IS 13832 : 1993
(Reaffirmed 1998)

भारतीय मानक

कीटनाशी — कृषि और खाद्य पदार्थों, मिट्टी एवं पानी में अवशिष्ट ज्ञात करने की पद्धति — डाईथियोकार्बमेट्स

Indian Standard

PESTICIDE — METHOD FOR DETERMINATION OF RESIDUES IN AGRICULTURAL AND FOOD COMMODITIES, SOIL AND WATER — DITHIOCARBAMATES

UDC 664 : 543 [632·95·028 DIT]

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NEW DELHI 110002

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Price Group 2

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Residue Analysis Sectional Committee had been approved by the Food and Agriculture Division Council.

Dithiocarbamate formulations are extensively used in agriculture for the control of several fungal and bacterial diseases of plants. This standard will enable the food, health authorities and others engaged in the fields to follow a uniform test procedure for the estimation of residues of dithiocarbamates in various food commodities.

In the preparation of this standard, due consideration has been given to the maximum limits of dithocarbamate residues laid under the provisions of *Prevention of Food Adulteration Act, 1955* and the Rules framed thereunder. The test method is restricted to the prescribed level of residues.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

PESTICIDE — METHOD FOR DETERMINATION OF RESIDUES IN AGRICULTURAL AND FOOD COMMODITIES, SOIL AND WATER — DITHIOCARBAMATES

1 SCOPE

1.1 This standard prescribes the spectrophotometric method for determination of residues of any of the following dithiocarbamate residues in agricultural and food commodities:

a) Ferbam;

b) Ziram;

c) Thiram;

d) Maneb;

e) Zineb;

f) Mancozeb; and

g) Nabam.

1.2 This method has a detection limit of 0·01 μg/g (0·01 ppm).

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
11380 : 1985	Method of sampling for the determination of pesticide residues in agricultural and food commodities

3 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

4 SAMPLING

The representative samples for the purpose of estimating dithiocarbamate residues in the samples shall be drawn in accordance with IS 11380 : 1985.

5 PRINCIPLE

A representative sample of the commodity is blended with deaerated ice-water in a predetermined ratio (normally 1 : 1, *m/v*) under nitrogen and an appropriate aliquot of the homogenised material is decomposed with sulphuric acid. The evolved carbon disulphide is absorbed in Vile's reagent. The intensity of the resulting colour complex is measured spectrophotometrically at 380 nm and the absorbance compared by means of a standard curve.

6 APPARATUS

6.1 Decomposition

Absorption train (*see* Fig. 1).

6.2 Spectrophotometer

7 REAGENTS

7.1 Vile's Reagent

Dissolve 0·05 g cupric acetate, monohydrate in 25 ml water in a 1 000 ml volumetric flask. Add 800 ml ethanol, 1 ml diethylamine and 20 ml triethanolamine. Make up the volume to the mark with ethanol.

7.2 Ethanol

95 percent (*v/v*), alternatively absolute alcohol may be used.

7.3 Lead Acetate Solution

30 percent aqueous solution (*m/v*).

7.4 Disodium Ethylenedinitrotrichloro Tetra Acetate (EDTA) Solution

Dissolve 33 g EDTA in 800 ml water in a 1 000 ml volumetric flask and dilute to mark with water.

