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Determination of nitrogen, phosphorus and potassium for fertilizers by auto analyzer

肥料中氮、磷、钾的自动分析仪测定法

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Standardization Administration of the People's Republic of China.

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Foreword

This standard was proposed by the China Petroleum and Chemical Industry Association.

This standard shall be under the jurisdiction of the National Standardization Technical Committee on Fertilizer and Soil Conditioner (SAC/TC 105).

The drafting organizations of this standard: National Fertilizer Quality Supervision and Inspection Center (Shanghai), Tianji Coal Chemical Industry Group Co., Ltd., Shanghai Shengsheng Automation Analysis Instrument Co., Ltd.

The main drafters of this standard: Zhang Minghong, Zhang Jiahong, Duan Lulu, Li Lihua, He Jianzhong, Wu Juan, Liu Wanqing.

This standard is the first release.

Determination of nitrogen, phosphorus and potassium for fertilizers by auto analyzer

1 Scope

This standard specifies the method for the determination of nitrogen, phosphorus and potassium in fertilizers using auto analyzers.

This standard applies to the determination of nitrogen, phosphorus and potassium in fertilizers.

Flow analyzer method is not applicable to the fertilizer containing organic matters, AND the nitrogen content determination method is not applicable to the fertilizer containing nitrogen of more than 40%.

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.

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

3 Test methods

The reagents, solutions and water used in this standard shall comply with the requirements of HG/T 2843, unless otherwise their specifications and preparation methods are indicated.

3.1 Determination of nitrogen content - Nitrogen analyzer method

3.1.1 Principle

The nitrate in the acidic medium is reduced into the ammonium salt, which is digested by concentrated sulfuric acid in the presence of a mixed catalyst to convert the amide nitrogen into an ammonium salt. Ammonia is distilled from the alkaline solution, AND the ammonia is absorbed with boric acid absorption solution AND then titrated with the sulfuric acid standard titration solution.

Automatic nitrogen analyzer can integrate such functions as distillation, titration, result display, or calculation, etc., to finish determination automatically and quickly.

WEIGH the sample of total nitrogen content of about 100 mg and nitrate nitrogen content of less than 25 mg (accurate to 0.0002 g) in the digestion tube; ADD 0.5 g of chromium powder; USE a small amount of water to wash the tube wall; PLACE the digestion tube in a ventilation cabinet; ADD 5 mL of hydrochloric acid in the digestion tube; LET it stand at room temperature for at least 5 min, (BUT not exceeding 10 min). PLACE the digestion tube in the digestion furnace which has been heated to 300 °C in advance; INSERT in the pear-shaped glass funnel; HEAT to boil it for 1 min ~ 2 min; PAY attention to avoid drying it; COOL it down; RINSE the funnel and digestion tube wall.

3.1.4.3 Treatment of sample containing amide nitrogen and ammonium nitrogen

WEIGH the sample containing nitrogen of about 100 mg (accurate to 0.0002 g) into the digestion tube; USE a small amount of water to wash the tube wall; PLACE the digestion tube in a ventilation cabinet; ADD 10 mL of sulfuric acid and 0.5 g of mixed catalyst; INSERT the pear-shaped glass funnel; HEAT it for 1 h in the digestion furnace at 340 °C; PAY attention to avoid drying it; COOL it down; RINSE the funnel and digestion tube wall.

3.1.4.4 Treatment of sample containing nitrate nitrogen and amide nitrogen

WEIGH the sample of total nitrogen content of about 100 mg and nitrate nitrogen content of less than 25 mg (accurate to 0.0002 g) in the digestion tube; ADD 0.5 g of chromium powder; USE a small amount of water to wash the tube wall; PLACE the digestion tube in a ventilation cabinet; ADD 5 mL of hydrochloric acid in the digestion tube; LET it stand at room temperature for at least 5 min, (BUT not exceeding 10 min). PLACE the digestion tube in the digestion furnace which has been heated to 300 °C in advance; INSERT in the pear-shaped glass funnel; HEAT to boil it for 1 min ~ 2 min; PAY attention to avoid drying it; COOL it down; carefully ADD 10 mL sulfuric acid and 0.5 g of mixed catalyst; PLACE it into the digestion furnace again; INCREASE the temperature to 340 °C; HEAT it until the solution color changes into purple; PAY attention to avoid drying it; COOL it down; RINSE the funnel and digestion tube wall.

3.1.4.5 Treatment of organic-inorganic compound fertilizer sample containing no nitrate nitrogen

WEIGH the sample containing nitrogen of about 100 mg (accurate to 0.0002 g) into the digestion tube which had been dried in advance; PLACE the digestion tube into the ventilation cabinet; ADD 10 mL of sulfuric acid and 0.5 g of mixed catalyst; INSERT the pear-shaped glass funnel; LET it stand for 10 min; PLACE the digestion tube over the digestion furnace to slowly heat it (if the reaction is intense to produce too much foam, MOVE it away from the digestion tube to cool it down for a moment); after the intense reaction finishes, INCREASE the temperature of the digestion furnace to 340 °C to continue digestion, until the solution is colorless or in light color. PAY attention to avoid drying it; COOL it down; RINSE the funnel and digestion tube wall.

