

SCHEDULE 2

METHODS OF ANALYSIS

PART I

26d.

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 iron and zinc 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 26a and 26b for which a declaration of total and/or water — soluble iron or zinc 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 means of 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 wavelength suitable for the trace element to be determined.

4 REAGENTS

4

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

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

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

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

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

This reagent is used for determinations of iron 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 11.73g 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.

Status: This is the original version (as it was originally made).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

Place 26.7 g of lanthanum chloride heptahydrate ($\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$) or 31.2 g of lanthanum nitrate hexahydrate ($\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$) or 26.2 g of lanthanum sulfate nonahydrate ($\text{La}_2(\text{SO}_4)_3 \cdot 9\text{H}_2\text{O}$) 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 methods of determination for each trace element.

5 APPARATUS

5. Atomic absorption spectrometer fitted with sources emitting radiation characteristic of 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 (e.g. Zn). The gases to be used are air and acetylene.

6 PREPARATION OF THE SOLUTION TO BE ANALYSED

6

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

See Method 26a and/or 26b and, if appropriate, 26c.

6.2. Treatment of the test solution

Dilute an aliquot portion of the extract obtained by Method 26a, 26b or 26c 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.

The final solution has to be obtained by placing an aliquot portion of the diluted extract in a 100 ml volumetric flask. Let the volume of this aliquot portion be (a) ml. Add 10 ml of the lanthanum salt solution (4.3). Make up to volume with 0.5 M hydrochloric acid solution (4.2) and mix thoroughly. Let D be the dilution factor.

7 PROCEDURE

7

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). Make up to volume with the 0.5 hydrochloric acid solution (4.2) and mix thoroughly.

7.3. Determination

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), 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/ml, along the abscissa.

From this curve, determine the concentrations of the 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

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

$$E(\%) = [(x_s x_b) \times V \times D] / (M \times 104)$$

If method 26c has been used:

$$E(\%) = [(x_s x_b) \times V \times 2D] / (M \times 104)$$

where:

E is the amount of the trace element determined, expressed as a percentage of the fertiliser;

x_s is the concentration of the Fe or Zn in the test solution (6.2), in µg/ml;

x_b is the concentration of the Fe or Zn in the blank solution (7.1) in µ/ml;

V is the volume of the extract obtained by Method 26a or 26b, in ml;

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

M the mass of the test sample taken in accordance with Method 26a or 26b, in grams.

Calculation of dilution factor D:

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

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