SCHEDULE 2

METHODS OF ANALYSIS

PART I

26e.

DETERMINATION OF BORON IN FERTILISER EXTRACTS BY MEANS OF ACIDIMETRIC TITRATION

1 SCOPE

1. This method defines a procedure for determining the boron content of fertiliser extracts.

2 FIELD OF APPLICATION

2. This procedure is applicable to extracts from samples of fertilisers obtained by Method 26a or Method 26b and for which a declaration for the total and/or water — soluble boron content is required.

3 PRINCIPLE

3. A mannitoboric complex is formed by the following reaction of the borate with mannitol:

C6H8(OH)6+H3BO3→C6H15O8B+H2O

The complex is titrated with sodium hydroxide solution to a pH of 6.3.

4 REAGENTS

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4.1. Methyl red indicator solution

Dissolve 0.1 g of methyl red ($C_{15}H_{15}N_3O_2$) in 50 ml of ethanol (95% in a 100 ml volumetric flask. Make up the volume to 100 ml with water. Mix thoroughly.

4.2. Diluted hydrochloric acid solution, about 0.5 M

Mix 1 volume of hydrochloric acid HCI, (ρ = 1.18 g/ml) with 20 volumes of water.

4.3. Sodium hydroxide solution, about 0.5 M

Must be free of carbon dioxide. Dissolve 20 g of sodium hydroxide (NaOH) in pellet form in a 1 litre volumetric flask containing about 800 ml of boiled water. When the solution has cooled, make up to 1000 ml with boiled water and mix thoroughly.

4.4. Standard sodium hydroxide solution, about 0.025 M

Must be free of carbon dioxide. Dilute the 0.5 M sodium hydroxide solution (4.3) 20 times with boiled water and mix thoroughly. The value of the solution expressed as boron (B) is to be determined (see paragraph 9).

4.5. Boron calibration solution (100 µg/ml B)

Dissolve 0.5719 g of boric acid (H_3BO_3), weighed to the nearest 0.1 mg, in water in a 1 litre volumetric flask. Make up to volume with water and mix thoroughly. Transfer to a plastic bottle for storage in a refrigerator.

4.6. D-mannitol ($C_6H_{14}O_6$) powder.

4.7. Sodium chloride (NaCl).

5 APPARATUS

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5.1. pH meter with glass electrode

5.2. Magnetic stirrer

5.3. 400 ml beaker with teflon rod

6 PREPARATION OF THE SOLUTION TO BE ANALYSED

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6.1. Preparation of the boron solution

See Methods 26a, 26b and, where appropriate, 26c.

7 PROCEDURE

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7.1. Determination Place in a 400 ml beaker (5.3) an aliquot portion (a) of the extract (6.1) containing 2 to 4 mg B. Add 150 ml of water.

Add several drops of the methyl red indicator solution (4.1).

In the case of extraction with Method 26b, acidify by adding 0.5 M hydrochloric acid (4.2) up to the point of change of the indicator solution, then add a further 0.5 ml of 0.5 M hydrochloric acid (4.2).

After adding 3 g of sodium chloride (4.7), bring to boiling to drive off the carbon dioxide. Allow to cool. Place the beaker on the magnetic stirrer (5.2) and insert the precalibrated pH meter electrodes (5.1).

Adjust the pH to exactly 6.3, first with the 0.5 M sodium hydroxide solution (4.3), then with the 0.025 M solution (4.4).

Add 20 g of D-mannitol (4.6), dissolve completely and mix thoroughly. Titrate with the 0.025 M sodium hydroxide solution (4.4) to pH 6.3 (at least 1 minute stability). Let x_1, be the volume required.

8 BLANK SOLUTION

8. Prepare a blank solution by repeating the whole procedure from the preparation of solution stage, omitting only the fertiliser. Let x_0 be the volume required.

9 BORON (B) VALUE OF THE SODIUM HYDROXIDE SOLUTION (4.4)

9. Transfer by pipette 20 ml (2.0 mg B) of the calibration solution (4.5), into a 400 ml beaker and add several drops of methyl red indicator solution (4.1). Add 3g of sodium chloride (4.7) and the hydrochloric acid solution (4.2) up to the point of change of the indicator solution (4.1).

Make up the volume to about 150 ml and bring gradually to the boil so as to eliminate carbon dioxide. Allow to cool. Place the beaker on the magnetic stirrer (5.2), and insert the precalibrated pH meter electrodes (5.1). Adjust the pH to exactly 6.3, first with the 0.5 M sodium hydroxide solution (4.3), then with the 0.025 M solution (4.4).

Add 20 g of D – mannitol (4.6), dissolve completely and mix thoroughly. Titrate with the 0.025 M sodium hydroxide solution (4.4) to pH 6.3 (at least 1 minute stability). Let V_1 be the volume required.

Prepare a blank solution in the same way, substituting 20 ml of water for the calibration solution. Let V_0 be the volume required.

The boron value (F) in mg/ml of the standard NaOH solution (4.4) is as follows:

F(inmg/ml)=2/(V1V0)

1 ml of exactly 0.025 M sodium hydroxide solution corresponds to 0.27025 mg B.

10 EXPRESSION OF RESULTS

10. The percentage of boron in fertiliser is given by:

B(%)=(x1x0)×F×V10×a×M

where:

B(%) is the percentage of boron in the fertiliser;

x_1 is the volume, in ml, of the 0.025 M sodium hydroxide solution (4.4);

x_0 is the volume, in ml, of the 0.025 M sodium hydroxide solution M (4.4);

F is the boron (B) value, in mg/ml, of the 0.025 M sodium hydroxide solution (4.4);

V is the volume, in ml, of the extract solution obtained in accordance with Method 26a or 26b;

a is the volume, in ml, of the aliquot portion (7.1) taken from the extract solution (6.1);

M is the mass, in grams, of the test sample taken in accordance with Method 26a or 26b.