3.2.1.3.2 Automatic digestion furnace, the temperature can be controlled within the range of 340 °C ± 5 °C;

3.2.1.3.3 Electric heating plate, the power is about 1.8 kW ~ 2.4 kW;

3.2.1.3.4 Digestion tube, the volume is about 350 mL;

3.2.1.3.5 Flow analyzer, it has the ammonium nitrogen channel and automatic injection device, with the ammonium nitrogen detection lower limit of 0.2 mg/L.

3.2.1.4 Preparation of sample solution

MAKE parallel determinations against two set of materials.

PREPARE laboratory samples in accordance with the corresponding product standards.

3.2.1.4.1 Treatment of sample containing ammonium nitrogen only: WEIGH the sample containing about 100 mg of nitrogen (accurate to 0.0002 g) into a 250-mL conical flask; ADD about 100 mL of water and 5 mL of sulfuric acid; HEAT to boil it for 15 min; COOL it down; quantitatively TRANSFER it to a 250-mL volumetric flask; USE water to dilute it to the mark; MIX it uniformly; dry FILTER it; DISCARD the initial filtrate.

3.2.1.4.2 Treatment of sampling containing amide nitrogen and ammonium nitrogen: Treatment of sample containing ammonium nitrogen only: WEIGH the sample containing about 100 mg of nitrogen (accurate to 0.0002 g) into a digestion tube; USE a small amount of water to rinse the tube wall; PLACE the digestion tube into the ventilation cabinet; ADD 5 mL of sulfuric acid; PLACE the digestion tube on the digestion furnace; HEAT it at 340 °C for 1 h; quantitatively TRANSFER it to a 250 mL volumetric flask; USE water to dilute it to the mark; MIX it uniformly; dry FILTER it; DISCARD the initial filtrate.

3.2.1.4.3 Preparation of blank solution: except for not adding sample, the rest procedures are same as the preparation of sample solution.

3.2.1.5 Analytical procedures

3.2.1.5.1 Drawing of working curves

PIPETTE 0 mL, 0.5 mL, 1.0 mL, 2.0 mL and 4.0 mL of ammonium nitrogen (NH_4^+-N) standard solution respectively into five 100 mL volumetric flask; USE the corresponding blank solution to dilute it to the mark; MIX it uniformly. AND the concentrations of this standard solution are 0, 50 mg/L, 100 mg/L, 200 mg/L, and 400 mg/L, respectively.

Before making the determination, with reference to the instrument instruction manual, SELECT the best working parameters.

Then, PLACE the solution sucker of the flow analyzer into the buffer solution, sodium salicylate solution, and dichloroisocyanic acid solution respectively. At

PREPARE laboratory samples in accordance with the corresponding product standards.

WEIGH the sample contain the nitrate nitrogen of about 50 mg (accurate to 0.0002 g); PLACE it into a 250mL-conical flask; ADD about 100 mL of water; HEAT to boil it for 15 min; COOL it down; quantitatively TRANSFER it to a 250-mL volumetric flask; USE water to dilute it to the mark; MIX uniformly; dry FILTER it; DISCARD the initial part of the filtrate.

Preparation of blank solution: except for not adding sample, the rest procedures are same as the preparation of sample solution.

3.2.2.5 Analytical procedures

3.2.2.5.1 Drawing of working curves

PIPETTE 0 mL, 1.0 mL, 2.0 mL，4.0 mL，and 8.0 mL of nitrate nitrogen (NO_3^+-N) standard solution respectively into five 100 mL volumetric flask; USE the corresponding blank solution to dilute it to the mark; MIX it uniformly. AND the concentrations of this standard solution are 0, 25 mg/L, 50 mg/L, 100 mg/L, and 200 mg/L, respectively.

Before making the determination, with reference to the instrument instruction manual, SELECT the best working parameters.

Then, PLACE the solution sucker of the flow analyzer into the sodium hydroxide solution, sulfuric acid ammonia solution and the color developing reagent respectively. At the wavelength of 550 nm, DETERMINE the absorbance of each standard solution after the baseline is stable.

USE the nitrate nitrogen (NO_3^+-N) concentration of the standard solution as the abscissa AND the corresponding absorbance as the ordinate, to draw the working curve or regression linear equation.

3.2.2.5.2 Determination

At the conditions same as the standard solution determination, DETERMINE the absorbance of the sample solution; from the working curve or the linear equation, CALCULATE the corresponding nitrate nitrogen concentration.

