

## SCHEDULE 2

### METHODS OF ANALYSIS

#### PART II

9a.

#### *DETERMINATION OF BORON — TITRIMETRIC METHOD*

##### **1 SCOPE AND FIELD OF APPLICATION**

1. This method is applicable to all fertilisers where the levels of boron are greater than 1,000 mg/kg.

##### **2 PRINCIPLE**

2. The sample is dissolved in acid, the solution treated with lead nitrate in order to remove phosphate, and the borate in the filtrate is titrated potentiometrically in the presence of mannitol.

##### **3 REAGENTS**

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3.1 Calcium oxide.

3.2 Mannitol.

3.3 Sodium carbonate.

3.4 Hydrochloric acid solution 50% (V/V): dilute 50 ml concentrated hydrochloric acid (d = 1.18 g/ml) with water to 100 ml.

3.5 Hydrochloric acid, 0.5 N solution.

3.6 Lead nitrate solution, 10 g per 100 ml.

3.7 Sodium hydroxide, 0.5 N solution.

3.8 Sodium hydroxide, 0.05 N solution, carbonate free.

3.9 Methyl red indicator solution: dissolve 0.1 g of methyl red in 50 ml 95% ethanol, make up to 100 ml with water and filter if necessary.

3.10 Phenolphthalein indicator solution: dissolve 0.25 g phenolphthalein in 1.50 ml 95% alcohol and dilute with water to 250 ml.

##### **4 APPARATUS**

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4.1 pH meter.

##### **5 PREPARATION OF SAMPLE**

5. See Method 1.

*Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.*

## 6 PROCEDURE

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### *Preparation of the solution for analysis*

#### *In the absence of organic matter*

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6.1.—(6.1.1) Weigh to the nearest 0.001 g, 2g of the prepared sample if the boron content is 0.5% or less, or 1 g if the boron content is from 0.5 – 1.0%, and place in a 400 ml beaker. Add 100 ml water, a few drops of phenolphthalein indicator solution (3.10) and sufficient sodium carbonate (3.3) to make the solution slightly alkaline. Boil gently and keep the boiling solution alkaline, adding more sodium carbonate (3.3) as necessary until all the ammonia which may be present has been evolved. Cool the solution and add 12 ml hydrochloric acid solution (3.4).

#### *In the presence of organic matter*

(6.1.2) Weigh to the nearest 0.001 g, 2 g of the prepared sample if the boron content is 0.5% or less, or 1 g if the boron content is from 0.5 – 1.0%, and place it in a silica dish. Add 0.2 g calcium oxide (3.1) for each 1 g of sample, moisten with water, mix thoroughly, evaporate the mixture to dryness and transfer the crucible to a cold muffle furnace. Raise the temperature slowly to  $4.50 \pm 10^\circ\text{C}$  and then ignite for about 3 hours. Remove the crucible from the furnace, cool and moisten the ash with 10 ml of hydrochloric acid solution (3.4). Warm the solution on a steam bath for 15 minutes, covering the dish with a watch glass. Transfer the contents of the dish quantitatively into a 400 ml beaker, add a few drops of phenolphthalein indicator solution (3.10) and dilute to about 120 ml with water.

### *Determination*

6.2 To the prepared solution (6.1.1 or 6.1.2), add 20 ml lead nitrate solution (3.6) for each 12%  $\text{P}_2\text{O}_5$  in the sample if 2 g of the sample has been used; add 10 ml lead nitrate solution for each 12%  $\text{P}_2\text{O}_5$  in the sample if 1 g of the sample has been used. Heat to boiling, remove from the source of heat, and make slightly alkaline by addition of sodium carbonate (3.3). Warm the solution on a steam bath for five minutes, cool and transfer the solution quantitatively into a 200 ml graduated flask. Make up to the mark with water, mix and filter through a 24 cm filter paper(1), rejecting the first 10 — 21 ml of the filtrate.

Transfer 100 ml of the filtrate into a 250 ml beaker, add a few drops of methyl red indicator (3.9) and acidify the solution with 0.5 N hydrochloric acid solution (3.5). Heat almost to boiling, stir vigorously to remove carbon dioxide, keeping the solution acidic, by adding if necessary more 0.5 N hydrochloric acid solution (3.5). Neutralise the solution with 0.5 N sodium hydroxide solution (3.7), and then make just acid by addition of 0.5 N hydrochloric acid solution (3.5). Cover the beaker with a watch glass and boil the solution gently for 5 minutes in order to expel any remaining carbon dioxide.

Cool the solution rapidly and using the pH meter (4.1), adjust the pH of the solution to 6.3 by the addition of 0.05 N sodium hydroxide solution (3.8). Add 10 g mannitol(3.2) and titrate the solution with 0.05 N sodium hydroxide solution to a pH of 6.3. Continue to add further 10 g portions of mannitol(3.2) and to re-adjust the pH to 6.3 until after the final addition of mannitol the pH remains constant at 6.3. The total amount of 0.05 N sodium hydroxide solution used after the first addition of mannitol corresponds to the amount of boron present in the sample solution. Allow a standard value of 0.1 ml 0.05 N sodium hydroxide solution as 'blank' value.

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(1) Whatman 42 or equivalent.

## 7 EXPRESSION OF THE RESULT

7. The percentage boron content of the sample is given by the formula

$$\frac{0.1082 \times (T - 0.1)}{M}$$

where:

T = ml of 0.05 N sodium hydroxide the mannitol

M = weight of the sample in grams.