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भारतीय मानक मसौदा

डाईमेथोएट ईमलसीफ़ाईएबल कान्सन्ट्रेट (ई सी) — विशिष्टि

(आइ एस 3903 का तीसरा पुनरीक्षण)

Draft Indian Standard

**DIMETHOATE EMULSIFIABLE CONCENTRATE (EC) —
SPECIFICATION**

(Third Revision of IS 3903)

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 emulsifiable concentrate (EC) is largely used in the control of pests of agricultural and veterinary importance. It is generally formulated to contain 30 percent (*m/m*) of dimethoate.

This standard was published in 1966 and first revised in 1975. In the second revision issued in 1984, various requirements of the standard were updated.

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 emulsifiable concentrates (EC).

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:

<i>IS No.</i>	<i>Title</i>
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 1448 (Part 20) : 2019	Methods of test for petroleum and its products: Part 20 Determination of flash point – Abel closed – Cup method (<i>third revision</i>)
IS 6940 : 1982	Methods of test for pesticides and their formulations (<i>first revision</i>)
IS 8190 (Part 2) : 1988	Requirements for packing of pesticides: Part 2 Liquid pesticides (<i>second revision</i>)
IS 10627 : 1983	Methods for sampling of pesticidal formulations

3 REQUIREMENTS

3.1 Constituent

The material shall consist of dimethoate, technical, dissolved in suitable solvent, together with emulsifying agent(s), and with or without stabilizer(s). It shall conform to IS 3902.

3.2 Physical

The material shall comply with the physical requirements specified in 3.2.1 to 3.2.4.

3.2.1 Description

The material shall be a stable homogeneous liquid and shall be free from sediment. Suspended matter shall be negligible. On dilution with water it shall readily form an emulsion, suitable for spray.

NOTE – Though initially, the product may be homogeneous, a slight separation or resinous matter might occur which settles at the bottom, but this does not in any manner affect other characteristics of the material.

3.2.2 Cold Test

No turbidity or separation of solid or oily matter or both shall occur when the material is subjected to the cold test at 10 °C as prescribed in IS 6940 or any other lower temperature as agreed to between the purchaser and the vendor. Introduction of a seeding crystal is not necessary for the test.

3.2.3 Flash Point (Abel)

When determined by the method prescribed in IS 1448 (Part 20), the flash point of the material shall be above 24.5 °C

3.2.4 Emulsion Stability

Any separation, including creaming at the top and sedimentation at the bottom of 100 ml of emulsion prepared in standard hard water with 2 ml of concentrate for agriculture use, shall not exceed 2 ml when tested by one of the methods prescribed in IS 6940.

3.3 Chemical - The material shall comply with the chemical requirements specified in 3.3.1 and 3.3.2.

3.3.1 Dimethoate Content

When determined by the method prescribed in Annex A, the observed dimethoate content, percent (*m/m*) of any of the samples shall not differ from the declared nominal value by more than tolerance limits indicated below:

<i>Nominal Value, percent</i>	<i>Tolerance limit, percent</i>	
Up to 9	+10 - 5	} of the nominal value
Above 9 and below 50	±5	
50 and above	+5 - 3	

The actual value of dimethoate content shall be calculated to two decimal places and then rounded off to one decimal place before applying the tolerance.

3.3.2 Acidity

When tested by the method prescribed in IS 6940, acidity (as H₂SO₄) shall be not more than 1.0 percent by mass.

4 PACKING

The material shall be packed as per requirements given in IS 8190 (Part 2).

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

When freshly manufactured material in bulk quantity is offered for inspection, representative samples of the material shall be drawn and tested as prescribed in IS 10627 within 90 days of its manufacture. When the material is offered for inspection after 90 days of its manufacture, sampling shall be done as prescribed in IS 10627. However, the criteria for conformity of the material when tested, shall be the limits of tolerances, as applicable over the declared nominal value and given under clause **3.3.1** of the standard.

7 TESTS

Tests shall be carried out by appropriate methods as referred in Tests shall be carried out by the appropriate methods referred to in **3.2.2** to **3.2.4**, **3.3.1** and **3.3.2**.

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 ALKALINE HYDROLYSIS METHOD

A-3.1 Principle

The procedure is based on the alkaline hydrolysis of dimethoate to form volatile methylamine which is collected and determined quantitatively by titrating with standard acid.

A-3.2 Apparatus

An assembly of the apparatus is illustrated in Fig. 1. Minor modifications in the illustrative assembly with regard to joints might be made. However, it should be ensured that all joints are leak-proof.

A-3.3 Reagents

A-3.3.1 Potassium Hydroxide Solution – 1.0 N solution in diethylene glycol.

A-3.3.2 Boric Acid Solution

Dissolve 20 g of boric acid in distilled water and dilute to 1 litre. Heat at 70 °C, swirl and cool to room temperature. Add 10 ml or 0.1 percent bromocresol green indicator and neutralise to a green end point with 0.1 N hydrochloric acid.

A-3.3.3 Standard Hydrochloric Acid Solution – 0.1 N standardized with sodium carbonate previously dried at 260 to 270 °C for 30 min.

