the volume of the test solution taken in 7.3.4.2, expressed in (mL);

V---the volume of the test solution taken in 7.3.4.1, expressed in (mL);

w_2---the moisture content determined in 7.4;

3.37---the coefficient for converting carbonate to potassium nitrate;

w_6---the mass fraction of carbonate determined in 7.8.

Take the arithmetic mean of the parallel determination results as the determination result. The

Transfer all into a 500 mL (V_1) volumetric flask, use water to dilute to the scale and shake it well. This solution is test solution A, which is used for the determination of chloride, sulfate, carbonate and ammonium salt content.

7.6.1.4.2 Preparation of reference solution

Add 50 mL of water to a 250 mL conical flask, add 3 g of urea, and heat to dissolve it. At a slight boil, dropwise add nitric acid (1 + 1) solution, until no fine bubbles are generated, then, cool it. Add 2 ~ 3 drops of bromophenol blue indicator solution, use sodium hydroxide (1 mol/L) solution to adjust the solution, until it turns blue, then, use nitric acid (1 mol/L) solution to adjust the solution, until it turns from blue to yellow, then, add an overdose of 2 ~ 6 drops. Add 1.0 mL of phenylazoformic acid 2-phenylhydrazide indicator solution, and use a microburette to titrate it with a mercury nitrate standard titration solution with a concentration c [1/2 $Hg(NO_3)_2$] of 0.05 mol/L, until the solution turns purple red. Record the volume (V_0) of the mercury nitrate standard titration solution used. Prepare this solution before use.

7.6.1.4.3 Test

Use a pipette to transfer 50 mL (V_2) of test solution A (see 7.6.1.4.1), place it in a 250 mL conical flask. Add 3 g of urea and heat to dissolve it. At a slight boil, dropwise add nitric acid (1 + 1) solution, until no fine bubbles are generated, then, cool it. Add 2 ~ 3 drops of bromophenol blue indicator solution, use sodium hydroxide (1 mol/L) solution to adjust the solution, until it turns blue. Then, use nitric acid (1 mol/L) solution to adjust the solution, until it turns from blue to yellow, then, add an overdose of 2 ~ 6 drops. Add 1.0 mL of phenylazoformic acid 2-phenylhydrazide indicator solution, and use a mercury nitrate standard titration solution with a concentration c [1/2 $Hg(NO_3)_2$] of 0.05 mol/L to titrate it, until the solution turns from yellow to purple red, which is the same as the reference solution, which is the end point. The treatment of mercury-containing waste liquid shall be carried out in accordance with Appendix D of GB/T 3051-2000.

7.6.1.5 Test data processing

Chloride, which is calculated as the mass fraction w_4 of chlorine (Cl), shall be calculated in accordance with Formula (4):

$$w_4 = \frac{(V - V_0)cM \times 10^{-3}}{m(V_2/V_1)} \times 100\% \qquad \cdots\cdots(4)$$

Where,

V---the volume of the mercury nitrate standard titration solution consumed during the determination, expressed in (mL);

V_0---the volume of the mercury nitrate standard titration solution consumed when preparing the reference solution, expressed in (mL);

c---the accurate value of the concentration of the mercury nitrate standard titration solution,

expressed in (mol/L);

M---the numerical value of the molar mass of chloride (calculated as Cl), expressed in (g/mol) [M(Cl) = 35.45];

m---the mass of the sample contained in test solution A (see 7.6.1.4.1) in 7.6.1.4.1, expressed in (g);

V_2---the volume of test solution A (see 7.6.1.4.1) transferred in 7.6.1.4.3, expressed in (mL);

V_1---the volume of test solution A (see 7.6.1.4.1) in 7.6.1.4.1, expressed in (mL).

Take the arithmetic mean of parallel determination results as the determination result. The absolute difference between two parallel determination results shall not be greater than 0.002%.

7.6.2 Visible turbidimetric method

7.6.2.1 Principle

Same as Chapter 3 of GB/T 23945-2009.

7.6.2.2 Reagents or materials

Same as Chapter 6 of GB/T 23945-2009.

7.6.2.3 Test steps

Use a pipette to transfer 5 mL of test solution A (see 7.6.1.4.1) into a 50 mL colorimetric tube, add water to approximately 40 mL, add 1.0 mL of nitric acid solution and 1.0 mL of silver nitrate solution. Use water to dilute to the scale and shake it well, then, let it stand for 10 minutes. If the white turbidity generated is deeper than the standard turbidity solution, then, it does not comply with the indicator requirements specified in this document, otherwise, it complies with the indicator requirements specified in this document.

