

SCHEDULE 2

METHODS OF ANALYSIS

PART I

25g.

DETERMINATION OF COPPER IN FERTILISER EXTRACTS BY ATOMIC ABSORPTION SPECTROMETRY

1 SCOPE

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

2 FIELD OF APPLICATION

2. This procedure is applicable to analysing samples of fertilisers extracted by Methods 25a and 25b for which a declaration of total and/or water-soluble copper is required.

3 PRINCIPLE

3. After suitable treatment and dilution of the extracts, the copper content is determined by atomic absorption spectrometry.

4 REAGENTS

4

- 4.1. Hydrochloric acid solution, about 6 M

See Method 25d (4.1).

- 4.2. Hydrochloric acid solution, about 0.5 M

See Method 25d (4.2).

- 4.3. Hydrogen peroxide solution (30% H₂O, $\rho = 1.11$ g/ml), free from trace elements.

- 4.4. Copper calibration solutions

(4.4.1) Copper stock solution (1,000 µg/ml)

In a 250 ml beaker weigh to the nearest 0.1 mg, 1 g of copper, add 25 ml of 6M hydrochloric acid (4.1) add 5 ml hydrogen peroxide solution (4.3) and heat on a hotplate until the copper is completely dissolved. Transfer quantitatively to a 1 litre volumetric flask. Make up to volume with water and mix thoroughly.

(4.4.2) Copper working solution (100 µg/ml)

Place 20 ml of the stock solution (4.4.1) in a 200 ml volumetric flask. Make up to volume with 0.5 M hydrochloric acid solution (4.2) and mix thoroughly.

5 APPARATUS

5. Spectrometer equipped for atomic absorption: see Method 25.d (5). The instrument must be fitted with a source of rays characteristic of copper (324.8 nm).

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6 PREPARATION FOR THE SOLUTION TO BE ANALYSED

6

6.1. Copper extract solution

See Methods 25a and/or 25b and, if appropriate, 25c.

6.2. Preparation of the test solution

See Method 25d (6.2).

7 PROCEDURE

7

7.1. Preparation of blank solution

See Method 25d (7.1).

7.2. Preparation of calibration solutions

See Method 25d (7.2).

For an optimum determination range of 0 to 5 µg/ml of copper, place 0, 0.5, 1, 2, 3, 4 and 5 ml respectively of working solution (4.4.2) in a series of 100 ml volumetric flasks. If necessary adjust the hydrochloric acid concentration as closely as possible to that of the test solution (6.2). Make up to 100 ml with 0.5 M hydrochloric acid solution (4.2) and mix thoroughly. These solutions contain 0, 0.5, 1, 2, 3, 4, and 5 µg/ml respectively of copper.

7.3. Determination

See Method 25d (7.3). Prepare the spectrometer (5) for measurement at a wavelength of 324.8 nm.

8 EXPRESSION OF RESULTS

8. See Method 25d (8)

The percentage of copper in the fertiliser is given by:

$$\text{Cu}\% = [(x_s x_b) \times V_x D] / M x 104$$

If Method 25c is used:

$$\text{Cu}\% = [(x_s x_b) \times V_x 2D] / M x 104$$

where:

Cu is the quantity of copper expressed as percentage of the fertiliser;

x_s is the concentration in µg/ml of Cu in the test solution (6.2);

x_b is the concentration in µg/ml of Cu in the blank solution (7.1);

V is the volume in ml of extract obtained in accordance with Method 25a or 25b;

D is the factor of the dilution carried out in 6.2;

M is the mass in grams of the test sample taken in accordance with Method 25a or 25b.

Calculation of the dilution factor D: if (a_1) , (a_2) , (a_3) , . . . , (a_i) and (a) are aliquot portions and (v_1) , (v_2) , (v_3) , . . . , (v_i) and (100) are the volumes in ml corresponding to their respective dilution, the dilution factor D is given by:

$$D = (v_1/a_1) \times (v_2/a_2) \times (v_3/a_3) \dots \times (v_i/a_i) \times (100/a)$$