1994 No. 166

AGRICULTURE

Fertilisers (Sampling and Analysis) (Amendment) Regulations (Northern Ireland) 1994

Made	*27th April 1994*
Coming into operation . .	*27th May 1994*

The Department of Agriculture in exercise of the powers conferred on it by sections 66(1), 74A, 75(1), 76(1), 77, 78(2), (4) and (6), 79(1), (2) and (9), 84 and 86(1), (2), (3) and (9) of the Agriculture Act 1970(**a**) and of all other powers enabling it in that behalf, after consultation with such persons or organisations as appear to it to represent the interests concerned, hereby makes the following Regulations:—

Citation, commencement and interpretation

1.—(1) These Regulations may be cited as the Fertilisers (Sampling and Analysis) (Amendment) Regulations (Northern Ireland) 1994 and shall come into operation on 27th May 1994.

(2) The Interpretation Act (Northern Ireland) 1954(**b**) shall apply to these Regulations as it applies to a Measure of the Northern Ireland Assembly.

Amendment of the Fertilisers (Sampling and Analysis) Regulations (Northern Ireland) 1991

2. The Fertilisers (Sampling and Analysis) Regulations (Northern Ireland) 1991(**c**) (''the principal Regulations'') shall be further amended as provided in regulations 3 and 4.

3. For regulation 5(2) (*Application of the methods of analysis*) there shall be substituted:

> ''(2) Analytical constituents of materials listed in Groups 1(*a*), 2(*a*) and 3(*a*) of Section A, Groups 1 to 4 of Section B, Groups 1(*a*), 1(*b*) and 2 of Section C, Section D and Section E of the table in Schedule 1 to the Fertilisers Regulations (Northern Ireland) 1992(**d**) shall be determined by the methods prescribed in Part I of Schedule 2.''.

(**a**) 1970 c. 40; section 74A was inserted by s. 4(1) of, and paragraph 6 of Schedule 4 to the European Communities Act 1972 (c. 68) and there are other amendments not relevant to these Regulations
(**b**) 1954 c. 33 (N.I.)
(**c**) S.R. 1991 No. 540 as amended by S.R. 1992 No. 235
(**d**) S.R. 1992 No. 187

4. In Part I of Schedule 2 (*Methods of Analysis*)—

(1) in paragraph 3, after item 26 (Determination of the sodium extracted) there shall be added the provisions set out in Schedule 1;

(2) in paragraph 7 of method 10, (DETERMINATION OF EXTRACTED PHOSPHORUS), for the figure "0.03074" there shall be substituted the figure "0.032074";

(3) after the provisions relating to method 26, (DETERMINATION OF THE SODIUM EXTRACTED), there shall be added the provisions set out in Schedule 2.

Sealed with the Official Seal of the Department of Agriculture for Northern Ireland on 27th April 1994.

(L.S.) *I. C. Henderson*
Assistant Secretary

PROVISIONS TO BE INSERTED IN PART I OF SCHEDULE 2 TO THE PRINCIPAL REGULATIONS

"27. *a* Extraction of total trace elements

b Extraction of water-soluble trace elements

c Removal of organic compounds from fertiliser extracts

d Determination of trace elements in fertiliser extracts by atomic absorption spectrophotometry (general procedure)

e Determination of boron in fertiliser extracts by means of spectrophotometry with azomethine-h

f Determination of cobalt in fertiliser extracts by atomic absorption spectrophotometry

g Determination of copper in fertiliser extracts by atomic absorption spectrophotometry

h Determination of iron in fertiliser extracts by atomic absorption spectrophotometry

i Determination of manganese in fertiliser extracts by atomic absorption spectrophotometry

j Determination of molybdenum in fertiliser extracts by spectrophotometry of a complex with ammonium thiocyanate

k Determination of zinc in fertiliser extracts by atomic absorption spectrophotometry"

SCHEDULE 2 Regulation 4(3)

PROVISIONS TO BE ADDED TO PART I OF SCHEDULE 2 TO THE PRINCIPAL REGULATIONS

"27a. EXTRACTION OF TOTAL TRACE ELEMENTS

1. SCOPE

This method defines the procedure for extracting the following trace elements: total boron, total cobalt, total copper, total iron, total manganese, total molybdenum and total zinc. The aim is to carry out the minimum number of extractions, making use wherever possible of the same extract to determine the total level of each of the trace elements listed above.

