Doc: FAD 01(25485)WC
May 2024

BUREAU OF INDIAN STANDARDS

DRAFT FOR COMMENTS ONLY

भारतीय मानक मसौदा

डाईमेथोएट, तकनीकी — विशिष्टि

(*आइ एस 3902 का दूसरा पुनरीक्षण*)

Draft Indian Standard

DIMETHOATE, TECHNICAL — SPECIFICATION

(*Second Revision of IS 3902*)

ICS No. 65.100.10

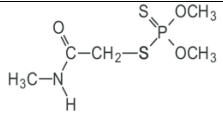
Pesticides Sectional Committee, FAD 01 **Last Date of Comments: 9 July 2024**

FOREWORD

(*Formal clauses would be added later*)

Dimethoate, technical is used in the preparation of formulations used in the control of a broad range of insect pests and mites. Dimethoate is primarily a systemic insecticide but also possesses properties of a contact insecticide and an acaricide.

Dimethoate is the accepted common name by the International Organization for Standardization (ISO) for the pesticidal chemical *O*, *O*-dimethyl *S*-(*N*-methylcarbamoylmethyl) phosphorodithioate as its active ingredient. The empirical and structural formulae and the molecular mass of this product are given below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_5H_{12}NO_3PS_2$		229.2

This standard was published in 1966 and the requirements were based on the material which was being imported into the country. With the commencement of production of dimethoate, technical in the country, the first revision of the standard was issued in 1975 and the requirements for various characteristics reviewed in the light of experience gained in the country. Also, the requirements for active ingredient content and acidity were modified.

In this revision, the standard has been brought out in the latest style and format of the Indian Standards, and references to Indian Standards wherever applicable have been updated. It also incorporates three amendments issued to the previous version of this standard.

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.

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 : 2022. 'Rules for rounding off numerical values (*second revision*)' This 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

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

2 REFERENCES

The following Indian Standards contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards 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*
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 6940 : 1982	Methods of test for pesticides and their formulations (*first revision*)
IS 8190 (Part 1) : 1988	Requirements for packing of pesticides: Part 2 Solid pesticides (*second revision*)
IS 10946 : 1996	Methods of sampling for technical grade pesticides (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall comprise *O*, *O*-dimethyl *S*-(*N*-methylcarbamoylmethyl) phosphorodithioate, in the form of white to off-white solid to semi-solid mass free from foreign impurities.

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

Table 1 Requirements of Dimethoate, Technical

(*Clause* 3.2)

Sl. No.	**Characteristic**	**Requirements**	**Method of Test, Ref to**
(1)	(2)	(3)	(4)
i)	Dimethoate content, percent by mass, *Min*	85	Annex A
ii)	Water content, percent by mass, *Max*	1.0	IS 6940
iii)	Material insoluble in acetone, percent by mass, *Max*	0.5	IS 6940
iv)	Acidity (as H_2SO_4), percent by mass, *Max*	3.0	IS 6940

4 PACKING

The material shall be packed as per requirements given in IS 8190 (Part 1).

5 MARKING

5.1 The containers shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name and address of the manufacturer;
c) Batch number;
d) Date of manufacture;
e) Date of expiry;
f) Net quantity;
g) Nominal dimethoate content, percent (*m*/*m*);
h) Cautionary notice as worded in the *Insecticides Act*, 1968, and Rules framed thereunder; and
j) Any other information required under the *Legal Metrology* (*Packaged Commodities*) *Rules*, 2011.

5.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 10946.

7 TESTS

Tests shall be carried out by appropriate methods as referred in col (4) of Table 1.

8 QUALITY OF REAGENTS

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

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

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF DIMETHOATE CONTENT

A-1 GENERAL

For the determination of dimethoate content, two methods have been prescribed. The Infrared Spectrophotometric method shall be referee method in case of dispute.

A-2 INFRARED SPECTROPHOTOMETRIC METHOD

A-2.1 Principle

The sample is dissolved in carbon disulphide. The absorbance of the extract is compared to that of the absorbance of an extract of a standard by infrared spectrophotometer.

A-2.2 Apparatus

A-2.2.1 *Infrared Spectrophotometer* **-** capable of reading in the region of 9.1 to 11.2 micron, with the slit width, gain and response time and scanning speed adjustable to produce satisfactory signal-to-noise ratio and adequate resolution under the conditions of the test.

A-2.2.2 *Absorption Cells* **–** sealed cesium iodide or potassium bromide absorption cells, having internal light path of about 0.1 mm.

A-2.2.3 *Hypodermic Syringe* **–** 5 and 2 ml capacity.

