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CCS G20



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 17767.3-2010

Replacing GB/T 17767.3-1999

**Determination of organic-inorganic compound fertilizers - Part
3: Total potassium content**

有机-无机复混肥料的测定方法 第3部分：总钾含量

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1 Scope

GB/T 17767.3-2010 is the Chinese national standard on determination of organic-inorganic compound fertilizers - part 3: total potassium content, in the field of agriculture. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 30 June 2010 by the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China; Standardization Administration of China, and has been in force since 1 January 2011. Classification: ICS 65.080, CCS G20. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This Part of GB/T 17767 specifies the determination method of total potassium content in organic-inorganic compound fertilizers. This Part is applicable to organic-inorganic compound fertilizers composed of organic fertilizers and chemical fertilizers; it is also applicable to the determination of total potassium content of various solid organic fertilizers.

2 Normative references

The provisions in following documents become the provisions of this Part through reference in this Part of

GB/T 17767. For the dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this Part; however, parties who reach an agreement based on this Part are encouraged to study if the latest versions of these documents are

applicable. For undated references, the latest edition of the referenced document applies.

GB/T 8571 Preparation of laboratory samples for compound fertilizers

HG/T 2843 Chemical fertilizer products - Standard volumetric, standard, reagent and indicator solutions for chemical analysis

3 Reagents and materials

Warning - Hydrogen peroxide, nitric acid, perchloric acid in the reagents are corrosive and oxidizing; sulfuric acid and sodium hydroxide solutions are corrosive. Related operations shall be carried out in a fume hood. This standard does not point out all possible safety issues. Users are responsible for taking appropriate safety and health measures and ensuring that the conditions specified in relevant national laws and regulations are met. The reagents, solutions and water used in this part shall comply with the provisions of HG/T 2843, when the specifications and preparation methods are not specified.

3.1 Sulfuric acid;

3.2 Hydrogen peroxide;

3.3 Nitric acid;

3.4 Perchloric acid;

3.5 Sodium tetraphenylborate solution precipitant: 15 g/L;

3.6 Sodium tetraphenylborate washing solution:

3.7 Disodium ethylenediaminetetraacetic acid (EDTA) solution: 40 g/L;

3.8 Sodium hydroxide solution: 400 g/L;

3.9 Phenolphthalein: 5 g/L ethanol solution; dissolve

0.5 g of phenolphthalein in 100 mL of 95% (volume fraction) ethanol;

3.10 Potassium oxide standard stock solution:

1.00 mg/mL. Weigh 1.5828 g of potassium chloride which was dried at 110 °C for 2 h. Dissolve it in water; adjust the volume to 1 L volumetric flask. Mix well. 1 mL of this solution contains

1.00 mg of potassium oxide (K₂O). Store it in a plastic bottle;

3.11 Potassium oxide standard solution: 100 µg/mL Pipette

25.0 mL of potassium oxide standard stock solution (3.10) into a 250 mL volumetric flask. Use water to dilute it to the mark. Mix well. 1 mL of this solution contains 100 µg of potassium oxide (K₂O).

4 Instruments and equipment

4.1 Common laboratory instruments;

4.2 Glass crucible filter: No.4, volume 30 mL;

4.3 Electric constant temperature drying oven: temperature can be controlled at (120 ± 5) °C;

4.4 Flame photometer.

5 Preparation of specimen solution

Make parallel determinations of two samples. Prepare laboratory samples according to GB/T 8571. When preparing the specimen, grind the sample until it passes through a 1 mm test sieve. If the sample is difficult to crush, grind it until it passes through a 2 mm test sieve. For specimens with potassium content (K_2O) < 2%, flame photometry is used for determination; weigh 2 g of specimen. For specimens with potassium content (K_2O) $\geq$ 2%, potassium tetraphenylborate gravimetric method is used for determination. When 2% $\leq$ potassium content (K_2O) < 5%, weigh 4 g of specimen. When potassium content (K_2O) $\geq$ 5%, weigh 2 g of specimen, accurate to 0.0002 g. Prepare the specimen solution by one of the following methods.

5.1 Nitric acid-perchloric acid digestion method Place the specimen in a 250 mL tall beaker. Add 20 mL of nitric acid. Shake carefully. Heat on an electric hot plate in a fume hood, until it is almost dry. Add 10 mL of perchloric acid after cooling slightly. Cover with a watch glass. Slowly heat until white smoke of perchloric acid is emitted. Continue heating until the solution is colorless or light-colored clear liquid (note that it cannot be evaporated dry!). Cool to room temperature. Transfer the digestion solution into a 250 mL volumetric flask. Use water to dilute it to the mark. Mix well. Dry filter it. Discard the first 50 mL filtrate.

5.2 Sulfuric acid-hydrogen peroxide digestion method Place the sample in a 500 mL conical flask. Add 20 mL of concentrated sulfuric acid and 3 mL ~ 5 mL of hydrogen peroxide. Shake carefully. Let it stand for 12 h ~ 15 h. Add 3 mL ~ 5 mL of hydrogen peroxide. Insert a pear-shaped funnel. Slowly heat to boiling in a fume hood with a 1500 W electric stove. Continue heating for 30 min. Remove it. If the solution is not clear, add 3 mL of hydrogen peroxide in batches after cooling slightly. Digest in batches, until the solution is colorless or light-colored clear liquid. Continue heating for 10 min. Cool to room temperature. Transfer the digestion solution into a 250 mL volumetric flask. Use water to dilute it to the mark. Mix well. Dry filter it. Discard the first 50 mL filtrate.

6 Analytical procedures

Make parallel determinations of two samples.

6.1 Potassium tetraphenylborate gravimetric method This method is used when the potassium oxide content in the specimen is greater than or equal to 2% (mass fraction).

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