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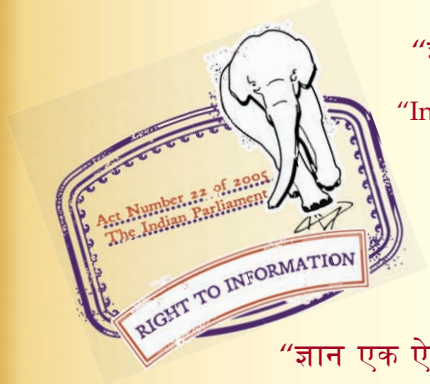
IS 6014 (1978): Emulsifiable Larvicidal Oil, Pyrethrum Based [FAD 1: Pesticides and Pesticides Residue Analysis]



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(Reaffirmed 2012)

Indian Standard

SPECIFICATION FOR EMULSIFIABLE LARVICIDAL OIL, PYRETHRUM BASED

(First Revision)

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(First Revision)

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Indian Standard

SPECIFICATION FOR EMULSIFIABLE LARVICIDAL OIL, PYRETHRUM BASED

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 31 January 1978, 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 The emulsifiable larvicidal oil is intended for use as a larvicide for mosquito abatement and control in breeding places like lakes, pools and other stagnant waters, and marshes.

0.3 Emulsifiable larvicidal oil, pyrethrin based is generally manufactured to contain 0·2 percent (*m/m*) of pyrethrin.

0.4 This standard was first issued in 1970. This has been revised to align with the statutory requirements regarding packing. This revision also incorporates certain modifications in the testing procedure based on the experience gained during its implementation.

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 the methods of sampling and tests for the emulsifiable larvicidal oil, pyrethrum based.

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

2. REQUIREMENTS

2.1 Constituents — The material shall consist of pyrethrum extract in a suitable mineral oil, and may have other additives excluding pesticides, incorporated into the oil, which increase its spreading and wetting characteristics over water and improve its larvicidal action (*see* **2.4**).

2.1.1 Pyrethrum extracts employed in the formulation of this material shall conform to IS : 1051-1980*.

2.2 Physical Requirements — The material shall comply with the physical requirements as described in **2.2.1** to **2.2.5**.

2.2.1 *Description and Identity*

2.2.1.1 *Discription* — The material shall be homogeneous liquid. Suspended matter shall be neglible.

2.2.1.2 *Identity* — The material shall comply with Identity Test as described under Appendix A of IS : 1051-1980* and shall not contain any synthetic pyrethroids. If any spots, in addition to those given by the standard reference samples are detected, it should be ensured that these additional spots are not due to any synthetic pyrethroids. If the presence of any synthetic pyrethroid is established in the TLC testing, the total pyrethrin content as described in Appendix B of IS : 1051-1980* should not be determined as synthetic pyrethroid would analyse wrongly as pyrethrins.

2.2.2 *Flash Point (Abel)* — When determined by the method prescribed in [P : 20] of IS : 1448-1960†, the flash point of the material shall be above 25°C.

2.2.3 *Emulsion Stability* — The creaming shall be between 20 ml and 50 ml when tested by any one of the methods prescribed in Appendix A.

2.2.4 *Relative Density* — The relative density of the material at 27°/27°C shall be reported by following the method as prescribed in [P : 32] of IS: 1448-1972‡.

2.2.5 *Spreading Pressure* — When determined by the method prescribed in Appendix B of IS: 588-1978§, the spreading pressure shall be more than 18×10^{-3}N/m (18 dynes/cm).

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*Specification for pyrethrum extracts (*second revision*).

†Methods of test for petroleum and petroleum products, P : 20 Flash point by Abel apparatus.

‡Methods of test for petroleum and its products, P:32 Density and relative density (*first revision*).

§Mosquito larvicidal oil (*second revision*).

2.3 Chemical Requirement — The material shall comply with the chemical requirement as described in **2.3.1.**

2.3.1 *Total Pyrethrin Content* — When determined by the method prescribed in Appendix B of IS : 1051-1980* the observed total pyrethrin content, of any of the samples shall not be less than 0·2 percent by mass.

2.4 Larvicidal Efficacy — The material shall have killing power of not less than 90 percent in case of *Anopheles stephensi* or *Anopheles fluviatilis* and not less than 80 percent in case of *Culex fatigans*, when tested by the method as prescribed in Appendix B.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed according to the requirements given in IS : 8190 (Part II)-1980*.

3.2 Marking — The containers shall bear legibly and indelibly the following information and any other information 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 volume of contents;

f) Nominal total pyrethrin content, percent (*m/m*); and

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

3.2.1 The containers 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 divised 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.

