SCHEDULE 2

METHODS OF ANALYSIS

PART I

6.

DETERMINATION OF CYANAMIDE NITROGEN

SCOPE

1. This method is for the determination of cyanamide nitrogen.

FIELD OF APPLICATION

2. Calcium cyanamide and calcium cyanamide/nitrate mixtures.

PRINCIPLE

3. Cyanamide nitrogen is precipitated as a silver complex and estimated in the precipitate by Kjeldahl's method.

4 REAGENTS

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4.1	Glacial acetic acid.
4.2	Ammonia solution: dilute one volume of ammonia (p=0.88 pg/ml) with 3 volumes of water.p
4.3	Ammoniacal silver solution, according to Tollens, freshly prepared: mix 500 ml silver nitrate solution (10 g per 100 ml) with 500 ml ammonia solution (4.2).

Do not expose unnecessarily to light, heat or air.

Safety precaution: when handling ammoniacal silver nitrate solution, safety goggles must be worn.

4.4	Concentrated sulfuric acid (p=1.84 g/ml).
4.5	Potassium sulfate.
4.6	Copper oxide (CuO), 0.3 – 0.4 g for each determination or an equivalent quantity of copper sulfate pentahydrate (0.95 – 1.25 g) for each determination.
4.7	Sodium hydroxide solution, 30 g per 100 ml, ammonia free.
4.8	Sulfuric acid, 0.05 M solution.

4.9	Sodium or potassium hydroxide, 0.1 M solution.
4.10	Indicator solutions:
	4.10.1 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 solution B.
	This indicator is violet in acid solution, grey in neutral solution and green in alkaline solution. Use 0.5 ml (10 drops).
4.10.2 Methyl red indicator:	
	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.
4.11	Anti-bump granules of pumice stone, washed in hydrochloric acid and ignited.
4.12	Potassium thiocyanate.

5 APPARATUS

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5.1 Distillation apparatus. See Method 2.

5.2 500 ml graduated flask (e.g. Stohmann).

5.3 Rotary shaker, 35 – 40 turns per minute.

PREPARATION OF THE SAMPLE

6. See Method 1.

7 PROCEDURE

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Preparation of the solution for analysis

7.1 Weigh, to the nearest 0.001 g, 2.5 g of the prepared sample into a small glass mortar. Grind the sample three times with water, pouring off the water after each grinding into the 500 ml graduated flask (5.2). Transfer the sample quantitatively into the flask, washing the mortar, pestle and funnel with water. Make up with water to approximately 400 ml. Add 15 ml acetic acid (4.1). Shake on the rotary shaker (5.3) for two hours.

Make up to 500 ml with water, mix and filter. Discard the first portion of the filtrate.

Proceed immediately to 7.2.

Determination

7.2 Transfer 50.0 ml of the filtrate to a 250 ml beaker. Add ammonia solution (4.2) until slightly alkaline and add 30 ml warm ammoniacal silver nitrate (4.3) to precipitate the yellow silver complex of cyanamide. Leave overnight, filter and wash the precipitate with cold water until completely free of ammonia.

Place the filter paper and the precipitate, still moist, in a Kjeldahl flask, add 10 – 15 g potassium sulfate (4.5), the catalyst (4.6) in the prescribed proportion, then 50 ml water and 25 ml concentrated sulfuric acid (4.4). Warm the flask slowly, whilst shaking it gently until the contents come to the boil. Increase the heat, boil until the contents of the flask become either colourless or pale green. Continue boiling for one hour, then leave to cool.

Transfer the liquid quantitatively from the Kjeldahl flask to the distillation flask, add a few anti-bump granules of pumice stone (4.11) and make up with water to a total volume of approximately 350 ml. Mix and cool. Add sufficient sodium hydroxide solution (4.7) to ensure a considerable excess.

Distil the ammonia and titrate the excess acid as described in Method 2 (variant (a)).

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, with an aliquot portion of a standard solution of potassium thiocyanate (4.12), corresponding to 0.05 g of nitrogen.

EXPRESSION OF RESULT

8. Express the result as the percentage of cyanamide nitrogen contained in the fertiliser as received for analysis.

$N\% = (50 - A) \times 0.56$

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