

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for Acephate 45% + Cypermethrin 5% DF.

2 REFERENCES

The standards, given below 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.

<i>IS No.</i>	<i>Title</i>
460 (Part 1) : 2020	Test Sieves — Specification Part 1 Wire Cloth Test Sieves (Fourth Revision)
1070 : 1992	Reagent grade water – Specification (<i>third revision</i>)
6940 : 1982	Methods of tests for pesticides and their formulations (first revision)
8190 (Part 1): 1988	Requirement for packing of pesticides: Part I Solid pesticides (second revision).
10627: 1983	Methods for sampling of pesticides formulations.

3 REQUIREMENTS

3.1 Constituents

3.1.1 The material shall consist of a homogeneous mixture of Acephate technical and Cypermethrin technical together with suitable carrier(s), stabilizer(s) and other necessary formulants(s).

3.1.2 Acephate technical and Cypermethrin technical employed in the formulation of this material shall conform to the specification submitted to the registration committee.

3.2 Description

3.2.1 The material shall be in the form of an off white to light brown coloured granules. Free from hard lumps and extraneous matter.

3.3 The material shall also comply with the requirements specified in Table 1.

Table 1 Requirements for Acephate 45% + Cypermethrin 5% DF

Sl. No.	Characteristic	Requirement	Method of test, Refer to	
(1)	(2)	(3)	(4)	
i)	Active content, percent by mass, <i>Min</i>	Acephate a.i	42.5 to 47.25	Annex A
		Cypermethrin a.i	4.75 to 5.5	Annex B
ii)	Moisture Content, percent by mass, <i>Max</i>	2.0	Annex C	
iii)	Wettability, <i>max</i> in sec	120	Annex D	
iv)	Wet sieve test (material passing through 200 BSS standard test sieve), percent by mass, <i>Min</i>	98	Annex E	
v)	Acidity (as H ₂ SO ₄) percent by mass, <i>Max</i>	0.15	Annex F	

3.3.1 Acephate and Cypermethrin Content

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

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

3.3.2 The actual value of Acephate and Cypermethrin content in the formulations shall be calculated to the second decimal place and then rounded off to the first decimal place before applying the tolerance given in **3.3.1**.

3.3.3 The average Acephate and Cypermethrin content of all samples taken shall not be less than the declared nominal content.

4 PACKING

The material shall be packed according to the general requirements given in IS 8190 (Part 1).

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 acephate content, percent (*m/m*);
- h) Nominal cypermethrin content, percent (*m/m*);
- j) Cautionary notice as worded in the *Insecticides Act*, 1968, and Rules framed thereunder; and
- k) 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

6.1 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**. 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

7.1 Tests shall be carried out by the methods referred to column 4 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS I 070) 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 ACEPHATE CONTENT

A-1 Method

Acephate is dissolved in dichloromethane containing di-isobutyl phthalate as internal standard, then separated by gas chromatography with flame ionization and quantified using peak area ratio.

A- 2 OPERATING PARAMETERS

Gas Chromatograph	
Column	Glass, 500×2mm (i.d.)
Column temperature	155°C
Injector	170 °C
Detector	250 °C
Gas Flow Rates	
Carrier gas	Nitrogen(30ml/min)
Hydrogen	As specified for detector
Zero air	As specified for detector

A-3 REAGENTS

Internal Standard – Di-isobutyl phthalate -Analytical Grade

Analytical Standard – Pure Acephate standard of known purity

Diluting Solvent – Dichloromethane - Analytical Grade

A-4 PROCEDURE

4.1 Preparation of Internal Standard Solution

Dissolve 1.2 g of Di-isobutyl phthalate in a 4 liter dichloromethane. Store at room temperature.

4.2 Preparation of Standard Solution

Weigh 0.1000 g +0.0100. g of Acephate analytical standard into a 100 ml standard volumetric flask and dilute to volume with internal standard solution, close the flask with cap and shake until the contents are dissolved. Prepare freshly just before analysis.

4.3 Preparation of Sample Solution

Weigh sufficient sample to contain 0.1000 g of acephate into a 100 ml standard volumetric flask and dilute to volume with internal standard solution, close the flask with cap and shake until the contents are dissolved.

