SCHEDULE 2

METHODS OF ANALYSIS

PART I

21.

DETERMINATION OF MAGNESIUM BY ATOMIC ABSORPTION SPECTROMETRY

1 SCOPE

1. This method is for the determination of magnesium in fertiliser extracts.

2 FIELD OF APPLICATION

2. This method applies to all fertiliser extracts obtained by methods 15 and 17, for which a declaration of the total magnesium and/or water-soluble magnesium is required, with the exception of kieserite, magnesium sulfate, magnesium chloride solution and kieserite with potassium sulfate to which method 22 applies.

3 PRINCIPLE

3. Determination of magnesium by atomic absorption spectrometry after appropriate dilution of the extract.

4 REAGENTS

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4.1 Hydrochloric acid, 1 M solution.

4.2 Hydrochloric acid, 0.5 M solution.

4.3 Standard solution of magnesium, 1.00 mg per ml.

(4.3.1) Dissolve 1.013 g of magnesium sulfate ($MgSO_4.7H_2O$) in 0.5 M hydrochloric acid solution (4.2).

or:

(4.3.2) weigh out 1.658 g of magnesium oxide (MgO), previously ashed to remove all traces of carbonate. Place in a beaker with 100 ml of water and 120 ml of 1 M hydrochloric acid (4.1). When it has dissolved, transfer quantitatively into a 1 litre graduated flask. Make up to the volume and mix.

or:

(4.3.3) Commercial standard solution.

The laboratory is responsible for testing such solutions.

4.4 Strontium chloride solution.

Dissolve 75 g of strontium chloride ($SrCl_2.6H_2O$) in the hydrochloric acid solution (4.2) and make up to 500 ml with the same acid solution.

5 APPARATUS

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5.1 Spectrometer fitted for atomic absorption, with a magnesium lamp, set at 285.2 nm.

5.2 Air-acetylene flame.

6 PREPARATION OF THE SOLUTION TO BE ANALYSED

6. See methods 15 and 17.

7 PROCEDURE

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7.1 If the fertiliser has a declared magnesium (Mg) content of more than 6% (i.e. 10% as MgO), take 25 ml (V_1) of the extraction solution (6). Transfer into a 100 ml graduated flask, and make up to volume with water and mix. The solution factor is $D_1 = 100/V_1$.

7.2 Using a pipette, take 10 ml of the extraction solution (6) or the solution (7.1). Transfer into a 200 ml graduated flask. Make up to the volume with the 0.5 M hydrochloric acid solution (4.2) and mix. The dilution factor is 200/10.

7.3 Dilute this solution (7.2) with the 0.5 M hydrochloric acid solution (4.2) so as to obtain a concentration in the optimum working field of spectrometer (5.1). V_2 is the volume of the sample in 100 ml. The dilution factor is $D_2 = 100/V_2$.

7.4 Preparation of blank solution.

Prepare a blank solution by repeating the whole procedure from the extraction (method 15 or 17), omitting only the test sample of fertiliser.

7.5 Preparation of calibration solutions.

By diluting the standard solution (4.3) with 0.5 M hydrochloric acid, prepare at least five calibration solutions in increasing concentration within the optimum measuring range of the apparatus (5.1).

These solutions should contain 10% v/v of the strontium chloride solution (4.4).

7.6 Measurement.

Set up the spectrometer (5.1) at a wavelength of 285.2 nm.

Spray, successively, the calibration solutions (7.5), the sample solution (7.3) and the blank solution (7.4), washing the instrument through with the solution to be measured next. Repeat this operation three times. Plot the calibration curve using the mean absorbances of each of the calibration solutions (7.5) as the ordinates and the corresponding concentration of magnesium in µg/ml as the abscissae. Determine the concentration of magnesium in the sample (7.3), x_s and blank (7.4), x_b, by reference to the calibration curve.

8 EXPRESSION OF RESULTS

8. Calculate the amount of magnesium (Mg) or magnesium oxide (MgO) in the sample by reference to the calibration solutions and taking into consideration the blank.

The percentage of magnesium (Mg) in the fertiliser is equal to:

Mg(%)=(xsxb)×D1×(200/10)×D2×500×1001,000×1,000M

Where:

x_s = the concentration of the solution as calculated from the calibration curve, in µg/ml.

x_b = the concentration of the blank solution as calculated from the calibration curve, in µg/ml.

D_1 = the dilution factor if the solution is diluted (7.1). It is equal to four if 25 millilitres are taken. It is equal to one when the solution is not diluted.

D_2 = the dilution factor in 7.3.

M = the mass of the test sample taken for the extraction.

MgO(%)=Mg(%)0.6