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.

2. FIELD OF APPLICATION

This procedure is applicable to all fertilisers contained 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, 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

The trace elements are extracted by shaking the fertiliser in water at 20°C ± 2°C.

Note: The extraction is empirical and may or may not be quantitative.

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.

5. APPARATUS

5.1 Rotary shaker set at between 35 to 40 rpm.

5.2 pH-meter.

Note: Where the boron content of the extract is to be determined, do not use borosilicate glassware. Teflon or silica is preferable for this extraction. Rinse the glassware thoroughly if it has been washed in detergents containing borates.

6. PREPARATION OF THE SAMPLE

See Method 1.

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 or 500 ml volumetric flask (according to the table).

7.2 *Preparation of the solution*

Add approximately 200 ml of water at 20°C ± 2° to the 250 ml flask or 400 ml of water at 20°C ± 2° to the 500 ml flask.

Stopper the flask well. Shake vigorously by hand to disperse the sample, then place the flask on the shaker and shake for 30 minutes.

Make up to volume with water and mix thoroughly.

7.3 *Preparation of the test solution*

Filter immediately into a clean, dry flask. Stopper the flask. Carry out the determination immediately after filtering.

Note: If the filtrate gradually becomes cloudy, make another extraction following 7.1 and 7.2 in a flask of volume Ve (250 or 500 ml — see table). Filter into a previously dried volumetric flask of volume W containing 5.00 ml of diluted hydrochloric acid (4.1). Stop the filtration at the exact moment when the calibration mark is reached. Mix thoroughly.

Under these conditions the value of V in the expression of results is:

$$V = Ve \times W/(W - 5).$$

The dilutions in the expression of results depend on this value of V.

8. DETERMINATION

The determination of each trace element is 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 by using Method 27c. In the case of determination by atomic absorption spectrophotometry, such removal may not be necessary.

27c. REMOVAL OF ORGANIC COMPOUNDS FROM FERTILISER EXTRACTS

1. SCOPE

This method defines a procedure for removing organic compounds from fertiliser extracts.

2. FIELD OF APPLICATION

This procedure is applicable to analysing samples of all fertilisers extracted by Methods 27a and 27b for which a declaration of the total and/or water-soluble element is required.

Note: The presence of small quantities of organic matter usually does not affect determinations by means of atomic absorption spectrophotometry.

3. PRINCIPLE

The organic compounds in an aliquot portion of the extract are oxidized with hydrogen peroxide.

4. REAGENTS

4.1 Diluted hydrochloric acid (HCl) solution, approximately 0.5 M:

Mix one volume of hydrochloric acid (ρ = 1.18 g/ml) with 20 volumes of water.

4.2 Hydrogen peroxide solution (30% H_2O_2 (100 volumes), $\rho = 1.11$ g/ml), free from trace elements.

5. APPARATUS

Electric hot plate with variable temperature control.

6. PROCEDURE

Pipette 25 ml of the extract solution obtained by Method 27a or Method 27b into a 100 ml beaker. In the case of Method 27b add 5 ml of the dilute hydrochloric acid solution (4.1). Then add 5 ml of the hydrogen peroxide solution (4.2). Cover with a watch glass. Allow oxidation to occur at room temperature for approximately one hour, then bring gradually to boiling and boil for half an hour. If necessary, add a further 5 ml of the hydrogen peroxide to the solution once it has cooled. Then boil to remove the excess hydrogen peroxide. Allow to cool and transfer quantitatively to a 50 ml volumetric flask and make up to volume. Filter where necessary.

Account should be taken of this dilution when taking aliquot portions and calculating the percentage of trace element in the product.

27d. DETERMINATION OF TRACE ELEMENTS IN FERTILISER EXTRACTS BY ATOMIC ABSORPTION SPECTROPHOTOMETRY (GENERAL PROCEDURE)

1. SCOPE

This method defines a general procedure for determining the levels of certain trace elements in fertiliser extracts by atomic absorption spectrophotometry.

2. FIELD OF APPLICATION

This procedure is applicable to analysing samples of all fertilisers extracted by Methods 27a and 27b for which a declaration of the total and/or water-soluble element is required.

Adaptations of this procedure for the various trace elements are detailed in the methods defined specifically for each element.

Note: In most cases the presence of small quantities of organic matter will not affect determinations by atomic absorption spectrophotometry.

3. PRINCIPLE

After the extract has been treated where necessary to reduce or eliminate interfering chemical species, the extract is diluted so that its concentration is in the optimum range of the spectrophotometer set to a wave-length suitable for the trace element to be determined.

4. REAGENTS

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

Mix one volume of hydrochloric acid ($\rho = 1.18$ g/ml) with 1 volume of water.

4.2 Diluted hydrochloric acid solution (HCl), approximately 0.5 M:

Mix one volume of hydrochloric acid ($\rho = 1.18$ g/ml) with 20 volumes of water.

4.3 Lanthanum salt solutions (10 g of La per litre).

This reagent is used in the determinations of cobalt, iron, manganese and zinc. It can be prepared either:

(*a*) with lanthanum oxide dissolved in hydrochloric acid (4.1). Place 11.73 g of lanthanum oxide (La_2O_3) in 150 ml of water in a 1 litre volumetric flask and add 120 ml of 6 M hydrochloric acid (4.1). Allow to dissolve and then make up to 1 litre with water and mix thoroughly. This solution is approximately 0.5 M in hydrochloric acid; or

(*b*) with solutions of lanthanum chloride, sulphate or nitrate. Dissolve 26.7 g of lanthanum chloride heptahydrate ($LaCl_3 . 7H_2O$) or 31.2 g of lanthanum nitrate hexahydrate [$La(NO_3)_3 . 6H_2O$] or 26.2 g of lanthanum sulphate nonahydrate [$La_2(SO_4)_3 . 9H_2O$] in 150 ml of water, then add 85 ml of 6 M hydrochloric acid (4.1). Allow to dissolve and then make up to 1 litre with water. Mix thoroughly. This solution is approximately 0.5 M in hydrochloric acid.

