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Controlled-release fertilizer

控释肥料

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Foreword

This Standard was drafted in accordance with the rules given in GB/T 1.1-2009 *Directives for standardization - Part 1: Structure and drafting of standards*.

Attention is drawn to the possibility that some of the elements of this Standard may be the subject of patent rights. The issuing authority shall not be held responsible for identifying any or all such patent rights.

Annex A, Annex B of this Standard are normative.

This Standard was proposed by China Petroleum and Chemical Industry Federation.

This Standard shall be under the jurisdiction of Subcommittee on New Fertilizer, National Technical Committee on Fertilizers and Soil Conditioning Agents of Standardization Administration of China (SAC/TC105 / SC5).

Main drafting organizations of this Standard: National Fertilizer Quality Supervision and Inspection Center (Shanghai), Shandong Kingenta Ecological Engineering Co., Ltd., Shandong Agricultural University, China Agricultural University.

Main drafters of this Standard: Liu Gang, Wan Lianbu, Cao Yiping, Xu Qiuming, Chen Hongkun, Yang Yi, Duan Lulu, Dong Maozhong.

This Standard was issued for the first time.

Controlled-release fertilizer

1 Scope

This Standard specifies the terms, requirements, test methods, inspection rules, marks, packaging, transport and storage of controlled release fertilizer.

This Standard applies to single, compound (combined), blending (BB) controlled release fertilizer made in various processes.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 6679, *General rules for sampling solid chemical products*

GB/T 8170-2008, *Rules of rounding off for numerical values & expression and judgement of limiting values*

GB 8569, *Packing of solid chemical fertilizers*

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

GB/T 17767.3-1999, *Determination of organic-inorganic compound fertilizers - Part 3: Total potassium content*

GB 18382, *Fertilizer marking - Presentation and declaration*

GB 21633, *Bulk Blending Fertilizer*

GB/T 22923, *Determination of nitrogen, phosphorus and potassium for fertilizers by auto analyzer*

GB/T 24891, *Determination of particle size for compound fertilizers*

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

and preparation methods are not specified.

6.1 Appearance

Use visual inspection method.

6.2 Determination of total chlorine content

Carry out according to the method specified in corresponding core product standard or the one specified in GB/T 22923. Take chemical method as the arbitration method.

6.3 Determination of water-soluble phosphorus and available phosphorus as well as calculation of mass fraction of water-soluble phosphorus accounting for available phosphorus

Carry out according to the method specified in corresponding core product standard or the one specified in GB/T 22923. Take chemical method as the arbitration method.

6.4 Determination of potassium content

Carry out according to the method specified in corresponding core product standard or the one specified in GB/T 22923. Take chemical method as the arbitration method.

6.5 Determination of moisture

Carry out according to the method specified in corresponding core product standard.

6.6 Determination of particle size

Choose the appropriate test sieve. Carry out according to the method specified in GB/T 24891.

6.7 Determination of release rate of nutrient

6.7.1 Method summary

Use water to soak the test material. At the specified temperature and time, the nutrients in the sample are dissolved from the fertilizer particles into the water. Use post-distillation titration or automatic analyzer to determine the total ammonia content. Use flame photometric method or gravimetric method or automatic analyzer method to determine the dissolved potassium content. The conductivity method in Annex A or the spectrophotometric method in Annex B can also be used to determine the dissolved nutrient content according to the controlled release nutrient in core product. The mass fraction

shall be same.

Determine the extraction temperature of cumulative release rate of nutrient of stated release longevity of nutrient as 25°C, extraction time as days of indicated stated release longevity of nutrient; or as the indicated extraction temperature (15°C, 60°C or 100°C) of simulated inspection as well as corresponding days or hours. The detection result under this condition is expressed by w_t.

6.7.3.3 Extraction of controlled release nutrient

6.7.3.3.1 Extraction at 25°C

Weigh about 10 g of the uncrushed test material in 7.4.2 (to the nearest 0.01 g) and put into a pouch made of nylon gauze with aperture of 150 μm (100 mesh). After sealing, place the pouch in a 250 mL glass or plastic bottle. Add into 200 mL of water. Cover it and seal. Place in a 25°C biochemical incubator. The sampling time is 24 h, 3 d, 5 d, 7 d, 10 d, 14 d, 28 d, 42 d, 56 d. The time interval for subsequent sampling shall be based on the release of controlled release nutrients (the maximum interval must not exceed 28 d) until the cumulative release rate of nutrient exceeds 80%. When sampling, make the bottle up and down three times to make the liquid concentration in the bottle consistent. Move them all into a 250 mL volumetric flask. Cool to room temperature and set volume to scale for the determination of nitrogen and potassium release contents according to 6.7.3.5, 6.7.3.6, respectively. Then, add 200 mL of water into the bottle loaded with test material pouch. Cover and seal. Then place into the biochemical incubator for continuous cultivation.

6.7.3.3.2 Extraction at 15°C, 60°C

The extraction temperatures shall be 15°C, 60°C. Carry out according to the steps of 6.7.3.3.1.