The standard turbidity solution is to pipette (Category I: 1.00 mL; Category II Type I, Category II Type II and Category III: 2.00 mL) chloride standard solution [1 mL contains 0.10 mg of chlorine (Cl)], and handle it at the same time in the same way as the test solution.

7.7 Determination of Sulfate Content

7.7.1 Gravimetric method (arbitration method)

7.7.1.1 Principle

Sulfate can react with barium ions to generate barium sulfate precipitate under acidic conditions. Weigh the mass of the generated barium sulfate. The main reaction formula is as follows:

$$Ba^{2+} + SO_4^{2-} = BaSO_4 \downarrow$$

m---the mass of the sample contained in test solution A (see 7.6.1.4.1) in 7.6.1.4.1, expressed in (g);

V_2---the volume of test solution A (see 7.6.1.4.1) transferred in 7.7.1.4, expressed in (mL);

V_1---the volume of test solution A (see 7.6.1.4.1) in 7.6.1.4.1, expressed in (mL).

Take the arithmetic mean of parallel determination results as the determination result. The absolute difference between two parallel determination results shall not be greater than 0.005%.

7.7.2 Visible turbidimetric method

7.7.2.1 Principle

Sulfate can react with barium ions to generate barium sulfate precipitate under acidic conditions. When the sulfate content is relatively low, a suspension is formed, which can be used for the determination of sulfate content. Its main reaction formula is as follows:

$$Ba^{2+} + SO_4^{2-} = BaSO_4 \downarrow$$

7.7.2.2 Reagents or materials

7.7.2.2.1 Hydrochloric acid solution: 1 + 4.

7.7.2.2.2 Barium chloride solution: 100 g/L.

7.7.2.2.3 Sulfate standard solution: 1 mL of solution contains 0.1 mg of sulfate (SO_4). Use a pipette to transfer 10 mL of the sulfate standard solution prepared in accordance with HG/T 3696.2, place it in a 100 mL volumetric flask, use water to dilute to the scale and shake it well.

7.7.2.3 Test steps

Use a pipette to transfer 5 mL of test solution A (see 7.6.1.4.1) into a 100 mL colorimetric tube, add water to approximately 75 mL, add 1 mL of hydrochloric acid solution and 2 mL of barium chloride solution. Use water to dilute to the scale and shake it well. If the white turbidity generated is deeper than the standard turbidity solution, then, it does not comply with the indicator requirements specified in this document, otherwise, it complies with the indicator requirements specified in this document.

The standard turbidity solution is to pipette (Category I and Category II Type I: 0.50 mL; Category II Type II and Category III: 1.00 mL) sulfate standard solution, transfer it into a 100 mL colorimetric tube, add water to approximately 75 mL, and handle it at the same time in the same way as the test solution of the same volume.

7.8 Determination of Carbonate Content

7.8.1.1 Principle

Take methyl orange as the indicator solution and use salt standard titration solution for titration.

7.8.1.2 Reagents or materials

7.8.1.2.1 Hydrochloric acid standard titration solution: c(HCl) ≈ 0.1 mol/L.

7.8.1.2.2 Hydrochloric acid standard titration solution: c(HCl) ≈ 0.5 mol/L.

7.8.1.2.3 Methyl orange indicator solution: 1 g/L.

7.8.1.3 Test steps

Use a pipette to transfer 50 mL or 5 mL (Category III) (V_2) of test solution A (see 7.6.1.4.1), place it in a 250 mL conical flask, and add 3 drops of methyl orange indicator solution. Use c(HCl) ≈ 0.1 mol/L or c(HCl) ≈ 0.5 mol/L hydrochloric acid standard titration solution to titrate it, until the solution turns orange.