7.5 Sulphuric Acid — 10 N.

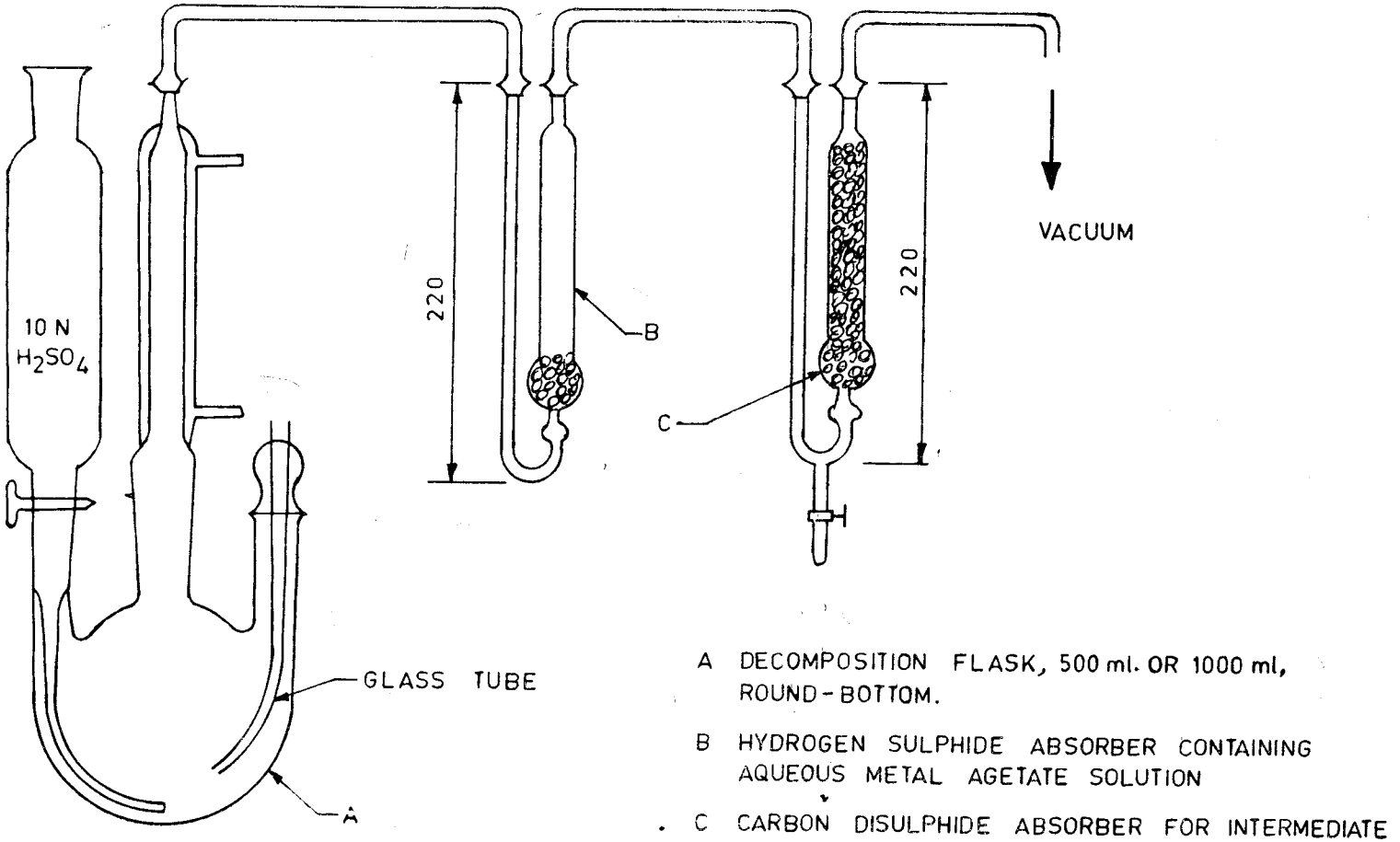
7.6 Reference Standard of the Dithiocarbamate — of known purity.

7.7 Chloroform — Glass re-distilled.

8 METHOD

8.1 Preparation of Standard Solution

Prepare a solution so as to contain 20 μg of the



All dimensions in millimetres.

FIG. 1 DECOMPOSITION-DISTILLATION APPARATUS FOR DETERMINATION OF DITHIOCARBAMATE RESIDUES IN AGRICULTURAL AND FOOD COMMODITIES

dithiocarbamate per millilitre of chloroform (*see* Notes 1, 2 and 3).

NOTES

1 If the dithiocarbamate is thiram/ferbam/ziram, 0·04 g of the active ingredient shall be dissolved in 100 ml chloroform and dilute the resultant solution to 100 ml with chloroform.

