

## FOREWORD

*(Formal clause would be added later)*

Ametoctradin 300 g/l + Dimethomorph 225 g/l (m/m) Suspension Concentrate is used as a Fungicide in Agriculture.

Ametoctradin + Dimethomorph Suspension Concentrate is generally manufactured to contain Ametoctradin 27.0% and Dimethomorph 20.27 percent by mass.

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 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 : 2022 '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.

## 1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for Ametoctradin + Dimethomorph Suspension 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:

<i>IS No.</i>	<i>Title</i>
1070 : 1992	Reagent grade water ( <i>third revision</i> )
6940 : 1982	Methods of test for pesticides and their formulation ( <i>first revision</i> )
8190 (Part 2) : 1988	Requirements for packing of pesticides : Part 2 Liquid pesticides ( <i>second revision</i> )
10627 : 1984	Method of sampling of pesticidal formulation

## 3 REQUIREMENTS

### 3.1 Constituents

The material shall consist of Ametoctradin technical and Dimethomorph technical, together with suitable ingredients.

### 3.2 Physical

The material shall comply with the physical requirements specified in **3.2.1** and **3.2.2**

#### 3.2.1 Description

The material shall be beige coloured, flowable liquid, free from agglomerates and extraneous matters, which on dilution with water readily forms a suspension, which is suitable for spray.

**3.2.2** The material shall also comply with the requirements given in Table 1.

**TABLE 1 REQUIREMENTS FOR ALFIDOPYROPEN DIAPERSIBLE CONCENTRATE**

*(Clause 3.2.2 and 7.1)*

Sl. No.	Characteristic	Requirement	Annex of this Standard
(1)	(2)	(3)	(4)
i)	Ametoctradin + Dimethomorph Suspension Concentrate, percent by mass, <i>Min</i>		A
ii)	Pourability, percent by mass, <i>Max</i>	3	B
iii)	Spontaneity of dispersion, percent by mass, <i>Min</i>	95	C
iv)	Suspensibility, percent by mass, <i>Min</i>	90	IS 6940
v)	Wet sieve test, percent by mass,	95	IS 6940
v)	Persistent Foam, <i>Max</i>	60 sec	D
vi)	Acidity (as H <sub>2</sub> SO <sub>4</sub> ), percent by mass, <i>Max</i>	0.15	IS 6940
vii)	pH		E

### 3.3 Chemical

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

#### 3.3.1 Ametoctradin and Dimethomorph content

When determined by the method prescribed (enclosed), the observed Ametoctradin and Dimethomorph content (*m/m*), of any of the samples shall not differ from the declared nominal value by more than the percent tolerances limits indicated below:

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

**3.3.1.1** The actual value of the Ametoctradin and Dimethomorph 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 Ametoctradin and Dimethomorph 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 mass of content, percent (*m/m*);
- g) Nominal Ametoctradin and Dimethomorph content, percent (*w/w*);
- 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**

**6.1** 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 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 appropriate methods referred to in Table 1

### **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**

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

### **DETERMINATION OF AMETOCTRADIN AND DIMETHOMORPH CONTENT**

#### **A-1 GC CHROMATOGRAPHY METHOD**

##### **A-1.1 REAGENTS**

**A-1.1.1** *Ametoctradin reference standard*

**A-1.1.2** *Dimethomorph reference standard*

**A-1.1.3** *Acetonitrile (HPLC grade)*

**A-1.1.4** *Water (HPLC grade)*

**A-1.1.5** *Triethyl amine (HPLC grade)*

**A-1.1.6** *Formic acid (AR grade)*

**A-1.1.7** *Distilled water/Deionised water*

**A-1.1.8** *CIPAC Standard water D/C*

**A-1.1.9** *Tap water*

#### **A-2 TEST METHODS**

##### **A-2.1 DESCRIPTION**

Visual observation

##### **A-2.2 ACTIVE CONTENT**

Chromatographic conditions:

