

SCHEDULE 2

METHODS OF ANALYSIS

PART I

19.

EXTRACTION AND DETERMINATION OF ELEMENTAL SULFUR

WARNING

This method of analysis involves the use of carbon disulfide (CS₂). Special safety measures must therefore be taken, in particular with regard to:

- the storage of CS₂,
- protective equipment for staff,
- occupational hygiene,
- prevention of fires and explosions,
- disposal of the reagent.

This method requires highly skilled staff and a suitably equipped laboratory.

1 SCOPE

1. This method is for the extraction and determination of the elemental sulfur content of fertilisers.

2 FIELD OF APPLICATION

2. This method applies to all fertilisers for which a declaration of the total sulfur in elemental form is required.

3 PRINCIPLE

3. After the removal of soluble compounds, elemental sulfur is extracted by using carbon disulfide, followed by gravimetric determination of the sulfur extracted.

4 REAGENTS

4. Carbon disulfide.

5 APPARATUS

5

- 5.1 100 ml extraction flask with ground glass stopper.
- 5.2 Soxhlet apparatus.
- 5.3 Vacuum rotary evaporator.
- 5.4 Electric oven, fan assisted, set at 90+2° C.
- 5.5 Petri dishes, five to seven centimetres in diameter, not exceeding five centimetres in height.

Status: This is the original version (as it was originally made).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

5.6 Electric hot plate with adjustable temperature.

6 PREPARATION OF THE SAMPLE

6. See method 1.

7 PROCEDURE

7

7.1 Test sample. Weigh out 5 – 10 g of the sample to an accuracy of 1 mg and place in the thimble of the Soxhlet apparatus (5.2).

7.2 Extraction of the sulfur.

Wash thoroughly the contents of the thimble with hot water to remove all soluble compounds. Dry in the oven at 90°C (5.4) for at least one hour. Place the thimble in the Soxhlet apparatus (5.2).

Place a few glass beads in the flask of the apparatus (5.1) and weigh (P_0), then add 50 ml of carbon disulfide (4.1).

Connect the apparatus, switch on and leave for six hours. Switch off the heat and, after cooling, disconnect the flask. Connect the flask to the rotary evaporator (5.3) and evaporate until the contents of the flask have solidified in a spongy mass.

Dry the flask in the oven at 90° C (5.4) (generally one hour if necessary) until a constant weight is obtained (P_1).

7.3 Determination of the purity of the elemental sulfur.

Certain substances may have been extracted by the carbon disulfide at the same time as the elemental sulfur. The purity of the elemental sulfur is determined as follows:

homogenize the contents of the flask as thoroughly as possible and remove 2 – 3 g, weigh to an accuracy of 1 mg (n). Place in the petri dish (5.5). Weigh dish and contents together (P_2). Place on the hot plate (5.6), set at a temperature not exceeding 220° C so as not to cause combustion of the sulfur. Continue sublimation for three to four hours until a constant weight is obtained (P_3).

(Note) For some fertilisers, it may not be necessary to determine how pure the sulfur is. In this case, omit step 7.3.

8 EXPRESSION OF RESULTS

8. The percentage elemental sulfur (S) content of the fertiliser is as follows:

$$\text{ImpureS(\%ofthefertiliser)} = P_1 P_0 m \times 100$$

$$\text{PurityofSextracted(\%)} = P_2 P_3 n \times 100$$

$$\text{PureS(\%ofthefertiliser)} = (P_1 P_0) (P_2 P_3) m \times n \times 100$$

Where:

m = the mass of the test sample of fertiliser in grams,

P_0 = the mass of the Soxhlet flask in grams,

P_1 = the mass of the Soxhlet flask and the impure sulfur after drying,

n = the mass of impure sulfur to be purified in grams,

P_2 = the mass of the Petri dish and the sample,

P_3 = the mass of the Petri dish after sublimation of the sulfur.