7.8.1.4 Test data processing

The carbonate content, which is calculated as the mass fraction w_6 of carbonate (CO_3), shall be calculated in accordance with Formula (6):

$$w_6 = \frac{VcM \times 10^{-3}}{m(V_2/V_1)} \times 100\% \qquad \cdots\cdots(6)$$

Where,

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

c---the accurate value of the concentration of hydrochloric acid standard titration solution, expressed in (mol/L);

M---the numerical value of the molar mass of carbonate, expressed in (g/mol) [M(1/2 CO_3) = 29.99];

m---the mass of the sample contained in test solution A (see 7.6.1.4.1) in 7.6.1.4.1, expressed in (g);

V_2---the volume of test solution A (see 7.6.1.4.1) transferred in 7.8.1.3, expressed in (mL);

V_1---the volume of test solution A (see 7.6.1.4.1) in 7.6.1.4.1, expressed in (mL).

Take the arithmetic mean of parallel determination results as the determination result. The absolute difference between two parallel determination results shall not be greater than 0.002% for Category I and Category II products, and not greater than 0.02% for Category III products.

7.9 Determination of Ammonium Salt Content

7.9.2 Visual colorimetric method

7.9.2.1 Principle

In an alkaline solution, free ammonia or combined ammonium reacts with Nessler's reagent and generates a light yellow to brown colloidal compound. When the ammonium content is relatively high, the product is a reddish-brown precipitate. When the ammonium content is relatively low, then, a stable suspension is formed, which can be used for the visual colorimetric determination of ammonium salt. Its main reaction formula is as follows:

$$2[HgI_4]_2^- + 4OH^- + NH_4^+ = [Hg_2ONH_2]I\downarrow + 7I^- + 3H_2O$$

7.9.2.2 Reagents or materials

7.9.2.2.1 Sodium hydroxide solution: 320 g/L.

7.9.2.2.2 Nessler's reagent.

7.9.2.2.3 Ammonium standard solution: 1 mL of solution contains 0.1 mg of ammonium (NH_4). Use a pipette to transfer 10 mL of the ammonium standard solution prepared in accordance with HG/T 3696.2, place it in a 100 mL volumetric flask, use water to dilute to the scale and shake it well.

7.9.2.3 Test steps

Use a pipette to transfer 5 mL of test solution A (see 7.6.1.4.1) into a 100 mL colorimetric tube, add water to approximately 75 mL, add 3 mL of sodium hydroxide solution and 2 mL of Nessler's reagent. Use water to dilute to the scale and shake it well. If the generated color is deeper than the standard colorimetric solution, then, it does not comply with the indicator requirements specified in this document, otherwise, it complies with the indicator requirements specified in this document.

The standard colorimetric solution is to pipette ammonium standard solution [2.00 mL (Category I) and 7.00 mL (Category II Type I)], transfer into a 100 mL colorimetric tube, add water to approximately 75 mL, and handle it at the same time in the same way as the test solution of the same volume.

7.10 Determination of Moisture Absorption Rate

7.10.1 Principle

Place the sample after the moisture content determination in a desiccator containing a saturated potassium nitrate solution. At a certain temperature, place it for a certain period of time, then, examine the moisture absorption of the sample.

7.10.2 Reagents or materials

Saturated potassium nitrate solution.

7.10.3 Instruments and equipment

Desiccator: with a diameter of Φ140 mm. The bottom layer contains 100 mL of saturated potassium nitrate solution.

7.10.4 Test steps

Place the sample that has been dried to a constant mass in 7.4, together with the weighing bottle, in a desiccator, remove the cap of the weighing bottle, and properly cover the desiccator. Place it in an environment of 20 °C ± 2 °C for 6 hours, take out the weighing bottle and the cap, use filter paper to dry the surface moisture, and weigh it.

7.10.5 Test data processing

The moisture absorption rate, which is calculated as the mass fraction w_8, shall be calculated in accordance with Formula (8):

$$w_8 = \frac{m - m_1}{m} \times 100\% \qquad \cdots\cdots (8)$$

Where,

m---the mass of the sample that has been dried to a constant mass in 7.4, expressed in (g);

m_1---the mass of the sample after absorbing moisture, expressed in (g).

Take the arithmetic mean of two parallel determination results as the determination result. The absolute difference of parallel determination results shall not be greater than 0.05%.

7.11 Determination of Looseness

7.11.1 Method summary

Freely drop bagged samples that have been stacked for a certain period of time onto a hard surface from a height of 1 m. After sieving, weigh the mass of the sample left on the sieve.

