SCHEDULE 2

METHODS OF ANALYSIS

PART I

14g.

DETERMINATION OF COPPER

1 SCOPE AND FIELD OF APPLICATION

1. This method defines the procedure for the determination of the copper content of straight ammonium nitrate fertilisers containing more than 28% nitrogen by weight.

2 PRINCIPLE

2. The sample is dissolved in dilute hydrochloric acid and the copper content is determined by atomic absorption spectrometry.

3 REAGENTS

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3.1 Hydrochloric acid (density at 20° C ρ = 1.18 g/ml).

3.2 Hydrochloric acid, 6 M solution.

3.3 Hydrochloric acid, 0.5 M solution.

3.4 Ammonium nitrate.

3.5 Hydrogen peroxide, 30%.

3.6 Copper solution(**1**) (stock): weigh, to the nearest 0.001 gram, 1 gram of pure copper, dissolve in 25 ml of 6 M hydrochloric acid solution (3.2), add 5 ml of hydrogen peroxide (3.5) in portions and dilute to 1 litre with water. 1 ml of this solution contains 1,000 µg of copper (Cu).

(3.6.1) Copper solution (dilute): dilute 10 ml of stock solution (3.6) to 100 ml with water and then dilute 10 ml of the resulting solution to 100 ml with water. 1 ml of the final dilution contains 10 µg of copper (Cu).

Prepare this solution at the time of use.

4 APPARATUS

4. Atomic absorption spectrometer with a copper lamp (324.8 nm).

5 PROCEDURE

5

(**1**) Commercially available standard copper solution may be used.

Preparation of the solution for analysis

5.1 Weigh 25 grams, to the nearest 0.001 gram, of the sample into a 400 ml beaker, add carefully 20 ml of hydrochloric acid (3.1) (there may be a vigorous reaction due to carbon dioxide formation). Add more hydrochloric acid, if necessary. When effervescence has stopped, evaporate to dryness on a steam bath, stirring occasionally with a glass rod. Add 15 ml 6 M hydrochloric acid solution (3.2) and 120 ml of water. Stir with the glass rod, which should be left in the beaker, and cover the beaker with a watch glass. Boil the solution gently until dissolution is complete and then cool.

Transfer the solution quantitatively into a 250 ml graduated flask, by washing the beaker with 5 ml 6 M hydrochloric acid (3.2), and twice with 5 ml of boiling water, cool and make up to the mark with 0.5 M hydrochloric acid (3.3) and mix carefully.

Filter through a copper-free filter paper(**2**), discarding the first 50 ml.

Blank solution

5.2 Prepare a blank solution from which only the sample has been omitted and allow for this in the calculation of the final result.

Determination

5.3.—(5.3.1) *Preparation of sample and blank test solutions*

Dilute the sample solution (5.1) and the blank test solution (5.2) with 0.5 M hydrochloric acid solution (3.3) to a concentration of copper within the optimal measuring range of the spectrometer. Normally no dilution is needed.

(5.3.2) *Preparation of the calibration solutions*

By diluting the standard solution (3.6.1) with 0.5 M hydrochloric acid solution (3.3), prepare at least five standard solutions corresponding to the optimal measuring range of the spectrometer (0 to 5.0 µg/l Cu). Before making up to the mark, add ammonium nitrate (3.4) to every solution to give a final concentration of 100 mg per ml.

Measurement

5.4 Set up the spectrometer (4) at a wavelength of 324.8 nm and use an oxidising air-acetylene flame. Spray, in triplicate, the calibration solutions (5.3.2), the sample solution and the blank solution (5.3.1), washing the instrument through with distilled water between each spraying. Plot the calibration curve using the mean absorbances of every standard used as the ordinates and the corresponding concentrations of copper in µg/ml as the abscissae.

Determine the concentration of copper in the final sample and blank solutions by reference to the calibration curve.

6 EXPRESSION OF RESULTS

6. Calculate the copper content of the sample taking into account the weight of the test sample, the dilutions carried out in the course of the analysis and the value of the blank. Express the result as mg Cu/kg.

(**2**) Whatman 541 or equivalent.