4.4 *Calibration solutions*

For the preparation of these, see the individual method of determination for each trace element.

5. APPARATUS

Atomic absorption spectrophotometer fitted with sources emitting radiation characteristic of the trace elements to be determined.

The analyst must follow the manufacturer's instructions and be familiar with the apparatus. The apparatus must allow background correction so that it can be used whenever necessary (Co and Zn). The gases to be used are air and acetylene.

6. PREPARATION OF THE SOLUTION TO BE ANALYSED

6.1 Preparation of extract solutions of the trace elements to be determined.

See Methods 27a and/or 27b and, if appropriate, 27c.

6.2 *Treatment of the test solution*

Dilute an aliquot portion of the extract obtained by Method 27a, 27b or 27c with water and/or hydrochloric acid (4.1) or (4.2) so as to obtain, in the final solution for measurement, a concentration of the element the level of which is to be determined that is appropriate to the calibration range used (7.2) and a hydrochloric acid concentration of at least 0.5 M and not more than 2.5 M. This operation may require one or more successive dilutions.

Take an aliquot portion of the final solution obtained by dilution of the extract, let (a) be its volume in ml, and pour into a 100 ml volumetric flask. Add 10 ml of the lanthanum salt solution (4.3) when determining the cobalt, iron, manganese or zinc content. Make up to volume with 0.5 M hydrochloric acid solution (4.2) and mix thoroughly. This is the final solution for measurement. Let D be the dilution factor.

7. PROCEDURE

7.1 *Preparation of a blank solution*

Prepare a blank solution by repeating the whole procedure from the extraction stage, omitting only the test sample of fertiliser.

7.2 *Preparation of calibration solutions*

From the working calibration solution prepared using the method given for each individual trace element, prepare in 100 ml volumetric flasks a series of at least five calibration solutions of increasing concentration within the optimum measuring range of the spectrophotometer. If necessary, adjust the concentration of hydrochloric acid to bring it as close as possible to that of the diluted test solution (6.2). Add 10 ml of the same lanthanum salt solution (4.3) as used in 6.2 for determining cobalt, iron, manganese or zinc. Make up to volume with the 0.5 M hydrochloric acid solution (4.2) and mix thoroughly.

7.3 *Determination*

Prepare the spectrophotometer (5) for the determination and adjust to the wavelength given in the method for the individual trace element concerned.

Spray three times in succession the calibration solutions (7.2), and the test solution (6.2) and the blank solution (7.1), noting each result and flushing the instrument with distilled water between individual sprayings.

Construct the calibration curve by plotting the average spectrophotometer reading for each calibration solution (7.2) along the ordinate and the corresponding concentration of the element, expressed in μg per ml, along the abscissa.

From this curve, determine the concentrations of relevant trace element in the test solution x_s (6.2) and in the blank solution x_b (7.1), expressing these concentrations in μg per ml.

8. Expression of Results

The percentage of trace element (E) in the fertiliser is given by:

$$E\% = [(x_s - x_b) \times V \times D]/(M \times 10^4).$$

If Method 27c has been used:

$$E\% = [(x_s - x_b) \times V \times 2D]/(M \times 10^4),$$

where:

- E is the amount of the trace element determined, expressed as a percentage of the fertiliser;
- x_s is the concentration in μg/ml of the element in the test solution (6.2);
- x_b is the concentration in μg/ml of the element in the blank solution (7.1);
- V is the volume of the extract obtained by Method 27a or 27b, in ml;
- D is the factor corresponding to the dilution carried out in (6.2);
- M is the mass of the test sample taken in accordance with Method 27a or 27b, in grams.

Calculation of dilution factor D:

If (a1), (a2), (a3), ., ., ., (ai) and (a) are the aliquot portions and (v1), (v2), (v3), ., ., ., (vi) and (100) are the volumes in ml corresponding to their respective dilutions, the dilution factor D will be equal to:

$$D = (v1/a1) \times (v2/a2) \times (v3/a3) \times . \times . \times . \times (vi/ai) \times (100/a).$$

27e. DETERMINATION OF BORON IN FERTILISER EXTRACTS BY MEANS OF SPECTROPHOTOMETRY WITH AZOMETHINE-H

1. SCOPE

This method describes a procedure for determining boron in fertiliser extracts.

2. FIELD OF APPLICATION

This procedure is applicable to analysing samples of all fertilisers extracted by Methods 27a and 27b for which a declaration of the total and/or water-soluble boron is required.

3. PRINCIPLE

In an azomethine-H solution, borate ions form a yellow complex, the concentration of which is determined by molecular absorption spectrophotometry at 410 nm. Interfering ions are masked with EDTA.

4. REAGENTS

4.1 EDTA buffer solution

Place in a 500 ml volumetric flask containing 300 ml of water:

— 75 g of ammonium acetate (NH_4OOCCH_3);

— 10 g of disodium salt of ethylene diamine tetraacetic acid (Na_2EDTA);

— 40 ml of acetic acid (CH_3COOH, $\rho = 1.05$ g/ml).

