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भारतीय मानक मसौदा

डीडीटी इमल्सीफाइबल कॉन्सेंट्रेट (ई सी) — विशिष्टि

(15 633 का तीसरा पुनरीक्षण)

Draft Indian Standard

DDT EMULSIFIABLE CONCENTRATE (EC) - SPECIFICATION

(Third Revision of IS 633)

ICS 65.100.10

Pesticides Sectional Committee, FAD 01 Last date of comments: 20 October 2025

FOREWORD

(Adoption clauses will be added later)

DDT (Dichloro Diphenyl Trichloroethane) emulsifiable concentrate (EC) is largely used in the control of insect in public health programmes. It is generally manufactured to contain 25 percent by mass of DDT, technical.

This standard was first published in 1956 and revised in 1975. In the second revision issued in 1985, the requirements for the various characteristics were updated.

In this revision, the test method for identity test and absence of any other chlorinated products other than DDT has been included. The standard has also 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 two amendments issued to the previous version of this standard.

In year 2006 vide S.O. 295 (E) dated 8 March 2006, the use of DDT was banned in agriculture except public health programmes by Government of India.

In the preparation of this standard due consideration has been given to the provisions of the *Insecticides Act*, 1968 and the Rules framed there-under. 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*)'. 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

This standard prescribes the requirements and the methods of sampling and test for DDT emulsifiable concentrate (EC).

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provisions 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 563 : 202X | DDT, technical — Specification (third revision) [Under preparation Doc No. FAD 01(28712)WC] |
| IS 1070 : 2023 | Reagent grade water — Specification (fourth revision) |
| IS 1448 (Part 20) : 2024/ ISO 13736:2021 | Petroleum and its products - Test methods: Part 20 Determination of flash point - Abel closed - Cup method (<i>fourth revision</i>) |
| IS 6940 : 2025 | Pesticides and their formulations – Methods of test (second revision) |
| IS 8190 (Part 2): 1988 | Requirements for packing of pesticides: Part 1 Liquid pesticides (second revision) |
| IS 10627 : 1983 | Methods for sampling of pesticidal formulations |

3 REQUIREMENTS

- **3.1 Constituents** The material shall consist of DDT, technical, dissolved in suitable solvent(s), together with emulsifying agent(s) with or without stabilizer(s) and coupler (s).
- **3.1.1** DDT, technical, used in the manufacture of DDT emulsifiable concentrates shall conform to IS 563.
- **3.1.2** *Identity* The material shall comply with identity test as prescribed in Annex A and shall not contain any chlorinated pesticide other than DDT, technical.

3.2 Physical

The material shall comply with the following physical requirements.

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

- **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 at any other lower temperature as agreed to between the purchaser and the supplier. 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 5 ml of concentrate for public health use and with 2 ml of concentrate for agriculture use, shall not exceed 2.0 ml when tested by one of the methods prescribed in IS 6940.

3.3 Chemical

The material shall also comply with the following chemical requirements.

3.3.1 *DDT Content*

When determined by the method prescribed in Annex B, the observed DDT content, percent by mass, of any of the samples shall not differ from the nominal value by more than the tolerance limits indicated below:

3.3.1.1 The actual value of DDT, technical content shall be calculated to the second decimal place for rounding off to the first decimal place be-fore applying the tolerance as given in **3.3.1**.

3.3.2 *Acidity or Alkalinity*

When tested by the methods prescribed in IS 6940, the acidity, if any, calculated as sulphuric acid (H₂SO₄), or alkalinity, if any, calculated as sodium hydroxide (NaOH), shall not be more than 0.05 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 of the manufacturer;
 - c) Date of manufacture;
 - d) Batch Number;
 - e) Net mass of contents;
 - f) Nominal DDT, technical content, percent (m/m);
 - g) Cautionary notice as worded in the *Insecticides Act*, 1968, and Rules framed thereunder; and
 - h) 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 **3.3.1**.

7 TESTS

7.1 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**.

7.2 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

(*Clause* 3.1.2)

IDENTITY TEST FOR DDT

A-1 PRINCIPLE

The method is based on the thin layer chromatographic separation of DDT from other added chlorinated pesticides, if any.

