IS 4320 (1982): Thiram, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]

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Indian Standard

SPECIFICATION FOR THIRAM, TECHNICAL

(First Revision)

UDC 632·951 THIRAM

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3

May 1983

Indian Standard

SPECIFICATION FOR THIRAM, TECHNICAL

(First Revision)

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Indian Standard

SPECIFICATION FOR THIRAM, TECHNICAL

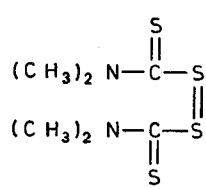
(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 10 November 1982, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Thiram, technical is used in the preparation of various formulations for the control of diseases of agricultural crops.

0.3 Thiram, is the accepted common name by the International Organization for Standardization (ISO) for the pesticide chemical containing *bis* (dimethylthiocarbamoyl) disulphide as its active ingredient. The structural and empirical formulae and the molecular mass of this compound are as indicated below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_6H_{12}N_2S_4$		240·4

0.4 This standard was first published in 1967 and subsequently one amendment was issued. As this standard was prepared more than a decade ago, it was considered desirable to issue a revised version of the standard in order to make it up to date. The present revision incorporates latest packing and marking requirements along with the modified carbon disulphide evolution method and the amine method which have been found to give comparable results.

0.5 In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act*, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS:2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for thiram, technical.

2. REQUIREMENTS

2.1 Description — The material shall be white to off white powder. It shall comprise essentially tetramethyl thiuram disulphide (TMTD) and shall be free from extraneous matter.

2.2 The material shall also comply with the requirements specified in Table 1.

TABLE 1 REQUIREMENTS FOR THIRAM, TECHNICAL

(*Clause* 2.2)

Sl No.	Characteristic	Requirements	Method of Test (Ref to Appendix)
(1)	(2)	(3)	(4)
i)	*Bis* (dimethylthiocarbamoyl) disulphide, percent by mass, *Min*	95·0	A
ii)	Loss on drying at 60°C, percent by mass, *Max*	0·5	B
iii)	Ash content, percent by mass, *Max*	0·5	C

3. PACKING AND MARKING

3.1 Packing — The material shall be packed as per requirements given in IS:8190 (Part I)-1980†.

*Rules for rounding off numerical values (*revised*).

†Requirements for packing of pesticides: Part I Solid pesitcides (*first revision*).

3.2 Marking — The containers shall bear legibly and indelibly the following information and any other information as is necessary under the Insecticides Act and Rules:

a) Name of the material;

b) Name of the manufacturer;

c) Date of manufacture;

d) Batch number;

e) Net mass of the contents;

f) Thiram content, percent (*m/m*); and

g) A cautionary notice as worded in *Insecticides Act* and Rules.

3.2.1 Each container may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The representative samples of the material shall be drawn as prescribed in the 'Indian Standard Methods for sampling of pesticides and their formulations' (*under prepration*).

NOTE — Till such time the standard under preparation is published, the samples shall be drawn as agreed to between the parties concerned.

5. TESTS

5.1 Tests shall be carried out as prescribed in the appropriate appendices referred to in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in the tests.

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

*Specification for water for general laboratory use (*second revision*).

APPENDIX A

[*Table* 1, *Item* (i)]

DETERMINATION OF THIRAM CONTENT

A-0. GENERAL

A-0.1 For the determination of thiram content, two methods, namely, the carbon disulphide evolution method (*see* **A-1**) and the amine method (*see* **A-2**) have been specified. Any one of these methods may be employed in determining thiram content but carbon disulphide evolution method shall be the referee method in case of dispute.

A-1. CARBON DISULPHIDE EVOLUTION METHOD

A-1.1 Principle — Thiram on digestion with boiling acetic acid/zinc oxide solution, yields dimethyl amine, carbon-disulphide, and a small amount of carbonyl sulphide. The gaseous mixture is carried by a stream of air through two absorbers, the first containing cadmium sulphate to remove hydrogen sulphide, and the second absorber containing methanolic potassium hydroxide to afford potassium methyl xanthate and potassium methyl monothiocarbamate which are neutralized with dilute acetic acid and titrated with standard iodine.

A-1.2 Reagents

A-1.2.1 *Acetic Acid/Zinc Oxide Solution* — Dissolve 2·5 g of zinc oxide in 100 ml of aqueous acetic acid (1:1).

A-1.2.2 *Cadmium Sulphate Solution* — 18·5 percent (*m/v*).

A-1.2.3 *Potassium Hydroxide* — 2 N in methanol.

A-1.2.4 *Dilute Acetic Acid* — 8 percent solution.

