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Compound fertilizer

复合肥料

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Compound fertilizer

1 Scope

This standard specifies the terms and definitions, technical requirements, sampling, test methods, inspection rules, marking and quality certificates, packaging, transportation, storage of compound fertilizers.

This standard applies to compound fertilizers (including ternary or binary solid fertilizers, which use nitrogen, phosphorus, potassium as basic nutrients, under various names). This standard does not apply to compound fertilizer products, such as monoammonium phosphate and diammonium phosphate.

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 3597 Fertilizer - Determination of nitrate nitrogen content - Nitron gravimetric method

GB/T 6274 Fertilizers and soil conditioners - Vocabulary

GB/T 6679 General rules for sampling solid chemical products

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

GB/T 8569 Packing of solid chemical fertilizers

GB/T 8571 Preparation of laboratory samples for compound fertilizers

GB/T 8572-2010 Determination of total nitrogen content for compound fertilizers titrimetric method after distillation

GB/T 8573 Determination of available phosphorus content for compound fertilizers

GB/T 8574 Determination of potassium content for compound fertilizers - Potassium tetraphenylborate gravimetric method

5.2 Sample reduction

Quickly mix the collected aggregate sample uniformly. Use a divider or quartering method, to reduce the sample to about 1 kg. Then divide it into two parts, which are divided into two clean and dry glass or plastic bottles with ground stoppers (the quality inspection department of the manufacturer can use a clean and dry plastic Ziplock bag, to hold the sample). Seal and label it. Indicate the name of the manufacturer, product name, batch number or production date, sampling date and the name of the sampler. One bottle is used for product inspection; the other bottle is retained for two month, for future reference.

6 Test methods

6.1 General provisions

Except for appearance and particle size, make parallel determinations of two sets of samples. The reagents, solutions, water, which is used in this standard, shall comply with the provisions of HG/T 2843, when the specifications and preparation methods are not specified.

6.2 Visual inspection

Use the sample in 5.2, to make inspection visually.

6.3 Total nutrients

6.3.1 Determination of total nitrogen content

6.3.1.1 Method 1: Post-distillation titration (arbitration method)

It is carried out, in accordance with GB/T 8572.

6.3.1.2 Method 2: Automatic analyzer method

After the specimen is prepared, according to the provisions of GB/T 8571 (if the sample is difficult to pulverize, it can be ground, until it passes through a 1.00 mm aperture test sieve). It is determined, according to GB/T 22923.

6.3.1.3 Method 3: Dumas combustion method

It is carried out, in accordance with 3.2 of NY/T 1977-2010.

6.3.2 Determination of available phosphorus content and calculation of the percentage of water-soluble phosphorus in available phosphorus

6.3.2.1 Method 1: Quinoline phosphomolybdate gravimetric method (Arbitration method)

It is carried out, in accordance with Appendix A.

6.3.2.2 Method 2: Quinoline phosphomolybdate gravimetric method or plasma emission spectrometry

It is carried out, in accordance with GB/T 8573.

6.3.2.3 Method 3: Automatic analyzer method

After the specimen is prepared, according to the provisions of GB/T 8571 (if the sample is difficult to pulverize, it can be ground, until it passes through a 1.00 mm aperture test sieve). It is determined according to GB/T 22923.

6.3.3 Determination of potassium content

6.3.3.1 Method 1: Potassium tetraphenylborate gravimetric method (Arbitration method)

It is carried out, in accordance with GB/T 8574.

6.3.3.2 Method 2: Automatic analyzer method

After the specimen is prepared, according to the provisions of GB/T 8571 (if the sample is difficult to pulverize, it can be ground until, it passes through a 1.00 mm aperture test sieve). It is determined according to GB/T 22923.

6.3.3.3 Method 3: Plasma emission spectrometry

Prepare the specimen solution, according to 4.3.2 of NY/T 2540-2014. Then it is determined, in accordance with 5.3 of NY/T 2540-2014.