Make up to volume with water and mix thoroughly. The pH of the solution, checked by means of a glass electrode, must be 4.8 ± 0.1.

4.2 *Azomethine-H solution*

Place in a 200 ml volumetric flask

— 10 ml of the buffer solution (4.1);

— 400 mg of azomethine-H ($C_{17}H_{12}NNaO_8S_2$);

— 2 g of ascorbic acid ($C_6H_8O_6$).

Make up to volume and mix thoroughly. Do not prepare large quantities of this reagent as it is stable for only a few days.

4.3 *Boron calibration solutions*

4.3.1 Boron stock solution (100 μg/ml)

Dissolve 0.5719 g of boric acid (H_3BO_3) in water in a 1000 ml volumetric flask. Make up to volume with water and mix thoroughly. Transfer to a plastic bottle for storage in a refrigerator.

4.3.2 Boron working solution (10 μg/ml)

Place 50 ml of stock solution (4.3.1) in a 500 ml volumetric flask. Make up to volume with water and mix thoroughly.

5. APPARATUS

Spectrophotometer fitted for molecular absorption and set to a wavelength of 410 nm with cells having a 10 mm optical path.

6. PREPARATION OF THE SOLUTION TO BE ANALYSED

6.1 *Preparation of the boron solution*

See Methods 27a and/or 27b and, if appropriate, 27c.

6.2 *Preparation of the test solution*

Dilute an aliquot portion of extract (6.1) to obtain a boron concentration as specified in 7.2. Two successive dilutions may be necessary. Let D be the dilution factor.

6.3 *Preparation of the correct solution*

If the test solution (6.2) is coloured, prepare a corresponding correction solution by placing in a plastic flask 5 ml of test solution (6.2), 5 ml of EDTA buffer solution (4.1) and 5 ml of water and mix thoroughly.

7. PROCEDURE

7.1 *Preparation of the blank solution*

Prepare a blank solution by repeating the whole procedure from the extraction stage, omitting only the test sample of fertiliser.

7.2 *Preparation of the calibration solutions*

Transfer 0, 5, 10, 15, 20 and 25 ml of the working calibration solution (4.3.2) to a series of 100 ml volumetric flasks. Make up to 100 ml with water and mix thoroughly. These solutions contain between 0 and 2.5 μg/ml of boron.

7.3 *Colour development*

Transfer 5 ml of the calibration solutions (7.2), test solutions (6.2) and blank (7.1) to a series of plastic flasks. Add 5 ml of the EDTA buffer solution (4.1). Add 5 ml of the azomethine-H solution (4.2).

Mix thoroughly and allow the colour to develop in the dark for 2½ to three hours.

7.4 *Determination*

Measure the absorbance of the solutions obtained at 7.3 and if appropriate the correction solution (6.3) against water at a wavelength of 410 nm. Rinse the cells before each new reading.

8. EXPRESSION OF RESULTS

Plot a calibration curve of the concentration of the calibration solutions (7.2) along the abscissa and the absorbance given by the spectrophotometer (7.4) along the ordinate.

Read off the calibration curve the concentration of boron in the blank (7.1), the concentration of boron in the test solution (6.2) and, if the test solution is coloured, the corrected concentration of the test solution. To calculate the latter, subtract the absorbance of the correction solution (6.3) from the absorbance of the test solution (6.2) and determine the corrected concentration of the test solution. Note the concentration of the test solution (6.2), with or without correction, (x_s) and of the blank (x_b).

The percentage of boron in the fertiliser is given by:

$$B\% = [(x_s - x_b) \times V \times D]/(M \times 10^4).$$

If Method 27c is used:

$$B\% = [(x_s - x_b) \times V \times 2D]/(M \times 10^4)$$

where:

B is the quantity of boron expressed as a percentage of the fertiliser;

x_s is the concentration in μg/ml of the element in the test solution (6.2), with or without correction;

x_b is the concentration in μg/ml of the element in the blank solution (7.1);

V is the volume in ml of extract obtained in accordance with Method 27a or 27b;

D is the factor corresponding to the dilution carried out in (6.2);

M is the mass in grams of the test sample taken in accordance with Method 27a or 27b.

Calculation of dilution factor D: if (a1) and (a2) are successive aliquot portions and (v1) and (v2) are the volumes corresponding to their respective dilutions, the dilution factor is given by:

$$D = (v1/a1) \times (v2/a2).$$

27f. DETERMINATION OF COBALT IN FERTILISER EXTRACTS BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

1. SCOPE

This method describes a procedure for determining cobalt in fertiliser extracts.

2. FIELD OF APPLICATION

This procedure is applicable to analysing samples of all fertilisers extracted by Methods 27a and 27b for which a declaration of the total and/or water-soluble cobalt is required.

3. PRINCIPLE

After suitable treatment and dilution of the extracts, the cobalt content is determined by atomic absorption spectrophotometry.

4. REAGENTS

4.1 Hydrochloric acid solution, approximately 6 M.
See Method 27d (4.1).

4.2 Hydrochloric acid solution, approximately 0.5 M.
See Method 27d (4.2).

4.3 Lathanum salt solutions (10 g of La per litre)
See Method 27d (4.3).

4.4 *Cobalt calibration solutions*

4.4.1 Cobalt stock solution (1,000 μg/ml)
In a 250 ml beaker, weigh to the nearest 0.1 mg, 1 g of cobalt, add 25 ml of 6 M hydrochloric acid (4.1) and heat on a hotplate until the cobalt is completely dissolved. When cool, transfer quantitatively to a 1,000 ml volumetric flask. Make up to volume with water and mix thoroughly.

