

SCHEDULE 2

METHODS OF ANALYSIS

PART I

3a.

DETERMINATION OF NITRIC AND AMMONIACAL NITROGEN — ULSCH METHOD

SCOPE

1. This method is for the determination of nitric and ammoniacal nitrogen with reduction according to Ulsch.

FIELD OF APPLICATION

2. All nitrogenous fertilisers, including compound fertilisers, in which nitrogen is found exclusively in nitrate form, or in ammoniacal and nitrate form.

PRINCIPLE

3. Reduction of nitrates and nitrites to ammonia by means of metallic iron in an acidic medium and displacement of the ammonia thus formed by the addition of an excess of sodium hydroxide: distillation of the ammonia and determination of the ammonia absorbed in a known volume of standard sulfuric acid solution. Titration of the excess sulfuric acid with a standard solution of sodium or potassium hydroxide.

4 REAGENTS

4

4.1	Hydrochloric acid solution, 50% (V/V): dilute an appropriate volume of hydrochloric acid ($\rho=1.18$ g/ml) with an equal volume of water.
4.2	Sulfuric acid, 0.05 M solution.
4.3	Sodium or potassium hydroxide, 0.1 M solution, carbonate free.
4.4	Sulfuric acid solution, approximately 30% H ₂ SO ₄ (W/V), ammonia free.
4.5	Powdered iron reduced in hydrogen. (The prescribed quantity of iron must be able to reduce at least 0.05 g nitrate nitrogen.)
4.6	Sodium hydroxide solution, 30 g per 100 ml, ammonia free.
4.7	Indicator solutions: 4.7.1 Mixed indicator:

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	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 solution A and 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).
	4.7.2 Methyl red indicator solution:
	Dissolve 0.1 g methyl red in 50 ml 95% ethanol, make up to 100 ml with water and filter if necessary.
	This indicator may be used (4 – 5 drops) instead of the preceding one.
4.8	Anti-bump granules of pumice stone, washed in hydrochloric acid and ignited.
4.9	Sodium nitrate.

APPARATUS

5. See Method 2.

6 PREPARATION OF SAMPLE

6. See Method 1.

7 PROCEDURE

7

Preparation of the solution

- 7.1 See Method 2.

Determination

7.2 Place in the receiving flask an exactly measured quantity of standard sulfuric acid (4.2) as indicated in the Table of Method 2 (variant (a)) and add the appropriate quantity of indicator solution (4.7.1 or 4.7.2).

The end of the extension tube of the condenser must be below the surface of the standard acid in the receiving flask.

Using a pipette, transfer an aliquot part of the clear solution as indicated in the Table of Method 2 (variant (a)) to the distillation flask of the apparatus. Add 350 ml water, 20 ml 30% sulfuric acid solution (4.4), stir, and add 5 g of reduced iron (4.5). Wash the neck of the flask with several ml of water, and place a small, long-stemmed funnel in the neck of the flask. Heat in a boiling water

bath for an hour and then wash the stem of the funnel with a few ml of water. Allow to cool to room temperature.

Taking care to avoid any loss of ammonia, add 50 ml concentrated sodium hydroxide solution (4.6) to the contents of the distillation flask, or in the cases where 20 ml of hydrochloric acid (4.1) has been used to dissolve the sample, add 60 ml of concentrated sodium hydroxide solution (4.6). Assemble the distillation apparatus. Distil the ammonia according to the procedure given in Method 2. Titrate the excess acid with the standard solution of sodium or potassium hydroxide (4.3).

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 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 freshly prepared solution of sodium nitrate (4.9) containing 0.045 g to 0.05 g of nitrogen.

EXPRESSION OF RESULTS

8. Express the results of analysis as a percentage of nitric nitrogen, or combined ammoniacal and nitric nitrogen, contained in the fertiliser as received for analysis.