

SCHEDULE 2  
METHODS OF ANALYSIS

PART II

8.

*DETERMINATION OF TOTAL MAGNESIUM*

*EXTRACTION OF TOTAL MAGNESIUM*

(8.1)

**SCOPE AND FIELD OF APPLICATION**

1.—(1.1) This method is applicable to all fertilisers.

**PRINCIPLE**

2.—(2.1) Solubilisation by boiling in dilute hydrochloric acid.

**REAGENTS**

3.—(3.1) Diluted hydrochloric acid:  
one volume of hydrochloric acid (d=1.18) plus one of water.

**APPARATUS**

4.—(4.1) Electric hot plate with adjustable temperature.

**PREPARATION OF THE SAMPLE**

5.—(5.1) See method 1.

**PROCEDURE**

*Test sample*

*Test sample*

6.—(6.1) Magnesium is extracted from a test sample of five grams weighed to within one milligram.

*Preparation of the solution*

(6.2) Add approximately 400 millilitres of water and, taking care when the sample contains a significant quantity of carbonates, 50 millilitres of dilute hydrochloric acid (4. 1) a small amount at a time. Bring to the boil and maintain for 30 minutes. Allow to cool, stirring occasionally. Decant quantitatively into a 500 millilitre graduated flask. Make up to volume with water and mix. Pass through a dry filter into a dry container, discarding the initial portion. The extract must be completely transparent. Stopper if the filtrate is not used immediately.

*DETERMINATION OF MAGNESIUM BY ATOMIC ABSORPTION SPECTRO-PHOTOMETRY*

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(8.2)

## SCOPE AND FIELD OF APPLICATION

1. This method applies to all fertiliser extracts obtained by method 8.1.

## PRINCIPLE

2.—(2.1) Determination of magnesium by atomic absorption spectrophotometry after appropriate dilution of the extract.

## REAGENTS

3.—(3.1) Hydrochloric acid, I M solution.

(3.2) Hydrochloric acid, 0.5 M solution.

(3.3) Standard solution of magnesium, 1.00 mg/ml.

(3.3.1) Dissolve 1.013 grams of magnesium sulphate ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) in the 0.5 M hydro-chloric acid solution (3.2).

(3.3.2) Weigh out 1.658 grams of magnesium oxide ( $\text{MgO}$ ), previously calcined to remove all traces of carbonation. Place in a beaker with 100 ml of water and 120 ml of I M hydrochloric acid (3. 1). When it has dissolved, decant quantitatively into a 1,000 ml graduated flask. Make up the volume by adding and mix

or

*Commercial standard solution*

(3.3.3) The laboratory is responsible for testing such solutions.

*Strontium chloride solution*

(3.4) Dissolve 75 grams of strontium chloride ( $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$ ) in a hydrochloric acid solution (3.2) and make up to 500 ml with the same acid solution.

## APPARATUS

4.—(4.1) Spectrophotometer fitted for atomic absorption, with a magnesium lamp, set at 285.2 nm.

(4.2) Air-acetylene flame.

## PREPARATION OF THE SAMPLE

5.—(5.1) See Method 8.1.

## PROCEDURE

6.—(6.1) If the fertiliser has a declared magnesium (Mg) content of more than 6% (ie 10% as  $\text{MgO}$ ), take 25 ml ( $V_1$ ) of the extraction solution (5). Transfer into a 100 ml graduated flask, and make up to volume with water and mix. The dilution factor is  $D_1=100/V_1$ .

(6.2) Using a pipette, take 10 millilitres of the extraction solution (5) or the solution (6.1). Transfer into a 200 ml graduated flask. Make up to volume with the 0.5 M hydrochloric acid solution (3.2) and mix. The dilution factor is 200/10.

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(6.3) Dilute this solution (6.2) with the 0.5 M hydrochloric acid solution (3.2) so as to obtain a concentration in the optimum working field of the spectrophotometer (4.1).  $V_2$  is the volume of the sample in 100 ml. The dilution factor is  $D_2 = 100/V_2$ .

The final solution should contain 10% v/v of the strontium chloride solution (3.4).

#### *Preparation of the blank solution*

(6.4) Prepare a blank solution by repeating the whole procedure from the extraction (method 8.1), omitting only the test sample of fertiliser.

#### *Preparation of calibration solutions*

(6.5) By diluting the standard solution (3.3) with the 0.5 M hydrochloric acid, prepare at least five calibration solutions of increasing concentration within the optimum measuring range of the apparatus (4.1).

These solutions should contain 10% v/v of the strontium chloride solution (3.4).

#### *Measurement*

(6.6) Set up the spectrophotometer (4.1) at a wavelength of 285.2 nm.

Spray, successively, the calibration solutions (6.5), the sample solution (6.3) and the blank solution (6.4), 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 calibrations (6.5) as the ordinates and the corresponding concentration of magnesium in  $\mu\text{ml}$  as the abscissae. Determine the concentration of magnesium in the sample (6.3),  $X_s$ , and blank (6.4),  $X_b$ , by reference to the calibration curve.

## **EXPRESSION OF RESULTS**

7. 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:

$$\text{Mg (\%)} = \frac{(X_s - X_b) D_1 (200/10) D_2 500.100}{1,000.1,000 \text{ M}}$$

where:

$X_s$  = the concentration of the solution to be analysed recorded on the calibration curve, in  $\mu\text{ml}$ .

$X_b$  = the concentration of the blank solution as recorded on the calibration curve, in  $\mu\text{ml}$ .

$D_1$  = the dilution factor when the solution is diluted (6. 1).

It is equal to four if 25 ml are taken.

It is equal to one when the solution is not diluted.

$D_2$  = the dilution factor in 6.3.

M = the mass of the test sample at the time of extraction.

$\text{MgO(\%)} = \text{Mg(\%)} / 0.6$ .