SCHEDULE 2

METHODS OF ANALYSIS

PART I

25e.

DETERMINATION OF BORON IN FERTILISER EXTRACTS BY MEANS OF SPECTROMETRY WITH AZOMETHINE-H

1 SCOPE

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

3 PRINCIPLE

3. In an azomethine — H solution, borate ions form a yellow complex the concentration of which is determined by molecular absorption spectrometry at 410 nm. Interfering ions are masked with EDTA.

4 REAGENTS

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4.1. EDTA buffer solution

Place in a 500 ml volumetric flask containing 300 ml of water:

— 75 g of ammonium acetate (NH_4OOCCH_3);

— 10 g of disodium salt of ethylene diamine tetraacetic acid (Na_2EDTA);

— 40 ml of acetic acid (CH_3COOH, p = 1.05 g/ml).

Make up to volume with water and mix thoroughly. The pH of the solution, checked by means of a glass electrode, must be 4.8 0.1.

4.2. Azomethine-H solution

Place in a 200 ml volumetric flask

— 10 ml of the buffer solution (4.1);

— 400 mg of azomethine-H ($C_{17}H_{12}NNaO_8S_2$);

— 2 g of ascorbic acid ($C_6H_8O_6$).

Make up to volume and mix thoroughly. Do not prepare large quantities of this reagent as it is stable for only a few days.

4.3. Boron calibration solutions

(4.3.1) Boron stock solution (100 μg/ml)

Dissolve 0.5719 g of boric acid (H_3BO_3) in water in a 1,000 ml volumetric flask. Make up to volume with water and mix thoroughly. Transfer to a plastic bottle for storage in a refrigerator.

(4.3.2) Boron working solution (10 µg/ml)

Place 50 ml of stock solution (4.3.1) in a 500 ml volumetric flask. Make up to volume with water and mix thoroughly.

5 APPARATUS

5. Spectrometer fitted for molecular absorption with cells having a 10 mm optical path and set to a wavelength of 410 nm.

6 PREPARATION OF THE SOLUTION TO BE ANALYSED

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6.1. Preparation of the boron solution

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

6.2. Preparation of the test solution

Dilute an aliquot portion of extract (6.1) to obtain a boron concentration as specified in 7.2. Two successive dilutions may be necessary. Let D be the dilution factor.

6.3. Preparation of the correction solution.

If the test solution (6.2) is coloured, prepare a corresponding correction solution by placing in a plastic flask 5 ml of test solution (6.2), 5 ml of EDTA buffer solution (4.1) and 5 ml of water and mix thoroughly.

7 PROCEDURE

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7.1. Preparation of the blank solution

Prepare a blank solution by repeating the whole procedure from the extraction stage, omitting only the test sample of fertiliser.

7.2. Preparation of the calibration solutions

Transfer 0, 5, 10, 15, 20 and 25 ml of the working calibration solution (4.3.2) to a series of 100 ml volumetric flasks. Make up to 100 ml with water and mix thoroughly. These solutions contain between 0 and 2.5 µg/ml of boron.

7.3. Colour development

Transfer 5 ml of the calibration solutions (7.2), test solutions (6.2) and blank (7.1) to a series of plastic flasks. Add 5 ml of the EDTA buffer solution (4.1). Add 5 ml of the azomethine-H solution (4.2).

Mix thoroughly and allow the colour to develop in the dark for 2½ to 3 hours.

7.4. Determination

Measure the absorbance of the solutions obtained at 7.3 and if appropriate the correction solution (6.3) against water at a wavelength of 410 nm. Rinse the cells with water before each new reading.

8 EXPRESSION OF RESULTS

8. Plot a calibration curve of the concentration of the calibration solutions (7.2) along the abscissa and the absorbance given by the spectrophotometer (7.4) along the ordinate.

Read off the calibration curve the concentration of boron in the blank (7.1), the concentration of boron in the test solution (6.2) and, if the test solution is coloured, the corrected concentration of the test solution. To calculate the latter, subtract the absorbance of the correction solution (6.3) from the absorbance of the test solution (6.2) and determine the corrected concentration of the test solution. Note the concentration of the test solution (6.2), with or without correction, (x_s) and of the blank (x_b).

The percentage of boron in the fertiliser is given by:

B%=[(xsxb)×V×D]/(M×104)

If Method 25c is used:

B%=[(xsxb)×V×2D]/(M×104)

where:

B is the quantity of boron expressed as a percentage of the fertiliser;

x_s is the concentration μg/ml) in the test solution (6.2) with or without correction;

Xb is the concentration (μg/ml) in the blank (7.1);

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

D is the factor corresponding to 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) and (a_2) are successive aliquot portions and (v_1) and (v_2) are the volumes corresponding to their respective dilutions, the dilution factor is given by:

D=(v1/a1)×(v2/a2).