Instrument		HPLC with PDA detector and LC solutionsoftware.	
Column		Chromatopack, C18 (250mm (L) x 4.6 mm (i.d.) x 5 µm particle size)	
Mobile phase		Acetonitrile: 0.2% v/v Triethyl amine in HPLC water (3.0 pH adjusted by formic acid)	
Detection wavelength		254 nm	
Flow rate		1.5 ml/min	
Column Temperature		Ambient	
Injection volume		25 µl	
Diluent		Acetonitrile	
Retention time	Dimethomorph approx.	Cis	9.74 min
		Trans	10.74 min
	Ametoctradin approx.		18.60 min

### A-3 PROCEDURE

#### A-3.1 Preparation of Standard Solution:

10 mg (±1mg) of Ametoctradin standard and 10 mg (±2mg) of Dimethomorph referencstandard were weighed respectively into a 50 ml volumetric flask and made up to the mark with acetonitrile.

#### A-3.2 Preparation of Sample solution:

38 mg (±10 mg) of formulation were weighed into two different 50 ml volumetric flask. and made up to the mark with acetonitrile.

### A-4 CALCULATION

#### A-4.1 Calculation for Ametoctradin

**Estimation:** 25 µL of standard and sample solution was injected into HPLC. From areaobtained calculated the % active content by using the following formula.

$$\text{Ametoctradin content, \% } m/m = \frac{H_W \times M \times P}{H_S \times w}$$

where

$H_W$  = Peak area of Ametoctradin in sample solution

$H_S$  = Peak area of Ametoctradin in standard solution

$M$  = Mass of Ametoctradin in standard (mg)

$w$  = Weight of sample taken (mg)

$P$  = % purity of Ametocetradin reference standard.

Ametocetradin content, %  $m/m$  = Active content, %  $m/m$  x density

Ametocetradin content, g/l = Active content, %  $m/m$  x 10

#### A-4.2 Calculation for Dimethomorph

**Estimation:** 25  $\mu$ L of standard and sample solution was injected into HPLC. From area obtained calculated the % active content by using the following formula.

$$\text{Dimethomorph content, \% } m/m = \frac{H_w \times M \times P}{H_s \times w}$$

Where

$H_w$  = Peak area of Dimethomorph isomers in sample solution

$H_s$  = Peak area of Dimethomorph isomers in standard solution

$M$  = Mass of Dimethomorph in standard (mg)

$w$  = Weight of sample taken (mg)

$P$  = % purity of Dimethomorph.

Dimethomorph content, %  $m/m$  = Active content, %  $m/m$  x density

Dimethomorph content, g/l = Active content, %  $m/m$  x 10

## ANNEX B

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

### DETERMINATION OF POURABILITY IN AMETOCTRADIN AND DIMETHOMORPH CONTENT

#### B-1 PROCEDURE

The empty test cylinder (500 mL) and stopper ( $W_0$  g) were weighed and enough of the suspension concentrate was taken from a recently mixed bulk sample to leave approximately 20% of the volume of the test cylinder (400 mL) as ullage. The stopper was replaced and the test cylinder ( $W_1$  g) was reweighed. The cylinder with sample was allowed to stand undisturbed for 24 hours. The suspension concentrate was poured out within 60 seconds by keeping the measuring cylinder at an angle of  $45^\circ$  and then finally the measuring cylinder was inverted to transfer the entire samples within 60 seconds. The cylinder with stopper ( $W_2$  g) was reweighed. Distilled water at  $20^\circ\text{C}$  (a volume of 80 % of that of the container) was added and the stopper was replaced. The cylinder was inverted 10 times and was emptied and the cylinder and stopper ( $W_3$  g) were reweighed. The residue (R) and the rinsed residue (R') was calculated in %.

