NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 8574-2024

Determination of potassium content for compound fertilizers

复合肥料中钾含量的测定

Issued on: September 29, 2024 **Implemented on: April 1, 2025**

Issued by: State Administration for Market Regulation;
Standardization Administration of the PRC

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1 Scope

GB/T 8574-2024 gives the determination of potassium in compound fertilisers. The K of the NPK figure printed on the bag is one of the three numbers a farmer buys on, and short-weighting it is the commonest fertiliser adulteration in China, so the reference method is the basis of both contract and enforcement testing. The difficulty is the extraction as much as the measurement, since potassium in a compound fertiliser is not all readily soluble. The standard covers heating and boiling extraction and ultrasonic extraction, and three determinations: the potassium tetraphenylborate gravimetric method, inductively coupled plasma atomic emission spectrometry, and the flow analyser method. It sets the samples, the preparation of the specimen solutions and the determination, with normative annexes on the gravimetric and ICP-AES methods and an informative annex giving the interlaboratory test statistics. It takes effect on 1 April 2025.

This document describes the determination of potassium in compound fertilizers using the potassium tetraphenylborate gravimetric method, inductively coupled plasma atomic emission spectrometry, or flow analyzer method, through either heating and boiling extraction or ultrasonic extraction. For the heating and boiling extraction, the potassium tetraphenylborate gravimetric method is the arbitration method. This document applies to the determination of potassium content in compound fertilizers. The determination of potassium content in other potassium-containing inorganic fertilizers may take this document as a reference.

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 6274 Fertilizers and Soil Conditioners - Vocabulary

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

GB/T 8571 Preparation of Laboratory Samples for Compound Fertilizers

GB/T 15063 Compound Fertilizer

GB/T 22923-2008 Determination of Nitrogen, Phosphorus and Potassium for Fertilizers by Auto Analyzer

HG/T 2843 Chemical Fertilizer Products - Standard Volumetric, Standard Reagent and Indicator Solutions for Chemical Analysis

3 Terms and Definitions

The terms and definitions defined in GB/T 6274 and GB/T 15063 are applicable to this document.

4 Samples

Sample preparation shall be carried out in accordance with GB/T 8571.

5 Preparation of Specimen Solutions

5.1 Reagents or Materials Water shall comply with Grade 3 water specifications in GB/T 6682.

5.2 Instruments and Equipment

5.2.1 Common laboratory instruments and equipment.

5.2.2 Ultrasonic cleaner.

5.3 Preparation Procedures

5.3.1 Specimen weighing Perform two tests in parallel. For samples to be determined for potassium content using the potassium tetraphenylborate gravimetric method (6.1), weigh-take approximately 400 mg of specimen containing potassium oxide, accurate to 0.0002 g. For samples to be determined for potassium content using the inductively coupled plasma atomic emission spectrometry (6.2), weigh-take 100 mg ~ 250 mg of

specimen containing potassium oxide, accurate to 0.0002 g.

5.3.2 Extraction method 1 (heating and boiling) In accordance with the requirements of 5.3.1, weigh-take the specimen and place it in a 250 mL conical flask. Add approximately 150 mL of water, and heat and boil it for 30 minutes. Cool, quantitatively transfer it to a 250 mL volumetric flask, use water to dilute to the scale, mix it well, dry filter it, and discard the first 50 mL of the filtrate.

5.3.3 Extraction method 2 (ultrasonic extraction) In accordance with the requirements of 5.3.1, weigh-take the specimen and place it in a 250 mL volumetric flask. Add 150 mL of water, place the volumetric flask in an ultrasonic cleaner and ultrasonically extract it for 8 minutes (the ultrasonic cleaner liquid level is not lower than the liquid level in the volumetric flask). Cool to room temperature, use water to dilute to the scale, mix it well, dry filter it, and discard the first 50 mL of the filtrate.