A-2.3 Reagents

A-2.3.1 *Dimethoate* **–** standard of known purity.

A-2.3.2 *Carbon Disulphide* **–** analytical grades reagent.

A-2.4 Procedure

A-2.4.1 *Calibration*

A-2.4.1.1 Weigh accurately a quantity of standard dimethoate to give a 10 mg per ml solution with carbon disulphide in a suitable volumetric flask.

A-2.4.1.2 Fill the absorption cell with carbon disulphide. Adjust the spectrophotometer for the optimum instrument setting with respect to gain, slit width, response, speed and drum drive. Make scan with carbon disulphide in the cell over the wave length region 9.1 to 11.2 micron.

A-2.4.1.3 Turn the wave length scale back to its original setting. Fill the cell with solution (*see* **A-2.4.1.1).** Make a scan in the wave length region of 9.1 to 11.2 microns, with the same setting as previously used (*see* **A-2.4.1.2).**

A-2.4.1.4 With the base line drawn from 9.1 to 11.2 microns, measure the absorbance of the solution at 9.8 micron.

A-2.4.2 *Estimation of Active Ingredient in the Material*

Weigh accurately a quantity of sample to give a 10 mg per ml solution of active ingredient with carbon disulphide in a suitable volumetric flask.

A-2.4.2.1 Make a scan of carbon disulphide and the carbon disulphide solution of the material (*see* **A-2.4.2**) as prescribed under **A-2.4.1.2** and **A-2.4.1.3** using the same cell and setting. Measure the absorbances as prescribed in **A-2.4.1.4.**

A-2.5 Calculation

$$\text{Dimethoate content, percent by mass} = \frac{A \times M}{A_1 \times M_1} \times P$$

where,

A = absorbance of the sample solution (*see* **A-2.4.2.1**);

M = mass, in g, of standard dimethoate taken for the test (*see* **A-2.4.1.1**);

P = purity of standard dimethoate;

A_1 = absorbance of the standard solution (*see* **A-2.4.1.4**); and

M_1 = mass, in g, of the sample taken for the test (*see* **A-2.4.2**).

A-3 CHROMATOGRAPHIC METHOD

A-3.1 Principle

The active ingredient is separated by liquid partition chromatography using isopropyl ether petroleum ether mixture as moving phase and water as static phase on kieselguhr. *O*, *O*, *S*-trimethyl-phosphorodithioate precedes dimethoate; dimethylphosphorodithioic acid and other more hydrophilic impurities follow the 'active' peak. Dimethoate in the eluted fraction is determined spectrophotometrically after conversion into phosphate.

A-3.1.1 The behaviour of the chromatographic column can at times be erratic. Therefore, several columns should be packed and checked before indulging in their use. The reproducibility should be determined in triplicate with at least three samples of dimethoate having different purities. The triplicate determination should agree within ± 1 percent.

A-3.2 Apparatus

A-3.2.1 *Chromatographic Column* **-** a glass tube of 14 mm inside diameter and 400 mm length constricted to 5 mm diameter at the lower end and fitted with a B 19 ground-glass joint at the upper end. A solvent reservoir of 250 ml in capacity is provided with a B 19 joint to make with the column joint.

A-3.2.2 *Pipette* **-** graduated, one millilitre capacity**.**

A-3.3 Reagents

A-3.3.1 *Hyflo-Super-Gel* **-** analytical grade.

A-3.3.2 *Petroleum Ether* **-** aromatic free, boiling range from 60 to 80 °C. Can be rendered aromatics free by shaking with concentrated sulphuric acid.

A-3.3.3 *Mixed Solvent*

Prepared by mixing equal volumes of technical isopropyl ether and petroleum ether (aromatic-free, boiling range from 60 to 80 °C) and filtering through ether-extracted cotton wool.

A-3.3.4 *Diethyl Ether* **-** reagent grade.

A-3.3.5 *Nitric Acid* **-** sp. gr. 1.42.

A-3.3.6 *Perchloric Acid* **-** 60 percent

A-3.3.7 *Ammonium Molybdate Solution* **-** prepared by dissolving 50 g of ammonium molybdate in one litre of water,

A-3.3.8 *Ammonium Vanadate Solution* **-** prepared by dissolving 2.5 g of ammonium vanadate 1:49 (*v*/*v*) nitric acid and made to one litre.

