

SCHEDULE 2

METHODS OF ANALYSIS

PART I

9g.

EXTRACTION OF PHOSPHORUS BY ALKALINE AMMONIUM CITRATE (JOLIE'S METHOD)

1 SCOPE

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This method is for the determination of phosphorus soluble in Joulie's alkaline ammonium citrate.

2 FIELD OF APPLICATION

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Applicable to all the straight and compound phosphatic fertilisers, in which the phosphate occurs in an aluminocalcic form.

3 PRINCIPLE

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Extraction by shaking vigorously with an alkaline solution of ammonium citrate of defined specification (and where appropriate in the presence of oxine), at about 20°C.

4 REAGENTS

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4.1. Joulie's alkaline solution of ammonium citrate:

This solution contains 400 g of citric acid monohydrate and 153 g of NH₃ per litre. Its free ammonia content is approximately 55 g per litre. It is prepared by one of the methods described below:

(4.1.1) In a 1 litre graduated flask, dissolve 400 g of citric acid monohydrate in approximately 600 ml ammonia solution (p=0.925 g/ml), containing 200 g NH₃ per litre; this may be prepared by diluting 760 ml ammonia solution (p=0.880 g/ml) from a freshly opened bottle with water to 1 litre. The citric acid is added successively in quantities of 50 to 80 g maintaining the temperature below 50 C. Make up the volume to 1 litre with ammonia solution (p=0.925 g/ml).

(4.1.2) In a litre graduated flask, dissolve 432 g of diammonium citrate in 300 ml of water. Add 440 ml of ammonia solution (p=0.925 g/ml) (see 4.1.1 above). Make up the volume to 1 litre with water.

Verification of the total ammonia content:

Transfer a 10 ml sample of the citrate solution to a 250 ml flask. Make up the volume with distilled water. Determine the ammoniacal nitrogen content on 25 ml of this solution using Method 2. In these conditions the reagent is considered to be correct when the volume of 0.25 M sulfuric acid required is between 17.7 and 18.0 ml (1 ml 0.25 M H₂SO₄=0.008516g NH₃).

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If the titre is too low add 4.25 ml of ammonia ($\rho=0.925$ g/ml) per 0.1 ml below the 18 ml indicated above.

4.2 8-Hydroxyquinoline (oxine), powdered.

5 APPARATUS

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5.1 Rotary shaker, 35 – 40 turns per minute.

5.2 500 ml graduated flask with a wide neck (Stohmann).

6 PREPARATION OF THE SAMPLE

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See Method 1.

7 PROCEDURE

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Extraction

7.1 Weigh to the nearest 0.0005 g, 1 g of the prepared sample and place in a small glass or porcelain mortar. Add about ten drops of ammonium citrate solution (4.1) to moisten it and then break it up very carefully with a pestle. Add 20 ml ammonium citrate solution (4.1), mix to a paste and leave to settle for about 1 minute.

Decant the liquid into a 500 ml graduated flask (5.2). Add 20 ml ammonium citrate solution (4.1) to the residue, grind as above and decant the liquid into the graduated flask. Repeat the process four times, so that by the end of the fifth time all the product can be poured into the flask. The total quantity of ammonium citrate solution used for these processes must be approximately 100 ml.

Rinse the residue from the pestle and mortar into the graduated flask with 40 ml of distilled water.

Stopper the flask and shake for three hours on the rotary shaker (5.1).

Leave the flask standing for fifteen to sixteen hours and then shake it again under the same conditions for three hours. The temperature during the whole process should be kept at $20^{\circ}\pm 2^{\circ}\text{C}$.

Make up to volume with distilled water and mix. Filter through a dry filter, discard the first portion of the filtrate and collect the clear filtrate in a dry flask.

Determination

7.2 Determine the phosphorus using Method 10 on an aliquot portion of the clear filtrate.

NOTE

8. The use of oxine makes it possible to apply this method to fertilisers containing magnesium. This is recommended when the ratio of magnesium to phosphorus pentoxide is higher than 0.03 ($\text{Mg}/\text{P}_2\text{O}_5 > 0.03$). If this is the case, add 3 g of oxine to the moistened sample for analysis. The use of oxine in the absence of magnesium is not, moreover, likely to interfere subsequently with the determination. In the known absence of magnesium, oxine may be omitted.