A-2 APPARATUS

- A-2.1 Thin Layer Chromatography Apparatus
- **A-2.2 Ultraviolet Apparatus**
- **A-3 REAGENTS**
- **A-3.1 Adsorbent** Silica gel or aluminium oxide (G).
- A-3.2 Mobile Solvents
- **A-3.2.1** *n-hexane*
- **A-3.2.2** n-hexane + acetone (98 + 2).
- **A-3.2.3** n-hexane + alcohol (98 + 2).
- A-3.3 Standard Reference Solution
- **A-3.3.1** *Standard DDT Solution* 1 mg/1 ml.
- **A-3.3.2** *Standard BHC Solution* 1 mg/1 ml.
- **A-3.3.3** *Standard DDT* + *BHC Solutions* —1:1 (*see* **A-3.3.1** and **A-3.3.2**).
- **A-3.4** Extract of amount of sample containing 0.1 g of the active ingredient in acetone and make up the volume to 100 ml in a volumetric flask. This solution will give a concentration of 1 mg/1 ml.
- **A-3.5 Preparation of Plates** Dissolve 0.1 to 0.15 g of silver nitrate in 1 to 2 ml distilled water in 100 ml beaker, add 58 ml methyl alcohol and mix. Weigh 40 g adsorbent (*see* **A-3.1**) in 250 mi flask, add silver nitrate-methyl alcohol solution and shake vigorously for 20 sec. Apply slurry at 0.2 mm thick layer, to five 20×20 cm plates positioned on plastic mounted boards. After plates appear dry, store in desiccator over desiccant. When plates are dry, scrap 1 cm strip from side edges to ensure even solvent front. Use plates immediately after removal from desiccator.
- **A-3.6** Pour *n*-hexane into glass chromatograph tank to depth of 10 to 20 mm, place two paper blotter [7.5 x 22 cm] on each side of tank or large blotter covering back of tank and let in equilibrate approximately 2 h before use.
- **A-3.7 Detection** Spot 10 μ l sample extract with a 100 μ l syringe. Do not disturb adsorbent layer. Also spot standard solutions of DDT, BHC and mixture of DDT + BHC. Spot would be approximately less than 6 mm diameter and placed less than 30 mm from bottom of plate. Place in chromatographic tank and let plate develop approximately greater than 10 cm. Remove plate

and expose to short-wave UV. Chlorinated organic pesticides should be visible as direct spots against white or light-grey background. Expose plate for approximately 1 h. Longer exposure will not harm plate. To confirm identification of pesticides, repeat TLC with different mobile solvents (*see* **A-3.2.2** and **A-3.2.3**).

A-4 Observations — The sample shall be considered free from added chlorinated pesticide other than DDT if the chromatogram of the standard DDT and the sample are alike.

ANNEX B

(*Clause* 3.3.1)

DETERMINATION OF DDT CONTENT

B-1 GENERAL

There are two methods for the determination of the DDT, technical content, namely, the total organic chlorine method and the hydrolysable chlorine method. Whereas for routine tests both the methods are prescribed, the latter method shall be used as a referee method in case of dispute.

B-2 TOTAL ORGANIC CHLORINE METHOD

B-2.1 Reagents

- **B-2.1.1** *Isopropyl Alcohol* of two concentrations, namely, 99 percent, dry, and 50 percent (v/v), aqueous solution.
- **B-2.1.2** *Metallic Sodium* pure, in the form of ribbon or cut to small pieces.
- **B-2.1.3** *Phenolphthalein Indicator Solution* One percent (m/u) in rectified spirit.
- **B-2.1.4** *Dilute Nitric Acid* 50 percent by volume.
- **B-2.1.5** *Standard Silver Nitrate Solution* 0.1 N.
- **B-2.1.6** *Ferric Ammonium Sulphate Solution* Saturated, aqueous, freshly prepared.
- **B-2.1.7** *Standard Potassium Thiocyanate Solution* 0.1 N.
- **B-2.1.8** *Nitric Acid* Concentrated.

