

## SCHEDULE 2

### METHODS OF ANALYSIS

#### 13b.

#### DETERMINATION OF TOTAL MAGNESIUM —EDTA METHOD

### 1 SCOPE

1. This method is for the determination of total magnesium.

### 2 FIELD OF APPLICATION

2. Exclusively to the fertiliser magnesium ammonium nitrate in Group 1(a) of Section A of the Table in Schedule 1 of the Fertilisers Regulations (Northern Ireland) 1990 in respect of which the indication of total magnesium is required.

### 3 PRINCIPLE

3. Solution of magnesium by boiling a test sample in dilute acid. Titration of calcium and magnesium with EDTA in the presence of eriochrome black-T, followed by titration with EDTA of calcium in the presence of calcein or of calcon carbonic acid. Determination of magnesium by difference.

### 4 REAGENTS

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- 4.1 Magnesium solution, 0.05 M: weigh out 2.016 g of magnesium oxide previously calcined at 600°C for 2 hours, place in a beaker with 100 ml of water and stir in 120 ml of approximately 1 N hydrochloric acid. After dissolution, transfer quantitatively into a 1 litre graduated flask, make up the volume with water and mix. Check the strength of the solution gravimetrically by precipitation as ammonium-magnesium phosphate.

1 ml of the solution should contain 1.216 mg of magnesium (Mg) (= 2.016 mg of magnesium oxide (MgO)).

- 4.2 EDTA solution 0.05 M: dissolve 18.61 g of the dihydrated disodium salt of ethylenediaminetetra-acetic acid in 600-800 ml water contained in a 1 litre beaker. Transfer the solution quantitatively into a 1 litre graduated flask, make up to volume with water and mix. Check this solution with solution (4.1) by taking a sample of 20 ml of the latter and titrating following analytical procedure 7.3.1.

1 ml of the EDTA solution should correspond to 1.216 mg of Mg or 2.016 mg of MgO and to 2.004 mg of Ca or 7.804 mg of CaO.

- 4.3 Calcium solution 0.05 M: weigh out 5.004 g of dry calcium carbonate and place in a beaker with 100 ml of water. Progressively stir in 120 ml of approximately N hydrochloric acid. Bring to the boil in order to drive off the carbon dioxide, cool, transfer quantitatively into a 1 litre graduated flask, make up to volume with water and mix. Check this solution against the EDTA solution (4.2) following analytical procedure 7.3.2.

One ml of this solution should contain 2.004 mg of Ca (= 2.804 mg of CaO) and should correspond to 1 ml of the 0.05 molar EDTA solution.

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4.4 Calcein indicator: carefully mix in a mortar 1 g of calcein with 100 g of sodium chloride. Use 10 mg of this mixture. The indicator changes from green to orange. Titration must be carried out until an orange colour is obtained which is free from green tinges.

4.5 Calcon carbonic acid indicator: dissolve 400 mg of calcon carbonic acid in 100 ml of methanol. Use three drops of this solution. The indicator changes from red to blue. Titration must be carried out until a blue colour is obtained which is free from red tinges.

4.6 Eriochrome black-T indicator: dissolve 300 mg of eriochrome black-T in a mixture of 25 ml of propan-1-ol and 15 ml of triethanolamine. Use three drops of this solution. This indicator turns from red to blue and titration must be carried out until a blue colour is obtained which is free from red tinges. It changes colour only when magnesium is present. If necessary add 0.1 ml of standard solution (4.1).

4.7 Potassium cyanide solution, 2 g per 100 ml.

4.8 Solution of potassium hydroxide and potassium cyanide: dissolve 280 g potassium hydroxide and 66 g potassium cyanide in water, make up the volume to one litre and mix.

4.9 pH 10.5 buffer solution: dissolve 33 g ammonium chloride in 200 ml of water, add 207 ml ammonia solution ( $d = 0.880 \text{ g/ml}$ ) from a freshly opened bottle (or an equivalent amount of diluted ammonia, for example if  $d = 0.91 \text{ g/ml}$ , use 250 ml). Make up the volume to 500 ml with water and mix. Check the pH of this solution regularly.

4.10 Hydrochloric acid solution: 50% (V/V): dilute an appropriate volume of hydrochloric acid ( $d = 1.18 \text{ g/ml}$ ) with an equal volume of water.

4.11 Sodium hydroxide solution, 5 N.

## 5 APPARATUS

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5.1 Magnetic or mechanical stirrer.

5.2 pH meter.

## 6 PREPARATION OF THE SAMPLE

6. See Method 1.

## 7 PROCEDURE

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### *Extraction*

7.1 Weigh to the nearest 0.001 g, 5 g of the prepared sample and place in a 500 ml graduated flask. Add about 200 ml water and 20 ml hydrochloric acid (4.10) and boil for half an hour. Cool, make up to volume with water, mix and filter.

### *Control test*

7.2 Carry out a determination on aliquot parts of solutions (4.1) and (4.3) such that the Ca/Mg ratio is equal to that expected from the sample. For this purpose take (a) ml of standard solution (4.3) and (b - a) ml standard solution (4.1), where (a) and (b) are the numbers of ml EDTA solution used in the two titrations when analysing the sample. This procedure is correct only if the standard solutions

of EDTA, calcium and magnesium are exactly equivalent. If this is not the case, it is necessary to make the appropriate corrections.

#### *Determination*

##### *Titration in the presence of eriochrome black-T*

##### *Titration in the presence of eriochrome black-T*

7.3.—(7.3.1) Transfer by pipette 50 ml of the solution to be analysed into a 300 ml beaker. Neutralise the excess acid with the 5 N sodium hydroxide solution (4.11) using the pH meter (5.2). Dilute with water to 100 ml. Add 5 ml buffer solution (4.9). The pH measured by the meter must be  $10.5 \pm 0.1$ . Add 2 ml potassium cyanide solution (4.7) and three drops eriochrome black-T indicator (4.6). Titrate with the EDTA solution (4.2); stirring gently with the stirrer (5.1). Let “b” be the number of ml of 0.05 molar EDTA solution.

#### *Note:*

For titration with eriochrome black-T, the titration must not exceed 25 ml of EDTA otherwise the volume of the aliquot part must be reduced.

##### *Titration in the presence of calcein or of calcon carbonic acid*

(7.3.2) Place an aliquot part of the solution to be analysed equal to that taken for the above titration in a 300 ml beaker. Neutralise the excess acid with 5 N sodium hydroxide solution (4.11) using the pH meter (5.2).

Dilute with water to about 100 ml. Add 10 ml potassium hydroxide — potassium cyanide solution (4.8) and the indicator (4.4) or (4.5). Stir gently and titrate with the EDTA solution. Let “a” be the number of ml of 0.05 molar EDTA solution.

## **8 EXPRESSION OF THE RESULTS**

$$\% \text{ MgO} = \frac{(b - a) \times 0.2016}{M}$$

$$\% \text{ Mg} = \frac{(b - a) \times 0.1216}{M}$$

M = weight of the sample, expressed in grams, present in the aliquot part.