A-3.4 Procedure

A-3.4.1 Introduce into the kettle, a quantity of sample equivalent to about 0.4 g of dimethoate accurately weighed. Introduce about 4 to 5 mg of hydrated copper sulphate into the kettle, by means of a graduated cylinder. Add 50 ml of the potassium hydroxide solution to the kettle and add a few glass beads to ensure smooth ebullition. Apply silicone grease to the joint and connect the kettle and condenser using a clamp. Before start of boiling (in order to avoid overheating, a thermostatically controlled heating mantle shall be used), ensure that the joints are secured against leakages. Check for leakages during the experiment with pH paper.

Methylamine is alkaline in nature and as such its leakage can be easily detected with pH paper 7 to 8.5 range. By means of a graduated cylinder, add to the receiver 150 ml of the boric acid solution. Clamp the nitrogen inlet tube securely to the glass inlet tube on the flask and start the nitrogen flow so as to get slow and uniform bubbling in the receiver containing boric acid. Apply sufficient heat to the kettle so that the contents start boiling. Continue boiling for 90 min. Stop heating while continuing to pass nitrogen for next 5 min.

NOTE – Heating of the kettle should be controlled so that proper refluxing is effected. Carry over of condensate to the receiver, which may result from vigorous distillation may give higher result to the extent of about one percent. So precaution should always be taken to avoid such carryover. Low heating rate results in low results for dimethoate and further heating than 90 min does not help.

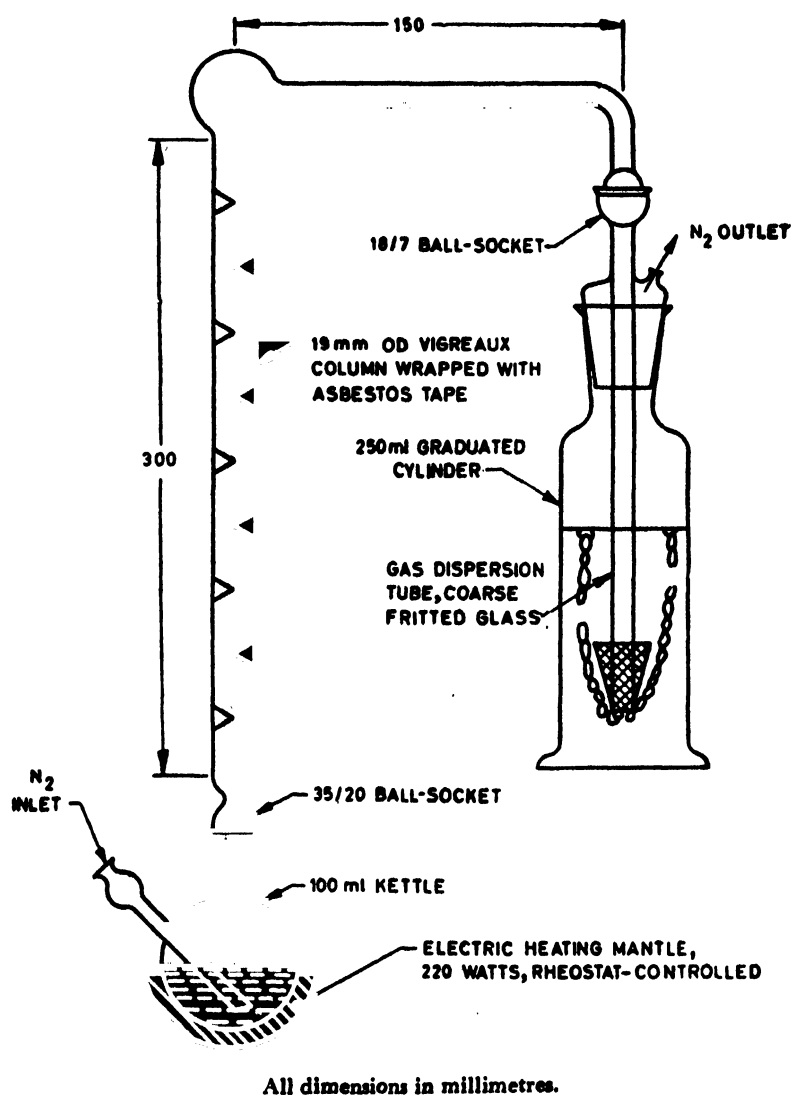


FIG 1 APPARATUS FOR DETERMINATION OF DIMETHOATE CONTENT (ALKALINE HYDROLYSIS METHOD)

A-3.4.2 Disconnect the receiver first and then nitrogen supply. Transfer the contents of the receiver into a 500 ml Erlenmeyer flask. Rinse the inside and outside of the gas delivery tube and the inside of the receiver with boric acid solution adding the washings to the Erlenmeyer flask.

A-3.4.3 Titrate the contents of the Erlenmeyer flask with the standard hydrochloric acid solution to the original green colour of the boric acid solution. The end point is best determined by comparing the colour to that of a blank solution of the boric acid and bromocresol green.

A-3.5 Calculation

$$\text{Dimethoate content, percent by mass} = \frac{22.93 \times V \times N}{M}$$

where,

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

M = mass, in g, of sample taken for test; and

N = normality of standard hydrochloric acid solution;