

SCHEDULE 2

METHODS OF ANALYSIS

2.

DETERMINATION OF AMMONIACAL NITROGEN

1 SCOPE

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

2 FIELD OF APPLICATION

2. All nitrogenous fertilisers, including compound fertilisers, in which nitrogen is found exclusively either in the form of ammonium salts, or ammonium salts together with nitrates.

It is not applicable to fertilisers containing urea, cyanamide or other organic nitrogenous compounds.

3 PRINCIPLE

3. Displacement of ammonia by means of an excess of sodium hydroxide; distillation; determining the yield of ammonia in a given volume of a standard sulphuric acid and titration of the excess acid by means of 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 ($d = 1.18 \text{ g/ml}$) with an equal volume of water.

4.2 Sulphuric acid, 0.1 N solution. for variant (a)

4.3 Sodium or potassium hydroxide, 0.1 N solution, carbonate free.

4.4 Sulphuric acid, 0.2 N solution. for variant (b) (see *Note* on Page 18)

4.5 Sodium or potassium hydroxide, 0.2 N solution, carbonate free.

4.6 Sulphuric acid, 0.5 N solution. for variant (c) (see *Note* on Page 18)

4.7 Sodium or potassium hydroxide, 0.5 N solution, carbonate free.

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

4.9 Indicator solutions:

Mixed indicator:

(4.9.1) Solution A: dissolve 1 g methyl red in 37 ml sodium hydroxide solution 0.1 N 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 3 volumes of solution 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) of this indicator solution.

Methyl red indicator solution:

(4.9.2) dissolve 0.1 g methyl red in 50 ml ethanol (95%) make up to 100 ml with water and filter if necessary. This indicator may be used (4 to 5 drops) instead of the preceding one.

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

4.11 Ammonium sulphate.

5 APPARATUS

5

5.1 Distillation apparatus consisting of a round-bottomed flask of suitable capacity connected to a condenser by means of a splash head.

Examples of the different types of equipment recommended for this determination are reproduced in Figures 1, 2, 3 and 4 in the Appendix.

5.2 Rotary shaker, 35 to 40 turns per minute.

6 PREPARATION OF SAMPLE

6. See Method 1.

7 PROCEDURE

Solubility test

Solubility test

(7.1.1) Carry out a solubility test on the sample in water at room temperature in the proportion of 2 g per 100 ml.

Preparation of the solution

(7.1.2) Weigh to the nearest 0.001 g, according to the indications in the Table, a quantity of 5, 7 or 10 g of the prepared sample and place it in a 500 ml graduated flask. From the result of the solubility test, proceed as follows:

Products completely soluble in water

(a) Add to the flask the quantity of water needed to dissolve the sample; shake, and when completely dissolved, make up the volume and mix thoroughly.

Products not completely soluble in water

(b) Add to the flask 50 ml water and then 20 ml hydrochloric acid solution (4.1). Shake and leave undisturbed until the evolution of carbon dioxide has ceased. Add 400 ml water and shake for half an hour with the rotary shaker (5.2). Make up to volume with water, mix and filter through a dry filter into a dry receiver.

Determination

7.2 According to the variant chosen, place in the collecting flask a measured quantity of standard sulphuric acid as indicated in the Table on page 19. Add the appropriate quantity of the chosen

indicator solution (4.9.1 or 4.9.2) and, if necessary, water in order to obtain a volume of at least 50 ml. The condenser outlet must be below the surface of the standard acid in the collecting flask.

Transfer by pipette, according to the details given in the Table, an aliquot portion of the clear solution into the distillation flask of the apparatus. Add water to obtain a volume of about 3.50 ml and several grains of pumice in order to control the boiling.

Assemble the distillation apparatus, and taking care to avoid any loss of ammonia, add to the contents of the distillation flask 10 ml of concentrated sodium hydroxide solution (4.8) or 20 ml of the reagent in the cases where 20 ml hydrochloric acid (4.1) have been used in order to dissolve the sample. Warm the flask gently and when boiling commences distil at such a rate that about 250 ml are distilled in 30 minutes.