*Requirements for packing of pesticides : Part II Liquid pesticides (*first revision*).

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in IS : 10627-1983*.

5. TESTS

5.1 Tests shall be carried out by the appropriate methods referred to in **2.2.1** to **2.2.5**, **2.3.1** and **2.4.**

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

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

APPENDIX A

(*Clause* 2.2.3)

DETERMINATION OF EMULSION STABILITY

A-1. METHODS

A-1.1 Two methods are prescribed for the determination of emulsion stability of the material. The method to be employed shall depend upon the manufacturer's instructions for the preparation of diluted emulsion from the material. Method A shall be employed in the case of the material which is required to be added to standard hard water for the preparation of the diluted emulsion while Method B shall be employed in the case of a material to which standard hard water is to be added to prepare the diluted emulsion.

A-2. METHOD A

A-2.1 Reagent

A-2.1.1 *Standard Hard Water* — Dissolve 2·740 g of calcium carbonate and 0·276 g of magnesium oxide in the minimum of dilute hydrochloric acid (approximately 2 N). Expel the excess acid by evaporation to dryness on a water-bath and dissolve the residue in a small quantity of water. Transfer this solution quantitatively to a 100-ml graduated flask, make up

*Methods for sampling of pesticidal formulations.

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

the volume with water and mix. Pipette 10 ml of this solution into a one-litre graduated flask, make up the volume with water and mix.

NOTE — Standard hard water is defined as water which provides a hardness of 342 parts per million, calculated as calcium carbonate and has the following composition:

Calcium chloride, anhydrous	0·304 g
Magnesium chloride hexahydrate	0·139 g
Distilled water	To make 1 000 ml

A-2.2 Apparatus

A-2.2.1 *Beaker* — Capacity 250 ml with an internal diameter of 6·0 to 6·5 cm and marked at 100 ml.

A-2.2.2 *Mohr-Type Pipette*

A-2.2.3 *Glass Rod* — 4 to 6 mm in diameter and of a convenient length.

A-2.2.4 *Graduated Cylinder* — 100-ml capacity.

A-2.3 Procedure — Add 80 ml of the standard hard water at a temperature of 30 ± 1°C in the stoppered cylinder. With the help of the graduated pipette, add 20 ml of the material to the stoppered cylinder. Stopper the cylinder and shake vigorously for one minute. Allow it to stand undisturbed for one hour.

A-2.4 Report — Report the total volume in ml of the creamed material at the top.

A-3. METHOD B

A-3.1 Procedure — Place 20 ml of the material in the stoppered cylinder. Add 80 ml of the standard hard water (*see* **A-2.1.1**) to the cylinder. Stopper the cylinder and shake vigorously for one minute. Allow the cylinder to stand undisturbed for one hour.

A-3.2 Report — Report the total volume in ml of the creamed material at the top.

APPENDIX B

(*Clause* 2.4)

DETERMINATION OF LARVICIDAL EFFICACY

B-1. APPARATUS

B-1.1 Tray — white enamelled, top dimensions 46 × 31 × 7 cm and bottom dimensions 45 × 29·75 cm.

B-1.2 Glass Partition — of dimensions 28 × 7 cm.

B-1.3 Slotted Wooden Blocks — suitable for placing the glass partition across the tray.

B-1.4 Stainer for Larvae — suitable device for introduction and removal of larvae below the surface of water.

B-1.5 Beaker — of any convenient size.

B-2. TYPES OF INSECTS

B-2.1 The test shall be carried out using the types of insects given under **B-2.1.1** or **B-2.1.2.**