A-5 CALCULATION

$$\text{Percent of acephate} = \frac{RR \text{ of Sample} \times Wt. \text{ of Std.} \times \text{Purity of Std.}}{RR \text{ of Std} \times Wt. \text{ of Sample}}$$

ANNEX B

[Table 1, Sl. No. (i)]

DETERMINATION OF CYPERMETHRIN CONTENT

A-1 Method

Cypermethrin is dissolved in 4-methylpentan-2-one containing di-(2-ethylhexyl) phthalate as internal standard, then separated by gas chromatography with flame ionization and quantified using peak area ratio.

A-2 OPERATING PARAMETERS

Gas Chromatograph	
Column	Glass, 1.0m×4mm (i.d.)
Column temperature	235°C
Injector	250 °C
Detector	250 °C
Gas Flow Rates	
Carrier gas	Nitrogen(0.05l/min)
Hydrogen	As specified for detector
Zero air	As specified for detector

A-3 REAGENTS

Internal Standard – Di-(2-ethylhexyl) phthalate-Analytical Grade

Analytical Standard – Pure cypermethrin standard of known purity

Diluting Solvent – 4-methylpentan-2-one(MIBK)-Analytical Grade

A-4 PROCEDURE

4.1 Preparation of Internal Standard Solution

Dissolve 10 g of Di-(2-ethylhexyl) phthalate in a 500 ml 4-methylpentan-2-one. Store at room temperature.

4.2 Preparation of Standard Solution

Weigh 0.2000 g \pm 0.0100 g of cypermethrin analytical standard into a 50 ml standard volumetric flask and dissolve in a few ml of MIBK. Add by pipette 10 ml of internal standard solution and dilute to 50 ml with MIBK.

4.3 Preparation of Sample Solution

Weigh sufficient sample to contain 0.2000 g of Cypermethrin into a 5.0 ml standard volumetric flask and dissolve in a few ml of MIBK. Add by pipette 10 ml of internal standard solution and dilute to 50 ml with MIBK.

A-5 CALCULATION

$$\text{Percent of cypermethrin} = \frac{RR \text{ of Sample} \times Wt. \text{ of Std.} \times \text{Purity of Std.}}{RR \text{ of Std} \times Wt. \text{ of Sample}}$$

ANNEX C

[Table 1, Sl. No. (ii)]

DETERMINATION OF MOISTURE CONTENT

C-1 OUTLINE OF METHOD

The sample is dispersed in methanol, and titrated with standard Karl Fischer reagent of known water equivalent.

C-2 REAGENTS

Methanol (CH₃OH) containing not more than 0.03% water (*m/m*)

Karl Fischer reagent

Deionized Water

C-3 STANDARDIZATION

Add methanol (about 20 ml) to the titration vessel and titrate to the null point with Karl Fischer reagent without recording the volume required. Introduce an accurately 10 μ l of deionized water into the apparatus and titrate to the null point with the Karl Fischer reagent. Calculate the water equivalent (*e*) of the reagent in mg of water per ml.

C-4 PROCEDURE

Add the methanol (about 20 ml) to the titration vessel and titrate to the null point with Karl Fischer reagent. Transfer quickly a suitable amount of acephate 45% + cypermethrin 5% DF sample (about 1 g), accurately weighed (*w* g), into the titration vessel. Stir for 1 min and titrate again with the standardized Karl Fischer reagent (*t* ml) of known water equivalent.

$$\text{Water Content, percent by mass} = \frac{t \times e}{10w}$$

Where,

e = water equivalent in mg/ml of Karl Fischer reagent

ANNEX D

[Table 1, Sl. No. (iii)]

DETERMINATION OF WETTABILITY CONTENT

D-1 SCOPE

The method describes a procedure for the determination of the time of complete wetting of acephate 45% + cypermethrin 5% DF.

D-2 OUTLINE OF METHOD

A weighed amount of the sample is dropped on water in a beaker from a specified height. The time for complete wetting is determined.

D-3 REAGENT

Standard Water of 342 ppm

D-4 APPARATUS

Beaker 250 ml, internal diameter 6.5 ± 0.5 cm, height 9.0 ± 0.5 cm

Stop watch accurate to the nearest second

Measuring cylinder 100 ml

D-5 PROCEDURE

Pour Standard water (100 ± 1 ml) into the beaker. Weigh out 2 ± 0.1 g of a representative sample of the acephate 45% + cypermethrin 5% DF formulation, taking care that it remains in a non-compacted state. Add the entire sample at once, by dropping it on the water from a position level with the rim of the beaker, without undue agitation of the liquid surface and spreading it uniformly on the water surface. When the sample is added, start the stop watch and note the time taken (to the nearest second) for it to become completely wetted. Report the time, to the nearest second, required for complete wetting of the sample as the wetting time.

