Doc: FAD 01 (20166) WC
January 2024

BUREAU OF INDIAN STANDARDS

DRAFT FOR COMMENTS ONLY

भारतीय मानक मसौदा

एफ़िडोपाइरोपेन डिस्पर्सिबल कॉन्सेंट्रेट – विशिष्टि

Draft Indian Standard

AFIDOPYROPEN DISPERSIBLE CONCENTRATE – SPECIFICATION

ICS No. 65.100.10

Pesticides Sectional Committee, FAD 01 Last Date of Comments: **4 March 2024**

FOREWORD

(*Formal clause would be added later)*

Afidopyropen dispersible concentrate is used as an insecticide in agriculture.

Afidopyropen dispersible concentrate is generally manufactured to contain 50 g/l of afidopyropen on *w*/*v* basis.

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.

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 afidopyropen dispersible concentrate.

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.

IS No.	*Title*
IS 460 (Part 1) : 2020	Test Sieves — Specification part 1 wire cloth test sieves (*fourth revision*)
IS 1070 : 2023	Reagent grade water - Specification (*fourth revision*)
IS 8190 (Part 2) : 1988	Requirements for packing of pesticides Part 2 liquid pesticides (*second revision*)
IS 10627 : 1983	Methods for sampling of pesticidal formulation

3 REQUIREMENTS

3.1 Constituents

The material shall consist of Afidopyropen technical, together with suitable ingredients.

3.2 Physical

The material shall comply with the following physical requirements:

3.2.1 *Description*

The material shall be homogeneous, clear yellow liquid with no distinct colour.

3.2.2 *Density* – When determined by the method prescribed in Annex B, density at 20 °C is 1.02 to 1.03 g/ml.

3.2.3 *Suspensibility* – When determined by the method prescribed in Annex C, the suspensibility shall be minimum 85 percent (*m*/*m*).

3.2.4 *Wet Sieve Test* – Not more than 1 percent by mass of the material shall pass through 75 microns IS sieve [*see* IS 460 (Part 1)], when determined by the method prescribed in IS 6940.

3.2.5 *Persistent Foam* – The persistent foaming shall be 60 ml, maximum after 1 minute, when determined by the method prescribed in Annex D.

3.2.6 *p*H – When determined by the method prescribed in Annex E, the *p*H of 1 % aqueous solution of the material shall be in the range 7.2 – 7.6.

3.3 Chemical

The material shall comply with the chemical requirements specified in **3.3.1.**

3.3.1 *Afidopyropen Content*

When determined by the method prescribed in Annex A, the observed afidopyropen content (*w/v*), of any of the samples shall not differ from the declared nominal value by more than the percent tolerances limits indicated below:

Nominal Value, *percent*	*Tolerance, percent*	
Up to 9	+10 -5	of the nominal value
Above 9 and below 50	±5	
50 and above	+5 -3	

3.3.1.1 The actual value of the afidopyropen content in the formulations shall be calculated to the second decimal place for rounding off to the first decimal place before applying the tolerances given in **3.3.1.**

3.3.1.2 The average Afidopyropen content of all samples taken shall not be less than the declared nominal content.

4 PACKING

The material shall be packed in mild steel or tin plate containers suitably and properly lacquered with epoxy resin from inside. Closure provided shall be such as not to allow any material to leak through them. Containers holding 5 litres or more of liquid shall be provided with suitable pouring device with each container. It shall also conform to the general requirements given in IS 8190 (Part 2).

5 MARKING

5.1 The containers shall be securely closed and shall be bear legibly and indelibly the following information in addition to any other information as required 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 quantity;
g) Nominal afidopyropen content, percent by volume;
h) Cautionary notice as worded in the *Insecticides Act*, 1968, and Rules framed thereunder; and
j) 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 he 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 appropriate methods referred to in **3.2.2 to 3.2.6.**

7.2 Quality of Reagent

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 AFIDOPYROPEN CONTENT

A-1 GENERAL

Determine afidopyropen content in afidopyropen DC formulation samples by HPLC method. The identity of the active ingredient is established by comparison with the equivalent authentic standard.

