SCHEDULE 2

METHODS OF ANALYSIS

9e.

EXTRACTION OF PHOSPHORUS BY ALKALINE AMMONIUM CITRATE (PETERMANN'S METHOD) AT 65°C

1 SCOPE

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

2 FIELD OF APPLICATION

2. Exclusively to precipitated dihydrated dicalcium phosphate ($CaHPO_4.2H_2O$).

3 PRINCIPLE

3. Extraction of phosphorus at a temperature of 65°C with an alkaline solution of ammonium citrate (Petermann) under specified conditions.

4 REAGENTS

4

4.1 Petermann’s solution

Characteristics:

citric acid monohydrate, 173 g per litre. Ammonia, 42 g per litre ammoniacal nitrogen, pH between 9.4 and 9.7.

Preparation ,from diammonium citrate:

dissolve 941 g diammonium citrate in about 3.500 ml water in a 5 litre graduated flask. Stand the flask in a bath of running water, mix and cool. Add in small amounts, 430 ml of ammonia solution (d = 0.880 g/ml), from a freshly opened bottle, (or an equivalent amount of diluted ammonia, for example if d = 0.906 g/ml then 502 ml are required). Adjust the temperature to 20°C, make up to volume with water and mix.

Preparation from citric acid and ammonia:

dissolve 865 g citric acid monohydrate in about 2,500 ml distilled water in a container of about 5 litres capacity. Place container in an ice bath and add in small amounts, shaking continually, 966 ml of ammonia solution (d = 0.880 g/ml), from a ,freshly opened bottle, (or an equivalent amount of diluted ammonia, for example if d = 0.906 g/ml, then 1,114 ml are required). Adjust the temperature to 20°C, transfer to a 5 litre graduated flask, make up to the mark with distilled water and mix.

Check the ammoniacal nitrogen content as follows:

transfer 25 ml of the solution into a 250 ml graduated flask, make up to volume with distilled water and mix. Determine the ammoniacal nitrogen content on 25 ml of this solution following Method 2. If

the solution is correct. 15 ml 0.5 N H_2SO_4 are consumed. Calculate the concentration of ammoniacal nitrogen in the reagent solution (1 ml 0.5 N H_2O = 0.007 g nitrogen).

If the concentration of ammoniacal nitrogen is greater than 42 g/litre, ammonia can be expelled by a stream of inert gas or by moderate heating to bring back the pH to 9.7. Carry out a second determination.

If the concentration of ammoniacal nitrogen is less than 42 g/litre, calculate the volume of ammonia solution required to achieve this level (1 ml ammonia solution. d = 0.880 g/ml contains approximately 0.22 g ammoniacal nitrogen). For each ml of ammonia solution required add 0.173 g of citric acid.

Whenever corrections are made to this reagent solution, it is imperative that the final concentration of both the citric acid and ammoniacal nitrogen are as specified.

5 APPARATUS

5

5.1 Water bath which can be maintained at a temperature of 65° ± 1°C.

5.2 500 ml graduated flask (for example Stohmann flask).

6 PREPARATION OF SAMPLE

6. See Method I.

7 PROCEDURE

7

Extraction

7.1 Weigh up to the nearest 0.001 g, 1 g of the prepared sample and transfer to the 500 ml graduated flask (5.2). Add 200 ml alkaline ammonium citrate solution (4.1). Stopper the flask and shake vigorously by hand to avoid the formation of lumps and to prevent any adherence of the substance to the sides.

Place the flask in the water bath at 65°C and shake every 5 minutes during the first half an hour. After each shaking, raise the stopper to equilibrate the pressure. The level of water in the water bath should be above the level of solution in the flask. Allow the flask to remain in the water bath a further hour at 65°C and shake every ten minutes. Remove the flask, cool to a temperature of about 20°C, make up to a volume of 500 ml with water. Mix and filter through a dry fluted filter paper, rejecting the first portion of filtrate.

Determination

7.2 Determine the phosphate according to Method 10 on an aliquot part of the clear filtrate.