7.11.2 Instruments

7.11.2.1 Test sieve: 950 mm long, 600 mm wide, with a wooden frame about 120 mm high, and the mesh size is 4.75 mm.

7.11.2.2 Stopwatch.

7.11.2.3 Platform scale: 10 kg, with a division value of 0.1 kg.

7.11.3 Analysis steps

magnesium (Mg), barium (Ba), zinc (Zn), manganese (Mn), copper (Cu), cadmium (Cd), chromium (Cr) and lead (Pb), 10 μg each. Use a pipette to respectively transfer 10 mL of iron (Fe), calcium (Ca), magnesium (Mg), barium (Ba), zinc (Zn), manganese (Mn), copper (Cu), cadmium (Cd), chromium (Cr) and lead (Pb) standard solutions, place them in 100 mL volumetric flasks, use water to dilute to the scale and shake them well.

7.12.3 Instruments and equipment

Inductively coupled plasma optical emission spectrometer (ICP-OES): determine 1 mg/L or 10 mg/L multi-element mixed standard solution and repeat the determination 10 times with an RSD ≤ 0.5%. See Appendix A for the commonly used ICP-OES spectral lines and detection limits of each determined ion.

7.12.4 Test steps

7.12.4.1 Preparation of test solution

Weigh-take about 10 g of sample, accurate to 0.01 g. Place it in a 250 mL beaker, add about 150 mL of water, heat to boiling to completely dissolve the specimen, then, cool it to room temperature. Transfer all into a 250 mL (V_1) volumetric flask, use water to dilute to the scale and shake it well.

7.12.4.2 Test

Use a pipette to respectively take 5 portions of 20 mL (V_2) of test solution and place them in five 100 mL volumetric flasks. Respectively add 5 mL of nitric acid solution to each, then, respectively add 0.0 mL, 1.00 mL, 2.00 mL, 3.00 mL and 4.00 mL of the metal ion standard mixed solution, use water to dilute to the scale and shake them well.

Turn on the inductively coupled plasma optical emission spectrometer (ICP-OES), and after the operation is stable, under the selected optimized conditions, respectively determine the emission spectrum intensity of different masses of each metal ion. Take the mass value A of the determined metal ion as the x-coordinate, and the corresponding emission spectrum intensity I as the y-coordinate to draw a working curve. Intersect the reverse extension line of the curve with the x-coordinate, and the distance between the intersection point and the origin is the mass of the determined ion (see Figure 1). Thus, the content of each ion can be obtained through calculation.

- When there is an update of key production processes;
- When there are changes in the main raw materials;
- When production is suspended and resumed;
- When there are relatively significant differences from the previous type inspection;
- When it is stipulated in the contract.

b) In the requirements, potassium nitrate, moisture, water-insoluble matter, chloride, sulfate, carbonate, ammonium salt content and moisture absorption rate are exit-factory inspection items, which shall be inspected batch by batch.

8.2 Industrial potassium nitrate of the same specifications and continuously produced or by the same team using the same materials and basically the same production conditions is considered as one batch. Each batch of products shall not exceed 100 t.

8.3 In accordance with the stipulations of GB/T 6678, determine the number of sampling units. During sampling, insert the sampler from the top of the packaging bag to 3/4 depth of the material layer for sampling. Evenly mix the collected samples and cut them into quarters to about 800 g, respectively put them into two dry, clean jars or plastic bags, and seal. Attach a label to the jars or bags, indicating manufacturer's name, product name, type, grade, batch No., sampling date and sampler's name. One is used as laboratory sample, and the other is kept for future reference. The retention time is determined by the manufacturer based on actual demands.

8.4 If any indicator in the inspection results does not comply with the requirements of this document, sampling shall be conducted from twice the amount of packaging for a re-inspection. When even one indicator in the re-inspection results does not comply with the requirements of this document, then, the entire batch of products shall be deemed disqualified.

8.5 Adopt the rounding-off comparison method specified in GB/T 8170 to determine whether the inspection results comply with the document.

9 Marking and Labeling

9.1 The packaging bags of potassium nitrate for industrial use must have solid and clear markings, including manufacturer's name and address, product name, category, type, net content, batch No. or production date, serial No. of this document, as well as the "Oxidizing Substance" mark stipulated in Chapter 3 of GB 190-2009, and the "Avoid Sun Exposure" and "Keep Dry" marks stipulated in Chapter 2 of GB/T 191-2008.

9.2 Each batch of potassium nitrate for industrial use exiting the factory shall be accompanied by a quality certificate, including manufacturer's name and address, product name, category, type, net content, batch No. or production date, and serial No. of this document.

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