SCHEDULE 2

METHODS OF ANALYSIS

PART II

9b.

DETERMINATION OF BORON — SPECTROPHOTOMETRIC METHOD

1 SCOPE AND FIELD OF APPLICATION

1. This method is applicable to all fertilisers for levels of boron up to 1,000 mg/kg .

2 PRINCIPLE

2. The sample is ashed in the presence of calcium oxide and the residue is dissolved in hydrochloric acid. The resulting solution is treated with carmine to form a coloured complex with boron, the absorption of which is measured at 625 nm.

3 REAGENTS

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3.1 Calcium oxide.

3.2 Sulphuric acid (d = 1.84 g/ml).

3.3 Carminic acid solution: dissolve 0.025 g carminic acid in sulphuric acid (3.2) and dilute to 100 ml with sulphuric acid (3.2).

3.4 Hydrochloric acid solution 20% (V/V): dilute 20 ml hydrochloric acid (d = 1.18 g/ml) with water to 100 ml.

Boron solution (stock):

(3.5.1) weigh to the nearest 0.001 g, 1.905 g boric acid, dissolve in water and dilute to 1 litre with water.

1 ml of this solution = 0.333 mg boron.

Boron solution (working standard):

(3.5.2) dilute 10 ml of boric acid stock solution (3.5.1) with water to 100 ml.

Transfer 5, 10, 15, 20 and 25 ml respectively into separate 100 ml graduated flasks and dilute to the marks with water. These solutions contain 5, 10, 15, 20 and 25 µg of boron per 3 ml of solution.

3.6 Hydrazine hydrate (approximately 60% W/W solution).

WARNING:

Hydrazine hydrate is toxic and corrosive, causing burns; avoid contact with eyes and skin.

4 APPARATUS

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4.1 Spectrophotometer with 10 mm cells.

5 PREPARATION OF THE SAMPLE

5. See Method 1.

6 PROCEDURE

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Preparation of the solution for analysis

6.1 Weigh to the nearest 0.001 g, 5 g of the prepared sample and place it in a silica dish. Add 1 g calcium oxide (3.1)) moisten with water, mix thoroughly, evaporate the mixture to dryness and then transfer the crucible to a cold muffle furnace. Raise the temperature slowly to 450 ± 10°C and ignite for about 3 hours. Remove the crucible from the furnace, cool and add hydrochloric acid solution (3.4) until the resulting mixture is acid, then add 5 ml hydrochloric acid solution (3.4) in excess. Heat the mixture at 70°C for 15 minutes, cool and filter through a filter paper(**1**) into a suitable graduated flask washing both the dish and the filter with water. Make up to the mark with water and mix. Dilute an aliquot of this solution so that 3 ml contains between 5 and 25 kg of boron.

Blank test

6.2. Carry out a blank test omitting only the sample.

Determination

6.3. Transfer 3 ml of the prepared solution (6.1) to a small conical flask, add cautiously 15 ml sulphuric acid (3.2), swirl the flask and add 10 ml carminic acid solution (3.3). Cool the flask rapidly to room temperature, mix well and allow to stand for 2 hours. Measure the absorbance in the spectrophotometer (4.1) at 625 nm with water as reference. Determine the quantity of boron in the solution by reference to the calibration curve (6.4).

Calibration curve

6.4 Transfer 3 ml of each working standard solution (3.5.2) into a series of small conical flasks and proceed as described in 6.3 commencing at " ... ... add cautiously 15 ml sulphuric acid (3.2) ... ... ". Plot a calibration curve of the absorbance of the solutions against the corresponding quantities of boron, in µg.

7 EXPRESSION OF THE RESULTS

7. The boron content in mg/kg is given by the formula:

$$\frac{A \times V \times F}{3 \times M}$$

where:

A = weight of boron in the 3 ml aliquot taken for colour development after allowing for the blank reading (pg)

V = volume of prepared solution before dilution

(**1**) Whatman 42 or equivalent.

F = factor allowing for dilution under 6.1

M = weight of the sample in grams.

Note

The colour of the boron-carmine complex is affected by nitrate nitrogen. When this form of nitrogen is known to be present in the sample, the "Determination" procedure (6.3) should be modified by the addition of 0.5 ml hydrazine hydrate (3.6) before the addition of the sulphuric acid which should be carried out under conditions of extreme caution because of the (3.2) violent nature of the reaction. (The use of a burette for the addition of the sulphuric acid is recommended).

The hydrazine hydrate (3.6) has no influence on the absorption of the boron-carmine complex and therefore is not added to the standard solutions in the preparation of the calibration curve. However, in order to equalise the volumes of the solutions of both samples and standards, add 0.5 ml water to the latter.