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**Water-soluble fertilizers - Determination of humic-acids
content**

水溶肥料 腐植酸含量的测定

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Water-soluble fertilizers - Determination of humic-acids content

1 Scope

This standard specifies the test method for the determination of humic-acid content, in water-soluble fertilizers, by redox titration after acid precipitation.

This standard applies to the determination of humic-acid 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

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 Principles

The humic-acid in the specimen solution is quantitatively precipitated, under acidic conditions. Other non-humic-acid carbon, chloride ions, low-valent metal ions that interfere with the determination, remain in the solution. After the solution is discarded, the organic carbon in the precipitate is oxidized by a quantitative potassium dichromate-sulfuric acid solution. The remaining potassium dichromate is titrated, by a standard titration solution of ferrous sulfate. Taking the reagent blank as the benchmark, according to the consumption of oxidant before and after the oxidation of the specimen, the organic carbon content is calculated. The humic-acid content of the specimen is obtained, through the conversion of the carbon coefficient.

c_2 - The concentration of the ferrous sulfate standard titration solution, in moles per liter (mol/L);

c_1 - The concentration of potassium dichromate standard solution, in moles per liter (mol/L);

V_1 - The volume of the potassium dichromate standard solution, which is pipetted, in milliliters (mL);

V_2 - The volume of ferrous sulfate standard solution, which is consumed during titration, in milliliters (mL).

5 Instruments

5.1 General laboratory instruments.

5.2 Centrifuge: 4000 r/min, which is equipped with a 50 mL polytetrafluoroethylene or round-bottom glass centrifuge tube.

5.3 Constant temperature water bath: The temperature can reach $100\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

6 Analytical procedures

6.1 Preparation of specimens

After the solid sample has been divided several times, take out about 100 g. Quickly grind it, until it all passes through a 0.50 mm sieve (if the sample is wet, it can pass through a 1.00 mm sieve). Mix it evenly. Place it in a clean and dry container. After the liquid samples is shaken several times, quickly remove about 100 mL. Place it in a clean, dry container.

6.2 Preparation of specimen solution

6.2.1 Solid specimen

Weigh about 0.5 g of the specimen (accurate to 0.0001 g), in a 50 mL beaker. Add about 10 mL of water. Use a glass rod to stir it. Let it stand for a while. Transfer the solution part into a 100 mL volumetric flask. Add about 10 mL of water to the beaker. Repeat this step 3 times. Add 1 mL of sodium hydroxide solution (4.7) to the residue. Stir to dissolve it. Transfer it into a volumetric flask. Use water to make the volume reach to the mark. Mix well.

6.2.2 Liquid specimen

Weigh 2 g ~ 3 g of the specimen (accurate to 0.0001 g), into a 100 mL volumetric flask. Add 1 mL of sodium hydroxide solution (4.7) and a small amount of water. Dissolve it

fully. Make the volume reach to the mark. Mix well.

6.3 Precipitation of humic-acid in specimen solution

Accurately pipette 5.0 mL of uniform specimen solution into a centrifuge tube. Add 5 mL of sulfuric acid solution (4.6). Mix well. Put it into a centrifuge, to centrifuge it for 10 min, at 3000 r/min ~ 4000 r/min (if there are still solid floating substances in the solution, the centrifugation time shall be extended, until all the solids are precipitated). Pour off the supernatant liquid.

6.4 Oxidation of humic-acid

Add 5.0 mL of potassium dichromate solution (4.8) to the centrifuge tube. Slowly add 5 mL of sulfuric acid (4.3). Gently shake the centrifuge tube, to mix the contents evenly. Put the centrifuge tube on the tube rack. Cover the funnel. Heat it in a boiling water bath for 30 min. Take it out. Cool it. Transfer the contents into a 250 mL conical flask. The volume shall be controlled at 60 mL ~ 80 mL.

6.5 Titration

Add 3 ~ 5 drops of o-phenanthroline indicator (4.5) to the conical flask. Use the standard titration solution of ferrous sulfate (4.10), to titrate the remaining potassium dichromate. When the color of solution changes from orange-yellow → blue-green → brown-red, it reaches the end point. If the volume, which is consumed by the titration, is less than 1/3 of the volume, which is consumed by the titration blank, the specimen weighing amount shall be reduced, to make measurement again.

6.6 Blank test

Except that no specimen is added, other steps are the same as the determination of the specimen solution. When the absolute difference of the titration, between the two blank tests is ≤ 0.06 mL, the average value can be taken and substituted into the calculation formula.

7 Presentation of analysis results

The humic-acid content, w , is expressed in mass fraction (%). It is calculated according to formula (2):

$$w = \frac{(V_1 - V_2)cD \times 1.724 \times 0.003 \times 1.43}{m} \times 100 \dots \dots \dots (2)$$

Where:

c - The concentration of ferrous sulfate standard titration solution, which is used for determination of the specimen and blank test, in mole per liter (mol/L);

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