SCHEDULE 2

METHODS OF ANALYSIS

PART II

10.

DETERMINATION OF COBALT

SCOPE AND FIELD OF APPLICATION

1. This method is applicable to all fertilisers.

PRINCIPLE

2. The sample is dissolved in hydrochloric acid (after ashing if necessary) and the solution is treated with citric acid in order to prevent precipitation of iron and phosphate. Cobalt is extracted as its 2-nitroso-1-naphthol complex into toluene. The cobalt content is measured at 367 nm, by reference to a calibration curve.

REAGENTS

3.—(3.1) Sodium sulphate, anhydrous.

(3.2) Toluene.

(3.3) Hydrochloric acid, 2 N solution.

(3.4) Hydrochloric acid solution, 50— (V/V): dilute 50 ml concentrated hydrochloric acid solution (d = 1.18 g/ml) to 100 ml with water.

(3.5) Hydrogen peroxide solution, 3% (10 volume).

(3.6) Nitric acid solution, 30% (V/V): dilute 30 ml nitric acid (d = 1.42 g/ml) with water to 1OOml.

(3.7) 2-nitroso-1-naphthol solution:

dissolve 1 g of 2-nitroso-l-naphthol in 100 ml glacial acetic acid and add 1 g activated carbon.

Shake the solution before use and filter off the required amount.

(3.8) Sodium citrate solution: 40 g per 100 ml.

(3.9) Sodium hydroxide, 2 N solution.

(3.10) Cobalt solution (stock):

weigh to the nearest 0.001 g, 0.670 g ammonium cobaltous sulphate, $[(NH_1)_2CO(SO_4)_2.6H_2O]$

dissolve in water and make up to 100 ml with water. 1 ml of this solution contains 1,000µ cobalt.

(3.11) Cobalt solution (working standard):

dilute the stock colbalt solution (3.10) as required so that 1 ml contains 1µ cobalt. Prepare the solution freshly before use.

APPARATUS

4.—(4.1) Spectrophotometer with 10 mm cells.

PREPARATION OF THE SAMPLE

5. See Method 1.

PROCEDURE

Preparation of the solution for analysis

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In the absence of organic matter

(6.1.1) Weigh to the nearest 0.001 g, 5 g of the prepared sample, place in a 100 ml beaker, add 10 ml hydrochloric acid solution (3.4) and evaporate to dryness on a steam bath. Extract the soluble salts with three successive 10 ml portions of boiling 2 N hydrochloric acid solution (3.3), decanting the solution each time through the same filter paper(**1**) into a 50 ml graduated flask. Wash the filter paper with a little water, cool the solution to room temperature and make up to the mark with water.

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 dish and place a silica cover on top. Transfer the dish to a cold mute furnace, raise the temperature to about 475°C (do not exceed 500°C). Maintain at this temperature for at least 16 hours and then open the furnace and allow the crucible to cool. Add 10 ml hydrochloric acid solution (3.4) and evaporate to dryness on a steam bath. Extract the soluble salts with two successive 10 ml portions of boiling 2 N hydrochloric acid solution (3.3), decanting the solution each time through the same filter paper(**2**) into a 50 ml graduated flask. Add 5 ml hydrochloric acid solution (3.4) and 5 ml nitric acid solution (3.6) to the residue in the dish and evaporate the mixture to dryness on a hot plate at low heat. Add 10 ml boiling hydrochloric acid solution (3.3) to the residue and filter the solution through the same filter paper into the 50 ml graduated flask. Wash the filter paper with water, cool the solution to room temperature and make up to the mark with water.

Determination

(6.2.1) (6.2) (6.2.1) Transfer a suitable aliquot of the solution prepared in 6.1 (containing not more than 15 µ cobalt) to a 100 ml beaker, add 15 ml sodium citrate solution (3.8), dilute to about 50 ml with water and adjust the pH to between 3 and 4 by adding 2 N hydrochloric acid solution (3.3). (A precipitate of ferric hydroxide may form but this can be dissolved by heating the solution.) Cool to room temperature, add 10 ml hydrogen peroxide solution (3.5) and, after 5 minutes, 1 ml 2-nitroso-l-naphthol solution (3.7). Heat the solution to about 90°C and then allow to stand for 30 minutes at room temperature. Transfer the solution to a 125 ml separating funnel, add I 0 ml toluene (3.2), shake vigorously for 2 minutes, allow the phases to separate and discard the lower aqueous phase.

(6.2.2) To the toluene extract add 20 ml 2 N hydrochloric acid solution (3.3), shake for 1 minute and discard the lower aqueous phase. Add 20 ml 2 N sodium hydroxide solution (3.9), shake for 1 minute and again discard the aqueous phase. Repeat the washing with a further 20 ml of 2 N sodium hydroxide solution (3.9). Finally run off the toluene solution through a little anhydrous sodium sulphate (3.1) into a clean dry stoppered tube. Carry out a blank determination repeating the procedure, but omitting the sample. Measure the absorbance of the magenta coloured solutions at a wave length of 367 nm in the spectrophotometer

(**1**) Whatman 541 or equivalent.
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(4.1) with toluene (3.2) as reference. Determine the quantity of cobalt in the solution by reference to the calibration curve (6.3).

Calibration curve

(6.3) Measure amounts of cobalt working standard solution (3.1 1) corresponding to 3, 6, 9, 12 and 15μ cobalt into five separate 100 ml beakers and proceed as described in 6.2 commencing at ". . . add 15 ml sodium citrate solution (3.8). . .". Plot a calibration graph of the absorbance of the solutions against the corresponding amounts of cobalt (μ).

EXPRESSION OF RESULTS

7. The cobalt content in mg/kg is given by formula:

$$\frac{A \times 50}{V \times M}$$

where:

A = weight of cobalt taken for colour development as read from the calibration graph after allowing for the blank reading (mgr;)

V = volume in ml of sample taken for colour development

M = weight of sample in grams.