SCHEDULE 2

METHODS OF ANALYSIS

14.

DETERMINATION OF CHLORIDES IN THE ABSENCE OF ORGANIC MATERIAL

1 SCOPE

1. This method is for the determination of chloride, in the absence of organic material.

2 FIELD OF APPLICATION

2. All fertilisers which are free from organic material, except ammonium nitrate fertilisers of a nitrogen content greater than 28% by weight.

3 PRINCIPLE

3. The chlorides, dissolved in water, are precipitated in an acid medium by an excess of standard solution of silver nitrate. The excess is titrated with a solution of ammonium thiocyanate in the presence of ferric ammonium sulphate. (Volhard's method).

4 REAGENTS

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4.1 Nitrobenzene or diethyl ether.

4.2 Nitric acid, 10 N solution.

4.3 Indicator solution: dissolve 40 g of ferric ammonium sulphate [$Fe_2(SO_4)_3.(NH_4)_2SO_4.24H_2O$] in water and make up to 1 litre.

4.4 Silver nitrate, 0.1 N solution.

4.5 Ammonium thiocyanate, 0.1 N solution.

Preparation: since this salt is hygroscopic and cannot be dried without risk of decomposition, it is advisable to weigh out approximately 9 g, dissolve in water and make up the volume to one litre. Standardise by titration against 0.1 N silver nitrate solution.

5 APPARATUS

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5.1 Rotary shaker, 35 — 40 turns per minute.

6 PREPARATION OF SAMPLE

6. See Method 1.

7 PROCEDURE

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Extraction

7.1 Weigh to the nearest 0.001 g, 5 g of the prepared sample and place in a 500 ml graduated flask and add 450 ml water. Mix for half an hour on the shaker (5.1); make up to 500 ml with distilled water, mix and filter into a beaker.

Determination

7.2 Take an aliquot part of the filtrate containing not more than 0.150 g of chloride. If the sample taken is smaller than 50 ml it is necessary to make up the volume to 50 ml with distilled water. Add 5 ml 10 N nitric acid (4.2), 20 ml indicator solution (4.3), and two drops ammonium thiocyanate standard solution (taken from a burette adjusted to zero). From a burette then add silver nitrate solution (4.4) until there is an excess of 2 to 5 ml. Add 5 ml nitrobenzene or 5 ml diethyl ether (4.1) and shake well to agglomerate the precipitate. Titrate the excess silver nitrate with 0.1 N ammonium thiocyanate (4.5) until a red-brown colour appears which remains after the flask has been shaken slightly.

Note:

Nitrobenzene or diethyl ether (especially the former) prevents the silver chloride from reacting with thiocyanate ions, thus a clear colour change is obtained.

Blank test

7.3 Make a blank test under the same conditions (omitting only the sample) and allow for it when calculating the final result.

Control test

7.4 Carry out the determination on an aliquot part of a freshly prepared solution of potassium chloride, containing 0.100 g as chloride.

8 EXPRESSION OF THE RESULT

8. Express the result of the analysis as a percentage of chloride contained in the sample as it has been received for analysis.

Calculation: calculate the percentage of chloride (Cl) with the formula:

$$\% \text{ Cl} = \frac{0.003546 \times (V_z - V_{cz}) - (V_a - V_{ca}) \times 100}{M}$$

where:

V_z = number of millilitres of silver nitrate added

V_{cz} = number of millilitres of silver nitrate used in the blank test

V_a = number of millilitres of ammonium thiocyanate used for the titration of the sample

V_{ca} = number of millilitres of ammonium thiocyanate used for the titration of the blank

M = weight in grams of the sample in aliquot volume taken for titration