#### B-2 CALCULATION

$$R = \frac{W_2 - W_0}{W_1 - W_0} \times 100 \%$$
$$R' = \frac{W_3 - W_0}{W_1 - W_0} \times 100 \%$$



## ANNEX C

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

### DETERMINATION OF SPONTANEITY IN AMETOCTRADIN AND DIMETHOMORPH CONTENT

#### C-1 PRINCIPLE

The density of the formulation was determined and the mass of formulation equivalent to 12.5 mL (w g) was calculated. CIPAC Standard water D/C (237.5 mL) was poured at the required temperature into the graduated cylinder keeping it on a top-pan balance; the calculated mass of formulation from a small beaker was added to it. The addition was completed within 15 seconds. As soon as the formulation was added, the cylinder was stoppered and inverted once. The cylinder was kept in an upright position on a bench free from vibration or direct sources of heat for 5 minutes. At the end of the 5 minutes of settling period, the nozzle of a glass tube was dipped into the supernatant liquid in the cylinder and nine-tenths (225 mL) of the suspension was withdrawn with minimum disturbance within 10 to 15 seconds, using suction tube and vacuum pump maintaining the nozzle of the glass tube just below the liquid level.

The remaining 1/10<sup>th</sup> (25 mL) suspension was measured for its active ingredient by method used for active content determination using HPLC Method.

#### Chromatographic conditions:

Instrument	HPLC with PDA detector attached to LC solution software.		
Column	Chromatopack, C18 (250mm (L) x 4.6 mm(i.d.) x 5 µm particle size)		
Mobile phase	Acetonitrile: 0.2% v/v Triethyl amine in HPLC water (3.0 pH adjusted by formic acid)		
Detection wavelength	254 nm		
Flow rate	1.5 mL/min		
Column Temperature	Ambient		
Injection volume	25 µL		
Diluent	Acetonitrile		
Retention time	Dimethomorph approx.	Cis	9.74 min
		Trans	10.74 min
	Ametoctradin approx.		18.60 min

### C-3 PROCEDURE

**C-3.1 Preparation of Standard Solution** — 10 mg ( $\pm 2$ mg) of Ametoctradin standard and 10 mg ( $\pm 2$ mg) of Dimethomorph reference standard were weighed respectively into a 50 ml volumetric flask and made up to the mark with acetonitrile.

**C-3.2 Preparation of Sample Solution** — Remaining 1/10<sup>th</sup> (25 ml) sample after Spontaneity test was taken into separate 50 ml volumetric flask and made up to the mark using acetonitrile. 3.5 ml of this solution was taken into 100 ml volumetric flask and made up to the mark with acetonitrile.

**Estimation** — 25  $\mu$ l of standard and sample solution was injected into HPLC. From area obtained % spontaneity of dispersion was calculated as per below formula.

### C-4 CALCULATION

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

where

a = percent by mass of the a.i. in the sample

w = mass of sample added to the cylinder (g)

c = mass (e.g. a.i.'s) in the whole cylinder =  $\frac{a \times b}{100}$  (g)

Q = mass (e.g. a.i.'s) of the 25 ml remaining in the cylinder (g).

## **ANNEX D**

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

### **DETERMINATION OF PERSISTENT FOAM IN AMETOCTRADIN AND DIMETHOMORPH CONTENT**

#### **D-1 PROCEDURE**

180 ml of CIPAC Standard Water D was taken into a 250-ml measuring cylinder standing on a top pan balance and about 0.5 g of sample was weighed into it. Finally, the cylinder was made up to the 200-ml with CIPAC Standard Water D and recorded the temperature  $25\pm 5^{\circ}\text{C}$ . The cylinder was stoppered and inverted 30 times by hand through 180 degrees and back to its original position. The stoppered cylinder was placed upright on the bench and immediately the stopwatch was started. The volume of foam produced and remaining after 1 minute's  $\pm 10$  seconds was noted.

## **ANNEX E**

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

### **DETERMINATION OF pH CONTENT AT $25^{\circ}\text{C}$ IN AMETOCTRADIN AND DIMETHOMORPH CONTENT**

#### **E-1 PROCEDURE**

About 1 gm of sample was weighed into a measuring cylinder containing about 50-ml of water made up the volume to 100-ml with water and shaken vigorously until completely mixed or dispersed. The solution was transferred into a beaker (200-ml) and allowed any suspended material to settle for 1 min. The electrode was immersed into the liquid and immediately started the stop watch. The pH values after 1 min from immersing the electrode was recorded and reported at  $25^{\circ}\text{C}$ . (Two values should not differ by more than 0.1 pH units).