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Determination of calcium, magnesium and sulphur content for compound fertilizer

复混肥料中钙、镁、硫含量的测定

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Foreword

This Standard refers to the International standard ISO 10084:1992 *Solid fertilizers - Determination of mineral-acid-soluble sulfate content - Gravimetric method* for the determination of sulphur content for compound fertilizer. The determination of calcium and magnesium content uses EDTA volumetric method.

This Standard was proposed by China Petroleum and Chemical Industry Association.

This Standard shall be under the jurisdiction of National Technical Committee on Fertilizers and Soil Conditioning Agents of Standardization Administration of China.

The drafting organizations of this Standard: Shanghai Chemical Industry Research Institute, China-Arab Fertilizer Company Limited.

Main drafters of this Standard: Fan Bin, Wang Lianjun, Zhang Qiuzhen, Wang Yin, Du Xianlan, Liu Gang, Liu Yun.

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Determination of calcium, magnesium and sulphur content for compound fertilizer

1 Scope

This Standard specifies the determination of total calcium, magnesium and sulphur content in compound fertilizer.

This Standard is applicable to the determination of total calcium, magnesium and sulphur content in all kinds of compound fertilizers.

2 Normative references

The following standards contain the provisions which, through reference in this Standard, constitute the provisions of this Standard. For dated references, subsequent amendments (excluding corrections) or revisions do not apply to this Standard. However, the parties who enter into agreement based on this Standard are encouraged to investigate whether the latest versions of these documents are applicable. For undated reference documents, the latest versions apply to this Standard.

GB/T 8571 *Preparation of laboratory samples for compound fertilizers*

HG/T 2843-1997 *Chemical fertilizer Products-standard volumetric, standard, reagent and indicator solutions for chemical analysis*

3 Test methods

3.1 General provisions

The preparation of reagents, water and solutions used in this Standard, in the absence of specifications and preparation methods, it shall be in accordance with the provisions of HG/T 2843-1997.

3.2 Laboratory sample preparation

Prepare the laboratory sample according to the provisions of GB/T 8571.

3.3 Preparation of sample solution

3.3.1 Reagents and materials

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3.4.2.6 Ammonia-ammonium chloride buffer solution: pH ≈ 10; prepare it according to the method in 9.5.1 of HG/T 2843-1997;

3.4.2.7 Disodium edetate (EDTA) standard titration solution: c(EDTA) = 0.02 mol/L;

3.4.2.8 Malachite green indicator solution: 1 g/L;

3.4.2.9 Calcein - methyl thymol blue indicator (referred to as calcein indicator): grind and well mix 0.10 g of calcein and 0.10 g of methyl thymol blue (or methyl thymol blue), 0.03 g of thymol phenolphthalein, 5 g of potassium chloride; store in grinding mouth bottle, it is ready for later use;

3.4.2.10 Acid chrome blue K-naphthol green B mixed indicator (referred to as K-B indicator).

3.4.3 Apparatus

General laboratory instruments.

3.4.4 Analysis steps

3.4.4.1 Determination of total calcium content

3.4.4.1.1 Content determination

Accurately pipet a certain amount of sample solution (less than 15 mg, in Ca) in an Erlenmeyer flask. Add 50 mL of water, 10 mL of starch solution, 8 mL of triethanolamine solution, 1 mL of ethylenediamine, 1 drop of malachite green indicator solution. Drop-add potassium hydroxide solution till it is colorless. And add an excess of 10 mL. Add 0.1 g of hydroxylamine hydrochloride (each reagent must be well shaken when it is added). Add 0.1 g ~ 0.3 g of calcium yellow indicator. On a black background, immediately use disodium edetate (EDTA) standard titration solution to titrate till green fluorescence disappears. It shall be the end of titration when it appears in purple-red.

3.4.4.1.2 Blank test

Except that sample is not added, it must use reagents, dosage and analysis steps which are identical with sample determination to carry out the parallel test.

3.4.5 Expression of analysis results

3.4.5.1 Total calcium (in Ca) content w_1, expressed in mass fraction (%), shall be calculated according to equation (1):

$$w_1 = \frac{c_1 (V_1 - V_{01}) \times 40.08}{1\,000 \times m_1 \times V_2/250} \times 100 \qquad \cdots\cdots (1)$$

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