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“I am a knowledge, I am a knowledge”
Mazdoor Kisan Shakti Sangathan
“The Right to Information, The Right to Live”

“Know the new, leave the old”
Jawaharlal Nehru
“Step Out From the Old to the New”

Indian Standard

CHLORPYRIFOS + CYPERMETHRIN EMULSIFIABLE CONCENTRATE — SPECIFICATION

ICS 65.100.99
FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Division Council.

Chlorpyrifos + cypermethrin emulsifiable concentrate are used for control of insect pests of agriculture crops.

Chlorpyrifos + cypermethrin EC formulation is generally manufactured to contain a mixture of 50 percent (m/m) of chlorpyrifos and 5 percent (m/m) of cypermethrin.

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 the Act and Rules, 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.
Indian Standard

CHLORPYRIFOS + CYPERMETHRIN EMULSIFIABLE CONCENTRATE — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for chlorpyrifos + cypermethrin emulsifiable concentrate.

2 REFERENCES

The following Indian 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:

<table>
<thead>
<tr>
<th>IS No.</th>
<th>Title</th>
</tr>
</thead>
<tbody>
<tr>
<td>1070 : 1992</td>
<td>Reagent grade water (third revision)</td>
</tr>
<tr>
<td>6940 : 1982</td>
<td>Methods of test for pesticides and their formulations (first revision)</td>
</tr>
<tr>
<td>8963 : 1978</td>
<td>Chlorpyrifos technical — Specification</td>
</tr>
<tr>
<td>9445 : 1980</td>
<td>Aluminium containers for packing of liquid pesticides (capacity 5 litres and above)</td>
</tr>
<tr>
<td>9503 : 1988</td>
<td>Aluminium bottles for liquid pesticides (first revision)</td>
</tr>
<tr>
<td>10627 : 1983</td>
<td>Methods for sampling of pesticidal formulations</td>
</tr>
<tr>
<td>12015 : 1987</td>
<td>Cypermethrin technical — Specification</td>
</tr>
</tbody>
</table>

3 REQUIREMENTS

3.1 Constituents

3.1.1 The material shall consist of chlorpyrifos technical and cypermethrin technical dissolved in suitable solvent(s) together with emulsifying agent(s).

3.1.2 Chlorpyrifos technical and cypermethrin technical employed in the manufacture of the material, shall conform to IS 8963 and IS 12015 respectively.

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 homogeneous and stable liquid, free from sediment and/or suspended matter. It shall readily form an emulsion on dilution with water.

3.2.2 Cold Test

No turbidity or separation of solid or oily matter shall occur when the material is subjected to the cold test at 10 °C as prescribed in 13.1 of IS 6940 or any other lower temperature as agreed to between the purchaser and the supplier.

3.2.3 Flash Point (Abel)

When determined by the method prescribed in IS 1448 [P : 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 emulsion prepared in standard hard water with 2.0 ml of EC, shall not exceed 2.0 ml when tested by the method prescribed in 13.3 of 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 Chlorpyrifos and Cypermethrin Content

When determined by the method prescribed in Annex A of this standard, the observed chlorpyrifos and cypermethrin content of any of the samples shall not differ from the declared nominal value by more than the percent tolerance limits given below:

<table>
<thead>
<tr>
<th>Nominal Value, Percent</th>
<th>Tolerance Limits, Percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Up to 9</td>
<td>+ 10</td>
</tr>
<tr>
<td></td>
<td>- 5</td>
</tr>
<tr>
<td>Above 9 and below 50</td>
<td>± 5</td>
</tr>
<tr>
<td>50 and above</td>
<td>+ 5</td>
</tr>
<tr>
<td></td>
<td>- 3</td>
</tr>
<tr>
<td></td>
<td>of the nominal value</td>
</tr>
</tbody>
</table>
3.3.1.1 The actual value of chlorpyrifos and cypermethrin content in formulation shall be calculated to the second decimal place and then rounded off to first decimal place before applying the tolerance limits.

3.3.2 Acidity
When tested by the method prescribed in 11.3 of IS 6940, the acidity (as H₂SO₄) of the material shall be not more than 0.25 percent by mass.

4 PACKING
The material shall be packed in PET and or aluminium containers (see IS 9445 and IS 9503). In addition, the material shall comply with general requirements as given in IS 8190 (Part 2).

5 MARKING
5.1 The container shall be securely closed and shall bear legibly and indelibly the following in addition to any other information as is necessary under the Insecticides Act, 1968 and Rules framed thereunder:
   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 mass of contents;
   g) Nominal value: Chlorpyrifos content percent m/m; and cypermethrin content percent m/m; and

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 provisions of the 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 the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING
When freshly manufactured material in bulk quantity and/or the retail pack of the formulated product is/are offered for inspection, representative sample of the material shall be drawn and tested as prescribed in IS 10627 and if tested within 90 days of its date of manufacture, the criteria for conformity shall be the contents in percent (m/m), shall not be less than the declared nominal value. The upper limit for conformity shall be the same as those given in 3.3.1 of this standard.

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 of this standard.

7 TESTS
7.1 Tests shall be carried out by the methods referred in 3.2.1 to 3.2.4 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 3.3.1)

DETERMINATION OF CHLORPYRIFOS AND CYPERMETHRIN CONTENT IN CHLORPYRIFOS AND CYPERMETHRIN FORMULATION SAMPLE

A-0 METHOD OF ANALYSIS FOR CHLORPYRIFOS AND CYPERMETHRIN

A-1 PRINCIPLE

The chlorpyrifos and cypermethrin content are determined simultaneously by gas chromatographically using the internal standard technique.

