

## SCHEDULE 2

### METHODS OF ANALYSIS

#### PART II

##### 4.

#### DETERMINATION OF UREA

### 1 SCOPE AND FIELD OF APPLICATION

1. This method is applicable to fertilisers in Group I(c) of Section A, Group 5 of Section B and Group I(d) of Section C of the Table in Schedule 1 of the Fertilisers Regulations (Northern Ireland) 1990.

### 2 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-dimethylamino-benzaldehyde (4-DMAB) by measuring the absorbance at 435 nm.

### 3 REAGENTS

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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 N.

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

3.6 4-dimethylamino-benzaldehyde solution:

dissolve 1.6 g of 4-dimethylamino-benzaldehyde (4-DMAB) in 100 ml 96% ethanol and add 10 ml of hydrochloric acid ( $d = 1.18 \text{ g/ml}$ ).

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

### 4 APPARATUS

4. Spectrophotometer with 10 mm cells.

### 5 PREPARATION OF SAMPLE

5. See Method 1.

### 6 PROCEDURE

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**Status:** This is the original version (as it was originally made).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*

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 N hydrochloric acid solution (3.4), shake for 30 minutes 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 2.50 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 flasks; add 75 ml 0.02 N 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.

**7 EXPRESSION OF THE 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:

$$(\text{mg urea} \times 0.4665 = \text{mg ureic nitrogen}).$$