SCHEDULE 2

METHODS OF ANALYSIS

PART II

4.

DETERMINATION OF UREA

SCOPE AND FIELD OF APPLICATION

1. This method is applicable to fertilisers in Group 1(c) of Section A, Group 5 and 6 of Section B and Group 1(d) of Section C of the Table in Schedule 1 of the Fertilisers Regulations 1991.

PRINCIPLE

2. The sample is suspended in acid solution with a clarifying agent and filtered. The urea content of the filtrate is determined after the addition of 4-dimethylaminobenzaldehyde (4-DMAB) by measuring the absorbance at 435 nm.

REAGENTS

3

3.1 Activated charcoal.

3.2 Carrez solution I:

dissolve 21.9 g zinc acetate dihydrate in water, add 3 ml glacial acetic acid and dilute to 100 ml with water.

3.3 Carrez solution II: 10.6 g potassium ferrocyanide per 100 ml.

3.4 Hydrochloric acid solution, 0.02 M.

3.5 Sodium acetate solution: 136 g sodium acetate trihydrate per litre.

3.6 4-dimethylaminobenzaldehyde solution:

dissolve 1.6 g of 4-dimethylaminobenzaldehyde (4-DMAB) in 100 ml 96% ethanol and add 10 ml of hydrochloric acid ($\rho = 1.18$ g/ml)

3.7 Urea standard solution: 1.0 g per 100 ml (1 ml of this solution = 10 mg urea).

APPARATUS

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4.1 Mechanical shaker.

4.2 Spectrometer with 10 mm cells.

PREPARATION OF SAMPLE

5. See Method 1.

PROCEDURE

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Preparation of the solution for analysis

6.1 Weigh to the nearest 0.001 g, 2 g of the prepared sample, or a suitable amount expected to contain between 50 and 500 mg of urea and transfer it to a 500 ml graduated flask. Add 150 ml 0.02 M hydrochloric acid solution (3.4), shake for 30 minutes using a mechanical shaker (4.1) then add 10 ml sodium acetate solution (3.5) and mix well. Add 2 g activated charcoal (3.1) to the flask, shake well and allow to stand for a further 15 minutes. Add 5 ml Carrez solution I (3.2), followed by 5 ml Carrez solution II (3.3), mixing well between additions. Dilute to volume with water and mix well. Filter a portion of the solution through a dry filter paper into a clean dry 250 ml beaker.

Determination

6.2 Transfer 10 ml of the filtrate (6.1) to a 50 ml graduated flask, add 10 ml 4-DMAB solution (3.6), dilute to 50 ml with water, mix well and allow to stand for 10 minutes. Measure the absorbance of the solution at 435 nm, in a 10 mm cell against a reference solution prepared by diluting 10 ml 4-DMAB solution (3.6) to 50 ml with water.

Calibration curve

6.3 Transfer amounts of standard urea solution (3.7) corresponding to 50, 100, 150 and 250 mg of urea into a series of 250 ml graduated flask; add 75 ml 0.02 M hydrochloric acid solution (3.4) and proceed as described above (6.1) commencing at ". . . shake for 30 minutes . . .". Measure the absorbance of the solutions and construct a calibration graph relating the absorbances to the amounts of urea present.

EXPRESSION OF RESULTS

7. Determine the amount of urea in the sample by reference to the calibration graph. Express the result in terms of percentage ureic nitrogen of the sample:

(mgurea×0.4665=mgureicnitrogen).