A-1.2.5 *Standard Iodine Solution* — 0·1 N.

A-1.2.6 *Starch Indicator Solution* — freshly prepared.

A-1.2.7 *Phenolphthalein Indicator Solution* — 1 percent *m/v* in ethyl alcohol.

A-1.3 Apparatus — The apparatus shall be as shown in Fig. 1.

A-1.4 Procedure — Assemble the apparatus as shown in Fig. 1 with 30 ml cadmium sulphate in the first absorber, and 25 ml methanolic potassium hydroxide in the second absorber and 5 ml in each bubblers. Maintain the water bath surrounding the first absorber at 70-80°C throughout the determination. Keep cool the second absorber containing methanolic potassium hydroxide by immersing it in a beaker of melting ice.

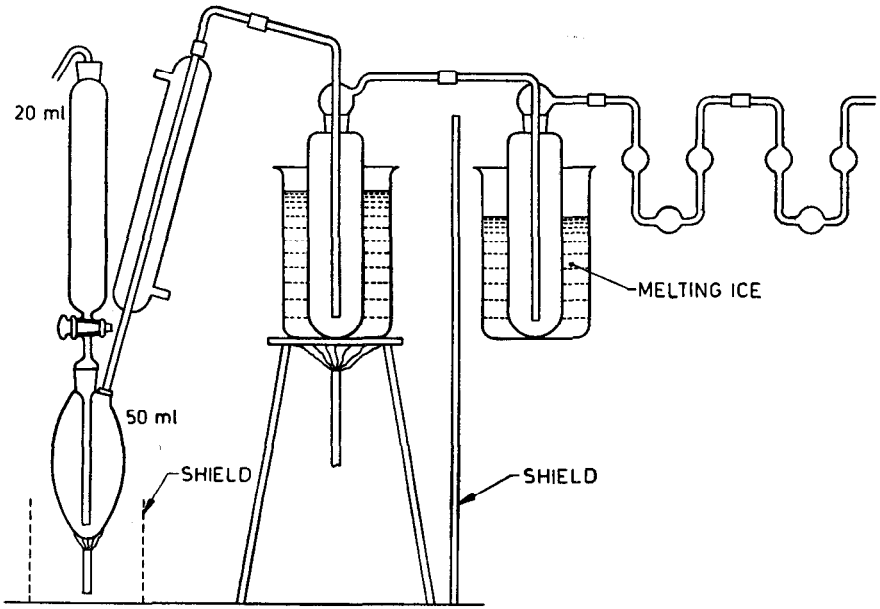


FIG. 1 APPARATUS FOR DETERMINATION OF THIRAM BY CARBON DISULPHIDE EVOLUTION METHOD

A-1.4.1 Weigh accurately a quantity of the sample containing about 0·3 g of thiram and transfer to the digestion flask. Add 20 ml of the acid mixture to the digestion flask containing thiram through the dropping funnel. Apply suction at a rate that approximately three bubbles of air per second pass through the absorbers.

A-1.4.2 Boil the contents of the flask and maintain a moderate rate of refluxing for 60 minutes. Turn off the condenser cooling water, for about 2-3 minutes, to flush the condenser and cadmium sulphate absorber with steam, to ensure complete removal of carbon disulphide (*see* Note 1 under **A-1.5**). Stop heating and immediately disconnect the absorbers.

A-1.4.3 Carefully wash the contents of the methanolic potassium hydroxide absorber and bubblers into the beaker using 300 to 400 ml distilled water. Add 2 drops of phenolphthalein, neutralize with acetic acid from a burette until the phenolphthalein, is just decolorized and add about 0·1 ml in excess. Titrate the solution immediately (*see* Note 2 under **A-1.5**), while stirring, with iodine solution nearly to the end point, add starch indicator solution and complete titration till the colour changes.

A-1.5 Calculation

$$\text{Thiram content, percent by mass} = \frac{V \times N \times 12{\cdot}02}{M}$$

where

V = volume, in ml, of standard iodine solution used for sample;

N = normality of iodine solution; and

M = mass, in g, of the sample taken for test.

NOTE 1 — The residue in the digestion flask is used for the determination of the thiram content by the amine method (*see* **A-2**).

NOTE 2 — The iodine titration must be done, immediately and rapidly, after neutralization, as decomposition of the mixed xanthate/monothiocarbamate solution is extremely rapid under acidic conditions, hence this exercise should be completed within 2 minutes.