A-3.4 Procedure

A-3.4.1 *Preparation of Column*

Weigh 20 g of Hyflo-Super-Gel and transfer the same to a mortar. Add dropwise, constantly stirring, 10 ml of distilled water with the help of a pipette. Triturate gently but thoroughly to homogenize the mixture. Add sufficient quantity of the mixed solvent to form a thin slurry and mix well. Plug the bottom of the column with a small wad of ether extracted cotton wool. In order to help the easy rotation of the tube and at the same time to keep it firm in its vertical position under pressure, fix the tube with the help of two clamps; one placed just below the constriction of the tube and another near the top of the column. Pour the slurried Hyflo-Super-Gel into the tube and pack the first 0.5 cm of the column relatively firmly in order to provide a sound base fur the column. Gently lower the packer disc from a position 3 to 4 cm above the packed surface so as to compress 1 to 2 cm of the material. Rotate the tube and with short strokes and applying minimum pressure, consolidate the edge of the column taking care that only a small amount of the slurry is pushed down and no air enters into the column during operation. Repeat this process until the whole of the Hyflo-Super-Cell has been packed in 1 to 2 mm sections and the column is uniformly built. Wash the column with 100 ml of the mixed solvent applying nitrogen pressure.

A-3.4.2 *Standardization of Column*

Dissolve 1 g of dimethoate in 100 ml of ether. Transfer 1 ml of the solution to the column from a l ml graduated pipette. By applying nitrogen pressure at the top of the column, force the solvent carefully through the column until its surface coincides with the surface of the packing. Carefully release the pressure and wash the solution on to the column with two 1 ml portions of mixed solvent. Elute with mixed solvent at the rate of 2.5 to 3.0 ml per min by application of nitrogen pressure to the top of the reservoir, collecting l0 ml fractions. Transfer the fractions quantitatively to, 50 ml Kjeldahl flasks, add 5 to 7 ml water and evaporate the organic layer on a steam-bath blowing off the last traces with a hand bulb. Determine the total phosphorus as described under **A-3.4.3** from an average column) dimethoate is eluted in fraction 10, 11 and 12.

A-3.4.3 *Analysis of Sample*

A-3.4.3.1 *Preparation of solution*

Weigh 0.5 g of the technical material into a 50 ml volumetric flask. Dissolve in diethyl ether and make up to the mark with diethyl ether. Mix well and continue as given in **A-3.4.3.2**.

A-3.4.3.2 *Chromatographic separation of dimethoate*

Pipette 1 ml of the diethyl ether solution on to the column and wash in with two l ml portions of mixed solvent as described under **A-3.4.2**. Great care shall be taken to avoid errors in pipetting 1 ml of solution. The temperature of the aliquot taken shall be the same as the temperature at which the 50 ml was measured. All possibilities of evaporation of solvent before and during pipetting shall be avoided. Elute with mixed solvent at the rate of 2.5 to 3.0 ml per min collecting a 50 ml fraction as determined through standardization steps to contain all the dimethoate and separately 10 ml fractions from either side of the 50 ml fraction. Transfer the 50 ml fraction to a 250 ml conical flask, add 5 ml of water, and evaporate the solvent on a steam-bath. Blow off last traces of solvent with a hand bulb. Transfer the residue quantitatively to a 50 ml Kjeldahl flask, and 4 ml perchloric acid and 1 ml nitric acid. Evaporate to fumes of perchloric acid and reflux gently for 3 to 5 min (*see* Note 1). Cool, add 10 ml water and boil gently for about 5 min. Cool, dilute with water to about 35 ml in a 50 ml volumetric flask, add 5 ml ammonium molybdate solution. Swirl, add 5 ml ammonium molybdate solution, then dilute to the mark with water and shake.

Read the optical density against a reagent blank in a 1 cm cell at 470 nm after 30 to 40 min (*see* Note 2). Read, the equivalent phosphorus from a graph obtained using pure potassium dihydrogen phosphate.

Transfer the 10 ml fractions directly to 50 ml Kjeldahl flask, add 5 ml of water, evaporate the solvent on a steam-bath, removing the last traces of solvent with a hand bulb. Proceed exactly as given in **A-3.4.3.2** (*see* Note 3).

Note

1. The evaporation has to be carried out in a fume cupboard and behind protective plastic screens. There is an explosive hazard if the organic solvent has not been completely removed prior to this stage.
2. The reagent blank shall be checked regularly against water. The optical density should not exceed 0.010.
3. Neither of the fraction should contain phosphorus (if it does, it should be within 0.002 of reagent blank). If phosphorus is found, the column should be re-standardized and, if necessary, discarded.

A-2.5 Calculation

$$\text{Dimethoate content, percent by mass} = \frac{37 \times P}{M}$$

where,

P = mass, in mg, of phosphorus determined; and

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

.