

SCHEDULE 2

METHODS OF ANALYSIS

PART II

7b.

DETERMINATION OF POTASSIUM — FLAME PHOTOMETRIC METHOD

1 SCOPE AND FIELD OF APPLICATION

1. This method is applicable to fertilisers in Groups 3(b), 3(c), 3(d) and 4(c) of Section A and Group 5 of Section B of the Table in Schedule 1 of the Fertilisers Regulations (Northern Ireland) 1990(1) in respect of which an indication of total potassium is required.

2 PRINCIPLE

2. The sample is ashed and dissolved in dilute hydrochloric acid or, if it contains no organic substances, it is dissolved directly in dilute hydrochloric acid. The solution is diluted and the potassium content of the extract is determined by flame photometry.

3 REAGENTS

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3.1 Ammonia solution (30% V/V): dilute 30 ml concentrated ammonia solution ($d = 0.88$ g/ml) to 100 ml.

3.2 Ammonium oxalate solution: saturated aqueous solution.

3.3 Hydrochloric acid ($d = 1.18$ g/ml).

3.4 Potassium dihydrogen phosphate: dried for one hour at 105°C .

3.5 Potassium solution (stock): dissolve 3.4807 g potassium dihydrogen phosphate (3.4) in water and dilute to 1 litre.

3.6 Potassium solution (dilute): dilute 50 ml stock solution (3.5) to 1 litre with water. 1 ml contains 50 μg potassium (K).

4 APPARATUS

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4.1 Flame photometer.

5 PREPARATION OF SAMPLE

5. See Method 1.

6 PROCEDURE

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(1) [S.R. 1990 No. 286](#)

Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.

Preparation of the solution for analysis

Fertilisers containing little or no organic matter

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6.1.—(6.1.1) Weigh to the nearest 0.001 g, 2.5 g of the prepared sample and transfer to a 400 ml beaker. Add 50 ml water and 5 ml hydrochloric acid (3.3) and evaporate to dryness on a steam bath. Add to the residue 125 ml water and 50 ml ammonium oxalate solution (3.2) and boil for 30 minutes. If necessary, a small quantity of potassium-free antifoaming agent may be added. Cool the mixture, add a slight excess of ammonia solution (3.1) and allow to cool. Transfer to a 250 ml graduated flask, dilute to the mark with water, mix and filter through a dry paper.

Fertilisers containing organic matter

(6.1.2) Weigh to the nearest 0.01 g, 10 g of the prepared sample into a suitable crucible and place in a cold muffle furnace. Gradually raise the temperature to about 475°C (not to exceed 500°C). Maintain at this temperature for at least 16 hours and then open the furnace and allow the crucible to cool. Grind the residue to eliminate any lumps, add 50 ml water and 10 ml hydrochloric acid (3.3) and evaporate to dryness on a steam bath. Add to the residue 125 ml water and 50 ml ammonium oxalate solution (3.2) and boil for 30 minutes. Cool the mixture, add a slight excess of ammonia solution (3.1) and allow to cool. Transfer to a 500 ml graduated flask, dilute to the mark with water, mix and filter through a dry paper.

Blank solution

6.2 Prepare a blank solution from which only the sample has been omitted and allow for this in the calculation of the final results.

Determination

Preparation of sample and blank test solutions

Preparation of sample and blank test solutions

6.3.—(6.3.1) Dilute sample solutions (6.1.1 or 6.1.2) and the blank solution (6.2) to a concentration within the optimal measuring range of the flame photometer.

Preparation of the calibration solutions

(6.3.2) By diluting the standard solution (3.6), prepare at least five standard solutions of increasing concentration corresponding to the optimal measuring range of the flame photometer.

Measurement

6.4 Set the flame photometer to measure the potassium emission according to the manufacturer's instructions. Spray successively, in triplicate, the standard solutions (6.3.2), the sample solution and the blank solution (6.3.1), washing the instrument through with distilled water between each spraying. Plot the calibration curve using the median emissions as the ordinates and the corresponding concentrations of potassium in µg/ml as the abscissae. Determine the concentration of potassium in the final sample solution by reference to the calibration curve.

The concentration of potassium in the final solution may be confirmed as follows:

prepare two further dilutions of the standard potassium solution to contain respectively 1 mg/litre more and 1 mg/litre less than the estimated potassium content of the diluted solution of the sample.

Successively spray the low standard solution, the diluted solution of the sample and the high standard solution. Take the median result of each of the three readings and calculate the potassium content of the sample solution.

7 EXPRESSION OF THE RESULTS

7. Calculate the percentage potassium content of the sample as K taking into account the weight of the test sample and the dilutions carried out in the course of the analysis. (Conversion factor K to $K_2O = 1.204$).