A-2. AMINE METHOD

A-2.1 Principle — The residue obtained after digestion with acetic acid/zinc oxide solution (*see* **A-1.1**) is treated with sodium hydroxide solution, the liberated dimethyl amine is distilled off, absorbed in saturated solution of boric acid, and titrated with standard sulphuric acid solution, using a mixed bromocresol green-methyl red indicator.

A-2.2 Reagents

A-2.2.1 *Standard Sulphuric Acid Solution* — 0·1 N.

A-2.2.2 *Boric Acid* — Saturated solution.

A-2.2.3 *Sodium Hydroxide Solution* — 40 percent (*m*/*v*).

A-2.2.4 *Bromocresol Green — Methyl Red Mixed Indicator Solution* — Dissolve 0·2 g of bromocresol green and 0·05 g of methyl red in about 250 ml ethanol.

A-2.3 Apparatus — The apparatus shall be as shown in Fig. 2.

A-2.4 Procedure — Wash the residue (*see* **A-1.4.2** and Note 1 under **A-1.5**) from the digestion flask into a 500 ml distillation flask using about 250 ml distilled water. Add a little pumice powder and assemble the apparatus as shown in Fig. 2 making sure that the lip of the condenser adopter is under the surface of the saturated boric acid solution.

A-2.4.1 Add 50 ml sodium hydroxide solution slowly from the dropping funnel and boil the contents of the flask and collect the distillate in 50 ml boric acid solution. Continue distillation until the distillate is neutral to red litmus (about 20 minutes).

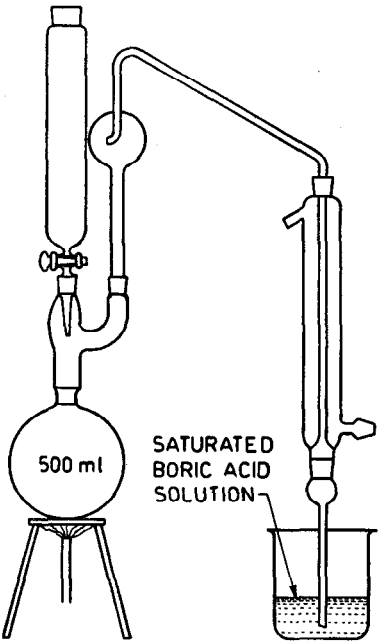


FIG. 2 APPARATUS FOR DETERMINATION OF THIRAM BY AMINE METHOD

A-2.4.2 When distillation is complete, remove the heating source, wash down the condenser walls and the adopter with small quantity of distilled water into the boric acid solution.

A-2.4.3 Titrate the solution with sulphuric acid solution using bromocresol green-methyl red mixed indicator solution to the first appearance of pink colour.

A-2.5 Calculation

$$\text{Thiram content, percent by mass} = \frac{V \times N \times 12{\cdot}02}{M}$$

where

V = volume, in ml, of standard sulphuric acid solution used;

N = normality of sulphuric acid solution; and

M = mass, in g, of the sample taken for test (*see* **A-1.4.1**).

APPENDIX B

[*Table* 1, *Item* (ii)]

DETERMINATION OF LOSS ON DRYING

B-1. PROCEDURE

B-1.1 Take about 5 g sample on a tared petri dish (5 to 6 cm diameter). Spread it over uniformly and weigh. Put the petri dish with the material in a thermostatic air-oven at 60°C for 4 hours. Cool the petri dish and the material in a desiccator and weigh. Repeat this operation till a constant weight is obtained.

B-2. CALCULATION

B-2.1 Loss on drying, percent by mass $= \frac{(m_2 - m_3)\ 100}{m_2 - m_1}$

where

m_2 = mass, in g, of the petri dish and sample before drying;

m_3 = mass, in g, of the petri dish and sample after drying; and

m_1 = mass in g of the petri dish.

APPENDIX C

[*Table* 1, *Item* (iii)]

DETERMINATION OF ASH CONTENT

C-1. PROCEDURE

C-1.1 Weigh accurately 2 to 3 g sample on a dry tared 10-ml silica basin, burn it slowly and carefully in a fume cupboard with good exhaust arrangement. The temperature should not exceed 555°C. Cool the basin and add a drop or two of concentrated sulphuric acid, heat the basin slowly to remove sulphur trioxide fumes and finally to 800°C to 900°C for about an hour. Cool the basin in a desiccator and weigh.

C-2. CALCULATION

C-2.1 Ash content, percent by mass $= \frac{m_2 - m_1}{m} \times 100$

where

m_2 = mass, in g, of the basin and sulphated ash;

m_1 = mass, in g, of the basin; and

m = mass, in g, of the sample.

(*Continued from page* 2)

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