6.3.4 Calculation of total nutrients

Total nutrients are the sum of total nitrogen, available phosphorus, potassium content.

6.4 Determination of nitrate nitrogen content

6.4.1 Method 1: Nitrogen reagent gravimetric method (arbitration method)

After the specimen is prepared, according to the provisions of GB/T 8571 (if the sample is difficult to pulverize, it can be ground, until it passes through a 1.00 mm aperture test sieve). Then it is determined, according to GB/T 3597.

6.4.2 Method 2: Automatic analyzer method

After the specimen is prepared, according to the provisions of GB/T 8571 (if the sample is difficult to pulverize, it can be ground, until it passes through a 1.00 mm aperture test sieve). Then it is determined, according to GB/T 22923.

7.3.1 The qualification of the product quality indicators, in this standard, shall be determined, by the "rounding-off value comparison method", in GB/T 8170.

7.3.2 Manufacturers shall carry out the exit-factory inspection and type inspection, according to the requirements of this standard. When all the inspection items meet the requirements of this standard, the batch of products is judged to be qualified.

7.3.3 If one of the indicators, in the results of the exit-factory inspection or type inspection, which is conducted by the manufacturer, does not meet the requirements of this standard, double amount of samples shall be taken from the same batch of packaging containers, for inspection. In the re-inspection results, even if there is one indicator does not meet the requirements of this standard, the batch of products is judged to be unqualified.

8 Identification and quality certificate

8.1 If the product contains nitrate nitrogen, it shall be marked with "nitrate nitrogen", on the packaging container.

8.2 When the products, which use the citrate-soluble phosphate fertilizers, such as calcium-magnesium-phosphate fertilizers, as the basic fertilizer, they shall be marked as "citrate-soluble phosphorus", in a prominent position of the packaging container.

8.3 For products whose mass fraction of chloride ions is greater than 3.0%, it shall, according to the "mass fraction of chloride ions" required in 4.2, clearly mark the Chinese characters "chlorine-contained (low chlorine)", "chlorine-contained (medium chlorine)", "chlorine-contained (high chlorine)", in prominent positions of the packaging container, rather than “chlorine", “Cl-contained” or “Cl”, etc. For products, which are marked with “Chlorine-contained”, it shall not have pictures of chlorine-sensitive crops on the packaging container, nor shall it have markings such as "potassium sulfate (type)", "potassium nitrate (type)", "sulfur group", "nitrosulfur group", which may easily lead users to misunderstand that the product does not contain chlorine. For products, which have the marking of "chlorine-contained (high chlorine)", it shall be marked of the warning words that "high chlorine content; improper use will cause damage to crop and soil".

8.4 For the products, which contain amide nitrogen (urea nitrogen), it shall be marked with the following warning words, in a prominent position on the packaging container: "Containing biuret; improper use will cause damage to crops". For products, which are not marked with this warning, the inspection and testing agency may select a method for measuring the total nitrogen content for detection and determination, based on the fact that the product does not contain amide nitrogen (urea nitrogen).

8.5 If medium elements and (or) trace elements are added, the content of each single element can be indicated separately, according to the medium elements and (or) trace

elements (both are calculated as elemental elements); the content of medium elements and trace elements shall not be included in the total nutrients. When the content of available calcium and available magnesium, in a single medium element, is less than 1.0%, the total sulfur content is less than 2.0%, the content of a single trace element is less than 0.02%, it shall not be marked.

8.6 There shall be warnings, precautions for use, etc., on the outer packaging container of the product. Some product information, such as production date or batch number, certificate of conformity, instructions for use, can be marked with easily identifiable QR codes or barcodes.

8.7 If fertilizer additives, other than the requirements of this standard, are marked on the product packaging, it shall mark the name, function, content, corresponding testing method standards of the additives, on the packaging container.

8.8 The marking of nutrient content shall be labelled on the basis of the total material; the material in the packaging container shall not be separated and labelled separately.

