

SCHEDULE 2

METHODS OF ANALYSIS

PART I

12.

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 sulfate (Volhard's method).

4 REAGENTS

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- 4.1 Nitrobenzene or diethyl ether.

- 4.2 Nitric acid, 10 M solution.

- 4.3 Indicator solution: dissolve 40 g of ferric ammonium sulfate $[\text{Fe}_2(\text{SO}_4)_3 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O}]$ in water and make up to 1 litre.

- 4.4 Silver nitrate, 0.1 M solution.

- 4.5 Ammonium thiocyanate, 0.1 M solution.

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

- 4.6 Potassium Chloride solution: Dissolve 2.103g of potassium chloride, previously dried at 130°C for one hour, in water and make up to 500ml.

5 APPARATUS

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- 5.1 Rotary shaker, 35 – 40 turns per minute.

PREPARATION OF SAMPLE

6. See Method 1.

Status: This is the original version (as it was originally made). This item of legislation is currently only available in its original format.

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. Shake for half an hour on the rotary shaker (5.1); make up to 500 ml with distilled water, mix and filter into a beaker, discarding the first part of the filtrate.

Determination

7.2 Take an aliquot portion of the filtrate containing not more than 0.150 g of chloride. If the portion taken is smaller than 50 ml it is necessary to make up the volume to 50 ml with distilled water. Add 5 ml 10 M nitric acid (4.2), 20 ml indicator solution (4.3), and two drops of 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 M ammonium thiocyanate (4.5) until a red-brown colour just 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 Carry out a blank test under the same conditions (omitting only the sample) and allow for this in the calculation of the final result.

Control test

7.4 Carry out the determination using 50ml (equivalent to 0.100g of chloride) of the potassium chloride solution (4.6).

EXPRESSION OF 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:

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

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 = mass in grams of the sample in aliquot volume taken for titration.