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NY

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

NY/T 1117-2010

**Water-soluble fertilizers. Determination of calcium, magnesium,
sulphur and chlorine content**
(English Translation)

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1 Scope

This standard specifies the test methods for determining the calcium, magnesium, sulfur, chlorine content in water-soluble fertilizers.

This standard applies to the determination of calcium, magnesium, sulfur, chlorine content in liquid or solid water-soluble fertilizers.

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) is applicable to this standard.

GB/T 8170 Rules of rounding off for numerical values & expression and judgement of limiting values

GB/T 19203 Determination of calcium, magnesium and sulfur content in compound fertilizers

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

NY/T 887 Liquid fertilizers - Determination of density

3.1.1 Principle

Calcium in the specimen solution is atomized in a slightly acidic medium with a certain amount of strontium salt as a releasing agent in a lean-fuel air-acetylene flame. The resulting atomic vapor absorbs light, which has a characteristic wavelength of 422.7 nm emitted from a calcium hollow cathode lamp. The absorbance value is directly proportional

to the concentration of calcium ground-state atoms.

3.1.2 Reagents and materials

Unless otherwise specified, the preparation of reagents, water, solutions used in this standard shall comply with the provisions of HG/T 2843.

3.1.2.1

Hydrochloric acid.

3.1.2.2 Hydrochloric acid solution. 1 + 1

3.1.2.3 Strontium chloride solution. $\rho(\text{SrCl}_2) = 60.9 \text{ g/L}$. Weigh 60.9 g of $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$; dissolve it in 300 mL of water and 420 mL of hydrochloric acid solution (3.1.2.2); dilute to 1000 mL with water; then mix well.

3.1.2.4 Calcium standard stock solution. $\rho(\text{Ca}) = 1 \text{ mg/mL}$

3.1.2.5 Calcium standard solution. $\rho(\text{Ca}) = 100 \mu\text{g/mL}$. Pipette 10.00 mL of calcium standard stock solution (3.1.2.4) into a 100 mL volumetric flask; add 10 mL of hydrochloric acid solution (3.1.2.2); use water to dilute it to the mark; mix well.

3.1.2.6 Dissolved acetylene.

3.1.3 Instruments

3.1.3.1

General laboratory equipment.

3.1.3.2

Horizontal reciprocating shaker or a shaking device with equivalent function.

3.1.3.3 Atomic absorption spectrophotometer equipped with an air-acetylene burner

and a calcium hollow cathode lamp.

3.1.4.1 Specimen preparation

After multiple reductions of solid sample, take approximately 100 g and quickly grind it until it passes through a 0.50 mm sieve (or a 1.00 mm sieve if the sample is moist).

Mix thoroughly and place in a clean, dry container. For liquid sample, after multiple shakings, quickly take approximately 100 mL and place in a clean, dry container.

3.1.4.2 Preparation of specimen solution

3.1.4.2.1

Solid specimen

Weigh 0.2 g ~ 3 g of specimen (accurate to 0.0001 g) into a 250 mL volumetric flask; add approximately 150 mL of water; place in a shaker at $(25 \pm 5) ^\circ\text{C}$. Shake at a frequency of (180 ± 20) r/min for 30 min. After removal, use water to dilute it to the mark; mix well; filter dry; discard the first few milliliters of filtrate; the filtrate is ready for analysis.

3.1.4.2.2

Liquid specimen

Weigh 0.2 g ~ 3 g of specimen (accurate to 0.0001 g) into a 250 mL volumetric flask; use water to dilute it to the mark; mix well; filter dry; discard the first few milliliters of filtrate; use the filtrate for analysis.

3.1.4.3 Plotting of working curve

Pipette 0 mL, 1.00 mL, 2.00 mL, 4.00 mL, 8.00 mL, 10.00 mL of calcium standard solution (3.1.2.5) into six 100 mL volumetric flasks, respectively. Add 4 mL of hydrochloric acid solution (3.1.2.2) and 10 mL of strontium chloride solution (3.1.2.3) to each flask; use water to dilute it to the mark; mix well. The standard series of calcium concentrations are 0 $\mu\text{g/mL}$, 1.0 $\mu\text{g/mL}$, 2.0 $\mu\text{g/mL}$, 4.0 $\mu\text{g/mL}$, 8.0 $\mu\text{g/mL}$, 10.0 $\mu\text{g/mL}$, respectively. Under optimal operating conditions, the absorbance of each standard solution is measured at a wavelength of 422.7 nm, using a lean air-acetylene flame, with the standard solution containing 0 $\mu\text{g/mL}$ of calcium as a reference solution.

A working curve is plotted, using the calcium concentration ($\mu\text{g/mL}$) of each standard solution as the abscissa and the corresponding absorbance value as the ordinate.

Note: The mass concentration of the standard curve can be adjusted according to the sensitivity of different instruments.

3.1.4.4 Determination

Pipette a certain volume of the specimen solution into a 100 mL volumetric flask; add 4 mL of hydrochloric acid solution (3.1.2.2) and 10 mL of strontium chloride solution (3.1.2.3). Dilute the mixture to the mark; mix it well. Under the same instrument conditions as the standard series solutions, measure the absorbance; find the corresponding calcium mass concentration ($\mu\text{g/mL}$) on the working curve.

3.1.4.5 Blank test

Except for not adding the specimen, the other steps are the same as for the specimen solution.

3.1.5 Expression of analytical results

Calcium (Ca) content w_1 is expressed as a mass fraction (%) and calculated according to formula (1).

Where.

ρ - Mass concentration of calcium in the specimen solution as obtained from the working curve, in micrograms per milliliter ($\mu\text{g/mL}$);

ρ_0 - Mass concentration of calcium in the blank solution as obtained from the working curve, in micrograms per milliliter ($\mu\text{g/mL}$);

D - Dilution factor of the specimen solution during the determination;

V - Volume of the specimen solution, in milliliters (mL);

m - Mass of the sample, in grams (g);

10^6 - Coefficient for converting grams to micrograms.

Tak the arithmetic mean of the parallel determination results as the determination result; the result is retained to two decimal places.

3.1.6 Permissible difference

The relative difference between parallel test results shall not exceed 10%.

The relative difference between test results from different laboratories shall not exceed 30%.

When the test result is less than 0.15%, the relative difference between parallel test results and test results from different laboratories shall not be considered.

Note: The relative difference is the ratio -- of the difference between two measurements TO the mean of the two measurements, the same below.

3.1.7 Conversion of mass concentration

The calcium (Ca) content ρ (Ca) of liquid fertilizer is expressed as mass concentration (g/L), which is calculated according to formula (2).

Where.