

## SCHEDULE 2

### METHODS OF ANALYSIS

#### PART I

##### 4a.

#### *DETERMINATION OF THE TOTAL NITROGEN IN CALCIUM CYANAMIDE—IN THE ABSENCE OF NITRATE*

#### SCOPE

1. This method is for the determination of total nitrogen in nitrate free calcium cyanamide.

#### FIELD OF APPLICATION

2. Exclusively to calcium cyanamide (nitrate free).

#### PRINCIPLE

3. After Kjeldahl digestion, the ammoniacal nitrogen formed is displaced by sodium hydroxide and collected in a standard solution of sulphuric acid. The excess sulphuric acid is titrated with a standard solution of sodium or potassium hydroxide.

#### REAGENTS

4.—(4.1) Sulphuric acid solution 50% (V/V): dilute an appropriate volume of sulphuric acid ( $d = 1.84 \text{ g/ml}$ ) with an equal volume of water.

(4.2) Potassium sulphate.

(4.3) Copper oxide (CuO)—0.3 to 0.4 g for each determination or an equivalent quantity of copper sulphate pentahydrate, from 0.95 to 1.25 g for each determination.

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

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

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

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

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

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

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

(4.11) Indicator solutions:

##### *Mixed indicator:*

(4.11.1) 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 A with 2 volumes of B.

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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:*

(4.11.2) 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 to 5 drops) may be used instead of the preceding one.

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

(4.13) Potassium thiocyanate.

## APPARATUS

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

## PREPARATION OF SAMPLE

6. See Method 1.

## PROCEDURE

*Preparation of the solution*

*Preparation of the solution*

7.—(7.1) Weigh to the nearest 0.001 g, 1 g of the prepared sample and place it in the Kjeldahl flask. Add 50 ml 50% sulphuric acid (4.1), 10-15 g potassium sulphate (4.2) and one of the prescribed catalysts (4.3). Heat slowly to drive off the water, boil gently for two hours, allow to cool, and dilute with 100-150 ml water. Cool again, transfer quantitatively the suspension to a 250 ml graduated flask, make up to volume with water, shake and filter through a dry filter into a dry flask.

*Determination*

(7.2) According to the variant chosen (see Method 2) transfer with a pipette 50, 100 or 200 ml of the solution to the distillation apparatus and add sufficient sodium hydroxide solution (4.4) to ensure a considerable excess. Distil the ammonia and titrate the excess acid as described in Method 2.

*Blank test*

(7.3) Make 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 standard solution of potassium thiocyanate (4.13), approximating to the concentration of nitrogen in the sample.

## EXPRESSION OF THE RESULT

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

Variant (a):  $N\% = (50 - A) \times 0.7$

Variant (b):  $N\% = (50 - A) \times 0.7$

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Variant (c):  $N\% = (35 - A) \times 0.875$

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