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NY/T 2272-2012

Soil amendment – Measurement of calcium, magnesium, and silicon content

土壤调理剂 钙、镁、硅含量的测定

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Foreword

This standard was drafted in accordance with the provisions of GB/T 1.1.

This standard was proposed by AND shall be under the jurisdiction of the Ministry of Agriculture of the People's Republic of China.

The drafting organizations of this standard: National Fertilizer Quality Supervision and Inspection Center (Beijing).

The main drafters of this standard: Fan Hongli, Sun Mi, Sun Qianfeng, Han Yansong, Zhang Yue.

Soil amendment – Measurement of calcium, magnesium, and silicon content

1 Scope

This standard specifies the test methods for the soil conditioner - calcium, magnesium, AND silicon content measurement.

This standard applies to measurement of soil conditioner - calcium, magnesium, AND silicon content.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this Standard.

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

NY/T 887 Density testing of liquid fertilizer

3 Measurement of calcium content

3.1 Atomic absorption spectrophotometric method (arbitration method)

3.1.1 Principle

As for calcium of the sample solution, in slightly acidic medium, USE a certain amount of strontium salt as the release agent, to make it atomization in the lean combustion air – acetylene flame; the generated atomic vapor absorbs the characteristic wavelength 422.7 nm light which is emitted from the calcium hollow cathode lamp, AND absorbance values is directly proportional to the calcium ground state atomic concentration.

3.1.2 Reagents and materials

The preparation of the reagents, water and solution which are used in this standard shall, unless otherwise indicated of specifications AND preparation methods, be in accordance with the provisions of HG/T 2843.

It shall follow the provisions of 3.1.4.2.

3.2.4.3 Drawing of working curve

Respectively PIPETTE 0 mL, 0.50 mL, 1.00 mL, 2.00 mL, 4.00 mL, AND 5.00 mL of calcium standard solution (3.2.2.1) into six 100 mL volumetric flasks; USE water to make it reach to constant volume; MIX it uniformly. The calcium's mass concentration of this standard series solution is respectively 0 μ g/mL, 5.0 μ g/mL, 10.0 μ g/mL, 20.0 μ g/mL, 40.0 μ g/mL, AND 50.0 μ g/mL.

Before measurement, based on the nature of the element to be measured and the instrument performance, OPTIMIZE the measurement conditions of argon flow rate, observation height, RF generator power, AND integration time. Then USE the plasma emission spectrometer, at a wavelength of 317.933 nm, to measure the emission intensity of each standard solution. USE the calcium's mass concentration of the standard series solution (μ g/mL) as the abscissa AND the corresponding emission intensity as the ordinate; DRAW the working curve.

Note: It can, based on the different instrument sensitivities, ADJUST the mass concentration of the standard series solution.

3.2.4.4 Measurement

USE the sample solution OR the solution which is diluted to certain ratio, under the same conditions of the standard series solution for measurement purposes, to measure the emission intensity of calcium; from the working curve, FIND the corresponding mass concentration of the calcium (µg/mL).

3.2.4.5 Blank test

Except for not adding of sample, the rest steps are same as those described in 3.2.4.4.

3.2.5 Analysis result presentation

It shall follow the provisions of 3.1.5.

3.2.6 Allowable difference

It shall follow the provisions of 3.1.6.

3.2.7 Conversion of mass concentration

It shall follow the provisions of 3.1.7.

4.1.4 Analysis procedures

4.1.4.1 Sample preparation

With respect to the solid samples, after multiple times of splitting, TAKE out about 100 g of sample; rapidly GRIND it to the grain which can all pass the 0.50 mm pore size sieve (if the sample is wet, it shall pass through the 1.00 mm pore size sieve); MIX it uniformly; PLACE it in a clean AND dry container; after the liquid sample is subject to multiple times of shaking, quickly TAKE out about 100 mL; PLACE it in a clean AND dry container.

4.1.4.2 Sample solution preparation

- Solid sample: WEIGH 0.2 g ~ 3 g of sample (accurate to 0.0001 g); PLACE into a 250 mL volumetric flask; ADD 150 mL of hydrochloric acid solution (4.1.2.5) which was pre-heated to 28 °C ~ 30 °C; tightly PLUG the volumetric flask; SHAKE the volumetric flask to disperse the sample in the liquid; MAINTAIN the solution temperature at 28 °C ~ 30 °C; USE the oscillator (4.1.3.2) the frequency of which is set to (180 ± 20) r/min to oscillate it for 30 min; then TAKE the volumetric flask out; COOL it to room temperature; USE water to dilute it to the mark; MIX it uniformly; dry FILTER it; DISCARD the first few milliliters of filtrate; USE the rest filtrate for measurement.
- Liquid sample: WEIGH 0.2 g ~ 3 g sample (accurate to 0.0001 g); PLACE it into a 250 mL volumetric flask; USE water to make it reach to constant volume; MIX it uniformly; dry FILTER it; DISCARD the first few milliliters of filtrate; USE the rest filtrate for measurement.

4.1.4.3 Drawing of working curve

Respectively PIPETTE 0 mL, 1.00 mL, 2.00 mL, 4.00 mL, 8.00 mL, AND 10.00 mL of magnesium standard solution (4.1.2.4) into six 100 mL volumetric flasks; respectively ADD 4 mL of hydrochloric acid solution (4.1.2.1) AND 10 mL of strontium chloride solution (4.1.2.2); USE water to make it reach to constant volume; MIX it uniformly. The magnesium's mass concentration of this standard series solution is respectively 0 μ g/mL, 1.00 μ g/mL, 2.00 μ g/mL, 4.00 μ g/mL, 8.00 μ g/mL, AND 10.00 μ g/mL. Under the selected optimum working conditions, at the wavelength of 285.2 nm, USE the lean combustion air – acetylene flame; and USE the magnesium content 0 standard solution as the reference solution to set zero, in order to measure the absorbance value of each standard solution. USE the magnesium's mass concentration of the standard series solution (μ g/mL) as the abscissa AND the corresponding absorbance value as the ordinate; DRAW the working curve.

USE the sample solution OR the solution which is diluted to certain ratio, under the same conditions of the standard series solution for measurement purposes, to measure the emission intensity of magnesium; from the working curve, FIND the corresponding mass concentration of the magnesium $(\mu g/mL)$.

4.2.4.5 Blank test

Except for not adding of sample, the rest steps are same as the sample solution measurement.

4.2.5 Analysis result presentation

It shall follow the provisions of 4.1.5.

4.2.6 Allowable difference

It shall follow the provisions of 4.1.6.

4.2.7 Conversion of mass concentration

It shall follow the provisions of 4.1.7.

5 Measurement of silicon content - Plasma emission spectrometric method

5.1 Principle

The silicon in the sample solution is atomized and excited to a high energy state in the ICP light source. The high energy state atom, when transited to ground state, generates the electromagnetic radiation of characteristic wavelength. AND the emission intensity is directly proportional to the silicon atom concentration.

5.2 Reagents and materials

The preparation of the reagents, water and solution which are used in this standard shall, unless otherwise indicated of specifications AND preparation methods, be in accordance with the provisions of HG/T 2843.

- **5.2.1** Silicon standard solution: ρ (Si) = 1000 μ g/mL.
- **5.2.2** Hydrochloric acid solution: c (HCl) = 0.5 mol/L.
- **5.2.3** High purity argon.

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