B-2.2 Procedure

- **B-2.2.1** Weigh accurately a quantity of the material containing about 0.1 g of DDT into a 250 ml Erlenmeyer flask. Add to it 25 ml of isopropyl alcohol (99 percent) and shake the flask to mix the contents. Add to the flask 2.5 g of metallic sodium, connect the flask to a reflux condenser and boil the contents gently for at least 2 h. Shake the flask occasionally. Dissolve the excess metallic sodium by cautiously adding 10 ml of isopropyl alcohol (50 percent) through the condenser at the rate of 1 to 2 drops per second. Boil for another 10 min and then add 60 ml of distilled water.
- **B-2.2.2** Cool, add 2 to 3 drops of phenolphthalein indicator solution. Neutralize by adding dilute nitric acid drop wise and then add 10 ml in excess. If necessary, cool the flask to room temperature; add a known volume of standard silver nitrate solution in slight excess and

coagulate the precipitated silver chloride by digesting on a steam bath for half-an-hour, with frequent stirring. Cool the flask and, if necessary, filter the contents of the flask through a fast qualitative filter paper collecting the filtrate quantitatively in Erlenmeyer flask. Add 5 ml of ferric ammonium sulphate solution either to the cooled unfiltered mixture or to the filtrate, as the case may be, and titrate the excess of silver nitrate with standard potassium thiocyanate solution. (The end point is the appearance of red ferric thiocyanate colour).

B-2.2.3 *Determination of Inorganic Chloride Content* — Weigh accurately about 1 g of the material and transfer with 100 ml distilled water to a 250 ml Erlenmeyer flask. Add 5 ml of concentrated nitric acid and 5 ml of standard silver nitrate solution. Coagulate the precipitate as before and titrate the excess silver nitrate with standard potassium thiocyanate solution. Carry out a blank determination on the reagents as before.

Inorganic chlorine content (c), percent by mass = $\frac{3.546 (B-A)N}{M}$

where,

B = volume, in ml, of standard potassium thiocyanate solution required for the blank;

A =volume, in ml, of standard potassium thiocyanate solution required for the sample;

N = normality of standard potassium thiocyanate solution; and

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

B-2.3 Calculation

DDT content, percent by mass = $\frac{7.092 (AN_1 - BN_2)}{M} - 2c$

where,

A = volume, in ml, of standard silver nitrate solution initially taken (see **B-2.2.2**);

 N_1 = normality of standard silver nitrate solution;

B = volume, in ml, of standard potassium thiocyanate solution used for titration (see **B-2.2.2**);

 N_2 = normality of standard potassium thiocyanate solution;

M = mass, in g, of the material taken for the test (see **B-2.2.1**); and

 $c = \text{inorganic chlorine content of the material, percent by the mass (see$ **B-2.2.3**).

B-3 HYDROLYSABLE CHLORINE METHOD

B-3.1 Reagents

B-3.1.1 *Acetone*

B-3.1.2 *Alcoholic Potassium Hydroxide Solution* —1 N.

B-3.1.3 *Nitric Acid* - Approximately 2 N.

B-3.1.4 *Standard Silver Nitrate Solution* — 0.1 N.

- **B-3.1.5** *Ferric Ammonium Sulphate Solution* Saturated, aqueous, freshly prepared.
- **B-3.1.6** *Standard Potassium Thiocyanate Solution* 0.1 N.

B-3.2 Procedure

B-3.2.1 Weigh accurately a quantity of the material containing about 0.5 g of DDT, into a 250 ml Erlenmeyer flask. Add 50 ml of acetone and 20 ml of alcoholic potassium hydroxide solution, keep it at 20 to 25 °C for 15 min and then add 50 ml of water. Add to the contents of the flask 20 ml of dilute nitric acid and exactly 25 ml of standard silver nitrate solution. Coagulate the precipitated silver chloride by digesting on a steam bath for half-an-hour, with frequent stirring. Cool the flask and, if necessary, filter the contents of flask through a fast qualitative filter paper, collecting the filtrate quantitatively in an Erlenmeyer flask. Add 5 ml of ferric ammonium sulphate solution either to the cooled unfiltered mixture or to the filtrate, as the case may be, and titrate the excess of silver nitrate, with standard potassium thiocyanate solution.

B-3.2.2 Carry out a blank determination using the method given under **B-3.2.1**.

B-3.3 Calculation

DDT content, percent by mass =
$$\frac{35.46 (B-A)N}{M} - 10c$$

Where,

B = volume, in ml, of standard potassium thiocyanate solution required for the blank determination (see **B-3.2.2**);

A = volume, in ml, of standard potassium thiocyanate solution required for the tests with the material (see **B-3.2.1**);

N = normality of standard potassium thiocyanate solution;

M = mass, in g, of the material taken for the test (see **B-3.2.1**); and

c = inorganic chlorine content of the material, percent by mass (see B-2.2.3).