

SCHEDULE 2

METHODS OF ANALYSIS

PART I

5.

DETERMINATION OF TOTAL NITROGEN IN UREA

SCOPE

1. This method is for the determination of total nitrogen in urea.

FIELD OF APPLICATION

2. The method is applicable exclusively to urea fertilisers which are nitrate free.

PRINCIPLE

3. Urea is transformed quantitatively into ammonia by boiling in the presence of sulfuric acid. The ammonia thus obtained is distilled from an alkaline medium and collected in an excess of standard sulfuric acid. The excess acid is titrated by means of a standard alkaline solution.

4 REAGENTS

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4.1	Sulfuric acid, concentrated (p = 1.84 g/ml).
4.2	Sodium hydroxide solution, 30 g per 100 ml, ammonia free.
4.3	Sulfuric acid, 0.05 M solution for variant (a) (see page 16)
4.4	Sodium or potassium hydroxide, 0.1 M solution, carbonate free
4.5	Sulfuric acid, 0.1 M solution for variant (b) (see page 16)
4.6	Sodium or potassium hydroxide, 0.2 M solution, carbonate free
4.7	Sulfuric acid, 0.25 M solution for variant (c) (see page 16)
4.8	Sodium or potassium hydroxide, 0.5 M solution, carbonate free
4.9	Indicator solutions: 4.9.1 Mixed indicator:

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	<p>Solution A: dissolve 1 g methyl red in 37 ml 0.1 M sodium hydroxide solution and make up to 1 litre with water.</p> <p>Solution B: dissolve 1 g methylene blue in water and make up to 1 litre. Mix 1 volume of A with 2 volumes of B.</p> <p>This indicator is violet in acid solution, grey in neutral solution and green in alkaline solution. Use 0.5 ml (10 drops).</p> <p>4.9.2 Methyl red indicator:</p> <p>Dissolve 0.1 g methyl red in 50 ml 95% ethanol, make up to 100 ml with water. Filter if necessary. This indicator (4 to 5 drops) may be used instead of the preceding one.</p>
4.10	<p>Anti-bump granules of pumice stone, washed in hydrochloric acid and ignited.</p>
4.11	<p>Urea.</p>

5 APPARATUS

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5.1 Distillation apparatus. See Method 2.

PREPARATION OF THE SAMPLE

6. See Method 1.

7 PROCEDURE

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Preparation of the solution

7.1 Weigh to the nearest 0.001 g, 2.5 g of the prepared sample into a 300 ml Kjeldahl flask and moisten with 20 ml water. Add with care 20 ml concentrated sulfuric acid (4.1) and a few anti-bump granules (4.10). To prevent splashing, place a long-stemmed glass funnel in the neck of the flask. Heat slowly at first, then increase the heat until white fumes are observed (30 – 40 minutes).

Cool and dilute with 100 – 150 ml water. Transfer quantitatively to a 500 ml graduated flask, discarding any sediment. Allow to cool to room temperature. Make up to volume with water, mix and, if necessary, filter through a dry paper into a dry receptacle. Discard the first portion of the filtrate.

Determination

7.2 According to the variant chosen (see Method 2) transfer with a pipette 25, 50 or 100 ml of the solution to the distillation apparatus and add sufficient sodium hydroxide solution (4.2) to ensure a considerable excess. Distil the ammonia and titrate the excess acid as described in Method 2.

Blank test

7.3 Carry out a blank test (omitting only the sample) under the same conditions and allow for this in the calculation of the final result.

Control test

7.4 Before carrying out the analysis, check that the apparatus is working properly and that the correct application of the method is used, with an aliquot portion of a freshly prepared solution of urea (4.11).

EXPRESSION OF RESULT

8. Express the result as the percentage of total nitrogen (N) contained in the fertiliser as received for analysis.

Variant (a): $N\% = (50 - A) \times 1.12$

Variant (b): $N\% = (50 - A) \times 1.12$

Variant (c): $N\% = (35 - A) \times 1.40$

Where A = millilitres of sodium or potassium hydroxide used for the titration.