

ICS 65.080

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

GB/T 22924-2008

**Determination of biuret content for compound fertilizers
(complex fertilizers)**

复混肥料(复合肥料)中缩二脲含量的测定

(English Translation)

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1 Scope

This standard specifies the test methods for the determination of the biuret content in the compound fertilizers (complex fertilizers). liquid chromatography, spectrophotometry.

This standard is applicable to the determination of biuret content in the compound fertilizers (complex fertilizers) in which the base fertilizer contains urea.

2 Normative references

The provisions in following documents become the provisions of this Standard through reference in this Standard. For the dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this Standard;

however, parties who reach an agreement based on this Standard 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 6682 Water for analytical laboratory use - Specification and test methods

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

3.1 General provisions

Unless otherwise stated, only the reagents identified as analytical reagents and the level III water as specified in GB/T 6682 are used in the analysis.

The standard titration solutions, preparations and products used in the analysis shall be prepared in accordance with HG/T 2843 unless otherwise specified.

3.2.1 Method summary

The biuret content is calculated by reverse phase liquid chromatography with UV detector and external standard method.

3.2.2 Reagents and solutions

3.2.2.1

Methanol. Chromatographic pure;

3.2.2.2

Potassium dihydrogen phosphate;

3.2.2.3

Biuret.

WEIGH about 15 g of biuret (chemical pure) in a beaker; ADD it into 500 mL of ethanol in volume fraction 95%; HEAT to dissolve it; FILTER it when it is hot;

CONDENSE the filtrate to 250 mL; COOL it to 5 °C to allow the crystals to precipitate; FILTER it and DRY it at 105 °C to prepare for use. AND it shall be free from impurity peaks under the conditions as specified in 3.2.6;

3.2.2.4

Phosphoric acid solution. 30 g/L;

3.2.2.5

Potassium hydroxide solution. 40 g/L;

3.2.2.6

Potassium dihydrogen phosphate solution. 10 g/L;

WEIGH 10 g of potassium dihydrogen phosphate; DISSOLVE it into 1 L of water; USE the phosphoric acid solution or potassium hydroxide solution to adjust the pH = 6.0; SHAKE it uniformly; USE the 0.45 µm filter membrane to filter it; PREPARE for use.

3.2.2.7 Mobile phase. methanol 5 mL + potassium dihydrogen phosphate

solution 95 mL; before use, USE the 0.45 µm filter membrane to filter it; MAKE it subjected to ultrasonic degassing;

3.2.2.8 Biuret standard solution. 0.5 mg/mL;

WEIGH 0.5 g (accurate to 0.0002 g) of biuret (3.2.2.3); DISSOLVE it into water which does not contain carbon dioxide; TRANSFER it into a 1 L volumetric flask; USE water to dilute it to the mark; SHAKE it uniformly; AND this solution shall be prepared before use.

3.2.2.9 Eluent. Water 10 mL + methanol 90 mL; before use, USE the 0.45 μm filter membrane to filter it; MAKE it subjected to ultrasonic degassing;

3.2.3 Instruments and equipment

The commonly used laboratory instruments and the following instruments.

3.2.3.1

Ultrasonic cleaner;

3.2.3.2

High performance liquid chromatography. with UV detector;

3.2.3.3

Microinjector. 10 μL ~ 50 μL ;

3.2.3.4 Needle filter. with aqueous microporous membrane of pore size 0.45

μm ;

wavelength of 550 nm, the absorbance is determined to obtain the biuret content in the sample.

3.3.2 Reagents and solutions

3.3.2.1

Anhydrous ethanol.

3.3.2.2

Hydrogen peroxide.

3.3.2.3 Potassium tartrate alkaline solution. 100 g/L; WEIGH 80 g of sodium

hydroxide; DISSOLVE it into 500 mL of water; COOL it down; ADD 100 g of sodium potassium tartrate ($\text{NaKC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$); after dissolving, USE water to dilute it to 1 L; MIX it uniformly; LET it standing overnight; FILTER it before use.

3.3.2.4 Copper sulfate solution. 30 g/L; WEIGH 30 g of copper sulfate

($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$); DISSOLVE it in water; FILTER it; USE water to dilute it to 1 L.

3.3.2.5 Biuret standard solution. 2 mg/mL; WEIGH 2 g (accurate to 0.0002 g)

of biuret (3.3.2.2); DISSOLVE it into water containing no carbon dioxide;

TRANSFER it into a 1 L volumetric flask; USE water to dilute it to the mark;

SHAKE it uniformly; PREPARE this standard solution before use.

3.3.3 Instruments and equipment

Commonly used laboratory instruments and the following instruments.

3.3.3.1

Electrically heated drying oven. the controlled temperature is at 105 °C

± 2 °C;

3.3.3.2

Ultrasonic cleaner;

3.3.3.3

Constant temperature water bath. the controlled temperature is at

30 °C ± 5 °C;

3.3.3.4 Spectrophotometer, with an optical path of 3 cm absorption cell AND it

can be measured at 550 nm.

3.3.4.1 Sample preparation

PREPARE samples in accordance with GB/T 8571.

3.3.4.2 Drawing of standard curves

3.3.4.2.1

Preparation of standard colorimetric solution

As shown in Table 3, respectively PIPETTE 0.00 mL (used as the compensation solution), 2.50 mL, 5.00 mL, 10.00 mL, 15.00 mL, 20.00 mL,

25.00 mL, and 30.00 mL biuret solution; PLACE it respectively into eight 100 mL volumetric flasks; USE water to dilute it to about 70 mL; and then ADD 10.0

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