When no more ammonia is likely to be evolved, lower the receiving flask so that the tip of the condenser is above the surface of the liquid.

Test the subsequent distillate by means of an appropriate reagent to ensure that all the ammonia is completely distilled. Wash the condenser extension with a little water and titrate the excess acid with the standard solution of sodium or potassium hydroxide prescribed for the variant adopted (see *Note*).

Note:

Standard solutions of different strengths may be used for the titration provided that the volumes used for the titration do not, as far as possible, exceed 40 to 45 ml.

Blank

7.3 Make a blank test under the same conditions (omitting only the sample) and allow for this in the calculation of the final result.

Control test

7.4 Before carrying out analyses, check that the apparatus is working properly and that the correct application of the method is used, using an aliquot part of a freshly prepared solution of ammonium sulphate (4.11) containing the maximum quantity of nitrogen prescribed for the chosen variant.

8 EXPRESSION OF THE RESULT

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

TABLE FOR METHOD 2

— Determination of the ammoniacal nitrogen and of the ammoniacal and nitrate nitrogen in fertilisers.

Table of the weighing, dilution and calculation to be carried out for each of the variants (a), (b) and (c) of the method.

Variant (a)

— Approximate maximum quantity of nitrogen to be distilled = 50 mg Sulphuric acid 0.1 N to be placed in the receiving flask = 50 ml Titration with sodium or potassium hydroxide, 0.1 N solution

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<i>Declaration N%</i>	<i>Amount to be weighed (g)</i>	<i>Dilution (ml)</i>	<i>Solution of sample to be distilled (ml)</i>	<i>Expression of the result (I) N % = (50-A) F</i>
0-5	10	500	50	$(50-A) \times 0.14$
5-10	10	500	25	$(50-A) \times 0.28$
10-15	7	500	25	$(50-A) \times 0.40$
15-20	5	500	25	$(50-A) \times 0.56$
20-40	7	500	10	$(50-A) \times 1.00$

Variant (b)

- Approximate maximum quantity of nitrogen to be distilled = 100 mg Sulphuric acid 0.2 N to be placed in the receiving flask = 50 ml Titration with sodium or potassium hydroxide, 0.2 N solution

<i>Declaration N%</i>	<i>Amount to be weighed (g)</i>	<i>Dilution (ml)</i>	<i>Solution of sample to be distilled (ml)</i>	<i>Expression of the result (I) N % = (50-A) F</i>
0-5	10	500	100	$(50-A) \times 0.14$
5-10	10	500	$(50-A) \times 0.28$	
10-15	7	500	50	$(50-A) \times 0.40$
15-20	5	500	50	$(50-A) \times 0.56$
20-40	7	500	50	$(50-A) \times 1.00$

Variant (c)

- Approximate maximum quantity of nitrogen to be distilled = 200 mg Sulphuric acid 0.5 N to be placed in the receiving flask = 35 ml Titration with sodium or potassium hydroxide, 0.5 N solution

<i>Declaration N%</i>	<i>Amount to be weighed (g)</i>	<i>Dilution (ml)</i>	<i>Solution of sample to be distilled (ml)</i>	<i>Expression of the result (I) N % = (50-A) F</i>
0-5	10	500	200	$(35-A) \times 0.175$
5-10	10	500	100	$(35-A) \times 0.350$
10-15	7	500	100	$(35-A) \times 0.500$
15-20	5	500	100	$(35-A) \times 0.700$
20-40	5	500	50	$(35-A) \times 1.400$

- (1) For the purposes of the formula for expression of the result:
 50 or 35 = millilitres of standard solution of sulphuric acid to be placed in the receiving flask;
 A = millilitres of sodium or potassium hydroxide used for the titration;
 F = factor taking into account the weight of sample, the dilution, the volume of the aliquot part distilled and the volumetric equivalent.