2. FIELD OF APPLICATION

This procedure is applicable to all fertilisers in Groups 1(*a*), 2(*a*) and 3(*a*) of Section A, Groups 1 to 4 of Section B, Groups 1(*a*) and 2 of Section C, Section D, and Section E, of the table in Schedule 1 to the Fertilisers Regulations (Northern Ireland) 1992(**a**), containing one or more of the following trace elements: boron, cobalt, copper, iron, manganese, molybdenum and zinc. It is applicable to each trace element, the declared content of which is less than or equal to 10%.

3. PRINCIPLE

Dissolution in boiling diluted hydrochloric acid.

Note: The extraction is empirical and may not be quantitative depending on the product or the other constituents of the fertiliser. In particular, in the case of certain manganese oxides, the quantity extracted may be substantially smaller than the total quantity of manganese which the product contains. It is the responsibility of fertiliser manufacturers to ensure that the declared content actually corresponds to the quantity extracted under the conditions pertaining to the method.

4. REAGENTS

4.1 Diluted hydrochloric acid (HCl) solution, approximately 6 M:

Mix 1 volume of hydrochloric acid (ρ = 1.18 g/ml) with 1 volume of water.

4.2 Concentrated ammonia solution (NH_4OH, ρ = 0.88 g/ml).

5. APPARATUS

Electric hot plate with variable temperature control.

Note: Where the boron content of an extract is to be determined, do not use borosilicate glassware. As the method involves boiling, teflon or silica is preferable. Rinse the glassware thoroughly if it has been washed in detergents containing borates.

6. PREPARATION OF THE SAMPLE

See Method 1.

(**a**) S.R. 1992 No. 187

7. PROCEDURE

7.1 *Test sample*

Take a quantity of fertiliser weighing between 2 and 10 g, depending on the declared content of the element in the product. The following table shall be used to obtain a final solution which, after appropriate dilution, will be within the measuring range for each method. Samples should be weighed to within 1 mg.

Declared content of trace element in the fertiliser (%)	<0.01	0.01 – <5	≧5 – 10
Mass of test sample (g)	10	5	2
Mass of element in the sample (mg)	1	0.5 – 250	100 – 200
Volume of extract V (ml)	250	500	500
Concentration of element in extract (mg/l)	4	1 – 500	200 – 400

Place the sample in a 250 ml beaker.

7.2 *Preparation of the solution*

If necessary moisten the sample with a little water, add carefully 10 ml of diluted hydrochloric acid (4.1) per gram of fertiliser, in small amounts, then add about 50 ml of water. Cover the beaker with a clock glass and mix. Bring to the boil on the hot plate and boil for 30 minutes. Allow to cool, stirring occasionally. Transfer quantitatively to a 250 or 500 ml volumetric flask (see table). Make up to volume with water and mix thoroughly. Filter through a dry filter into a dry container. Discard the first portion of the filtrate. The extract must be perfectly clear.

It is recommended that the determinations be carried out without delay on aliquot portions of the clear filtrate, if not the containers should be stoppered.

Note: For aliquot portions in which the boron content has to be determined: adjust the pH to between 4 and 6 with concentrated ammonia solution (4.2).

8. DETERMINATION

The determination of each trace element is to be carried out on the aliquot portions indicated in the method for each individual trace element.

If necessary, remove organic chelating or complexing substances from an aliquot portion of the extract by using Method 27c. In the case of determination by atomic absorption spectrophotometry, such removal may not be necessary.

27b. EXTRACTION OF WATER-SOLUBLE TRACE ELEMENTS

1. SCOPE

This method defines the procedure for extracting water-soluble forms of the following trace elements: boron, cobalt, copper, iron, manganese, molybdenum and zinc. The aim is to carry out the minimum number of extractions, making use wherever possible of the same extract to determine the level of each of the trace elements listed above.