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. This method is applied exclusively to urea fertilisers which are nitrate free.

PRINCIPLE

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

REAGENTS

4.—(4.1) Sulphuric acid, concentrated, (d = 1.84 g/ml).

(4.2) Sodium hydroxide solution, 30 g per 100 ml, ammonia free.

(4.3) Sulphuric acid, 0.1 N solution. } for variant (a) (page 15)

(4.4) Sodium or potassium hydroxide, 0.1 N solution, carbonate free. } for variant (a) (page 15)

(4.5) Sulphuric acid, 0.2 N solution. } for variant (b) (page 16) (see *Note* on page 15)

(4.6) Sodium or potassium hydroxide, 0.2 N solution, carbonate free. } for variant (b) (page 16) (see *Note* on page 15)

(4.7) Sulphuric acid, 0.5 N solution. } for variant (c) (page 16) (see *Note* on page 15)

(4.8) Sodium or potassium hydroxide, 0.5 N solution, carbonate free. } for variant (c) (page 16) (see *Note* on page 15)

(4.9) Indicator solutions:

Mixed indicator:

(4.9.1) Solution A: dissolve 1 g methyl red in 37 ml 0.1 N sodium hydroxide solution and make up to 1 litre with water.

Solution B: dissolve 1 g methylene blue in water and make up to 1 litre.

Mix 1 volume of solution A with 2 volumes of solution B.

This indicator is violet in acid solution, grey in neutral solution and green in alkaline solution.

Use 0.5 ml (10 drops).

Methyl red indicator solution:

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(4.9.2) dissolve 0.1 g methyl red in 50 ml 95% ethanol and make up to 100 ml with water. Filter if necessary. This indicator (4 — 5 drops) may be used instead of the preceding one.

(4.10) Anti-bump granules of pumice stone, washed in hydrochloric acid and ignited.

(4.11) Urea.

APPARATUS

5.—(5.1) Distillation apparatus. See Method 2.

PREPARATION OF THE SAMPLE

6. See Method 1.

PROCEDURE

Preparation of the solution

Preparation of the solution

7.—(7.1) Weigh to the nearest 0.001 g, 2.5 g of the prepared sample, place in a 300 ml Kjeldahl flask and moisten with 20 ml water. Stir in 20 ml concentrated sulphuric acid (4.1) and add a few glass beads to prevent bumping. 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. Quantitatively transfer 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 filter into a dry receptacle.

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, using an aliquot part of a freshly prepared solution of urea (4.11).

EXPRESSION OF THE RESULT

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

Variant (a): N% = (50 − A) × 1.12

Variant (b): N% = (50 − A) × 1.12

Variant (c): N% = (35 − A) × 1.40