A-2 CHROMOTOGRAPHIC METHOD

A-2.1 Reagents

A-2.1.1 *Acetonitrile*, HPLC grade

A-2.1.2 *Water*, HPLC grade

A-2.1.3 *Reference substance of known purity*

A-2.2 Apparatus

A-2.2.1 *Analytical Balance*

A-2.2.2 *Standard Glassware*

CHROMOTOGRAPHIC SEPARATION PARAMETER

<table>
<tr><td>Instrument</td><td>HPLC with Binary pump, with UV detector</td></tr>
<tr><td>Column length, mm</td><td>C18 column of 250 mm length, 4.6 mm internal diamter (i. d.), 5 µm particle size</td></tr>
<tr><td>Mobile Phase</td><td>Solvent A: 950 ml Water + 5 ml Acetonitrile
Solvent B : Acetonitrile</td></tr>
<tr><td>Detector</td><td>240 nm</td></tr>
<tr><td>Column temperature</td><td>Ambient</td></tr>
<tr><td>Injection Volume</td><td>10 µL</td></tr>
<tr><td>Gradient</td><td>
<table>
<tr><th>Time (min)</th><th>Solvent A (%)</th><th>Solvent B (%)</th><th>Flow (mL/min)</th></tr>
<tr><td>0.0</td><td>63</td><td>37</td><td>2.0</td></tr>
<tr><td>0.5</td><td>63</td><td>37</td><td>2.0</td></tr>
<tr><td>2.5</td><td>42</td><td>58</td><td>2.0</td></tr>
<tr><td>4.5</td><td>42</td><td>58</td><td>2.0</td></tr>
<tr><td>5.0</td><td>63</td><td>37</td><td>2.0</td></tr>
<tr><td>10.0</td><td>63</td><td>37</td><td>2.0</td></tr>
</table>
</td></tr>
<tr><td>Total Run Time</td><td>about 10 min</td></tr>
<tr><td>Retention Time</td><td>Afidopyropen ~ 4.8 min.
Diethyl phthalate (IS) ~ 6.2 min.</td></tr>
</table>

A-2.3 Procedure

A-2.3.1 *Preparation of Internal Standard Stock Solution*

Weigh 4 ml of diethyl phthalate into 1000 ml volumetric flask. Fill to the calibration mark with Methanol and mix well until dissolved.

A-2.3.2 *Preparation of Standard Solution*

Accurately weigh about 20 mg reference standard afidopyropen of known purity into a 25 ml volumetric flask. Add 2.0 ml internal standard and dilute up to the mark with methanol.

A-2.3.3 *Preparation of Sample Solution*

Accurately weigh sample to yield about 20 mg active content from Afidopyropen 50 g/l DC into a 25 ml volumetric flask. Add 2.0 ml internal standard, dissolve and dilute up to the mark with Methanol, Mix well. Filter through a 0.45 µm PTFE Syringe filter.

Estimation: Introduce separately 10 micro liters of the standard solution and the sample in to the HPLC and record chromatograms.

A-2.4 Calculation

Afidopyropen content, percent (*w/w*) = $\frac{M_1}{M_2} \times \frac{A_1}{A_2} \times \frac{A_4}{A_3} \times P$

where,

M_1 = Mass of reference standard taken for standard preparation in g

M_2 = Mass of sample in g

P = Percent by purity of reference standard

A_1 = Area of internal standard in the standard solution injected

A_2 = Area of active ingredient in the standard solution injected

A_3 = Area of internal standard in the sample solution injected

A_4 = Area of active ingredient in the sample solution injected

Afidopyropen content (percent by volume) = Afidopyropen content (percent by mass) × Specific gravity

ANNEX B
[*Clause* 3.2.2]
DETERMINATION OF RELATIVE DENSITY

B-1 PRINCIPLE

The relative density of the material shall be the ratio of the mass of a given volume of the material generally at 27°C to that of an equal volume of water at the same temperature.

B-2 APPARATUS

B-2.1 Density bottle closed type, made of glass and of a size and type suitable for use with the material under test.

B-2.2 Water bath thermostatically controlled with an accuracy of ± 0.1 °C

B-3 PROCEDURE

B-3.1 Clean and dry the density bottle and weigh it with its stopper to the nearest milligram. Fill the bottle with freshly boiled and cooled distilled water at the desired temperature and determine the apparent mass of the content.

B-3.2 Clean and dry the density bottle, fill it with the sample under test, and determine, in a similar manner the apparent mass of sample contained in the bottle at the same temperature.

B-4 CALCULATION

$$\text{Relative density of sample} = \frac{(C-A)}{(B-A)}$$

where

C = mass in g of the relative density bottle or the pyknometer filled with the material at 27°C,

A = mass in g of the dry relative density bottle or the pyknometer, and

B = mass in g of the relative density bottle or the pyknometer filled with water at 27°C.