6 Determination of Potassium in the Specimen Solution

6.1 Potassium Tetraphenylborate Gravimetric Method Proceed in accordance with Appendix A.

6.2 Inductively Coupled Plasma Atomic Emission Spectrometry Proceed in accordance with Appendix B.

6.3 Flow Analyzer Method Proceed in accordance with

3.4 of GB/T 22923-2008.

Appendix A

(normative) Potassium Tetraphenylborate Gravimetric Method A.1 Principle In a weakly alkaline solution, sodium tetraphenylborate solution reacts with potassium ions in the specimen solution to generate potassium tetraphenylborate precipitate. Filter, dry and weigh the precipitate. If the specimen contains cyanamides or organic matter, it can be treated by adding bromine water and activated carbon first. To prevent cationic interference, an appropriate amount of ethylenediaminetetraacetic acid disodium salt (EDTA) can be added in advance to allow the cations to complex with the EDTA. A.2 Reagents or Materials Unless otherwise specified, only analytically pure reagents shall be used. The standard titration solutions, standard solutions, reagent solutions, and indicator solutions used in this Method, unless the preparation method is specified, shall be prepared in accordance with HG/T 2843. A.2.1 Water, GB/T 6682, Grade 3. A.2.2 Sodium tetraphenylborate solution. 15 g/L. A.2.3 Ethylenediaminetetraacetic acid disodium salt (EDTA) solution. 40 g/L. A.2.4 Sodium hydroxide solution. 400 g/L. A.2.5 Aqueous bromine solution. approximately 5% (mass fraction). A.2.6 Sodium tetraphenylborate washing solution.

1.5 g/L. A.2.7 Phenolphthalein. 5 g/L ethanol solution, dissolve

0.5 g of phenolphthalein in 100 mL of 95% (mass fraction) ethanol. A.2.8 Activated carbon. shall not adsorb or release potassium ions. A.3 Instruments and Equipment A.3.1 Common laboratory instruments and equipment. A.3.2 Glass crucible filter. No. 4, 30 mL. A.3.3 Electrically heated constant-temperature drying oven. temperature controllable within the range of 120 °C 5 °C. A.4 Test Procedures A.4.1 Test solution preparation The specimen does not contain cyanamides or organic matter. pipette

25.0 mL of the specimen solution (

5.3.2 or 5.3.3) into a 200 mL beaker. Add 20 mL of EDTA solution (40 mL if there are a large number of cations), 2 ~ 3 drops of phenolphthalein solution, and dropwise add sodium hydroxide solution, until a red color appears. Add an excess of 1 mL. In a well- ventilated fume hood, slowly heat and boil for 15 minutes. Then, let it cool or cool to room temperature under running water. If the red color disappears, use sodium hydroxide solution to adjust the color to red again. If the specimen contains cyanamides or organic matter. pipette

25.0 mL of the specimen solution (

5.3.2 or 5.3.3) into a 200 mL ~ 250 mL beaker. Add 5 mL of aqueous bromine solution. Boil the solution, until all the bromine water is removed (no bromine color). If it contains other colors, after the solution cools, add

0.5 g of activated carbon, and thoroughly stir to adsorb it. Then, filter and wash it 3 ~ 5 times, using approximately 5 mL of water each time. Collect all the filtrate and add 20 mL of EDTA solution (40 mL if there are a large number of cations). The following steps are the same as for specimens without cyanamides or organic matter. A.4.2 Precipitation and filtration While continuously stirring, dropwise add sodium tetraphenylborate solution to the specimen solution (A.4.1) at a rate of

0.5 mL per 1 mg of potassium oxide, with an excess of approximately 7 mL. Continue stirring for 1 minute and let it stand for at least 15 minutes. Use the decantation method to filter the precipitate into a No. 4 glass crucible filter that has been pre-weighed to a constant mass at 120 °C. Use sodium tetraphenylborate washing solution to wash the precipitate 5 ~ 7 times, using approximately 5 mL each time. Finally, use water to wash it twice, using 5 mL each time. A.4.3 Drying Place the crucible containing the precipitate in an electrically heated constant-temperature drying oven at 120 °C 5 °C for