

SCHEDULE 2

METHODS OF ANALYSIS

PART I

16.

EXTRACTION OF TOTAL SULFUR

1 SCOPE

1. This method is for the extraction of total sulfur contained in fertilisers in elemental form and/or in other chemical combinations.

2 FIELD OF APPLICATION

2. This method applies to all fertilisers for which a declaration of the total sulfur present in various forms (elemental, thiosulfate, sulfite and sulfate) is required.

3 PRINCIPLE

3. Elemental sulfur is converted in an alkaline medium into polysulfides and thiosulfate; these, together with any sulfites which may be present, are then oxidised with hydrogen peroxide. The various forms of sulfur are thus converted into sulfate which is determined by precipitation as barium sulfate (method 23).

4 REAGENTS

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4.1 Diluted hydrochloric acid:

One volume of hydrochloric acid ($p = 1.18$ g/ml) plus one volume of water.

4.2 Sodium hydroxide solution, NaOH, 30% minimum ($p = 1.33$)

4.3 Hydrogen peroxide solution, 30% w/w.

4.4 Aqueous solution of barium chloride $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$, 122 gram per litre.

5 APPARATUS

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Electric hot plate with adjustable temperature.

6 PREPARATION OF THE SAMPLE

6. See method 1.

7 PROCEDURE

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7.1 Test sample.

Status: This is the original version (as it was originally made).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

Weigh out to within 1 mg a quantity of fertiliser containing between 80 and 350 mg of sulfur (S) or 200 and 875 mg SO_3 .

As a rule (where $S < 15\%$), weigh out 2.5 grams. Place the test sample in a 400 ml beaker.

7.2 Oxidation.

Add 20 ml of sodium hydroxide solution (4.2) and 20 ml of water. Cover with a watch glass. Boil for five minutes on the hot plate (5). Remove from the hot plate. Using a jet of hot water, collect any material sticking to the sides of the beaker and boil for 20 minutes. Leave to cool.

Add 2 ml increments of hydrogen peroxide (4.3) until no reaction is observed. Six to eight ml of hydrogen peroxide will be necessary. Allow oxidation to continue for one hour, then bring to the boil for half an hour. Leave to cool.

7.3 Preparation of the solution to be analysed.

Add approximately 50 ml of water and 50 ml of the hydrochloric acid solution (4.1).

— If the level of sulfur (S) is less than 5%:

filter into a 600 ml beaker. Wash the residue on the filter several times with cold water. After washing, check for the absence of sulfate in the last drops of the filtrate using the barium chloride solution (4.4). The filtrate must be perfectly clear. Sulfate is determined on the whole of the filtrate in accordance with method 23.

— If the level of sulfur (S) is at/above 5%:

transfer quantitatively into a 250 ml volumetric flask, make up to volume with water and mix. Filter through a dry filter into a dry container; the filtrate must be completely clear. Stopper if the solution is not to be used immediately. Determine sulfates in an aliquot portion of this solution by precipitation in the form of barium sulfate (method 23).