SCHEDULE 2

METHODS OF ANALYSIS

PART I

26g.

DETERMINATION OF COPPER IN FERTILISER EXTRACTS BY THE TITRIMETRIC METHOD

1 SCOPE

1. This method defines a procedure for determining copper in fertiliser extracts.

2 FIELD OF APPLICATION

2. This procedure is applicable to extracts from samples of fertilisers obtained by Method 26a or Method 26b for which a declaration of copper content is required.

3 PRINCIPLE

3. The cupric ions are reduced in an acidic medium with potassium iodide:

<2Cu+++4I→2CuI+I2

The iodine released in this way is titrated with a standard sodium thiosulfate solution in the presence of starch as an indicator in accordance with:

I2+2Na2S2O3→2NaI+Na2S4O6

4 REAGENTS

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4.1. Nitric acid (HNO_3, ρ = 1.40 g/ml).

4.2. Urea [$(NH_2)_2$ C O].

4.3. Ammonium bifluoride (NH_4HF_2) solution (10 % w/v)

Keep the solution in a plastic container.

4.4. Ammonium hydroxide solution (1 + 1)

Mix 1 volume of ammonia (NH_4OH, p =: 0.9 g/ml) with 1 volume of water.

4.5. Sodium thiosulfate standard solution

Dissolve 7.812 g of sodium thiosulfate pentahydrate ($Na_2S_2O_3.5H_2O$) with water in a litre volumetric flask. This solution must be prepared so that 1 ml = 2 mg Cu. For stabilization, add several drops of chloroform. The solution must be kept in a glass container and protected from direct light.

4.6. Potassium iodide (KI).

4.7. Potassium thiocyanate (KSCN) solution (25 % w/v)

Keep this solution in a plastic flask.

4.8. Starch solution (about 0.5 %)

Place 2.5 g of starch in a 600 ml beaker. Add about 500 ml of water. Boil while stirring. Cool to ambient temperature. The solution has a short preservation period. Its preservation can be extended by adding about 10 mg of mercury iodide.

5 PREPARATION OF THE SOLUTION TO BE ANALYSED

5. Preparation of the copper solution

See Methods 26a and 26b.

6 PROCEDURE

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6.1. Preparation of the solution for titration

Place an aliquot portion of the solution containing not less than 20 mg Cu in a 500 ml Erlenmeyer flask.

Drive off any excess oxygen present by boiling briefly. Make up to volume of about 100 ml water. Add 5 ml of nitric acid (4.1), bring to boiling and allow to boil for about half a minute.

Remove the Erlenmeyer flask from the heating apparatus, add about 3 g of urea (4.2) and resume boiling for about half a minute.

Remove from the heating apparatus and add 200 ml of cold water. Where necessary, cool the contents of the Erlenmeyer flask to ambient temperature.

Gradually add ammonium hydroxide solution (4.4) until the solution becomes blue, then add 1 ml in excess.

Add 50 ml of ammonium bifluoride solution (4.3) and mix.

Add 10 g of potassium iodide (4.6) and allow it to dissolve.

6.2. Titration of the solution

Place the Erlenmeyer flask on a magnetic stirrer. Insert the rod into the Erlenmeyer flask and adjust the stirrer to the desired speed.

Using a burette, add standard sodium thiosulfate solution (4.5) until the brown colour of the iodine released from the solution becomes less intense.

Add 10 ml of the starch solution (4.8).

Continue to titrate with the sodium thiosulfate solution (4.5) until the purple colour has almost disappeared.

Add 20 ml of the potassium thiocyanate solution (4.7) and continue titration until the violet blue colour has completely disappeared.

Note the volume of thiosulfate solution employed.

7 EXPRESSION OF RESULTS

7. 1 ml of standard sodium thiosulfate solution (4.5) corresponds to 2 mg Cu.

The percentage of copper in the fertiliser is given by:

$Cu(\%)=X\times Va\times M\times 5$

where:

X is the volume in ml of the sodium thiosulfate solution used;

V is the volume in ml of the extract solution in accordance with Methods 26a and 26b;

a is the volume in ml of the aliquot portion;

M is the mass in g of the test sample treated in accordance with Methods 26a and 26b.