2 For maneb, mancozeb and zineb, prepare the standard solution as described in Note 1 but use EDTA solution as solvent.

3 For nabam, prepare the solution as described in Note 1/Note 2, but use water as solvent.

8.2 Preparation of Blank Solution

Take 12·5 ml Vile's reagent and add 100 ml ethanol.

8.3 Preparation of Standard Curve and Calibration

8.3.1 Transfer 10 ml standard solution of the dithiocarbamate into a 500 ml distillation flask of the decomposition-absorption train. Add 10 ml lead acetate solution to the first absorption tower and 12·5 ml Vile's reagent to the second tower. Add 200 ml water to the distillation flask and assemble the train leaving the vacuum source disconnected. Heat the flask to 85-90°C temperature, leaving the contents just short of boiling. Apply gentle vacuum continuously and add 40 ml boiling sulphuric acid through the dropping funnel and reflux for 30-45 minutes (*see* Notes 1 and 2).

NOTES

1 When chloroform is used for preparation of reference standard solution of the dithiocarbamate, remove the solvent by passing a stream of nitrogen at room temperature. This step is not necessary if EDTA or water has been used for preparation of the reference standard solution of the dithiocarbamate.

2 Use 60 ml sulphuric acid for digestion for dithiocarbamates such as maneb, zineb, and mancozeb.

8.3.2 Drain the contents of the tower containing Vile's reagent to a 25 ml volumetric flask. Wash the tower with several 3-4 ml portions of the ethanol to ensure complete quantitative transfer and collect the washings in the flask. Make up the volume to mark with ethanol.

8.3.3 Transfer separately 2·0, 3·0, 5·0 and 8·0 ml portions of the standard solution of the dithiocarbamate into the 500 ml distillation flask and follow the digestion procedure described in **8.3.1** and **8.3.2**.

8.3.4 Measure the absorbances of the standard solution (*see* **8.3.2** and **8.3.3**) in 1 cm cell at 380 nm using blank solution prepared as described in **8.2**. Prepare the standard curve by plotting the absorbances in the graph against corresponding dithiocarbamate content.

8.4 Estimation

8.4.1 Select a sample to contain 20-160 μg of the dithiocarbamate. Transfer the sample to the 500 ml distillation flask of the decomposition absorption train. Add 10 ml lead acetate solution to the first absorption tower, 12·5 ml Vile's reagent to the second tower and 200 ml water to distillation flask and digest as described in **8.3.1** (*see* Notes given in **8.4.2**).

8.4.2 Prepare the solution of the evolved carbon disulphide according to the procedure described in **8.3.2** and measure the absorbance as described in **8.3.4**.

NOTES

1 Water is added to the distillation flask if thiram/ferbam/ziram/nabam residues have to be determined.

2 200 ml EDTA is added to the distillation flask if maneb/zineb/mancozeb residues have to be determined. 60 ml sulphuric acid is added and procedures as described in **8.3.1**, **8.3.2**, **8.3.3** and **8.3.4** have to be followed.

8.4.3 Determine the dithiocarbamate residue in the sample using the appropriate calibration curve.

8.5 Calculation

$$\text{Dithiocarbamate content } (\mu g/g) = \frac{\mu g \text{ of dithiocarbamate in the sample}}{\text{Mass in g of sample taken for test}}$$

9 DETERMINATION OF RECOVERY FACTOR

9.1 Fortify 500 g of the fresh commodity (not previously treated with dithiocarbamate) with about 200 μg of the dithiocarbamate. Blend with water (1 : 1, *m/v*) to obtain a homogeneous mixture.

9.2 Weigh 200 g of the blended material (containing about 40 μg of the dithiocarbamate) in a 500 ml distillation flask. Add 100 ml of EDTA and reflux with 40-60 ml of 10 N sulphuric acid by following the procedures described in **8.3** and measure the absorbance at 380 nm.

9.3 Calculation

$$\text{Recovery factor, percent} = \frac{\text{Mass in g of the dithiocarbamate in treated sample}}{\text{Mass in g of dithiocarbamate added to material}}$$

NOTE — 85-100 percent recoveries of 0·1-70 μg/g have been observed in a variety of substrates.

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