4.4.2 Cobalt working solution (100 μg/ml)
Place 10 ml of the stock solution (4.4.1) in a 100 ml volumetric flask. Make up to volume with 0.5 M hydrochloric acid solution (4.2) and mix thoroughly.

5. APPARATUS

Spectrophotometer fitted for atomic absorption, with a cobalt lamp, set at 240.7 nm: see Method 27d (5). The spectrophotometer must allow background correction to be made.

6. PREPARATION OF THE SOLUTION TO BE ANALYSED

6.1 *Cobalt extract solution*

See Methods 27a and/or 27b and, if appropriate, 27c.

6.2 *Preparation of the test solution*

See Method 27d (6.2). The test solution must contain 10% (v/v) of a lanthanum salt solution (4.3).

7. PROCEDURE

7.1 *Preparation of blank solution*

See Method 27d (7.1). The blank must contain 10% (v/v) of the lanthanum salt solution used in 6.2.

7.2 *Preparation of calibration solutions*

See Method 27d (7.2).

For an optimum determination range of 0 to 5 μg/ml of cobalt, place 0, 0.5, 1, 2, 3, 4 and 5 ml respectively of working solution (4.4.2) in a series of 100 ml volumetric flasks. If necessary adjust the hydrochloric acid concentration as closely as possible to that of the test solution. Add to each flask 10 ml of the lanthanum salt solution used in 6.2. Make up to 100 ml with 0.5 M hydrochloric acid solution (4.2) and mix thoroughly. These solutions contain 0, 0.5, 1, 2, 3, 4 and 5 μg/ml respectively of cobalt.

7.3 *Determination*

See Method 27d (7.3). Prepare the spectrophotometer (5) for measurement at a wavelength of 240.7 nm.

8. EXPRESSION OF RESULTS

See Method 27d (8).

The percentage of cobalt in the fertiliser is given by:

$$Co\% = [(x_s - x_b) \times V \times D]/(M \times 10^4)$$

If Method 27c is used:

$$Co\% = [(x_s - x_b) \times V \times 2D]/(M \times 10^4)$$

where:

Co is the quantity of cobalt expressed as a percentage of the fertiliser;

x_s is the concentration in μg/ml of the element in the test solution (6.2);

x_b is the concentration in μg/ml of the element in the blank solution (7.1);

V is the volume in ml of extract obtained in accordance with Method 27a or 27b;

D is the factor corresponding to the dilution carried out in (6.2);

M is the mass in grams of the test sample taken in accordance with Method 27a or 27b.

Calculation of the dilution factor D: if (a1), (a2), (a3), ., ., ., (ai) and (a) are aliquot portions and (v1), (v2), (v3), ., ., ., (vi) and (100) are the volumes in ml corresponding to their respective dilutions, the dilution factor D is given by:

$$D = (v1/a1) \times (v2/a2) \times (v3/a3) \times . \times . \times . \times . \times (vi/ai) \times (100/a).$$

27g. DETERMINATION OF COPPER IN FERTILISER EXTRACTS BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

1. SCOPE

This method describes a procedure for determining copper in fertiliser extracts.

2. FIELD OF APPLICATION

This procedure is applicable to analysing samples of all fertilisers extracted by Methods 27a and 27b for which a declaration of the total and/or water-soluble copper is required.

3. PRINCIPLE

After suitable treatment and dilution of the extracts, the copper content is determined by atomic absorption spectrophotometry.

4. REAGENTS

4.1 Hydrochloric acid solution, approximately 6 M

See Method 27d (4.1).

4.2 Hydrochloric acid solution, approximately 0.5 M

See Method 27d (4.2).

4.3 Hydrogen peroxide solution (30% H_2O_2 (100 Volumes), $\rho = 1.11$ g/ml), free from trace elements.

4.4 *Copper calibration solutions*

4.4.1 Copper stock solution (1,000 μg/ml)

In a 250 ml beaker, weigh to the nearest 0.1 mg, 1 g of copper, add 25 ml of 6 M hydrochloric acid (4.1) and 5 ml hydrogen peroxide solution (4.3) and heat on a hotplate until the copper is completely dissolved. Transfer quantitatively to a 1,000 ml volumetric flask. Make up to volume with water and mix thoroughly.

4.4.2 Copper working solution (100 μg/ml)

Place 20 ml of the stock solution (4.4.1) in a 200 ml volumetric flask. Make up to volume with 0.5 M hydrochloric acid solution (4.2) and mix thoroughly.

5. APPARATUS

Spectrophotometer fitted for atomic absorption, with a copper lamp, set at 324.8 nm: see Method 27d (5).

6. PREPARATION OF THE SOLUTION TO BE ANALYSED

6.1 *Copper extract solution*

See Methods 27a and/or 27b and, if appropriate, 27c.

6.2 *Preparation of the test solution*

See Method 27d (6.2).

7. PROCEDURE

7.1 *Preparation of blank solution*

See Method 27d (7.1).

7.2 *Preparation of calibration solutions*

See Method 27d (7.2).

For an optimum determination range of 0 to 5 μg/ml of copper, place 0, 0.5, 1, 2, 3, 4 and 5 ml respectively of working solution (4.4.2) in a series of 100 ml volumetric flasks. If necessary adjust the hydrochloric acid concentration as closely as possible to that of the test solution (6.2). Make up to 100 ml with 0.5 M hydrochloric acid solution (4.2) and mix thoroughly. These solutions contain 0, 0.5, 1, 2, 3, 4 and 5 μg/ml respectively of copper.