3.2.2.6 Expression of analytical results

3.2.2.6.1 Calculation of analytical results

The nitrogen content of the nitrate nitrogen is calculated based on the mass fraction w_3 of the nitrogen (N), AND the value is expressed in % and calculated in accordance with the equation (3):

$$w_3 = \frac{c_3 \times V_3}{m_3 \times 10^6} \times 100 \qquad \cdots\cdots (3)$$

Solution a: WEIGH 8.2 g of ammonium molybdate; DISSOLVE it into 300 mL of water;

Solution b: WEIGH 0.3 g of ammonium vanadate; DISSOLVE it into 200 mL of water; ADD 29 mL of perchloric acid;

While stirring, slowly POUR the solution a into the solution b; ADD 2 g of sodium dodecyl sulfate; after dissolving it, DILUTE it to 1000 mL; MIX it uniformly;

3.3.2.6 Pentoxide standard solution: 5 g/L, WEIGH 4.7900 g of potassium dihydrogen phosphate which had been dried at 105 °C for 2 h into a 50mL-beaker; USE water to dissolve it; TRANSFER it into a 500mL-volumetric flask; USE water to dilute it to the mark; MIX it uniformly.

3.3.3 Instrument

3.3.3.1 Commonly used laboratory equipment;

3.3.3.2 Constant temperature water bath oscillator, it is a reciprocating oscillator or rotary oscillator through which the temperature can be controlled at 60 °C ± 2 °C;

3.3.3.3 Flow analyzer, it has the phosphorus channel and automatic sampling device, with phosphorus pentoxide detection lower limit of 0.3 mg/L.

3.3.4 Preparation of sample solution

MAKE parallel determinations against two set of materials.

PREPARE laboratory samples in accordance with the corresponding product standards.

3.3.4.1 Extraction of water-soluble phosphorus: WEIGH the sample containing about 100 mg of phosphorus pentoxide (accurate to 0.0002 g); PLACE it into a 75mL-porcelain evaporator; ADD 25 mL of water and GRIND it; FILTER the clear solution into a 250mL-volumetric flask into which 5 mL of nitric acid solution is added in advance. CONTINUE using water to grind it for three times, each time using 25 mL of water; TRANSFER the water insoluble material onto the filter paper; USE water to wash the water insoluble, until the solution in the bottle reach up to about 200 mL. Finally USE water to dilute it to the mark; MIX it uniformly.

3.3.4.2 Extraction of available phosphorus: WEIGH the sample containing about 100 mg of phosphorus pentoxide (accurate to 0.0002 g); PLACE it on the filter paper; USE the filter paper to wrap the sample; INSER it into a 250 mL volumetric flask; ADD 150 mL of EDTA solution; tightly PLUG the flask; SHAKE the flask to break the filter paper and make the sample disperse in the solution; PLACE it in a 60 °C ± 2 °C constant temperature water bath oscillator; OSCILLATE it for 1 h at this temperature (oscillation frequency shall be such that the sample in the flask can be freely flip). TAKE out the volumetric flask;

3.4.3.3 Flow analyzer, it has the potassium channels and automatic sampling device, with the potassium detection lower limit of 0.36 mg/L;

3.4.3.4 Flame photometer.

3.4.4 Preparation of sample solution

MAKE parallel determinations against two set of materials.

PREPARE laboratory samples in accordance with the corresponding product standards.

3.4.4.1 Extraction of water-soluble potassium: WEIGH a certain amount of sample (accurate to 0.0002 g); PLACE it into a 250mL-conical flask; ADD about 100 mL of water; HEAT to boil it for 15 min; COOL it down; quantitatively TRANSFER it into a 250mL-volumetric flask; USE water to dilute it to the mark; dry FILTER it; DISCARD the initial part of the filtrate.

3.4.4.2 Preparation of blank solution: except for not adding sample, the rest procedures are same as the preparation of sample solution.

3.4.5 Analytical procedures

3.4.5.1 Drawing of working curves

PIPETTE 1.0 mL, 2.0 mL, 4.0 mL, 6.0 mL and 8.0 mL of potassium oxide standard solution respectively into five 100 mL volumetric flask; USE the corresponding blank solution to dilute it to the mark; MIX it uniformly. AND the concentrations of this standard solution are 25 mg/L, 50 mg/L, 100 mg/L, 150 mg/L, and 200 mg/L, respectively.

Before making the determination, with reference to the instrument instruction manual, SELECT the best working parameters.

Then, PLACE the solution sucker of the flow analyzer into the lanthanum oxide reagent. TURN on the flame photometer; DETERMINE the absorbance of each standard solution after the baseline is stable.

The concentration of potassium oxide in each standard solution is the abscissa, and the corresponding absorbance is the ordinate, and the working curve or regression linear equation is drawn.

USE the potassium oxide concentration of the standard solution as the abscissa AND the corresponding absorbance as the ordinate, to draw the working curve or regression linear equation.

3.4.5.2 Determination

At the conditions same as the standard solution determination, DETERMINE the absorbance of the sample solution without being diluted OR after being

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