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मानक

IS 15236 (2002): Profenofos + Cypermethrin Emulsifiable Concentrate [FAD 1: Pesticides and Pesticides Residue Analysis]



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Indian Standard

PROFENOFOS + CYPERMETHRIN EMULSIFIABLE CONCENTRATE—SPECIFICATION

ICS 65.100.10

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BUREAU OF INDIAN STANDARDS MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI 110002

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.

Profenofos + Cypermethrin emulsifiable concentrate are used as an insecticide in agriculture.

Profenofos + Cypermethrin EC formulation is generally manufactured to contain a mixture of 40 percent (m/m) of Profenofos and 4 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

PROFENOFOS + CYPERMETHRIN EMULSIFIABLE CONCENTRATE — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for Profenofos + 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:

IS No.	Title
1070 : 1992	Reagent grade water (third revision)
1448	Methods of test for petroleum and
[P:20]:1998	its products: [P:20] Determination
	of flash point by Abel apparatus
	(second revision)
6940 : 1982	Methods of test for pesticides and
	their formulations (first revision)
8190	Requirement for packing of
(Part 2) : 1988	pesticides: Part 2 Liquid pesticides
	(second revision)
9503:1988	Aluminium Bottles for packing of
	liquid pesticides (first revision)
10627 : 1983	Method for sampling of pesticides
	formulations
12340 : 1988	Round open top aluminium
	containers for liquid containers
15238 : 2002	Profenofos, technical - Specification

3 REQUIREMENTS

3.1 Constituents

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

3.1.2 Profenofos, technical and cypermethrin, technical employed in the manufacture of the material, shall conform to IS 15238 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 clear, homogeneous and stable

liquid, pale yellow to yellow brown in colour, 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 Profenofos and Cypermethrin Content

When determined by the method prescribed in Annex A of this standard, the observed profenofos 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:

Nominal Value,	Tolerance Limits,	
Percent	Percent	
Up to 9	+10`	
0000	-5	of the
Above 9 and below 50	±5	> nominal
50 and above	+5	value
bo und abort	-3]

3.3.2 Acidity

When tested by the method prescribed in 13.5 of IS 6940, the acidity (as H_2SO_4) of the material shall be not more than 0.6 percent by mass.

4 PACKING

The material shall be packed in Aluminium containers conforming to IS 9503 or IS 12340. It shall also

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 : Profenofos content, percent m/m; and Cypermethrin content, percent m/m; and
- h) A cautionary notice worded as in the Insecticides Act, 1968 and Rules framed thereunder; and the Standards of Weights and Measures (Packaged Commodities) Rules, 1977.

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 to in 3.2.1 to 3.2.4 and 3.3.1 to 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 PROFENOFOS AND CYPERMETHRIN CONTENT IN PROFENOFOS AND CYPERMETHRIN FORMULATION SAMPLE

A-0 METHOD OF ANALYSIS FOR PROFE-NOFOS AND CYPERMETHRIN

A-1 PRINCIPLE

The profenofos 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-integrater is used. The suggested operative parameters are as follows,

but can be changed if necessary, provided standardization is done.

Column	:	Glass/S.S, length-1m, ID 2 mm, packed with
		3 percent OV-101 or SE-30 on Gaschrome
		Q, 80-100 mesh.
Temperatures	:	190°C for 4 min then
		increased to 260° at
		10° C/min then hold
		at 260° for 4 min
Injector temperature	:	245°C
Detector temperature	:	270°C

Carrier gas	: Nitrogen : 30 ml/min
	Hydrogen : 30 ml/min
	gas flow
Air (FID)	: 300 ml/min

A-2.2 Microlitre Syringe — 5/10 µl capacity.

A-2.3 Standard Glassware

A-3 RETENTION TIMES (GUIDE VALUES):

Profenofos	:	4.7 min
Cypermethrin	:	10.8 min
Bis-(2 ethylhexyl) adipate	:	7.1 min
Total Run Time	:	15 min

A-4 REAGENTS

A-4.1 Dichloromethane AR Grade — or equivalent.

A-4.2 Profenofos and Cypermethrin Reference Standards — of known purity.

A-4.3 Internal Standard — Bis-(2 ethylhexyl) adipate AR grade or equivalent.

A-5 PROCEDURE

A-5.1 Preparation of Internal Standard Solution

Weigh out accurately 0.500-0.550 g of Bis-(2 ethylhexyl) adipate into a 100 ml volumetric flask. Dissolve in dichloromethane and make up to the volume to the mark with dichloromethane. Shake well to homogenize.

A-5.2 Preparation of Stock Solution of Cypermethrin

Weigh out accurately 0.100-0.110 g of cypermethrin reference standard into a 100 ml volumetric flask. Dissolve in dichloromethane and make up to the volume to the mark with dichloromethane. Shake well to homogenize.

A-5.3 Preparation of Reference Solution

Weigh out accurately 0.200-0.220 g of profenofos reference standard of known purity into a 50 ml volumetric flask. Add 20 ml of internal standard solution and 20 ml of stock solution of cypermethrin as prepared in A-5.2 and make up to the volume to the mark with dichloromethane. Shake well to homogenize.

A-5.4 Preparation of Sample Solution

Weigh out accurately quantity of a sample equivalent to 0.200 g of profenofos and 0.02 g of cypermethrin into a 50-ml volumetric flask. Add 20 ml of internal standard solution and make up the volume to the mark with dichloromethane. Shake well to homogenize.

A-5.5 Estimation

Inject 1 microlitre of reference standard solution until the internal standard/reference substance area quotients two successive chromatograms do not deviate from each other by more than 2 percent.

Then use the following injection sequence:

$$\dots CS_1S_1, CS_2S_2, CS_3S_3$$

where

C =Reference solution,

 $S = \text{Test solution } (1, 2, 3, \dots, n)$

From the chromatograms of the standard solution and sample solution, measure the peak areas of the internal standard and profenofos/cypermethrin peaks and compute the percentage of the profenofos/cypermethrin as given in A-6.

A-6 CALCULATION

Profenofos/cypermethrin
content, percent
$$w/w$$
 = $\frac{M_1 \times A_2 \times A_3}{M_2 \times A_1 \times A_4} \times P$

where

- M_1 = mass in g of standard profenofos/ cypermethrin in standard solution,
- $M_{2} = \text{mass in g of sample taken for test,}$
- A_1 = peak area of profenofos/cypermethrin in the chromatogram of standard solution,
- A_2 = peak area of profenofos/cypermethrin in the chromatogram of sample solution,
- A_3 = peak area of internal standard in the chromatogram of standard solution,
- A_4 = peak area of internal standard in the chromatogram of sample solution, and
- P = percent purity of profenofos/cypermethrin reference standard.

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