7.3 *Determination*

See Method 27d (7.3). Prepare the spectrophotometer (5) for measurement at a wavelength of 324.8 nm.

8. EXPRESSION OF RESULTS

See Method 27d (8).

The percentage of copper in the fertiliser is given by:

$$Cu\% = [(x_s - x_b) \times V \times D]/(M \times 10^4)$$

If Method 27c is used:

$$Cu\% = [(x_s - x_b) \times V \times 2D]/(M \times 10^4)$$

where:

Cu is the quantity of copper expressed as a percentage of the fertiliser;

x_s is the concentration in μg/ml of the element in the test solution (6.2);

x_b is the concentration in μg/ml of the element in the blank solution (7.1);

V is the volume in ml of extract obtained in accordance with Method 27a or 27b;

D is the factor of the dilution carried out in (6.2);

M is the mass in grams of the test sample taken in accordance with Method 27a or 27b.

Calculation of dilution factor D: if (a1), (a2), (a3) , ., ., ., (ai) and (a) are the aliquot portions and (v1), (v2), (v3),.,.,., (vi) and (100) are the volumes in ml corresponding to their respective dilutions, the dilution factor D is given by:

$$D = (v1/a1) \times (v2/a2) \times (v3/a3) \times . \times . \times . \times (vi/ai) \times (100/a).$$

27h. DETERMINATION OF IRON IN FERTILISER EXTRACTS BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

1. SCOPE

This method describes a procedure for determining iron in fertiliser extracts.

2. FIELD OF APPLICATION

This procedure is applicable to analysing samples of all fertilisers extracted by Methods 27a and 27b for which a declaration of the total and/or water-soluble iron is required.

3. PRINCIPLE

After suitable treatment and dilution of the extract, the iron content is determined by atomic absorption spectrophotometry.

4. REAGENTS

4.1 Hydrochloric acid solution, approximately 6 M
See Method 27d (4.1).

4.2 Hydrochloric acid solution, approximately 0.5 M
See Method 27d (4.2).

4.3 Hydrogen peroxide solution (30% H_2O_2 (100 Volumes), $\rho = 1.11$ g/ml), free from trace elements.

4.4 Lanthanum salt solutions (10 g of La per litre)
See Method 27d (4.3).

4.5 *Iron calibration solutions*

4.5.1 Iron stock solution (1,000 μg/ml)
In a 500 ml beaker, weigh to the nearest 0.1 mg, 1 g of pure iron wire, add 200 ml of 6 M hydrochloric acid (4.1) and 15 ml of hydrogen peroxide solution (4.3). Heat on a hotplate until the iron is completely dissolved. When cool, transfer quantitatively to a 1,000 ml volumetric flask. Make up to volume with water and mix thoroughly.

4.5.2 Iron working solution (100 μg/ml)
Place 20 ml of the stock solution (4.5.1) in a 200 ml volumetric flask. Make up to volume with the 0.5 M hydrochloric acid solution (4.2) and mix thoroughly.

5. APPARATUS
Spectrophotometer fitted for atomic absorption, with an iron lamp, set at 248.3 nm: see Method 27d (5).

6. PREPARATION OF THE SOLUTION TO BE ANALYSED

6.1 *Iron extract solution*
See Methods 27a and/or 27b and, if appropriate, 27c.

6.2 *Preparation of the test solution*
See Method 27d (6.2). The test solution must contain 10% (v/v) of a lanthanum salt solution.

7. PROCEDURE

7.1 *Preparation of blank solution*
See Method 27d (7.1). The test solution must contain 10% (v/v) of the lanthanum salt solution used in 6.2.

7.2 *Preparation of calibration solutions*
See method 27d (7.2).
For an optimum determination range of 0 to 10 μg/ml of iron, place 0, 2, 4, 6, 8 and 10 ml respectively of working solution (4.5.2) in a series of 100 ml volumetric flasks. If necessary adjust the hydrochloric acid concentration as closely as possible to that of the test solution. Add 10 ml of the lanthanum salt solution used in 6.2. Make up to volume with 0.5 M hydrochloric acid solution (4.2) and mix thoroughly. These solutions contain 0, 2, 4, 6, 8 and 10 μg/ml respectively of iron.

7.3 *Determination*
See Method 27d (7.3). Prepare the spectrophotometer (5) for measurement at a wavelength of 248.3 nm.

8. EXPRESSION OF RESULTS

See Method 27d (8).

The percentage of iron in the fertiliser is given by:

$$Fe\% = [(x_s - x_b) \times V \times D]/(M \times 10^4)$$

If method 27d is used:

$$Fe\% = [(x_s - x_b) \times V \times 2D]/(M \times 10^4)$$

where:

Fe is the quantity of iron expressed as a percentage of the fertiliser;

x_s is the concentration in μg/ml of the element in the test solution (6.2);

x_b is the concentration in μg/ml of the element in the blank solution (7.1);

V is the volume in ml of extract obtained in accordance with Method 27a or 27b;

D is the factor of the dilution carried out in (6.2);

M is the mass in grams of the test sample taken in accordance with Method 27a or 27b.

Calculation of dilution factor D: if (a1), (a2), (a3), ., ., ., (ai) and (a) are the aliquot portions and (v1), (v2), (v3), ., ., ., (vi) and (100) are the volumes in ml corresponding to their respective dilutions, the dilution factor D is given by:

$$D = (v1/a1) \times (v2/a2) \times (v3/a3) \times . \times . \times . \times (vi/ai) \times (100/a).$$

27i. DETERMINATION OF MAGANESE IN FERTILISER EXTRACTS BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

1. SCOPE

This method describes a procedure for determining manganese in fertiliser extracts.