6.7.3.3.3 Extraction at 100°C

Weigh about 10 g of the uncrushed test material in 7.4.2 (to the nearest 0.01 g) and put into a stainless steel pouch of constant temperature rapid extraction instrument. Put into the closed extraction chamber. Add 200 mL of water. When the temperature is constant at (100±1) °C, start timing. The sampling time is 1 h, 3 h, 5 h, 7 h, 10 h, 24 h, and the sampling interval shall be 6 h until the cumulative release rate of nutrient exceeds 80%. When sampling, move all the extraction liquid in the closed extraction chamber into a 250 mL volumetric flask. Cool to room temperature and set volume to scale for the determination of nitrogen and potassium release contents according to 6.7.3.5, 6.7.3.6, respectively. Add 200 mL of water. After sealing, continue the extraction of the next period.

n - can be 1 d, 10 d, 14 d, respectively;

m - can be 10 d, 21 d, 28 d, respectively.

6.9 The calculation of stated release longevity of nutrient of partial controlled release fertilizer shall be same with 6.8.

6.10 The release content w_s of partial controlled release fertilizer, in mass fraction (%), is calculated according to equation (7):

$$w_s = w - w_1 \quad \cdots\cdots (7)$$

where,

w - the mass fraction of total nitrogen, the mass fraction of total potassium or the sum of total nitrogen and potassium mass fraction measured according to 6.2 or 6.4, based on the marked controlled release nutrient type, in %;

w_1 - the release contents of nitrogen or potassium measured according to 6.7.3.4; the controlled release nutrient shall be consistent with w, in %.

7 Inspection rules

7.1 Inspection type and inspection items

The product inspection consists of exit-factory inspection and type inspection. The exit-factory inspection items include the total nutrients, the mass fraction of water-soluble phosphorus accounting for available phosphorus, moisture, particle size, initial release rate of nutrient, controlled release nutrient content in Table 1 and Table 2 as well as the exit-factory inspection items of core product. The type inspection items are all items in Table 1 and Table 2 and type inspection items of core product. The exit-factory inspection data of moisture shall prevail. Type inspection shall be performed in one of the following situations:

- when new products are put into operation;
- great changes in structure, material, technique in official production which may affect product performances;
- at least once a year in official production, regular or cumulative to a certain amount;
- required by national quality supervision organization at all levels.

7.2 Batching

7.3.3 Bulk products

In accordance with GB/T 6679.

7.4 Sample shrinkage and sample preparation

7.4.1 Sample shrinkage-division

Process the sample extracted according to the steps of sample shrinkage-division in GB 21633. Pack them in two clean, dry 500 mL polyethylene bottles or jar with a mouthpiece (the manufacturer can also use clean and dry plastic self-styled bag to contain samples). Tag the label after sealing. The label shall indicate manufacturer's name, product's name, level (if any), batch number or date of manufacture, batch, sampling date, name of sampling staff. One bottle is used for quality analysis. The shelf life of another bottle shall be at least two months beyond the stated release longevity of nutrient.

7.4.2 Sample preparation

Take a bottle of 500 g of shrank-divided sample in 7.4.1. Take out about 100 g after several times of mixing and shrinkage-division. Immediately ground them till all go through the 1.00 mm aperture sieve (if it is difficult, use 2.00 mm aperture sieve). Well mix and place in a clean, dry bottle for the determination of total nutrient, free water. Residual laboratory samples shall be used for the determination of release rate of nutrient, particle size.

7.5 Results determination

7.5.1 The qualification determination of product quality in this Standard shall use the "rounding-off value comparison method" in GB/T 8170-2008.

7.5.2 Should any item fail to meet requirements in type inspection, the whole batch of products shall be rejected.

7.5.3 During exit-factory inspection of manufacturer: when all exit-factory inspection items meet requirements, this batch of products shall be accepted. If one indicator fails to meet the requirements of this Standard, it shall extract sample double the amount in the packaging bag for inspection. In the re-inspection results, even only one indicator fails to meet the requirements of this Standard, the whole batch of products shall be rejected. If two or more indicators fail to meet the requirements of this Standard, this batch of products shall be rejected.

7.5.4 During the third-party inspection, should any item of cumulative release rate of nutrient of the 7^{th} day, the 28^{th} day and the indicated release period (actual measured or simulated result) fail to comply with the provisions

Annex A

(Normative)

Rapid determination of cumulative release rate of nutrient

- Conductivity method

A.1 Scope

This method is only applicable to controlled release mixed fertilizer (compound fertilizer), controlled release blend bulk fertilizer or controlled release potassium fertilizer.

This method is not applicable to the controlled release urea products.

A.2 Test methods

A.2.1 Preparation of standard series concentration

Accurately weigh 10.00 g of crushed test material into a 100 mL beaker. Use a small amount of water to dissolve. Filter in a 500 mL volumetric flask. Rinse repeatedly 5~6 times then set volume. Respectively pipet 0, 10 mL, 20 mL. 40 mL, 60 mL, 80 mL, 100 mL, 120 mL of solution and set volume in 250 mL volumetric flask. Use conductivity meter to measure the conductivity value. Make the standard curve with the total nutrient concentration values of standard series or make linear regression equation.

A.2.2 Determination of concentration of extract nutrient

Determine the conductivity value of extract under the test conditions same with for making the standard curve. Check the nutrient concentration in the extract from the standard curve or calculate it by the linear regression equation. Refer to 6.7.4 and calculate the cumulative release rate of nutrient. For controlled release mixed fertilizer (compound fertilizer), controlled release blend bulk fertilizer, w, w_1, w_7, w_{28}, w_t in the calculation equations in 6.7.4 refer to the mass fraction of total nutrient of nitrogen, phosphorus, potassium measured of each corresponding extract, in %.

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