8.9 The net content of each bag shall be marked with a single value, such as 50 kg.

8.10 Each batch of qualified exit-factory products shall be accompanied by a quality certificate, which includes: manufacturer's name, address, product name, batch number or date of manufacture, total nutrients, formula or main nutrient content, chloride ion content, biuret content, this standard number, as well as the content that shall be marked according to laws and regulations. For the products, which use the citrate-soluble phosphate fertilizers, such as calcium-magnesium phosphate fertilizers, as the basis, it shall be marked as "citrate-soluble phosphorus"; meanwhile, it shall indicate whether they are "nitrate nitrogen", "urea nitrogen", "organic nitrogen". Non-exit-factory inspection items are marked with the test results of the latest type inspection.

8.11 The rest shall be implemented, in accordance with the provisions of GB 18382.

9 Packaging, transportation, storage

9.1 The product is packaged, by the materials that meet the requirements of GB/T 8569. The packaging specifications are 1000 kg, 50 kg, 40 kg, 25 kg; the allowable range of the net content of each bag is (1000 ± 10) kg, (50 ± 0.5) kg, (40 ± 0.4) kg, (25 ± 0.25) kg; the average net content of each bag of each batch of products shall not be less than 1000 kg, 50.0 kg, 40.0 kg, 25.0 kg. Other packaging specifications, which are agreed upon by both parties in the contract, can also be used.

9.2 When there are additives in the product, within the stated net content range of each bag, they shall be mixed with the raw materials evenly; they shall not be put into the packaging bag, in the form of small packages.

9.3 On the premise of complying with the provisions of GB/T 8569, it should use the

Appendix A

(Normative)

Determination of available phosphorus content in compound fertilizers - Quinoline phosphomolybdate gravimetric method

A.1 Principle

Water-soluble phosphorus is extracted, by grinding with water or ultrasonically. Use an alkaline solution of disodium ethylenediaminetetraacetate (EDTA), which has a pH value between 12.0 and 12.5, to heat and boil it, to extract the available phosphorus in the compound fertilizer. The extract is boiled with nitric acid solution, for more than 10 min, to completely convert the non-orthophosphate in the test solution into orthophosphate. In acidic medium, the content of water-soluble phosphorus and available phosphorus is determined, by quinoline phosphomolybdate gravimetric method.

A.2 Reagents or materials

A.2.1 Sodium hydroxide solution: 200 g/L.

A.2.2 Disodium ethylenediaminetetraacetate (EDTA) alkaline solution: Weigh 37.5 g of disodium EDTA. Dissolve it in 1000 mL of water. Mix well. Adjust the pH value to 12.0 ~ 12.5, by sodium hydroxide solution (A.2.1), before use.

A.2.3 Nitric acid solution: 1 + 1.

A.2.4 Quinmolybdenum limonene reagent.

A.3 Instruments and equipment

A.3.1 Usually laboratory instruments are used.

A.3.2 Ultrasonic cleaning instrument: It can control the temperature (30 ± 2) °C.

A.3.3 Electric heating constant temperature drying oven: The temperature can be controlled at (180 ± 2) °C.

A.3.4 Glass crucible filter: No.4, 30 mL.

A.4 Sample

A.4.1 Specimen preparation

It is carried out, according to the provisions of GB/T 8571 (if the sample is difficult to

pulverize, it can be ground to pass the 1.00 mm aperture test sieve).

A.4.2 Weighing of specimen

Weigh the sample, which contains 100 mg ~ 180 mg of phosphorus pentoxide, from A.4.1, accurate to 0.0002 g.

A.5 Test procedure

A.5.1 Extraction of water-soluble phosphorus

A.5.1.1 Extraction method 1 (grinding by adding water): Weigh the sample, according to the requirements of A.4.2. Place it in a 75 mL porcelain evaporating dish. Add 25 mL water to grind it. Pour the clear liquid into the 250 mL volumetric flask, which was pre-filled with 5 mL of nitric acid solution. Continue to grind with water three times, using 25 mL of water each time. Then transfer the water-insoluble matter to the filter paper. Use water to wash the water-insoluble matter, until the solution in the volumetric flask is about 200 mL. Finally, use water to dilute it to the mark. Mix well, to obtain the solution A, which is used for the determination of water-soluble phosphorus.