2. FIELD OF APPLICATION

This procedure is applicable to analysing samples of all fertilisers extracted by Methods 27a and 27b for which a declaration of the total and/or water-soluble manganese is required.

3. PRINCIPLE

After suitable treatment and dilution of the extracts, the manganese content is determined by atomic absorption spectrophotometry.

4. REAGENTS

4.1 Hydrochloric acid solution, approximately 6 M

See Method 27d (4.1).

4.2 Hydrochloric acid solution, approximately 0.5 M

See Method 27d (4.2).

4.3 Lanthanum salt solutions (10 g of La per litre)

See Method 27d (4.3).

4.4 *Manganese calibration solutions*

4.4.1 Manganese stock solution (1,000 μg/ml)

In a 250 ml beaker, weigh to the nearest 0.1 mg, 1 g of manganese, add 25 ml of 6 M hydrochloric acid solution (4.1). Heat on a hotplate until the

manganese is completely dissolved. When cool, transfer quantitatively to a 1,000 ml volumetric flask. Make up to volume with water and mix thoroughly.

4.4.2 Manganese working solution (100 μg/ml)

Dilute 20 ml of the stock solution (4.4.1) in the 0.5 M hydrochloric acid solution (4.2) in a 200 ml volumetric flask. Make up to volume with the 0.5 M hydrochloric acid solution (4.2) and mix thoroughly.

5. APPARATUS

Spectrophotometer fitted for atomic absorption, with a manganese lamp, set at 279.6 nm: see Method 27d (5).

6. PREPARATION OF THE SOLUTION TO BE ANALYSED

6.1 *Manganese extract solution*

See Methods 27a and/or 27b and, if appropriate, 27c.

6.2 *Preparation of the test solution*

See Method 27d (6.2). The test solution must contain 10% by volume of lanthanum salt solution (4.3).

7. PROCEDURE

7.1 *Preparation of the blank solution*

See Method 27d (7.1). The blank solution must contain 10% by volume of the lanthanum salt solution used in 6.2.

7.2 *Preparation of calibration solutions*

See Method 27d (7.2).

For an optimum determination range of 0 to 5 μg/ml manganese, place 0, 0.5, 1, 2, 3, 4 and 5 ml respectively of the working solution (4.4.2) in a series of 100 ml volumetric flasks. Where necessary, adjust the hydrochloric acid concentration to bring it as close as possible to that of the test solution. To each flask add 10 ml of the lanthanum salt solution used in 6.2. Make up to 100 ml with 0.5 M hydrochloric acid solution (4.2) and mix thoroughly. These solutions contain 0, 0.5, 1, 2, 3, 4 and 5 μg/ml respectively of manganese.

7.3 *Determination*

See Method 27d (7.3). Prepare the spectrophotometer (5) for measurements at a wavelength of 279.6 nm.

8. EXPRESSION OF RESULTS

See Method 27d (8).

The percentage of manganese in the fertiliser is given by:

$$Mn\% = [(x_s - x_b) \times V \times D]/(M \times 10^4)$$

If Method 27c is used:

$$Mn\% = [(x_s - x_b) \times V \times 2D]/(M \times 10^4)$$

where:

Mn is the quantity of manganese expressed as a percentage of the fertiliser;

x_s is the concentration in μg/ml of the element in the test solution (6.2);

x_b is the concentration in μg/ml of the element in the blank solution (7.1);

V is the volume in ml of extract obtained using Method 27a or 27b;

D is the factor corresponding to the dilution performed in (6.2);

M is the mass in grams of the test sample taken using Method 27a or 27b.

Calculation of dilution factor D: where (a1), (a2), (a3), ., ., ., (ai) and (a) are the aliquot portions and (v1), (v2), (v3), ., ., ., (vi) and (100) are the volumes in ml corresponding to their respective dilutions, dilution factor D will be equal to:

$$D = (v1/a1) \times (v2/a2) \times (v3/a3) \times . \times . \times . \times (vi/ai) \times (100/a).$$

27j. DETERMINATION OF MOLYBDENUM IN FERTILISER EXTRACTS BY SPECTROPHOTOMETRY OF A COMPLEX WITH AMMONIUM THIOCYANATE

1. Scope

This method describes a procedure for determining molybdenum in fertiliser extracts.

2. Field of Application

This procedure is applicable to analysing samples of all fertilisers extracted by Methods 27a and 27b for which a declaration of the total and/or water-soluble molybdenum is required.

3. Principle

Molybdenum$^{(v)}$ forms a complex $[MoO(SCN)_5]^{--}$ in an acid medium with SCN^- ions. The complex is extracted with n-butyl acetate. Interfering ions such as those of iron remain in the aqueous phase. The yellow-orange colour is determined by molecular absorption spectrophotometry at 470 nm.

4. Reagents

4.1 Diluted hydrochloric acid solution (HCl), about 6 M

See Method 27d (4.1).

4.2 Copper solution (70 mg/l) in 1.5 M hydrochloric acid

Dissolve 275 mg of copper sulphate ($CuSO_4 . 5H_2O$) weighed to within 0.1 mg in 250 ml of the 6 M hydrochloric acid solution (4.1) in a 1,000 ml volumetric flask. Make up to volume with water and mix thoroughly.

4.3 Ascorbic acid solution (50 g/l)

Dissolve 50 g of ascorbic acid ($C_6H_8O_6$) in water in a 1,000 ml volumetric flask. Make up to volume with water, mix thoroughly and keep in a refrigerator.