B-2.1.1 *Anopheles Stephensi or Anopheles Flviatilis*

B-2.1.2 *Culex Fatigans*

B-3. PREPARATION OF STANDARD CULTURE

B-3.1 Procedure — Confine gravid female mosquitoes, to obtain eggs, in a small cage in which petri dishes or enamel bowls with water are put in. In each bowl or dish, float a paraffin coated ring just fitting into the inner diameter of the water surface so as to provide a foothold for oviposting females and at the same time to prevent eggs from being stranded on to the sides of the vessel (it may be necessary to start the colony from a batch of eggs laid by a single mosquito. In such a case, the isolated gravid female is introduced into a specimen tube 75 × 75 mm. Plug the mouth of the tube with moist filter paper on which the oviposition usually takes place). Remove the eggs with a camel hair brush and transfer to enamel basin 350 × 400 mm containing water (during hot months earthen basins are used). Feed the larvae with dry yeast powder or any other suitable rearing food (*see* **B-3.1.1**) sprinkled on the water surface every morning. Increase the quantity of yeast as the larvae grow, avoiding overcrowding of the larvae in the rearing pans. Keep not more than 750 larvae in each pan. Change the water in the pan on alternate days. Shake the water thoroughly inside a bottle before pouring it into the pans. Collect pupae as they are formed and put them into the emergence cage. Transfer the hatched out adults into the colony cage which is made of either wood or impressed fibreboard sheets and is of box-like construction measuring 50 × 50 × 50 cm. Provide the front of the cage with wire screen with a cloth sleeve entrance (this should be situated in the lower left or right quadrant of the front walls). Maintain a temperature between 25 to 30°C and relative humidity of 70 to 80 percent inside the room and the colony cage (this is generally done by hanging a wet cloth in the room and inside the colony cage). Keep cotton pads soaked in ten percent glucose water inside the cage for male mosquitoes to feed. Put every night a rabbit with shaved back into the cage after confining the animal in a smaller cage in order to provide the

blood feed for the female mosquitoes. Put an enamel bowl with water inside the cage for oviposition. Keep the colony cage in a dark room provided with an electric lamp of 60 watts.

B-3.1.1 Mosquito larvae may also be reared with any of the following foods:

a) Powdered dog biscuit (ground to a fine powder),

b) Powdered skimmed milk,

c) Mixture of dehydrated blood serum (one part) and litmus milk (two parts),

d) Brewers' yeast,

e) Dried toast or bread crumbs powdered,

f) Hay infusion — mature (shall be aerated vigorously every day),

g) Spirogyra, and

h) Chopped flies.

B-4. CLEANING OF APPARATUS

B-4.1 Rinse the tray, glass partition and beaker, first with petroleum ether (bp 60 to 80°C) and then clean with concentrated solution of chromic acid. Again rinse with distilled water to remove the last trace of acid and finally with acetone and then air dry.

B-4.2 Clean the strainer with petroleum ether (bp 60 to 80°C).

B-5. TEST TEMPERATURE

B-5.1 The test shall be carried out at 27 ± 1°C.

B-6. PROCEDURE

B-6.1 Pour 6 000 ml of tap water in the tray. Place the slotted wooden block and the glass partition in the tray in such a manner that the surface area on one side of the tray is 675 cm^2. Adjust the glass partition in such a manner that the clearance between the lower end of the glass partition and the bottom of the tray is 3 mm. This clearance is sufficient for introduction and removal of the larvae.

B-6.2 Prepare an emulsion of the material in the following manner:

Place 80 ml of standard hard water in a 100 ml stoppered cylinder. Add with the help of pipette, 20 ml of the material to the cylinder

and form an emulsion by shaking the cylinder vigorously for one minute. Immediately pipette out 1·2 ml of the emulsion gently on to the surface of the water in the adjusted portion of the tray.

B-6.3 Transfer 20 late 3rd instar/early 4th instar larvae from the standard culture to the beaker containing distilled water so as to free them from debris and then remove them by means of the strainer to the side of the tray, which has not been treated with the material. Now transfer these larvae from this side of the tray to the other by means of the strainer so that they are under the surface of water, which has been treated with the material. Lower the glass partition further so that there is no gap between the bottom of the tray and the lower end of the partition. Allow the larvae to remain under the surface for 30 minutes. Then remove to a clean beaker containing distilled water in the same manner as they were introduced. Any larvae not under the water surface may be disturbed with a needle inducing them to go to the bottom where they may be easily caught in the strainer. Wash in tap water twice and remove to a clean beaker containing distilled water with food (a pinch of yeast) and keep under observation for 24 hours undisturbed.

NOTE — The larvae should not be accidently oiled while transferring.

B-7. RECORDING AND REPORTING

B-7.1 The larvae shall be considered as dead if they show no sign of swimming movement even after touching with a glass rod at the end of 24 hours.

B-7.2 Report the average of 5 replicates of the individual test results.

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(First Revision)

(*Page* 7, *clause* **2.4**) — Substitute the following for the existing clause:

'2.4 Optional Requirement

2.4.1 *Larvicidal Efficacy* — The material should have killing power of not less than 90 percent in case of *Anopheles stephensi* or *Anopheles fluviatilis* and not less than 80 percent in case of *Culex fatigans,* when tested by the method as prescribed in Appendix B.'

(AFCDC 6)

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(First Revision)

(*Page* 8, *clause* **4.1**) — Substitute the following for the existing:

'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 : 1983 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 : 1983. 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 **2.3.1** of the standard.'

(FAD 1)

Reprography Unit, BIS, New Delhi, India