SCHEDULE 2

METHODS OF ANALYSIS

PART II

3.

DETERMINATION OF TOTAL NITROGEN — CHROMIUM POWDER REDUCTION METHOD

1 SCOPE AND FIELD OF APPLICATION

1. This method is applicable to fertilisers in Groups l(b), l(c), 3(b), 4(a) and 4(c) of Section A, Group 5 of Section B and Groups l(c) and I(d) of Section C of the Table in Schedule 1 of the Fertilisers Regulations (Northern Ireland) 1990(**1**) in respect of which the indication of total nitrogen is required.

2 PRINCIPLE

2. The nitrate is reduced to ammonia by chromium powder in an acid medium. Organic and ureic nitrogen is converted into ammonium sulphate by digestion with concentrated sulphuric acid using a catalyst. The ammonia is distilled from an alkaline solution and absorbed in a standard acid. The excess acid is titrated with standard alkali.

3 REAGENTS

3

3.1 Sodium hydroxide solution: 40 g per 100 ml, ammonia free.

3.2 Sulphuric acid, 0.1 N solution.

3.3 Sulphuric acid, 0.2 N solution.

3.4 Sulphuric acid, 0.5 N solution.

3.5 Sodium hydroxide, 0.2 N solution, carbonate free.

3.6 Chromium metal powder, 100 mesh, low nitrogen content.

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

3.8 Anti-foaming agent, paraffin wax.

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

3.10 Hydrochloric acid (d = 1.18 g/ml).

3.11 Catalyst mixture: 1,000 g potassium sulphate and 50 g copper sulphate pentahydrate. The ingredients must be ground and thoroughly mixed.

3.12 Indicator solutions:

Mixed indicator:

(3.12.1) mix 50 ml of 2 g/litre ethanolic solution of methyl red with 50 ml of 1g./litre ethanolic solution of methylene blue.

(**1**) S.R. 1990 No. 286

Methyl red indicator:

(3.12.2) dissolve 0.1 g methyl red in 50 ml ethanol. This indicator may be used instead of the preceding one.

3.13 pH indicator paper, wide range.

4 APPARATUS

4. Apparatus for mineral acid digestion and distillation according to Kjeldahl's method.

5 PREPARATION OF SAMPLE

5. See Method 1.

6 PROCEDURE

6

Reduction

6.1 Weigh, to the nearest 0.001 g, between 0.5 and

2.0 g of the prepared sample, containing not more than 0.06 g nitric nitrogen and 0.235 g total nitrogen, and transfer to a Kjeldahl flask. Add sufficient water to make the total volume 35ml. Allow the flask to stand for 10 minutes with occassional gentle swirling to ensure solution of all nitrate salts.

Add 1.2 g chromium powder (3.6) and 7 ml hydrochloric acid (3.10), mix well and allow the flask to stand for at least 5 minutes but not more than 10 minutes at ambient temperature. Heat the flask gently so that the contents just begin to boil in about 7 minutes. Continue boiling gently for 10 minutes, Remove the flask from the heat and allow to cool.

Hydrolysis, when the fertiliser is known not to contain organic matter

6.2 Place the flask (6.1) in a fume cupboard, add a small quantity of anti-bump granules (3.7) and then carefully add 25 ml sulphuric acid (3.9). Mix the contents of the flask and heat gently until boiling. Continue heating until dense white fumes of sulphuric acid are evolved for at least 15 minutes. Allow the mixture to cool and then carefully add 250 ml water. Allow to cool to room temperature and continue as described in 6.4.

Digestion, when the fertiliser is known to contain organic matter

6.3 Add a small quantity of anti-bump granules (3.7), 10 g of the catalyst mixture (3.11) and then carefully add 25 ml sulphuric acid (3.9) (see NOTE). Add 0.5g paraffin wax (3.8) to reduce foaming and mix. Heat the flask moderately at first, shaking from time to time until frothing ceases and the liquid is practically colourless. Continue the digestion for at least a further 60 minutes. Allow the mixture to cool and then carefully add 250 ml water. Allow to cool to room temperature, and continue as described in 6.4.

Note:

If organic matter other than urea exceeds 1 .O g add an additional 1.0 ml sulphuric acid for each 0.1 g organic matter in excess of 1.0 g.

Distillation

6.4 Transfer an appropriate volume of 0.1 N, or 0.2 N, or 0.5 N sulphuric acid (3.2,3.3,3.4) to the collecting flask of the distillation apparatus, according to the presumed level of nitrogen; add a few drops of indicator solution (3.12.1 or 3.12.2). Taking precautions against the loss of ammonia, carefully add to the contents of the Kjeldahl flask (6.2 or 6.3) 100 ml sodium hydroxide solution (3.1). Mix well and connect immediately to the distillation apparatus. Heat the flask so that approximately 150 ml of the liquid are distilled in 30 minutes. At the end of this time, lower the collecting flask so that the tip of the condenser is above the surface of the liquid. Test the subsequent distillate by means of the indicator paper (3.13) to ensure that all the ammonia is completely distilled. Remove the source of heat. Titrate the excess acid with 0.2 N sodium hydroxide solution (3.5) to the end point of the indicator.

Blank test

6.5 Carry out a blank test (omitting only the sample) under the same conditions and allow for this in the calculation of the final results.

7 EXPRESSION OF THE RESULTS

7. Determine the quantity of sulphuric acid consumed.

   1 ml 0.1 N sulphuric acid = 0.0014 g nitrogen.

   1 ml 0.2 N sulphuric acid = 0.0028 g nitrogen.

   1 ml 0.5 N sulphuric acid = 0.0070 g nitrogen.

Express the result as the percentage of nitrogen (N) contained in the fertiliser as received for analysis.