SCHEDULE 2

METHODS OF ANALYSIS

PART I

4a.

DETERMINATION OF 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 digestion using the Kjeldahl method, the ammoniacal nitrogen formed is displaced by sodium hydroxide and collected in a standard solution of sulfuric acid. The excess sulfuric acid is titrated with a standard solution of sodium or potassium hydroxide.

4 REAGENTS

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4.1	Sulfuric acid solution 50% (V/V): dilute an appropriate volume of sulfuric acid (p=1.84 g/ml) with an equal volume of water.
4.2	Potassium sulfate.
4.3	Copper oxide (CuO), 0.3 – 0.4 g for each determination or an equivalent quantity of copper sulfate pentahydrate (0.95 to 1.25g) for each determination.
4.4	Sodium hydroxide solution 30g per 100 ml, ammonia free.

4.5	Sulfuric acid, 0.05 M solution	for variant (a) (see page 16)
4.6	Sodium or potassium hydroxide, 0.1 M solution, carbonate free	
4.7	Sulfuric acid, 0.1 M solution	for variant (b) (see page 16)
4.8	Sodium or potassium hydroxide, 0.2 M solution, carbonate free	

4.9	Sulfuric acid, 0.25 M solution	for variant (c) (see page 16)
4.10	Sodium or potassium hydroxide, 0.5 M solution, carbonate free	

4.11	Indicator solutions: 4.11.1 Mixed indicator: Solution A: dissolve 1 g methyl red in 37 ml 0.1 M 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. This indicator is violet in acid solution, grey in neutral solution and green in alkaline solution. Use 0.5 ml (10 drops). 4.11.2 Methyl red indicator: Dissolve 0.1 g methyl red in 50 ml 95% ethanol, make up to 100 ml with water and 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.

5 APPARATUS

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5.1 Distillation apparatus. See Method 2.

PREPARATION OF SAMPLE

6. See Method 1.

7 PROCEDURE

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Preparation of the solution

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% sulfuric acid (4.1), 10-15 g potassium sulfate (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 the suspension quantitatively to a 250 ml graduated flask, make up to volume with water, shake and filter through a dry filter into a dry flask. Discard the first portion of the filtrate.

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, by taking an aliquot portion of a standard solution of potassium thiocyanate (4.13), approximating to the concentration of nitrogen in the sample.

EXPRESSION OF RESULT

8. The result of the analysis must be expressed as the percentage of nitrogen (N) contained in the fertiliser as received for analysis.

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

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

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

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