SCHEDULE 2

## METHODS OF ANALYSIS

2b.

*DETERMINATION OF WATER-SOLUBLE MAGNESIUM —EDTA METHOD*

### 1 SCOPE

1. This method is for the determination of water-soluble magnesium.

### 2 FIELD OF APPLICATION

2. Exclusively to straight fertiliser in Groups l(a) and 3(a) of Section A of the Table in Schedule 1 of the Fertiliser Regulations (Northern Ireland) 1990 in respect of which the indication of water-soluble magnesium, expressed as magnesium oxide, is required.

### 3 PRINCIPLE

3. Solution of magnesium by boiling a test sample in water. Titration with EDTA of calcium and magnesium 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 magnesium oxide, previously calcined at 600°C for 2 hours, place in a beaker with 100 ml water and stir in 120 ml of approximately 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 magnesium ammonium phosphate.

1 ml of the solution should contain 1.216 mg of Mg (= 2.016 mg of 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 to a 1 litre graduated flask, make up to volume with water and mix. Check this solution (4.1) by taking a sample of 20 ml of the latter and titrating as described under 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 2.804 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 water. Progressively stir in 120 ml 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.

4.4 Calcein indicator: carefully mix in a mortar 1 ,g calcein with 100 g 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 calcon carbonic acid in 100 ml 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 eriochrome black-T in a mixture of 25 ml propan-l-01 and 15 ml 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 1 litre and mix.

4.9 pH 10 buffer solution: dissolve 33 g ammonium chloride in 200 ml water, add 207 ml ammonia solution (d = 0.880 g/ml)*from a freshly opened bottle,*(or an equivalent amount of diluted ammonia, for example if d = 0.91 g/ml, use 250 ml). Make up the volume to 500 ml with water and mix. Check the pH of this solution regularly.

## 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 300 ml water and boil for half an hour. Cool, make up the volume, 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) Place an aliquot part of the solution to be analysed (see the Table) in a 300 ml beaker and dilute with water to about 100 ml. Add 5 ml buffer solution (4.9). The pH measured by

the meter (5.2) must be 10.5 ± 0.1. Add 2 ml potassium cyanide solution (4.7) and 3 drops of the eriochrome black-T indicator (4.6). Stir gently and titrate with the EDTA solution (4.2). 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 beaker. 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 (4.2). Let “a” be the number of ml of 0.05 molar EDTA solution.

## 8 EXPRESSION OF THE RESULTS

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

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

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

TABLE FOR METHOD 12

| *Type of fertiliser* | *Aliquot part to be taken as sample for each titration* | *Quantity of sample present in one aliquot part* |
|---|---|---|
| Nitrate of calcium and of magnesium | 20 ml | 0.200 g |
| Magnesium ammonium sulphate-nitrate | 50 ml | 0.500 g |
| Crude potassium salts | 25 ml | 0.250 g |
| Potassium magnesium chloride | 25 ml | 0.250 g |
| Sulphate of potassium and magnesium | 25 ml | 0.250 g |