SCHEDULE 2

METHODS OF ANALYSIS

12a.

DETERMINATION OF WATER-SOLUBLE MAGNESIUM — ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD

1 SCOPE

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

2 FIELD OF APPLICATION

2. Exclusively to fertilisers in Groups I(a) and 3(a) of Section A of the Table in Schedule 1 of the Fertilisers Regulations (Northern Ireland) 1990, in respect of which the declaration of water-soluble magnesium is required.

3 PRINCIPLE

3. Solution of magnesium by boiling the test sample in water, and determination by atomic absorption spectrophotometry.

4 REAGENTS

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4.1 Hydrochloric acid, N solution (approximately).

4.2 Hydrochloric acid, 0.5 N solution.

4.3 Magnesium standard solution: dissolve 1.013 g magnesium sulphate ($MgSO_4.7H_2O$) in 0.5 N hydrochloric acid solution (4.2) and dilute to 100 ml with this acid.

1 ml of this solution contains 1 mg of magnesium (Mg).

or

weigh out 1.658 g of magnesium oxide, previously calcined at 600°C for 2 hours, place in a beaker with 100 ml of water and 120 ml of approximately N hydrochloric acid (4.1). After dissolution, transfer quantitatively into a one litre graduated flask, make up the volume with water and mix.

1 ml of this solution contains 1 mg of magnesium (Mg).

4.4 Strontium chloride solution: dissolve 15 g strontium chloride ($SrCl_2.6H_2O$) in 0.5 N hydrochloric acid solution (4.2) and dilute to 100 ml with the same solvent.

5 APPARATUS

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5.1 Atomic absorption spectrophotometer with a magnesium lamp (285.2 nm).

6 PREPARATION OF 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. Allow to cool, dilute to the mark with water, mix and filter.

Preparation of the sample solution

7.2.—(7.2.1) If the fertiliser has a declared magnesium oxide (MgO) content greater than 10%, transfer by pipette 25 ml of the filtrate (7.1) into a 100 ml graduated flask, make up to the mark with water and mix.

(7.2.2) Transfer by pipette 10 ml of the filtrate (7.1) or the diluted filtrate (7.2.1) into a 200 ml graduated flask and make up to the mark with 0.5 N hydrochloric acid solution (4.2).

(7.2.3) Dilute solution (7.2.2) with 0.5 N hydrochloric acid solution (4.2) to a concentration within the working range of the spectrophotometer.

The final solution must contain 10% V/V of the strontium chloride solution (4.4).

Blank solution

7.3 Prepare a blank solution from which only the sample has been omitted.

Standard solutions for calibration

7.4 By diluting the standard solution (4.3) with 0.5 N hydrochloric acid solution (4.2), prepare at least 5 standard solutions of increasing concentration corresponding to the optimal measuring range of the spectrophotometer. The final solutions must contain 10% V/V of the strontium chloride solution (4.4).

Measurement

7.5 Set up the spectrophotometer (5.1), at a wavelength of 285.2 nm using an oxidising air-acetylene flame. Spray successively, in triplicate, the standard solutions (7.4), the sample solution (7.2) and the blank solution (7.3), washing the instrument through with distilled water between each spraying. Plot the calibration curve using the mean absorbances as the ordinates and the corresponding concentrations of magnesium in u/ml as the abscissae. Determine the concentration of magnesium in the sample and blank by reference to the calibration curve.

8 EXPRESSION OF THE RESULTS

8. Calculate the quantity of magnesium (Mg) or magnesium oxide (MgO) (conversion factor Mg to MgO = 1.66) in the sample taking into consideration the blank. Express the result as a percentage of the sample.