SCHEDULE 2

METHODS OF ANALYSIS

PART I

26f.

DETERMINATION OF COBALT IN FERTILISER EXTRACTS BY THE GRAVIMETRIC METHOD WITH 1-NITROSO-2-NAPHTHOL

1 SCOPE

1. This method defines a procedure for determining cobalt in fertiliser extracts

2 FIELD OF APPLICATION

2. This procedure is applicable to extracts from samples of fertilisers obtained by Method 26a or Method 26b for which a declaration of cobalt content is required.

3 PRINCIPLE

3. Cobalt^{III} combines with 1-nitroso-2-naphthol to give a red precipitate Co $(Cl_0H_6ONO)_3.2H_2O$. After the cobalt present in the extract has been brought to the cobalt 111 state, the cobalt is precipitated in an acetic acid medium by a solution of 1-nitroso-2-naphthol. After filtration, the precipitate is washed and dried to constant weight and then weighed as Co $(C_10H_6ONO)_3.2H_2O$.

4 REAGENTS

4

4.1. Hydrogen peroxide solution (H_2O_2 ρ = 1.11 g/ml) 30%

4.2. Sodium hydroxide solution, about 2 M

Dissolve 8 g of sodium hydroxide in pellet form in 100 ml of water.

4.3. Diluted hydrochloric acid solution, about 6 M

Mix one volume of hydrochloric acid (ρ = 1.18 g/ml) with 1 volume of water.

4.4. Acetic acid (99.7% CH_3COOH) (ρ = l.05 g/ml).

4.5. Acetic acid solution (1:2), about 6 M

Mix one volume of acetic acid (4.4) with 2 volumes of water.

4.6. Solution of l-nitroso-2-naphthol in 100 ml of acetic acid (4.4). Add 100 ml of lukewarm water. Mix thoroughly. Filter at once. The solution obtained must be used immediately.

5 APPARATUS

5

5.1. Filter crucible P 16/ISO 4793, porosity 4, capacity 30 or 50 ml

5.2. Drying oven at 130 2° C

6 PREPARATION OF THE SOLUTION TO BE ANALYSED

6

6.1. Preparation of the cobalt solution

See Methods 26a or 26b.

6.2. Preparation of the solution to be analysed

Place the aliquot portion of the extract containing not more than 20 mg Co in a 400 ml beaker. If the extract is obtained according to Method 26b, acidify with five drops of hydrochloric acid (4.3). Add about 10 ml of the hydrogen peroxide solution (4.1). Allow the oxidant to react in the cold state for 15 minutes, then make up to about 100 ml with water. Cover the beaker with a watchglass. Bring the solution to boiling point and allow to boil for about 10 minutes. Cool. Make alkaline with the sodium hydroxide solution (4.21) drop by drop until black cobalt hydroxide begins to precipitate.

7 PROCEDURE

7. Add 10 ml of acetic acid (4.4) and make up the solution with water to about 200 ml. Heat until boiling. Using a burette, add 20 ml of the 1-nitroso-2-naphthol solution (4.6) drop by drop, stirring constantly. Complete by vigorous stirring to make the precipitate coagulate.

Filter through a previously weighed filter crucible (5.1), taking care not to clog up the crucible. With this in mind, ensure that liquid is left above the precipitate throughout the filtration process.

Wash the beaker with dilute acetic acid (4.5) to remove all the precipitate, wash the precipitate on the filter with dilute acetic acid (4.5) and then three times with hot water.

Dry in a oven (5.2) at 130±2° C until constant weight is achieved.

8 EXPRESSION OF RESULTS

8. 1 mg of Co $(C_10H_6ONO)_3.2H_2O$ precipitate corresponds to 0.096381 mg Co.

The percentage of Cobalt (Co) in the fertiliser is given by:

$Co(\%)=X\times0.0096381\times V\times Da\times M$

where:

X is the mass in mg of the precipitate;

V is the volume in ml of the extract solution obtained in accordance with Method 26a or Method 26b;

a is the volume in ml of the aliquot taken from the last dilution;

D is the dilution factor of this aliquot;

M is the mass in g of the test sample.