SCHEDULE 2

METHODS OF ANALYSIS

4b.

DETERMINATION OF TOTAL NITROGEN IN CALCIUM CYANAMIDE — IN THE PRESENCE OF NITRATE

1 SCOPE

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

2 FIELD OF APPLICATION

2. The method is applicable to calcium cyanamide containing nitrates.

3 PRINCIPLE

3. The direct application of Kjeldahl's method cannot be applied to calcium cyanamides containing nitrates. For this reason the nitric nitrogen is reduced to ammonia with metallic iron and stannous chloride before Kjeldahl digestion. The ammoniacal nitrogen is that determined as in Method 4a.

4 REAGENTS

4

4.1 Sulphuric acid. (d = 1.84 g/ml).

4.2 Powdered iron reduced in hydrogen.

4.3 Potassium sulphate, finely pulverised.

4.4 Sulphuric acid. 0.1 N solution.	for variant (a) (page 19)
4.5 Sodium or potassium hydroxide, 0.1 N solution, carbonate free.	
4.6 Sulphuric acid, 0.2 N solution.	for variant (b) (See Note on page 18)
4.7 Sodium or potassium hydroxide, 0.2 N solution, (page 19) carbonate free.	
4.8 Sulphuric acid, 0.5 N solution.	for variant (c) (page 19) (See *Note* on page 18)
4.9 Sodium or potassium hydroxide, 0.5 N solution, carbonate free.	

Indicator solutions:

Mixed indicator:

Mixed indicator:

4.10.—(4.10.1) Solution A: dissolve 1 g of methyl red in 37 ml of the 0.1 N sodium hydroxide solution and make up to 1 litre with water.

Solution B: dissolve 1 g of methylene blue in water and make up to 1 litre. Mix 1 volume of A and 2 volumes of B.

This indicator is violet in acid solution, grey in neutral solution and green in alkaline solution. Take 0.5 ml (10 drops, of this indicator solution.

Methyl red indicator:

(4.10.2) dissolve 0.1 g of methyl red in 50 ml of 95% ethanol, make up to 100 ml with water and filter if necessary. This indicator (1 to 5 drops) may be used instead of the preceding one.

Solution of stannous chloride:

4.11 dissolve 120 g of stannous chloride ($SnC1_2.2H_2O$), in 400 ml concentrated hydrochloric acid (d = 1.18 g/ml) and make up to 1 litre with water. The solution must be completely clear and prepared immediately before use. It is essential to check the reducing power of the stannous chloride. Dissolve 0.5 g of stannous chloride in 2 ml concentrated hydrochloric acid (d = 1.18 g/ml) and make up to 50 ml with water. Then add 5 g of Rochelle salt (potassium sodium tartrate) and a sufficient quantity of sodium bicarbonate for the solution to show an alkaline reaction to a litmus paper test.

Titrate with 0.1 N iodine solution in the presence of a starch solution as an indicator.

1 ml of iodine solution 0.1 N corresponds to 0.01128 g $SnC1_2.2H_2O$.

At least 80% of the total tin present in the solution thus prepared must be in a bivalent form. For the titration at least 35 ml of 0.1 N iodine solution should be used.

4.12 Sodium hydroxide solution, 30 g per 100 ml, ammonia free.

Standard nitrate-ammoniacal solution:

4.13 Weigh out 2,500 g of potassium nitrate and 10.160 g of ammonium sulphate and place them in a 250 ml graduated flask. Dissolve in water and make up to 250 ml. 1 ml of this solution contains 0.010 g of nitrogen.

4.14 Anti-bump granules of pumice stone, washed in hydrochloric acid and ignited.

5 APPARATUS

5. Distillation apparatus. See Method 2.

6 PREPARATION OF THE SAMPLE

6. See Method 1.

7 PROCEDURE

7

Preparation of the solution

7.1 Weigh to the nearest 0.001 g, 1 g of the prepared sample and place in the Kjeldahl flask. Add 0.5 g of powdered iron (4.2) and 50 ml of the stannous chloride solution (4.1), stir and leave standing for half an hour. During the time it is left standing, stir again after 10 and 20 minutes. Then add 10 g of potassium sulphate (4.3) and 30 ml of sulphuric acid (4.1). Boil and carry on the process for an hour after the appearance of white fumes. Leave to cool and dilute with 100-150 ml of water.

Transfer the suspension quantitatively into a 250 ml graduated flask, cool and make up to volume with water, mix and filter through a dry filter into a dry container.

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 (4.12) 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 with a standard solution containing quantities of ammoniacal and nitrate nitrogen comparable to the quantities of cyanamide and nitrate nitrogen contained in nitrated calcium cyanamide.

For this purpose place 20 ml of the standard solution (4.13) in the Kjeldahl flask.

Carry out the analysis according to the method described in paragraph 7.

8 EXPRESSION OF THE RESULTS

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

N% Variant (a) = (50−A) × 0.7

N% Variant (b) = (50−A) × 0.7

N% Variant (c) = (35−A) × 0.875

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