ANNEX E

[Table 1, Sl. No. (iv)]

DETERMINATION OF WET SIEVE TEST CONTENT

E-1 SCOPE

The method is suitable for the determination of the amount of non-dispersible material in formulations, that are applied as dispersions in water.

E-2 OUTLINE OF METHOD

A sample of the formulation is dispersed in water and the suspension formed is transferred to a standard sieve and washed. The amount of the material retained on the sieve is determined by drying and weighing.

E-3 APPARATUS

Balance with an accuracy of at least 0.01g

Beaker 250 ml

Magnetic stirrer and magnetic flea (stirring bar) Rubber hose of approx. 10 mm internal diameter

Oven

Desiccator

Sieve 20 cm diameter, 75 µm mesh size (200 mesh), if not otherwise specified.

E-4 PROCEDURE

(a) **Wetting:** Weigh (to the nearest 0.1 g) 10 g (or if necessary a more appropriate quantity) of the acephate 45% + cypermethrin 5% DF sample into a beaker (250 ml) and add tap water (100 ml). Allow to stand for 60 sec. Then stir with the magnetic stirrer for 5 min, making no deliberate attempt to break up any lumps (The speed of rotation of the magnetic flea should be chosen such that a vortex just forms on the surface of the liquid. Care should be taken that the dispersion does not become aerated by over-vigorous dispersion of the sample).

(b) **Wet sieving:** Transfer the slurry to the sieve, rinsing with tap water, removing the magnetic flea (having washed any dispersed material from the flea into the sieve). Wash the material on the sieve with a jet of tap water (The tap water may contain solids, and should, if required, be screened before use) using a rubber hose of 10 mm i.d. delivering 4-5 litres of water per min. Continue the washing until the visible quantity of residue remains constant (max. 10 min). Direct the water from the circumference of the sieve towards the center and keep the end of the hose at a distance of 2 - 5 cm from the surface of the sieve. Transfer the residue to a tared glass dish with a jet of deionized water from a wash bottle. Dry to constant weight (A temperature of 60-70°C is recommended. If necessary, the temperature must be varied to avoid decomposition or volatilization at the drying temperature) and record the weight of the sample to the nearest 0.01 g.

E-5 CALCULATION

Express the weight of residue as a percentage of the sample weight and record the result as percentage retention on the stated test sieve.

$$\text{Percentage retention} = \frac{\text{Weight of formulation retained on the surface}}{\text{Total weight taken}} \times 100$$

ANNEX F

[Table 1, Sl. No. (v)]

DETERMINATION OF ACIDITY CONTENT

F-1 SCOPE

The method is suitable for determining the acidity of acephate + cypermethrin (45+5) DF

F-2 OUTLINE OF METHOD

The acidity is determined by titration with standard alkali in presence of methyl red indicator. Where this is impracticable, the end point is determined electrometrically.

F-3 REAGENTS

Acetone

Sodium hydroxide 0.05 M standardized solution

Hydrochloric acid 0.05 M standardized solution

Distilled water

Methyl red indicator solution

F-4 APPARATUS

Measuring cylinder 50ml and 100 ml

Conical flask 250 ml

Burette 25 ml

pH meter, capable of at least two-point calibration

Electrode system, e.g. glass electrode system conditioned and stored according to the manufacturer's instructions

Automatic titrated, alternative to the pH meter/electrode system

F-5 CALIBRATION

Operate the pH meter and the electrode system according to the manufacturer's instructions. Calibrate the measuring system (pH meter and electrode) according to the manufacturer's instructions using buffer solutions of 4.0 and 7.0 pH.

F-6 PROCEDURE

Weigh (to the nearest mg) 5 g of the sample (w g) into 250 ml conical flask and dissolve in acetone (25 ml) stir to homogenize. Add distilled water (75 ml) and titrate immediately with sodium hydroxide solution (t ml), in presence of methyl red. Carry out a blank determination on acetone (25 ml) and distilled water (75 ml) with sodium hydroxide (b ml).

F-7 CALCULATION

$$\text{Acidity, Calculated as H}_2\text{SO}_4 = \frac{4.904 \times C \times (t-b)}{w}$$

Where,

C = NaOH, mol/l (normality) of the sodium hydroxide solution