



IS 14551 : 1998

MEGHMANI INDUSTRIES LTD.

CIN U29199GJ1993PLC019013

THIOPHANATE METHYL, TECHNICAL – SPECIFICATION

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FOREWORD

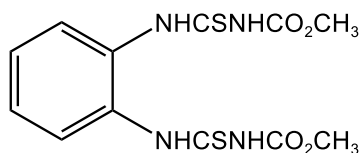
This Indian Standard was adopted by the Bureau of Indian Standard on 23 March 1990, after the draft finalized by the Pesticide Sectional Committee had been approved by the Food and Agriculture Council.

Thiophanate methyl is common name accepted by the International Organisation for Standardization (ISO) for dimethyl 4,4'(O-phenylene)bis(3-thioallophanate). The empirical and structural formulae, molecular mass and LD₅₀ values of thiophanate methyl are given below:

Empirical Formula.
C₁₂H₁₄N₄O₄S₂

Structural Formula

Molecular Mass
342.4



STRUCTURAL FORMULA

Acute Oral LD₅₀ for rats > 4987 mg/kg b.w.
Acute Dermal LD₅₀ for rats > 2000 mg/kg b.w.

Thiophanate methyl, technical is employed in the preparation of fungicide formulations.

The product has been registered for production and use in the country under the provisions of *Insecticides Act, 1968* and rules framed thereunder. In preparation of this standard, due consideration has, therefore, been given to the provision of the Act and Rules framed thereunder as well as the *Standards of Weights and Measures (Packaged Commodities) Rules, 1977*. However, this standard is subject to the restriction imposed under these wherever applicable.

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 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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ATRAZINE TECHNICAL – SPECIFICATION

1 SCOPE

This Standard prescribes the requirements and methods of sampling and test for thiophanate methyl technical.

2 REFERENCES

The following standard contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standard are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	Title
1070 : 1992	Reagent grade water (<i>Third revision</i>)
6940 : 1982	Methods of test for pesticide and their formulations (<i>first revision</i>)
8190 (Part 1) : 1988	Requirement for packing of pesticides: Part 1 Solid pesticides (<i>Second revision</i>)
10946 : 1996	Method of sampling for technical grade pesticide (<i>first revision</i>)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of grey brown powder.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING

The material shall be packed in mild steel drum, HDPE or fibreboard drum with polyethylene liner of thickness not less than 0.125 mm. It shall also confirm to the general requirements given in IS 8190 (Part 1).

5 MARKING

5.1 The drum shall be marked legibly and indelibly with the following information:

- Name of the material;
- Name and address of the manufacturer;
- Batch No. or Code No.;
- Date of expiry;
- Date of manufacture;
- Net mass in Kg;

Table 1 Requirements for Thiophanate Methyl, Technical

(Clause 3.2)

SI No	Characteristics	Requirement	Method of Tests, Ref to Annex of this Standard	CI No. of IS 6940
(1)	(2)	(3)	(4)	(5)
1	Thiophanate methyl content, percent by mass, <i>Min</i>	98.0	A	-
2	Moisture content, percent by mass, <i>Max</i>	0.5	-	4.1
3	Acidity (as H ₂ SO ₄), percent by mass, <i>Max</i>	0.5	-	11.3.2
4	Material insoluble in acetone, percent by mass, <i>Max</i>	0.5	D	9.1

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g) Thiophanate methyl content, percent (*m/m*), as registered under the provision of *Insecticides Act, 1968* and Rules framed thereunder; and
h) Any other information required under the *Insecticide Act, 1968* and Rules framed there under and *Standards of weights and measure (Packed commodities) Rules, 1977*.

5.2 BIS Certification Marking

The product may also be marked with the standard Mark.

5.2.1 The use of the Standard Mark is governed by the provision of *Bureau of Indian Standards Act,*

1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6. SAMPLING

Representative sample of the material shall be drawn according to IS 10946

7 QUALITY OF REAGENTS

Unless specified otherwise pure chemical and distilled water (*see IS 1070*) Shall be employed in Tests

ANNEX A

[Table 1, Item (i)]

DETERMINATION OF THIOPHANATE METHYL CONTENT

A-0 GENERAL

A-0.1 Either of the two methods namely, UV spectrophotometric method (*see A-1*) or HPLC method (*see A-2*) may be used for determination of thiophanate methyl content. However, in case of dispute, HPLC method will be the referee method.

A-1 UV SPECTROSCOPIC METHOD

A-1.1 Principle

The sample of thiophanate methyl is purified by passing through a silica gel column. Absorbance of the sample solution at 269 nm is compared with that of the standard of known purity to determine the content of thiophanate methyl.

A-1.2 Apparatus

A-1.2.1 UV Spectrophotometer

A-1.2.2 Quartz Cell-matched pair with path length of 1.000 cm

A-1.2.3 Volumetric Flask

A-1.2.4 Chromatography column suitable for holding about 30 ml solvent.

A-1.2.5 Long Tipped Pipette

A-1.2.6 Nitrogen cylinder with suitable regulator and tube.

A-1.3 Reagents

A-1.3.1 Acetone- Spectroscopic grade.

A-1.3.2 Ethyl Acetate- Spectroscopic grade.

A-1.3.3 *n*-Hexane- Spectroscopic grade

A-1.3.4 Methanol- Spectroscopic grade

A-1.3.5 Silica Gel- Column chromatography grade, 200 mesh particle size.