A.5.1.2 Extraction method 2 (ultrasonic extraction): Weigh the sample, according to the requirements of A.4.2. Place it in a 250 mL volumetric flask. Add 150 mL of water. Shake to make the sample evenly dispersed. Add the stopper tightly. Place the volumetric flask in the ultrasonic cleaner, to extract it for 10 min (the liquid level of the ultrasonic cleaner shall be higher than the liquid level of the volumetric flask). Use water to dilute it to the mark. Mix well. Perform dry filtration. Discard the initial part of the filtrate, to obtain the solution B, which is used for the determination of water-soluble phosphorus.

Note: The method A.5.1.1 is used to extract water-soluble phosphorus, during arbitration.

A.5.2 Extraction of available phosphorus

Weigh the sample, according to the requirements of A.4.2. Place it in a 400 mL beaker. Add 150 mL of EDTA alkaline solution. Cover with a watch glass. Place it on an electric hot plate, to heat for 15 min. Remove it. Cool it to room temperature. Quantitatively transfer it to a 250 mL volumetric flask. Use water to dilute it to the mark. Mix well. Perform dry filtration. Discard the initial part of the filtrate, to obtain the solution C, which is used for the determination of available phosphorus.

A.5.3 Determination of water-soluble phosphorus

Use a single-marked pipette, to pipette 25 mL of solution A or solution B. Transfer it into a 400 mL high beaker. Add 20 mL of nitric acid solution. Use water to dilute it to 100 mL. Add a glass rod and stir evenly. Cover with a watch glass. Heat on a hot plate for at least 10 min. Remove it. Use a small amount of water, to rinse the watch glass and the inner wall of the beaker. Add 35 mL of quinolybdenum limonene reagent. Cover

Appendix B

(Normative)

Determination of chloride ion content in compound fertilizers - Automatic potentiometric titration

B.1 Principle

Boil with water, to extract the chloride ion in the sample. Use the silver electrode as the indicator electrode. Use the silver nitrate standard titration solution, to titrate the chloride ion in the test solution. Use the sharp change of potential of the automatic potentiometric titrator, to determine the reaction end point. Use the consumed volume of silver nitrate standard titration solution, to calculate the chloride ion content.

B.2 Reagents or materials

B.2.1 Silver nitrate solution [$c(AgNO_3)$ = 0.01 mol/L]: Weigh 1.7 g of silver nitrate. Dissolve it in water. Add water to dilute it to 1000 mL. Store it in a brown bottle.

B.2.2 Chloride ion standard solution (1 mg/mL): Accurately weigh 1.6487 g of standard sodium chloride, which was dried at 270 °C ~ 300 °C for 4 h, in a 100 mL beaker. Use a small amount of water to dissolve it. Transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix well. Store it in a plastic bottle.

B.3 Instruments and equipment

B.3.1 General laboratory instruments.

B.3.2 Automatic potentiometric titrator, which is equipped with silver electrodes.

B.4 Sample

It is carried out, according to the provisions of GB/T 8571 (if the sample is difficult to pulverize, it can be ground to pass the 1.00 mm aperture test sieve).

B.5 Test procedure

B.5.1 Calibration of silver nitrate solution

Accurately pipette 3.0 mL of chloride ion standard solution, into the titration cup. Add water, until the liquid level covers the electrode. Then calibrate it. The relative difference, between the two calibration values, shall not be greater than 0.5%.

B.5.2 Preparation of specimen solution

Appendix C

(Normative)

Determination of available calcium and available magnesium content in compound fertilizer - Plasma emission spectrometry

C.1 Principle

Use citric acid solution at (30 ± 2) °C constant temperature, to perform oscillation or ultrasonic to extract available calcium and available magnesium in compound fertilizer. The calcium and magnesium in specimen solution are atomized and excited to high energy state, in plasma emission (ICP) light source. When the atoms in the high-energy state transition to the ground state, it generates electromagnetic radiation with characteristic wavelengths. The radiation intensity is proportional to the concentration of calcium and magnesium atoms.

