SCHEDULE 2

METHODS OF ANALYSIS

PART II

13.

DETERMINATION OF IRON

SCOPE AND FIELD OF APPLICATION

1. This method is applicable to all fertilisers.

PRINCIPLE

2. The sample is ashed and dissolved in dilute hydrochloric acid or, if it contains no organic substances, it is dissolved directly in dilute hydrochloric acid. The solution is diluted and the iron content of the extract is determined by atomic absorption spectrophotometry.

REAGENTS

3.—(3.1) Hydrochloric acid (d= 1.18 g/ml).

(3.2) Hydrochloric acid, 6 N solution.

(3.3) Hydrochloric acid, 0.5 N solution.

(3.4) Hydrogen peroxide, approximately 100 volume, 30% by weight.

(3.5.1) Iron solution(**1**) (stock):

weigh to the nearest 0.001 g, 1 g pure iron, dissolve in 200 ml 6 N hydrochloric acid solution (3.2), add 16 ml hydrogen peroxide solution (3.4) and dilute to 1 litre with water.

1 ml of this solution = 1,000 μ of iron (Fe).

(3.5.2) Iron solution (dilute):

dilute 10 ml of stock solution (3.5.1) to 100 ml with water.

1 ml of this solution = 100 μ of iron (Fe).

(3.6) Lanthanum chloride solution:

dissolve 12 g lanthanum oxide in 150 ml water, add 100 ml 6 N hydrochloric acid solution (3.2) and dilute to 1 litre with water.

APPARATUS

4.—(4.1) Atomic absorption spectrophotometer with an iron lamp (248.3 nm).

PREPARATION OF SAMPLE

5. See Method 1.

(**1**) Commercially available standard iron solution may be used.

PROCEDURE

Preparation of the solution for analysis

Preparation of the solution for analysis

In the absence of organic matter

(6.1.1) Weigh to the nearest 0.001 g, 5 g of the prepared sample, place it in a 400 ml beaker, add carefully 5 ml hydrochloric acid (3. 1) (there may be a vigorous reaction due to carbon dioxide formation). Add more hydrochloric acid, if necessary. When effervescence has stopped, evaporate to dryness on a steam bath, stirring occasionaly with a glass rod. Add 15 ml 6 N hydrochloric acid solution (3.2) and 120 ml water. Stir with the glass rod, which should be left in the beaker, and cover the beaker with a watch glass. Boil the solution gently until dissolution appears complete and then filter through a filter paper(**2**) into a 250 ml graduated flask. Wash the beaker and filter with 5 ml of hot 6 N hydrochloric acid solution (3.2) and twice with boiling water. Cool and make up to the mark with water. (The hydrochloric acid concentration of this solution should be about 0.5 N.)

In the presence of organic matter

(6.1.2) Weigh to the nearest 0.001 g, 5 g of the prepared sample into a silica or platinum crucible and place the crucible in a cold muffle furnace. Close the furnace and gradually raise the temperature to 450-475° over about 90 minutes. Maintain this temperature for at least 16 hours and then open the furnace and allow the crucible to cool. Moisten the ash with water and transfer it into a 250 ml beaker. Wash the crucible with about 5 ml hydrochloric acid (3.1) and add the latter slowly and carefully to the beaker (there may be a vigorous reaction due to carbon dioxide formation). If necessary, add more hydrochloric acid (3.1) with stirring, until all effervescence has stopped.

Evaporate the solution to dryness, occasionally stirring with a glass rod. Add 15 ml 6 N hydrochloric acid solution (3.2) and 120 ml water. Stir with the glass rod, which should be left in the beaker, and cover with a watch glass. Boil the solution gently until dissolution appears complete and filter through a filter paper(**3**) into a 250 ml graduated flask. Wash the beaker and filter with 5 ml of hot 6 N hydrochloric acid solution (3.2) and twice with boiling water. Cool and make up to the mark with water. (The hydrochloric acid concentration of this solution should be about 0.5 N.)

Blank solution

(6.2) Prepare a blank solution from which only the sample has been omitted and allow for this in the calculation of the final results.

Determination

Preparation of sample and blank test solutions

(6.3.1) Dilute the sample solutions (6.1.1 or 6.1.2) and the blank test solution (6.2) with 0.5 N hydrochloric acid solution (3.3) to a concentration within the optimal measuring range of the spectrophotometer. The final solution must contain 10% (V/V) of the lanthanum chloride solution (3.6).

Preparation of the calibration solutions

(**2**) Whatman 541 or equivalent.
(**3**) Whatman 541 or equivalent.

(6.3.2) By diluting the standard solution (3.5.2) with 0.5 N hydrochloric acid solution (3.3) prepare at least 5 standard solutions of increasing concentration corresponding to the optimal measuring range of the spectrophotometer. The final solutions must contain 10% (V/V) of the lanthanum chloride solution (3.6).

Measurement

(6.4) Set up the spectrophotometer (4.1), at a wave length of 248.3 nm using an oxidising air-acetylene flame. Spray successively, in triplicate, the standard solutions (6.3.2), the sample solution, and the blank test solution (6.3.1), washing the instrument through with distilled water between each spraying. Plot the calibration curve using the mean absorbances as the ordinates and the corresponding concentrations of iron in υ/ml as the abscissae. Determine the concentration of iron in the final sample and blank solutions by reference to the calibration curve.

EXPRESSION OF RESULTS

7. Calculate the iron content of the sample taking into account the weight of the test sample and the dilutions carried out in the course of the analysis. Express the result either as a percentage or as mg/kg.