SCHEDULE 2

METHODS OF ANALYSIS

# PART I

*25f.*

*DETERMINATION OF COBALT IN FERTILISER EXTRACTS BY ATOMIC ABSORPTION SPECTROMETRY*

## 1 SCOPE

**1.** This method describes a procedure for determining cobalt in fertilisers extracts.

## 2 FIELD OF APPLICATION

**2.** This procedure is applicable to analysing samples of fertilisers extracted by Methods 25a and 25b for which a declaration of total and/or water-soluble cobalt is required.

## 3 PRINCIPLE

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

## 4 REAGENTS

**4**

**4.1.** Hydrochloric acid solution, about 6 M.

See Method 25d (4.1).

**4.2.** Hydrochloric acid solution, about 0.5 M.

See Method 25d (4.2).

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

See Method 25d (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 hot plate 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

**5.** Atomic absorption spectrometer: see Method 25d (5). The instrument must be equipped with a source of rays characteristic of cobalt (240.7 nm). The spectrometer must allow background correction to be made.

## 6 PREPARATION OF THE SOLUTION TO BE ANALYSED

**6**

**6.1.** Cobalt extract solution

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

**6.2.** Preparation of the test solution

See Methods 25d (6.2). The test solution must contain 10% (v/v) of a lanthanum salt solution (4.3).

## 7 PROCEDURE

**7**

**7.1.** Preparation of blank solution

See Method 25d (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 25d (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 25d (7.3). Prepare the spectrometer (5) for measurement at a wavelength of 240.7 nm.

## 8 EXPRESSION OF RESULTS

**8.** See Method 25d (8).

The percentage of cobalt in the fertiliser is given by:

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

If Method 25c is used:

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

where:

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

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

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

V is the volume in ml of extract obtained in accordance with Method 25a or 25b;

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 25a or 25b.

Calculation of the dilution factor D: if ($a_1$), ($a_2$), ($a_3$), . . ., ($a_i$) and (a) are aliquot portions and ($v_l$), ($v_2$), ($v_3$), . . ., ($v_i$) and (100) are the volumes in ml corresponding to their respective dilutions, the dilution factor D is given by:

D=(vl/al)×(v2/a2)×(v3/a3)x...×(vi/ai)×(100/a).