4.4 n-butyl acetate

4.5 Ammonium thiocyanate solution, 0.2 M

Dissolve 15.224 g of NH_4SCN in water in a 1,000 ml volumetric flask. Make up to volume with water; mix thoroughly and store in a dark-coloured bottle.

4.6 Stannous chloride solution (50 g/l) in 2 M hydrochloric acid

Note: This solution must be perfectly clear and prepared immediately before use. Very pure stannous chloride must be used otherwise the solution will not be clear.

To prepare 100 ml of solution, dissolve 5 g of ($SnCl_2$. $2H_2O$) in 35 ml of 6 M HCl solution (4.1). Add 10 ml of the copper solution (4.2). Make up to volume with water and mix thoroughly.

4.7 *Molybdenum calibration solutions*

4.7.1 Molybdenum stock solution (500 μg/ml)

Dissolve 0.920 g of ammonium molybdate [$(NH_4)_6Mo_7O_{24}$. $4H_2O$] weighed to within 0.1 mg in the 6 M hydrochloric acid (4.1) in a 1,000 ml volumetric flask. Make up to volume with 6 M hydrochloric acid and mix thoroughly.

4.7.2 Molybdenum intermediate solution (25 μg/ml)

Place 25 ml of the stock solution (4.7.1) in a 500 ml volumetric flask. Make up to volume with 6 M hydrochloric acid (4.1) and mix thoroughly.

4.7.3 Molybdenum working solution (2.5 μg/ml)

Place 10 ml of the intermediate solution (4.7.2) in a 100 ml volumetric flask. Make up to volume with 6 M hydrochloric acid (4.1) and mix thoroughly.

5. APPARATUS

5.1 Spectrophotometer fitted for molecular absorption, set to a wavelength of 470 nm, with cells having a 20 mm optical path.

5.2 200 or 250 ml separating funnels.

6. PREPARATION OF THE SOLUTION TO BE ANALYSED

6.1 *Molybdenum extract solution*

See Methods 27a and/or 27b and, if appropriate, 27c.

6.2 *Preparation of the test solution*

Dilute an aliquot portion of the extract (6.1) with 6 M hydrochloric acid solution (4.1) so as to obtain an appropriate molybdenum concentration. Let D be the dilution factor.

Take an aliquot portion (a) from the extract solution containing 1 to 12 μg molybdenum and place it in the separating funnel (5.2). Make up to 50 ml with the 6 M hydrochloric acid solution (4.1).

7. PROCEDURE

7.1 *Preparation of blank solution*

Prepare a blank solution by repeating the whole procedure from the extraction stage, omitting only the test sample of fertiliser.

7.2 *Preparation of a series of calibration solutions*

Prepare a series of at least six calibration solutions of increasing concentration corresponding to the optimum response range of the spectrophotometer.

For an optimum determination range of 0−12.5 μg of molybdenum, place 0, 1, 2, 3, 4 and 5 ml respectively of the working solution (4.7.3) in the separating funnels (5.2). Make up to 50 ml with 6 M hydrochloric acid (4.1). The funnels contain, respectively, 0, 2.5, 5, 7.5, 10 and 12.5 μg molybdenum.

7.3 *Development and separation of the complex*

To each separating funnel (6.2, 7.1 and 7.2), add in the following order:

— 10 ml of the copper solution (4.2);
— 20 ml of the ascorbic acid solution (4.3);
mix thoroughly and wait for two or three minutes. Then add:
— 10 ml of n-butyl acetate (4.4), using a precision pipette;
— 20 ml of the thiocyanate solution (4.5).

Shake for one minute to extract the complex into the organic phase; allow to separate; after the separation of the two phases, draw off the entire aqueous phase and discard; then wash the organic phase with:
— 10 ml of the stannous chloride solution (4.6).

Shake for one minute. Allow to separate and draw off the entire aqueous phase. Collect the organic phase in a test tube; this will make it possible to remove the drops of water in suspension.

7.4 *Determination*

Measure the absorbencies of the solutions obtained in 7.3 at a wavelength of 470 nm using the 0 μg/ml molybdenum calibration solution (7.2) as a reference.

8. EXPRESSION OF RESULTS

Construct the calibration curve by plotting the corresponding masses of molybdenum in the calibration solutions (7.2) expressed in μg along the abscissa and the corresponding values of the absorbences (7.4) given by the spectrophotometer reading along the ordinate.

From this curve determine the mass of molybdenum in the test solution (6.2) and the blank solution (7.1). These masses are designated (x_s) and (x_b) respectively.

The percentage of molybdenum in the fertiliser is given by:

$$Mo\% = [(x_s - x_b) \times V/a \times D]/(M \times 10^4)$$

If Method 27c has been used:

$$Mo\% = [(x_s - x_b) \times V/a \times 2D]/(M \times 10^4)$$

where:

Mo is the quantity of molybdenum expressed as a percentage of the fertiliser;
a is the volume in ml of the aliquot taken from the last dilute solution (6.2);
x_s is the concentration in μg of the element in the test solution (6.2);
x_b is the concentration in μg of the element in the blank solution (7.1) the volume of which corresponds to the volume (a) of the aliquot of the test solution (6.2);
V is the volume in ml of the extract solution obtained in accordance with Method 27a or 27b;
D is the factor corresponding to the dilution performed in 6.2;
M is the mass in grams of the test sample taken in accordance with Method 27a or 27b.

Calculation of the dilution factor D: where (a1), (a2), are successive aliquot portions and (v1), (v2) are the volumes corresponding to their respective dilutions, the dilution factor D will be:

$$D = (v1/a1) \times (v2/a2).$$

27k. DETERMINATION OF ZINC IN FERTILISER EXTRACTS BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

1. SCOPE

This method describes a procedure for determining zinc in fertiliser extracts.

