COMMISSION DIRECTIVE

of 1 August 1989

supplementing and amending Directive 77/535/EEC on the approximation of the laws of Member States relating to methods of sampling and analysis for fertilizers

(89/519/EEC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Directive 76/116/EEC of 18 December 1975 on the approximation of the laws of the Member States relating to fertilizers (1), as last amended by Council Directive 89/284/EEC (2), and in particular Article 9 (2) thereof,

Whereas Directive 89/284/EEC supplements and amends Directive 76/116/EEC in respect of the calcium, magnesium, sodium and sulphur content of fertilizers;

Whereas Commission Directive 77/535/EEC (3), as last amended by Directive 87/566/EEC (4), provides for official controls for EEC fertilizers for the purpose of checking compliance with the requirements imposed by the Community provisions concerning the quality and composition of fertilizers; whereas that Directive should be supplemented so that fertilizers to which Council Directive 89/284/EEC relates can also be checked; whereas it is necessary to replace methods 5.1, 5.2, 5.3 and 5.4; whereas general provisions relating to methods for analysing fertilizers applicable to all methods should be added to the general observations of Annex II;

Whereas the measures provided for in this Directive are in accordance with the opinion of the Committee on the Adaptation to Technical Progress of the Directives for the Removal of Technical Barriers to Trade in Fertilizers,

HAD ADOPTED THIS DIRECTIVE:

Article 1

1. General provisions relating to methods for analysing fertilizers of Annex I to this Directive are added to the general observations of Annex II to Directive 77/535/EEC.

2. The methods 8.1 to 8.10 in Annex II to this Directive are added to Annex II to Directive 77/535/EEC.

3. Methods 5.1 to 5.4 set out in Annex II to Directive 77/535/EEC are deleted.

Article 2

1. Member States shall take measures necessary to comply with this Directive by 1 September 1990 and shall forthwith inform the Commission thereof.

The provisions adopted pursuant to the first indent shall make express reference to this Directive.

2. Member States shall communicate to the Commission the texts of the provisions of national law which they adopt in the field covered by this Directive.

Article 3

This Directive is addressed to the Member States.

Done at Brussels, 1 August 1989

For the Commission
Martin BANGEMANN
Vice-President

(1) OJ No L 24, 30. 1. 1976, p. 21.
(2) OJ No L 111, 22. 4. 1989, p. 34.
(3) OJ No L 213, 22. 8. 1977, p. 1.
(4) OJ No L 342, 4. 12. 1987, p. 32.

ANNEX I

'GENERAL PROVISIONS RELATING TO METHODS OF ANALYSING FERTILIZERS

1. **Reagents**

Unless otherwise specified in the method of analysing, all of the reagents must be analysis-pure (a. p.). Where trace elements are to be analysed the purity of the reagents must be checked by means of a blank test. Depending upon the result obtained, it might be necessary to conduct a further purification.

2. **Water**

The dissolution, dilution, rinsing or washing operations mentioned in the methods of analysis without stating precisely what the nature of the solvent or diluent is, imply the use of water. Normally, the water will have to be demineralized or distilled. In these specific instances, as mentioned in the method of analysis, that water will have to be subjected to specific purification processes.

3. **Laboratory equipment**

In view of the equipment normally used in inspection laboratories, the apparatus described in the methods of analysis is restricted to special instruments and apparatus or to such demanded by any specific requirements. This equipment must be perfectly clean, above all where small quantities are to be determined. The laboratory will have to ensure the accuracy of any graduated glassware used by referring to the standards.

ANNEX II

'Methods 8

SECONDARY NUTRIENTS

Method 8.1

EXTRACTION OF TOTAL CALCIUM, TOTAL MAGNESIUM, TOTAL SODIUM AND TOTAL SULPHUR IN THE FORM OF SULPHATES

1. SCOPE

This document defines the procedure for extracting total calcium, total magnesium and total sodium, and for extracting the total sulphur present in the form of sulphates, so that the same extract may be used for the determination of each nutrient required.

2. FIELD OF APPLICATION

This method applies to EEC fertilizers, for which a declaration of the total calcium, total magnesium, total sodium, and total sulphur in the form of sulphates is provided for in Council Directive 89/284/EEC (1).

3. PRINCIPLE

Solubilization by boiling in dilute hydrochloric acid.

4. REAGENTS

4.1 Diluted hydrochloric acid:

One volume of hydrochloric acid (d = 1,18) plus one volume of water.

5. APPARATUS

Electric hot plate with adjustable temperature.

6. PREPARATION OF THE SAMPLE

See method 1 in Commission Directive 77/535/EEC.

7. PROCEDURE

7.1 Test sample

Calcium, magnesium, sodium and sulphur in the form of sulphates are extracted from a test sample of five grams weighed to within one milligram.