C.2 Reagents or materials

C.2.1 Citric acid solution (20 g/L): Weigh 20 g of citric acid, in a 1000 mL beaker. Add water to dissolve it, to dilute it to 1000 mL. Mix well. Store it in a plastic bottle.

C.2.2 Calcium standard solution: ρ (Ca) = 1 mg/mL.

C.2.3 Magnesium standard solution: ρ (Mg) = 1 mg/mL.

C.2.4 High-purity argon.

C.3 Instruments and equipment

C.3.1 Laboratory instruments are usually used.

C.3.2 Constant temperature water bath oscillator: It can control the temperature at (30 ± 2) °C.

C.3.3 Ultrasonic cleaning instrument: It can control the temperature at (30 ± 2) °C.

C.3.4 Plasma emission spectrometer.

C.4 Sample

It is carried out, according to the provisions of GB/T 8571 (if the sample is difficult to pulverize, it can be ground to pass the 1.00 mm aperture test sieve).

C.5 Test procedure

C.5.1 Extraction of available calcium and available magnesium

C.5.1.1 Extraction method 1 (extraction by citric acid constant temperature oscillation): Weigh 1 g ~ 2 g of sample (accurate to 0.0002 g) from C.4. Put it in a 250 mL volumetric flask. Add 150 mL of citric acid solution. Shake it to make the specimen evenly dispersed. Close the bottle stopper. Place it in a constant temperature water bath oscillator, at (30 ± 2) °C. Perform oscillation extraction, at constant temperature for 1 h. Take it out. Cool it to room temperature. Use water to dilute it to the mark. Mix well. Perform dry filtration. Discard the initial part of the filtrate, to obtain the solution D, which is used for the determination of available calcium and available magnesium.

C.5.1.2 Extraction method 2 (extraction by citric acid ultrasonic): Weigh 1 g ~ 2 g of sample (accurate to 0.0002 g) from C.4. Put it in a 250 mL volumetric flask. Add 150 mL of citric acid solution. Shake to make the sample disperse evenly. Close the stopper. Place it in a (30 ± 2) °C ultrasonic cleaner, for constant temperature ultrasonic extraction, for 10 minutes. Take it out. Cool it to room temperature. Use water to dilute it to the mark. Mix well. Perform dry filtration. Discard the initial part of the filtrate, to obtain the solution E, which is used for the determination of available calcium and available magnesium.

C.5.2 Drawing of working curve

Draw 0 mL, 0.50 mL, 1.00 mL, 4.00 mL, 8.00 mL, 10.00 mL of calcium and magnesium standard solutions, respectively, into six 100 mL volumetric flasks. Use water to dilute it to the mark. Mix well. The mass concentrations of calcium and magnesium in this standard series are 0 μg/mL, 5.0 μg/mL, 10.0 μg/mL, 40.0 μg/mL, 80.0 μg/mL, 100.0 μg/mL, respectively.

Before the measurement, according to the properties of the element to be measured and the performance of the instrument, optimize the measurement conditions, such as argon gas flow, observation height, video generator power, integration time. Then, use a plasma emission spectrometer, to measure the radiation intensity of each standard solution, at wavelengths of 317.933 nm (calcium) and 285.213 nm (magnesium). Take the mass concentration of calcium and magnesium (μg/mL) of each standard solution as the abscissa AND the corresponding radiation intensity as the ordinate, to draw the working curve.

Note: The mass concentration of the standard curve can be adjusted, according to different instrument sensitivities.

C.5.3 Determination

After specimen solution D or specimen solution E is directly or properly diluted, under the same conditions as the standard series solutions, measure the radiation intensity of calcium and magnesium. Check the corresponding mass concentrations of calcium and magnesium (μg/mL), on the working curve.

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