

SCHEDULE 2

METHODS OF ANALYSIS

PART II

6.

DETERMINATION OF EXTRACTED PHOSPHORUS — SPECTROPHOTOMETRIC METHOD

1 SCOPE AND FIELD OF APPLICATION

1. This method is for the determination of the phosphorus extracted in Methods 5a and 5b.

2 PRINCIPLE

2. An acidic solution of the extracted phosphorus is treated with molybdo-vanadate reagent and the absorbance of the yellow solution is measured at 430 nm.

3 REAGENTS

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- 3.1 Nitric acid ($d = 1.42$ g/ml).

3.2 Molybdovanadate reagent: dissolve separately 20 g ammonium molybdate and 0.47 g ammonium vanadate in water, mix, acidify with 140 ml nitric acid (3.1) and dilute to 1 litre with water.

3.3 Phosphorus standard solution: dissolve 4.387 g potassium dihydrogen phosphate, previously dried at 105°C for 1 hour, in water and dilute to 1 litre.

1 ml of this solution contains 1 mg phosphorus (P) or 2.29 mg phosphorus pentoxide (P_2O_5).

- 3.4 Sodium hydroxide, approximately 5 N solution.

5 APPARATUS

5. Spectrophotometer with 10 mm cells.

5 PROCEDURE

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Determination

For Total Phosphorus

For Total Phosphorus

5.1.—(5.1.1) Dilute, if necessary, the prepared extract to obtain a phosphorus concentration of about 20 µg/ml. Transfer 10 ml of this solution to a glass stoppered test tube, add 10 ml freshly prepared molybdovanadate reagent (3.2) and mix. Allow to stand for 10 minutes at 20°C and then measure the absorbance of the solution at 430 nm against a freshly prepared reference solution made by adding 10 ml molybdovanadate reagent (3.2) to 10 ml water.

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For Water Soluble Phosphorus and Citric Acid Soluble Phosphorus

(5.1.2) Dilute, if necessary, the prepared extract to obtain a phosphorus concentration of about 80 µg/ml. Transfer 25 ml of this solution to a 100 ml conical flask, add 5 ml nitric acid (3.1) and boil gently for 30 minutes. Cool the solution and neutralise with sodium hydroxide solution (3.4). Cool the solution to 20°C, transfer quantitatively to a 100 ml graduated flask and make up to the mark with water. Transfer 10 ml of this solution to a glass stoppered test tube, add 10 ml freshly prepared molybdovanadate reagent (3.2) and mix. Proceed as described in 5.1.1 from “Allow to stand ...”.

Calibration

5.2 From the standard solution (3.3) prepare a series of solutions containing respectively 5, 10, 20, 30 and 40 µg/ml of phosphorus (P).

Transfer 10 ml of each solution into glass-stoppered test tubes, add 10 ml molybdovanadate reagent (3.2), mix and proceed as described in 5.1.1 from “Allow to stand ...”.

Construct a graph relating the absorbance to the amount of phosphorus present. The calibration curve should be prepared at the same time as the determination is carried out.

6 EXPRESSION OF THE RESULTS

6. Determine the amount of phosphorus in the sample by reference to the calibration curve. Express the result in terms of percentage phosphorus (P) or percentage phosphorus pentoxide (P₂O₅) of the sample:

$$\text{mg phosphorus (P)} \times 2.29 = \text{mg phosphorus pentoxide (P}_2\text{O}_5\text{)}$$