A-2 APPARATUS

A-2.1 Gas Liquid Chromatograph

A gas chromatograph equipped with flame ionization detector and a printer-plotter-cum-integrator is used. The suggested operative parameters are as follows, but can be changed if necessary, provided standardization is done:

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Column</td>
<td>Glass, length 100 cm, ID 2 mm, packed with 3% OV-101 on gaschrome Q or chromosorb WHP (80-100 mesh)</td>
</tr>
<tr>
<td>Oven temp. programme</td>
<td>170 °C- hold for 1 min, 10°C/min to 260 °C, hold for 5 min</td>
</tr>
<tr>
<td>Injector temperature</td>
<td>270 °C</td>
</tr>
<tr>
<td>Detector temperature</td>
<td>270 °C</td>
</tr>
<tr>
<td>Carrier gas</td>
<td>Nitrogen</td>
</tr>
<tr>
<td>Carrier gas flow</td>
<td>30 ml/min</td>
</tr>
<tr>
<td>Hydrogen gas flow</td>
<td>30 ml/min</td>
</tr>
<tr>
<td>Air (FID)</td>
<td>300 ml/min</td>
</tr>
</tbody>
</table>

A-2.2 Injection Volume: 1 μl capacity

A-2.3 Standard Glassware

A-2.4 Retention Time (Guide Values)

<table>
<thead>
<tr>
<th>Compound</th>
<th>Retention Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chlorpyrifos 4,3</td>
<td>8.9 min</td>
</tr>
<tr>
<td>Dioctyl Phthalate</td>
<td></td>
</tr>
<tr>
<td>Cypermethrin</td>
<td>10.9 min</td>
</tr>
<tr>
<td>Total run time</td>
<td>13 min</td>
</tr>
</tbody>
</table>

A-3 REAGENTS

A-3.1 Ethyl Acetate — AR grade or equivalent.

A-3.3 Dioctyl Phthalate — AR grade or equivalent.

A-4 PROCEDURE

A-4.1 Preparation of Internal Standard Solution

Weigh out (to the nearest of 0.1 mg) 2 g of dioctyl phthalate into a 1 litre volumetric flask. Dissolve it and make up to the volume with ethyl acetate.

A-4.2 Preparation of Standard Solution

Weigh out accurately (to the nearest of 0.1 mg) 0.15 g of chlorpyrifos reference standard and 0.015 g of cypermethrin reference standard into a 25-ml volumetric flask. Add 25 ml of internal standard solution and make up to 50 ml mark with ethyl acetate.

A-4.3 Preparation of Sample Solution

Weigh about 0.3 g of sample (to the nearest of 0.1 mg) into a 50-ml volumetric flask. Add 25 ml of internal standard solution and make up to 50 ml mark with ethyl acetate and shake well to mix.

A-4.4 Estimation

Inject standard solution until the area ratio of reference standard and internal standard of two successive injections do not deviate from each other by more than 2 percent. Then inject the sample solution. Then use the following injection sequence:

...C, S, C, S,......

C = standard solution, and
S = sample solution.

From the chromatograms of the standard solution and sample solution, measure the peak areas of the chlorpyrifos, cypermethrin and the internal standard peak and calculate the percentage of the chlorpyrifos and cypermethrin as given in A-5.

A-5 CALCULATION

\[
\text{Chlorpyrifos content, percent} \times \frac{M_1 \times A_2 \times A_3}{M_2 \times A_1 \times A_4} \times p_i
\]

where

\[
M_1 = \text{mass in g of chlorpyrifos in standard solution},
\]

3
\[ M_2 = \text{mass in g of sample taken for test}, \]
\[ A_1 = \text{area of chlorpyrifos peak in the chromatogram of standard solution}, \]
\[ A_2 = \text{area of chlorpyrifos peak in the chromatogram of sample solution}, \]
\[ A_3 = \text{area of internal standard peak in the chromatogram of standard solution}, \]
\[ A_4 = \text{area of internal standard peak in the chromatogram of sample solution}, \]
\[ P_1 = \text{percent purity of chlorpyrifos reference standard}. \]

Cypermethrin content, percent \( m/m \) is given by:
\[
\frac{M_2 \times A_3 \times A_5}{M_2 \times A_4 \times A_5} \times P_2
\]

where
\[ M_2 = \text{mass in g of sample taken for test}, \]
\[ M_1 = \text{mass in g of cypermethrin in standard solution}, \]
\[ A_3 = \text{area of internal standard peak in the chromatogram of standard solution}, \]
\[ A_4 = \text{area of internal standard peak in the chromatogram of sample solution}, \]
\[ A_5 = \text{area of cypermethrin peak in the chromatogram of standard solution}, \]
\[ A_6 = \text{area of cypermethrin peak in the chromatogram of sample solution}, \]
\[ P_2 = \text{percent purity of chlorpyrifos reference standard}. \]
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This Indian Standard has been developed from Doc: No. FAD 1 (1070).

Amendments Issued Since Publication

<table>
<thead>
<tr>
<th>Amend No.</th>
<th>Date of Issue</th>
<th>Text Affected</th>
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<tr>
<td></td>
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</tbody>
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