SCHEDULE 2

METHODS OF ANALYSIS

PART I

25d.

DETERMINATION OF TRACE ELEMENTS IN FERTILISER EXTRACTS BY ATOMIC ABSORPTION SPECTROMETRY (GENERAL PROCEDURE)

1 SCOPE

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

2 FIELD OF APPLICATION

2. This procedure is applicable to analysing samples of fertiliser extracts obtained by Methods 25a and 25b for which a declaration of 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 spectrometry.

3 PRINCIPLE

3. 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 spectrometer at a wave-length suitable for the trace element to be determined.

4 REAGENTS

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4.1. Dilute hydrochloric acid solution (HCl), about 6 M:

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

4.2. Dilute hydrochloric acid solution (HCl), about 0.5 M:

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

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

This reagent is used for determinations of cobalt, iron, manganese and zinc. Lanthanum is added to the extract to eliminate chemical interferences in the air-acetylene flame. 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, sulfate or nitrate. Place 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 sulfate nonahydrate La$_2$ (SO$_4$)$_3$.9H$_2$O] in 150 ml of water in a 1 litre volumetric flask, 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

5. Atomic absorption spectrometer 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

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

See Method 25a and/or 25b and, if appropriate 25c.

6.2. Treatment of the test solution

Dilute an aliquot portion of the extract obtained by Method 25a, 25b or 25c 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 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. When determining the cobalt, iron, manganese or zinc content, add 10 ml of the lanthanum salt solution (4.3). Make up to volume with the 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

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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 spectrometer. If necessary, adjust the concentration of hydrochloric acid to bring it as close as possible to that of the diluted test solution (6.2). For determining cobalt, iron, manganese or zinc add 10 ml of the same lanthanum salt solution (4.3) as used in 6.2. Make up to volume with the 0.5 M hydrochloric acid solution (4.2) and mix thoroughly.

7.3 Determination

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Prepare the spectrometer (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 spectrometer 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 xs (6.2) and in the blank solution xb (7.1), expressing these concentrations in µg per ml.

8 EXPRESSION OF RESULTS

8. The percentage of trace element (E) in the fertiliser is equal to:

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

If method (25c) 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 of the test solution (6.2), in µg/ml;

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

V is the volume of the extract obtained by Method 25a or 25b, 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 25a or 25b in grams.

Calculation of dilution factor D:

If (a_1), (a_2), (a_3), . . ., (a_i) and (a) are the aliquot portions and (v_1), (v_2), (v_3), . . ., (v_i) and (100) are the volumes in ml corresponding to their respective dilutions, the dilution factor D will be equal to:

$D = (v_1/a_1) \times (v_2/a_2) \times (v_3/a_3) \times ... \times (v_i/a_i) \times (100/a)$.