SCHEDULE 2

METHODS OF ANALYSIS

PART I

25i

DETERMINATION OF MANGANESE IN FERTILISER EXTRACTS BY ATOMIC ABSORPTION SPECTROMETRY

1 SCOPE

1. This method describes a procedure for determining manganese 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 manganese is required.

3 PRINCIPLE

3. After suitable treatment and dilution of the extracts, the manganese level 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. Lanthanum salt solutions (10 g of La per litre)

See Method 25d (4.3).

4.4. Manganese calibration solutions

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

In a 250 ml beaker, weigh to the nearest 0.1 mg, 1 g of manganese, add 25 ml of 6 M hydrochloric acid solution (4.1). Heat on a hotplate until the manganese is completely dissolved. When cool, transfer quantitatively to a 1,000 ml volumetric flask. Make up to volume with water and mix thoroughly.

(4.4.2) Manganese 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 the 0.5 M hydrochloric acid solution (4.2) and mix thoroughly.

5 APPARATUS

5. Atomic absorption spectrometer: see Method 25d (5). The apparatus must be fitted with a source of rays characteristic of manganese (279.6 nm).

6 PREPARATION OF THE SOLUTION TO BE ANALYSED

6

6.1. Manganese extract solution

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

6.2. Preparation of the test solution

See Method 25d (6.2). The test solution must contain 10% by volume of lanthanum salt solution (4.3).

7 PROCEDURE

7

7.1. Preparation of the blank solution

See Method 25d (7.1). The blank solution must contain 10% by volume of the lanthanum salt solution used in 6.2.

7.2. Preparation of the calibration solutions

See Method 25d (7.2).

For an optimum interval of 0 to 5 µg/ml manganese, place 0, 0.5, 1, 2, 3, 4 and 5 ml, respectively, of the working solution (4.4.2) in a series of 100 ml volumetric flasks. Where necessary, adjust the hydrochloric acid concentration to bring it as close as possible to that of the test solution. To each flask add 10 ml of the lanthanum salt solution used in 6.2. Make up to 100 ml with the 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 manganese respectively.

7.3. Determination

See Method 25d (7.3). Prepare the spectrometer (5) for measurements at a wavelength of 279.6 nm.

8 EXPRESSION OF RESULTS

8. See Method 25d (8).

The percentage of manganese in the fertiliser is as follows:

$Mn\% = [(x_s - x_b) \times V \times D] / (M \times 104)$

If Method 25c has been used:

$Mn\% = [(x_s - x_b) \times V \times 2D] / (M \times 104)$

where:

Mn is the quantity of manganese expressed as a percentage of the fertiliser;

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

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

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

D is the factor corresponding to the dilution performed in 6.2;

M is the mass in g of the test sample taken using Method 25a or 25b.

Calculation of dilution factor D: where (a_1), (a_2), (a_3), . . ., (a_i) and (a) are aliquot portions and (v_1), (v_2), (v_3), . . ., (v_i) and (100) the volumes in ml corresponding to their respective dilutions, dilution factor D will be equal to:

D=(v1/a1)×(v2/a2)×(v3/a3)×...×(vi/ai)×(100/a).