A-1.4 Procedure

A-1.4.1 Standard Preparation

Weigh accurately 100 mg of standard thiophanate methyl of known purity into a 100 ml volumetric flask, dissolve in methanol and dilute to mark with methanol. Pipette out 10 ml of this solution into a 100 ml volumetric flask and make up to mark with methanol. Pipette out 10 ml of this solution into 100 ml volumetric flask, add 7 ml developing solvent (*see A-1.4.2*) and make up the volume up to mark with methanol.

NOTE – Analytical reference of thiophanate methyl having purity more than 99 percent should be used. This can be prepared by recrystallizing the technical material thrice with fivefold quantity of hot acetone. Activated carbon may be used for removing the color.

A-1.4.2 Preparation of Developing Solvent – Mix 45 parts of ethyl acetate with 4 parts of methanol and 51 parts of *n*-hexane by volume.

A-1.4.3 Preparation of Chromatography Column

Pour into the column a slurry made from 6 g of silica gel and 20 ml of developing solvent. Drain the solvent by applying pressure of nitrogen gas till the level is just above that of the silica gel column. Wash down any carefully washing down with the

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Developing solvent so as not to disturb the silica gel column. Again drain the solvent so that its level is just above the silica gel by applying pressure of nitrogen gas.

A-1.4.4 Preparation of Sample Solution

Weigh accurately into a 50 ml volumetric flask a sample equivalent to about 175 mg thiophanate methyl. Add 40 ml of ethyl acetate and keep it for one hour at 70 °C with frequent shaking. Cool the solution to room temperature and dilute up to the mark with ethyl acetate.

A-1.4.5 Chromatography of Sample Solution

To the chromatography column prepared as in A-1.4.3 add carefully 5 ml of the sample solution (prepared in A-1.4.4) by means of a long tipped dropper. Wash the walls of the column twice with 1.5 ml of the developing solvent. Finally add 40 ml of the developing solvent without disturbing the silica gel bed and apply nitrogen pressure so that a flow rate of 2 ml/minute is maintained. Collect the elute in 50 ml volumetric flask till the solvent level in the column is just above the silica gel bed. Make up to mark with developing solvent. Again pipette out 10 ml of this solution in a 50 ml volumetric flask and make up to mark with the developing solvent.

A-1.4.6 Determination

Transfer 7 ml of the eluate obtained in A-1.4.5 to a 100 ml volumetric flask and dilute up to mark with methanol. Compare the absorbance of this solution with that of the standard solution (as prepared in A-1.4.1) at 269 nm.

A-1.5 Calculation

Thiophanate methyl content, percent by mass

$$= \frac{A_2}{A_1} \times \frac{m_1}{m_2} \times P \times \frac{25}{7}$$

Where

m_1 = mass in g of standard thiophanate methyl;
 m_2 = mass in g of sample taken for the test;
 A_1 = absorbance of standard thiophanate methyl;
 A_2 = absorbance of sample; and
 P = Purity of standard thiophanate methyl.

A-2 HPLC METHOD

A-2.1 Principle

A HPLC unit, equipped with a UV detector, is used for this assay. The solution of thiophanate methyl is compared with that of standard thiophanate methyl of known purity by using HPLC at 269 nm.

A-2.2 Apparatus

A-2.2.1 High Performance Liquid Chromatograph

A high performance liquid chromatograph equipped with printer/plotter-cum-integrator and UV detector. The suggestive HPLC operating condition are given below. However, these operating condition can be varied provided standardization is done.

Column : Lichrosorb RP-8, 10 μ m,
25 cm x 0.46 cm, Stainless steel
Or other equivalent column

Mobile phase : Acetonitrile: Methanol: Water
1:1:2 by volume

Sample size : 20 μ l

A-2.2.2 Volumetric Flask

A-2.2.3 Pipettes

A-2.3 Reagents

A-2.3.1 Acetonitrile – HPLC grade

A-2.3.2 Methanol – HPLC grade

A-2.3.3 Water – HPLC grade

A-2.3.4 Thiophanate methyl – HPLC grade

A-2.3.5 Internal Standard – HPLC grade

A-2.4 Procedure

A-2.4.1 Preparation of the Standard Solution

Weigh accurately about 125 mg of propyl-4-hydroxybenzoate into a 250 ml volumetric flask. Dissolve in 90 ml methanol and make up to the mark. Mix thoroughly. Weigh accurately about 125 mg of thiophanate methyl analytical standard into a 250 ml volumetric flask. Dissolve in 200 ml methanol and make up to mark. Mix thoroughly. Transfer by pipette 5.0 ml of this solution into a 50 ml volumetric flask, add by pipette 5 ml of internal standard solution and make up to volume with mobile phase. Mix thoroughly.

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A-2.6 Calculation

Introduce 20 µl of Standard Solution (A-2.4.1) into the HPLC system followed by sample solution (A-2.4.2). Note the areas of the thiophanate methyl and internal standard peaks. Calculate the thiophanate methyl content.

A-2.6 Calculation

Thiophanate methyl content,

$$\text{percent by mass} = \frac{A_2 \times M_1 \times A_3 \times P}{A_1 \times M_2 \times A_4}$$

where

M_1 = mass in g of thiophanate methyl standard,

M_2 = mass in g of the sample,

A_1 = area of thiophanate methyl peak in the standard,

A_2 = area of thiophanate methyl peak in the sample,

A_3 = area of internal standard peak in the standard,

A_4 = area of internal standard peak in the sample, and

P = purity of thiophanate methyl standard.

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