ANNEX C
[*Clause* 3.2.3]
DETERMINATION OF SUSPENSIBILITY

C-1 PRINCIPLE

It involves preparing 250 ml of diluted suspension, allowing it to stand in a measuring cylinder under defined conditions, and removing the top nine-tenths of suspension. The remaining tenth portion of suspension is then assayed chemically, gravimetrically or by solvent extraction, and calculate the suspensibility.

C-2 REAGENT

Standard hard water

C-3 APPARATUS

C-3.1 Beaker, 250 ml capacity

C-3.2 Suction Apparatus, connected with a glass tube

C-3.3 Stopwatch

C-3.4 Stoppered Measuring Cylinder, 250 ml capacity

C-3.5 Glass rod

C-4 PROCEDURE

Homogenize about 0.5 g formulation into 250 ml beaker and add 10 ml of the standard hard water at 30 ± 2 °C, then stir by hand for 30 seconds with a glass rod. Make up to 250 ml with standard water at 30 ± 2 °C and insert the stopper. Invert the cylinder 30 times. Place the cylinder in water bath in an upright position free from vibration and away from the direct sunlight. At the end of 30 minutes of settling period, dip the nozzle of a glass tube into the supernatant liquid contained and withdraw nine-tenths (225 ml) of the suspension with the minimum disturbance within 10-15 seconds, using suction tube and vacuum pump. The remaining one-tenth (25 ml) suspension is analyzed for its active ingredient content by method used for estimation of active content (*see* Annex A).

C-5 CALCULATION

$$\text{Suspensibility, percent by mass} = \frac{111 \times (C - Q)}{C}$$

where

$C = \frac{a \times W}{100}$ = mass of active ingredient in the whole cylinder, in g.

a = percentage of active ingredient by mass in formulation.

W = mass of formulation actually added to the cylinder, in g.

Q = mass of active ingredient in 25 ml sample at the bottom, in g.

ANNEX D
[*Clause* 3.2.5]
DETERMINATION OF PERSISTENT FOAM

D-1 PRINCIPLE

Dilute the Dispersible Concentrate in a measuring cylinder of standard dimensions which is inverted 30 times and create the amount of foam and measure the remaining after certain times.

D-2 APPARATUS

Graduated cylinder glass stoppered, 250 ml capacity with 2 ml graduations, the distance between the 0 mark and the 250 ml mark being 20-21.5 cm, and between the 250 ml mark and the bottom of the stopper, 4-6 cm.

D-3 REAGENT

Standard hard water

D-4 PROCEDURE

Take the mass of sample require to make 200 ml of a suspension with a concentration recommended in the directions for use supplied with the product. Where several concentrations are recommended, the maximum concentration shall be used. Put about 180 ml of standard water into the 250 ml measuring cylinder standing on a top pan balance and weigh in the required amount of suspension concentrate. Top up with standard water. Stopper the cylinder and invert 30 times. Place the stoppered cylinder upright on the bench and immediately start the stopwatch. Read the volume of foam produced and remaining after 60 seconds.

ANNEX E
[*Clause* 3.2.6]
DETERMINATION OF *p*H

E-1 PRINCIPLE

The *p*H value of a mixture of a sample with water or of an undiluted aqueous formulation is determined by means of a *p*H meter.

E-2 APPARATUS

E-2.1 Standard Measuring Flask, 100 ml capacity

E-2.2 Beaker, 100 ml capacity

E-2.3 Glass Rod

E-2.4 Weighing Balance

E-2.5 *p*H Meter

E-3 REAGENTS

E-3.1 *p*H 4.0 Standard Buffer Solution

E-3.2 *p*H 9.0 Standard Buffer Solution

E-4 PROCEDURE

E-4.1 Preparation of 1% Sample Solution

Homogenize and weigh accurately 1g of sample into a 100 ml standard flask and make up to the mark using distilled water.

E-4.2 Calibration of *p*H Meter

Calibrate the *p*H meter with *p*H 4.0 standard buffer solution, suitable to make measurement in acidic *p*H range and a *p*H 9.0 standard buffer solutions, suitable to make the measurement in alkaline *p*H range at 25 ± 1 °C.

E-4.3 *p*H Determination of the Sample at 25 ± 1 °C

Weight accurately about 1.0 g of the sample and add 100 ml of distilled water. Mix the sample thoroughly to get a uniform suspension in a beaker. Measure the pH of the suspension. Report result to the nearest 0.1 pH.