2. FIELD OF APPLICATION

This procedure is applicable to analysing samples of all fertilisers extracted by Methods 27a and 27b for which a declaration of the total and/or water-soluble zinc is required.

3. PRINCIPLE

After suitable treatment and dilution of the extracts, the zinc level is determined by atomic absorption spectrophotometry.

4. REAGENTS

4.1 Hydrochloric acid solution, approximately 6 M

See Method 27d (4.1).

4.2 Hydrochloric acid solution, approximately 0.5 M

See Method 27d (4.2).

4.3 Lanthanum salt solutions (10 g of La per litre).

See Method 27d (4.3).

4.4 *Zinc calibration solutions*

4.4.1 Zinc stock solution (1,000 μg/ml)

In a 1,000 ml volumetric flask dissolve 1 g of zinc powder or flakes weighed to within 0.1 mg in 25 ml of 6 M hydrochloric acid (4.1). When completely dissolved make up to volume with water and mix thoroughly.

4.4.2 Zinc working solution (100 μg/ml)

In a 200 ml volumetric flask, dilute 20 ml of the stock solution (4.4.1) in 0.5 M hydrochloric acid solution (4.2). Make up to volume with the 0.5 M hydrochloric acid solution and mix thoroughly.

5. APPARATUS

Spectrophotometer fitted for atomic absorption, with a zinc lamp, set at 213.8 nm: see Method 27d (5). The spectrophotometer must allow background correction to be made.

6. PREPARATION OF THE SOLUTION TO BE ANALYSED

6.1 *Zinc extract solution*

See Methods 27a and/or 27b and, if appropriate, 27c.

6.2 *Preparation of the test solution*

See Method 27d (6.2). The test solution must contain 10% by volume of lanthanum salt solution.

7. PROCEDURE

7.1 *Preparation of the blank solution*

See Method 27d (7.1). The blank solution must contain 10% by volume of the lanthanum salt solution used in 6.2.

7.2 *Preparation of calibration solutions*

See Method 27d (7.2).

For an optimum determination range of 0 to 5 μg/ml of zinc place 0, 0.5, 1, 2, 3, 4 and 5 ml respectively, of working solution (4.4.2) in a series of 100 ml volumetric flasks. Where necessary, adjust the concentration of hydrochloric acid to bring it as close as possible to that of the test solution. Add 10 ml of the lanthanum salt solution used in (6.2) to each volumetric

flask. Make up to 100 ml with the 0.5 M hydrochloric acid solution (4.2) and mix thoroughly. These solutions contain: 0, 0.5, 1, 2, 3, 4 and 5 μg/ml respectively of zinc.

7.3 *Determination*

See Method 27d (7.3). Prepare the spectrophotometer (5) for measurements at a wavelength of 213.8 nm.

8. EXPRESSION OF RESULTS

See Method 27d (8).

The percentage of zinc in the fertiliser is given by:

$$Zn\% = [(x_s - x_b) \times V \times D]/(M \times 10^4)$$

If Method 27c has been used:

$$Zn\% = [(x_s - x_b) \times V \times 2D]/(M \times 10^4)$$

where:

Zn is the quantity of zinc expressed as a percentage of the fertiliser;
x_s is the concentration in μg/ml of the element in the test solution (6.2);
x_b is the concentration in μg/ml of the element in the blank solution (7.1);
V is the volume in ml of the extract solution obtained in accordance with Method 27a or 27b;
D is the factor corresponding to the dilution performed in (6.2);
M is the mass in grams of the test sample taken in accordance with Method 27a or 27b.

Calculation of dilution factor D: where (a1), (a2), (a3) , ., ., ., (ai) and (a) are successive aliquot portions and (v1), (v2), (v3), ., ., ., (vi) and (100) are the volumes corresponding to their respective dilutions, the dilution factor D will be:

$$D = (v1/a1) \times (v2/a2) \times (v3/a3) \times . \times . \times . \times (vi/ai) \times (100/a).''$$

EXPLANATORY NOTE

(This note is not part of the Regulations.)

These Regulations further amend the Fertilisers (Sampling and Analysis) Regulations (Northern Ireland) 1991 (''the principal Regulations''). Regulation 3 amends regulation 5(2) of the principal Regulations (methods of analysis) so that reference is made to Section D of the table in Schedule 1 to the Fertilisers Regulations (Northern Ireland) 1992. Regulation 4(1) and (3) and Schedules 1 and 2 amend Part I of Schedule 2 to the principal Regulations by adding a number of methods of analysis concerning the trace elements boron, cobalt, copper, iron, manganese, molybdenum and zinc, where the declared content is less than or equal to 10%. Regulation 4(2) makes a minor amendment relating to the expression of the results of the determination of extracted phosphorus in fertiliser.

These Regulations implement as respects Northern Ireland Commission Directive 93/1/EEC (O.J. No. L113, 7.5.93, p. 17) which amends Commission Directive 77/535/EEC (O.J. No. L213, 22.8.77, p. 1) on the approximation of the laws of the Member States relating to methods of sampling and analysis of fertilisers.

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Road Races (Spelga Hill Climb) Order (Northern Ireland) 1994

This Order, being of a temporary character, is not printed at length in this volume.

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These Orders have been exempted from printing by the Statutory Rules (Northern Ireland) Order 1979. Summaries are given in the List of Statutory Rules of a Local Character under the heading ROADS.