SCHEDULE 2

METHODS OF ANALYSIS

PART I

24.

DETERMINATION OF THE SODIUM EXTRACTED

1 SCOPE

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

2 FIELD OF APPLICATION

2. This method applies to fertilisers for which a declaration of sodium is required.

3 PRINCIPLE

3. Following suitable dilution of the extract obtained via method 15 and/or 17 the sodium content of the solution is determined by flame-emission spectrometry.

4 REAGENTS

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4.1 Diluted hydrochloric acid:

One volume of hydrochloric acid (ρ = 1.18 g.ml) plus one volume of water.

4.2 Aluminium nitrate $Al(NO_3)_3.9H_2O$.

4.3 Caesium chloride, CsCl.

4.4 Anhydrous sodium chloride, NaCl.

4.5 Caesium chloride and aluminium nitrate solution.

Dissolve in water 50 g of caesium chloride (4.3) and 250 g of aluminium nitrate (4.2) in a 1 litre graduated flask. Make up to volume with water and mix.

4.6 Standard sodium solution of 1 mg/ml of Na.

Dissolve in water 2.542 g of sodium chloride (4.4) in a 1 litre graduated flask. Add 10 ml of hydrochloric acid (4.1). Make up to volume with water and mix.

5 APPARATUS

5. Spectrometer equipped for flame emission, set at 589.3 nm.

6 CALIBRATION SOLUTIONS

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6.1 Pipette 10 ml of standard solution (4.6) into a 250 ml graduated flask. Make up to volume and mix. Concentration of solution: 40 µg/ml of Na.

6.2 Using a burette place 0, 5, 10, 15, 20, 25 ml of the intermediate solution (6.1) in 100 ml graduated flasks. Add 10 ml of the solution (4.5). Make up to volume and mix. Concentration of solutions: 0, 2, 4, 6, 8, 10 µg/ml of Na.

7 PREPARATION OF SOLUTIONS TO BE MEASURED

7. Depending upon the expected sodium content of the extraction solution as in method 15 or 17 (5 g of fertiliser in 500 ml), carry out the dilutions in accordance with the following table:

		Intermediate dilution		*Final dilution*		
Na2O (%)	*Na (%)*	*Sample (ml) (v2)*	*Dilution to ml (v3)*	*Sample (ml) (v4)*	*Dilution to ml*	*Degree of dilution*
3 – 5	2.2 – 3.7	10	50	10	100	50
5 – 10	3.7 – 7.4	10	100	10	100	100
10 – 20	7.4 – 15	10	100	5	100	200
20 – 38	15 – 28	5	100	5	100	400

Make up the intermediate dilution with water. For the final dilution add 10 ml of the solution (4.5) to the 100 ml graduated flask.

For a test sample of 1 g multiply the volume of the final dilution (v_4) by five.

8 DETERMINATION

8. Prepare the spectrometer (5) for the measurements at 589.3nm. Calibrate the instrument by measuring the response of the calibration solutions (6.2). Then adjust the sensitivity of the instrument to use its entire scale when the most concentrated calibration solution is used. Then measure the response of the sample solution to be analysed (7). Repeat this operation twice.

9 CALCULATION OF RESULTS

9. Draw a calibration curve by plotting the average response for each calibration solution along the ordinate and the corresponding concentrations, expressed in µg per ml on the abscissa. Determine from this the sodium concentration of the test solution. Calculate the quantity of sodium from the standard solutions taking account of the levels of dilution. Express the results as a percentage of the sample.

The percentage sodium (Na) content of the fertiliser is as follows:

Na(%)=X×v3v4×v1v2×10-2m

Na2O(%)=Na(%)×1.348

Where:

X = the concentration of the solution introduced into the spectrometer in µg/ml,

v_1 = the volume of the extraction solution in millilitres,

v_2 = the aliquot volume in the intermediate dilution in millilitres,

v_3 = the volume of intermediate dilution in millilitres,

v_4 = the aliquot volume in ml of the final dilution (in 100 millilitres),

m = the mass of the test sample in grams.