

SCHEDULE 2

METHODS OF ANALYSIS

9d.

EXTRACTION OF PHOSPHORUS BY NEUTRAL AMMONIUM CITRATE

1 SCOPE

1. This method is for the determination of phosphorus soluble in neutral ammonium citrate.

2 FIELD OF APPLICATION

2. All fertilisers in Group 2(a) of Section A, and Groups 1,2 and 4 of Section B and Group 2 of Section C of the Table in Schedule 1 of the Fertilisers Regulations (Northern Ireland) 1990 in respect of which solubility in neutral ammonium citrate is laid down.

3 PRINCIPLE

3. Extraction of phosphorus at a temperature of 65°C using a neutral ammonium citrate solution (pH = 7.0) under specific conditions.

4 REAGENTS

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- 4.1 Neutral ammonium citrate solution (pH = 7.0).

This solution must contain per litre 185 g of citric acid monohydrate and must have a specific gravity of 1.09 at 20°C and a pH of 7.0. The reagent is prepared as follows:

dissolve 370 g citric acid monohydrate in about 1.5 litres of water and make an approximately neutral solution by adding 345 ml of ammonia solution (28 — 29% of NH₃). If the NH₃ concentration is lower than 28% add a correspondingly larger quantity of ammonia solution and dilute the citric acid in correspondingly smaller quantities of water.

Cool and make exactly neutral: by keeping the electrodes of the pH meter (5.1) immersed in the solution, add the ammonia solution (28-29% of NH₃) drop by drop, stirring continuously (with a mechanical stirrer) until a pH of exactly 7.0 at 20°C is obtained.

At this point make up the volume to 2 litres and test the pH again. Keep the reagent in a closed container and check the pH at regular intervals.

5 APPARATUS

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- 5.1 pH meter.

- 5.2 Water bath which can be set thermostatically at 65°C, equipped with a mechanically operated shaking tray (see Figure 8 in the Appendix).

6 PREPARATION OF THE SAMPLE

6. See Method 1.

Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.

7 PROCEDURE

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Extraction

7.1 Transfer 1(1) or 3(2) grams, as appropriate, of the fertilisers to be analysed into a 200 or 250 ml Erlenmeyer flask containing 100 ml of ammonium citrate solution previously heated to 65°C. Stopper the Erlenmeyer flask and shake in order to suspend the fertiliser without forming lumps. Remove the stopper for an instant in order to balance the pressure and close the Erlenmeyer flask again. Place the flask in the water-bath (5.2) set to maintain the contents of the flask at exactly 65°C. Shake mechanically for one hour so as to ensure complete suspension of the sample(3). The level of suspension in the flask must stay constantly below that of the water in the bath. After exactly one hour remove the Erlenmeyer flask from the water-bath. Cool immediately under running water to ambient temperature and quantitatively transfer the contents from the Erlenmeyer flask into a graduated 500 ml flask with a jet of water. Make up the volume with water. Mix thoroughly and filter through a dry fluted filter (medium speed) into a dry container, discarding the first part of the filtrate (about 50 ml).

About 100 ml of clear filtrate should be collected.

Determination

7.2 Determine the phosphorus according to Method 10 in an aliquot part of the clear filtrate.

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- (1) Where the fertiliser is normal superphosphate or concentrated superphosphate in Group 2(a) of Section A, or NPK fertiliser in Group 1, NP fertiliser in Group 2, or PK fertiliser in Group 4 of Section B or NPK fertiliser suspension, NP fertiliser suspension or PK fertiliser suspension in Section C of the Table in Schedule 1 of the Fertilisers Regulations (Northern Ireland) 1990.
 - (2) Where the fertiliser is triple superphosphate in Group 2(a) of Section A, or NPK fertiliser containing soft ground rock phosphate or partially solubilised rock phosphate in Group 1, or NP fertiliser containing soft ground rock phosphate or partially solubilised rock phosphate in Group 2, or PK fertiliser containing soft ground rock phosphate or partially solubilised rock phosphate in Group 4 of Section B of the Table in Schedule 1 of the Fertiliser Regulations (Northern Ireland) 1990.
 - (3) If no mechanical shaker is available, the flask may be shaken by hand every 5 minutes.