However, when the fertilizer contains more than 15 % of sulphur (S) i. e. 37,5 % SO_3, and more than 18,8 % of calcium (Ca) i. e. 26,3 % CaO, the extraction of calcium and sulphur is carried out on a test sample of one gram, weighed to within one milligram. Place the test sample in a 600 millilitre beaker.

7.2 Preparation of the solution

Add approximately 400 millilitres of water and, taking care when the sample contains a significant quantity of carbonates, 50 millilitres of dilute hydrochloric acid (4.1) a small amount at a time. Bring to the boil and maintain for 30 minutes. Allow to cool, stirring occasionally. Decant

(1) OJ No L 111, 22. 4. 1989, p. 34.

quantitatively into a 500 millilitre graduated flask. Make up to volume with water, and mix. Pass through a dry filter into a dry container, discarding the initial portion. The extract must be completely transparent. Stopper if the filtrate is not used immediately.

Method 8.2

EXTRACTION OF TOTAL SULPHUR PRESENT IN VARIOUS FORMS

1. SCOPE

This document defines the procedure for extracting the total sulphur contained in fertilizers in elemental form and/or in other chemical combinations.

2. FIELD OF APPLICATION

This method applies to EEC fertilizers for which a declaration of the total sulphur present in various forms (elemental, thiosulphate, sulphite, sulphate) is provided for in the Council Directive 89/284/EEC.

3. PRINCIPLE

Elemental sulphur is converted in an alkaline medium into polysulphides and thiosulphate; these, together with any sulphites which may be present, are then oxidized with hydrogen peroxide. The various forms of sulphur are thus converted into sulphate which is determined by precipitation of barium sulphate (method 8.9).

4. REAGENTS

4.1 Diluted hydrochloric acid:

One volume of hydrochloric acid (d = 1,18) plus one volume of water.

4.2 Sodium hydroxide solution, NaOH, 30 % minimum (d = 1,33).

4.3 Hydrogen peroxide solution, 30 % w/w.

4.4 Aqueous solution of barium chloride $BaCl_2$ $2H_2O$, 122 gram per litre.

5. APPARATUS

Electric hot plate with adjustable temperature.

6. PREPARATION OF THE SAMPLE

See method 1.

7. PROCEDURE

7.1 Test sample

Weigh out to within one milligram a quantity of fertilizer containing between 80 and 350 milligrams of sulphur (S) or 200 and 875 milligrams of SO_3.

As a rule (where S $\lhd$ 15 %), weigh out 2,5 grams. Place the test sample in a 400 millilitre beaker.

7.2 Oxidation.

Add 20 millilitres of sodium hydroxide solution (4.2) and 20 millilitres of water. Cover with a watch glass. Boil for five minutes on the hot plate (5.1). Remove from the hot plate. Using a jet of hot water, collect the sulphur sticking to the sides of the beaker and boil for 20 minutes. Leave to cool.

Add 2 millilitres increments of hydrogen peroxide (4.3) until no reaction is observed. Six to eight 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 millilitres of water and 50 millilitres of the hydrochloric acid solution (4.1).

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

filter into a 600 millilitre beaker. Wash the residue on the filter several times with cold water. After washing, check for the absence of sulphate in the last drops of the filtrate using a barium chloride solution (4.4). The filtrate must be perfectly clear. Sulphate is determined on the whole of the filtrate in accordance with method 8.9.

— *If the level of sulphur (S) is at above 5 %:*

transfer quantitatively into a 250 millilitre 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 sulphates on an aliquot of this solution by precipitation in the form of barium sulphate (method 8.9).

Method 8.3

EXTRACTION OF WATER-SOLUBLE CALCIUM, MAGNESIUM, SODIUM AND SULPHUR (IN THE FORM OF SULPHATES)

1. SCOPE

This document defines the procedure for extracting water-soluble calcium, magnesium, sodium and sulphur (in the form of sulphates), so that the same extract can be used to determine each nutrient required.

2. FIELD OF APPLICATION

This method applies solely to fertilizers for which a declaration of the water soluble calcium, magnesium, sodium and sulphur (in the form of the sulphates) is provided for in Council Directive 89/284/EEC.

3. PRINCIPLE

The nutrients are solubilized in boiling water.

4. REAGENTS

Distilled or demineralized water of equivalent quality.

5. APPARATUS

Electric hot plate with adjustable temperature.

6. PREPARATION OF THE SAMPLE

See method 1.

7. PROCEDURE

7.1 Test sample.

(a) Where fertilizers contain no sulphur or where they contain, at the same time, no more than 3 % of the sulphur (S) i. e. 7,5 % SO_3 and no more than 4 % of calcium (Ca) i. e. 5,6 % CaO, weigh out five grams of fertilizer to within one milligram.