

FOREWORD

(Formal clause would be added later)

Azoxystrobin 18.2 % *m/m* + Difenoconazole 11.4% *m/m* SC is a mixed suspension concentrate used as a fungicide in agriculture.

Azoxystrobin 18.2 % *m/m* + Difenoconazole 11.4% *m/m* SC is a mixed suspension concentrate and is generally manufactured to contain Azoxystrobin 18.2 % *m/m* + Difenoconazole 11.4% *m/m*.

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 Azoxystrobin 18.2 % *m/m* + Difenoconazole 11.4% *m/m* SC.

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>
8190 (Part 2) : 1988	Requirements for packing of pesticides Part 2 liquid pesticides (<i>second revision</i>)
9754:1981	High density polyethylene containers for packing of liquid pesticides up to one liter.
6940 : 1982	Methods of test for pesticide and their formulations (<i>first revision</i>)
10627 : 1983	Methods for sampling of pesticidal formulation
12512:1989	High density polyethylene containers for packing of liquid pesticides above one liter up to 5 liters.

3 REQUIREMENTS

3.1 Constituents

3.1.1 The material shall consist of Difenoconazole technical and Azoxystrobin technical together with suitable carrier(s), stabilizer(s) and other formulants.

3.1.2 Difenoconazole and Azoxystrobin technical employed in the manufacture of the material shall conform to specifications.

3.2 Description

The material shall be light yellow to yellow liquid. The material shall consists of a suspension of active ingredient complying with requirement of specification. After gentle agitation the material shall be homogenous and suitable for further dilution in water.

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

TABLE 1 REQUIREMENTS FOR AZOXYSTROBIN + DIFENOCONAZOLE SC

(Clause 3.3.2)

Sl. No.	Characteristic	Requirement	Method of test, Refer to
(1)	(2)	(3)	(4)
i.	Azoxystrobin content, percent by mass, <i>Min</i>	18.2	Annex A
ii.	Difenoconazole content, percent by mass, <i>Min</i>	11.4	Annex A
iii.	pH of 1% aq. solution	5-9	Annex B
iv.	Persistent foam (ml after 1 min), <i>Max</i>	60	Annex C
v.	Wet sieve, <i>Max</i>	2	IS 6940
vi.	Pourability, percent by mass, <i>Max</i>	5	Annex D
vii.	Spontaneity of Dispersion , percent by mass, <i>Min</i>	70	Annex E
viii.	Suspensibility, percent by mass, <i>Min</i>	70	Annex F

3.3 Difenoconazole and Azoxystrobin content

When determined by the method prescribed in Annex A the observed difenoconazole and azoxystrobin content percent (*m/m*) of any sample shall not differ from the declared nominal value by more than the tolerance limits given below:

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

4 PACKING (TO BE UPDATED)

4.1 Retail packs up to 20 liters:

4.1.1 The material up to one liter shall be packed in HDPE and PET containers conforming to IS 9754 or IS 13123 and above one liter and up to five liters in HDPE/ PET container conforming to IS 12512 or IS 13123.

The specifications for the containers shall be as agreed between the supplier & the manufacturer.

The material shall be packed in HDPE and PET containers in the pack sizes of 25ml, 100ml, 200ml, 500ml & 1 L.

4.2 Bulk Packing: up to 200 Ltr.

4.2.1 Material shall be packed in HDPE containers. The specifications for the containers shall be as agreed between the supplier & the manufacture.

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 azoxystrobin and difenoconazole content, percent (w/v);
- 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 bulk manufactured material is offered for inspection, representative sample of the material shall be drawn as prescribed in IS 10627 and if tested within 90 days of its 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 clause no. 3.3 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 shall be the limit of tolerance given under 3.3 of this standard.

7 TESTS

7.1 Tests shall be carried out by the appropriate methods referred to in Table 1 of Clause 3.2.

7.2 Quality of reagents

Unless specified otherwise, pure chemicals and reagent grade 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 and II) and (Clause 3.3)

Determination of Difenconazole and Azoxystrobin

A-1 PRINCIPLE

A-1.1 Difenconazole and Azoxystrobin Content is determined by using Gas Chromatography. Quantification is by using internal standard technique.

A-2 APPARATUS

A-2.1 Gas Chromatography equipment with Flame ionization detector coupled to a printer and plotter. The operative conditions suggested below are typical and can be changed provided validation is done.

Column:	Fused silica crosslinked 14% - phenyl 86% dimethyl polysiloxane CP-Sil 13 CB .Maximum operating temperature 300°C
Oven temperature programme:	
Initial temperature	270°C
Initial time	10 min
Programme rate 1	30°C
Final temperature 1	300°C
Final time 1	4.5 min
Detector temperature	325°C
Injector temperature	275°C
Carrier Gas	Helium
Constant pressure mode	
Average linear velocity	35 cm sec ⁻¹ (at 270°C)
Nominal pressure	15 psi
Make up gas Nitrogen	30 ml min ⁻¹
Hydrogen detector gas	30 ml min ⁻¹
Air	400 ml min ⁻¹
Split flow	100 ml min ⁻¹
Injection volume	1 µl

A-2.2 Microliter syringe - 5 µl capacity / 10 µl capacity

A-2.3 Ultrasonic bath

A-2.4 Standard glassware

A-3 REAGENTS

A-3.1 Acetone HPLC grade or equivalent

A-3.2 3-(2-Pyridyl)-516-diphenyl-1 ,2, 4-triazine

A-3.5 Azoxystrobin and Difenoconazole reference standards of known purity

A-4 PROCEDURE

A-4.1 Preparation of internal standard.

Weigh approximately 1.0 g of 3-(2-Pyridyl)-5,6-diphenyl-1,2,4-triazine in a 500 ml volumetric flask and add 400 ml of acetone and sonicate until completely dissolved. Cool and make up to volume with acetone.

A-4.2 Preparation of Standard Solution

Weigh (to the nearest 0.1 mg) 120 mg of Azoxystrobin and 75 mg of Difenoconazole reference standards into a 25 ml volumetric flask add 20.0 ml of internal standard solution and sonicate for 2 minutes or until completely dissolved. Mix thoroughly.

A-4.3 Preparation of Sample (test) solution

Weigh (to the nearest 0.1 mg) sufficient sample to contain about 120 mg of Azoxystrobin and 75 mg of Difenoconazole into a 25 ml volumetric flask add 20.0 ml of internal standard solution. Sonicate for 10 minutes. Mix thoroughly. Filter using 0.45 micron filter and inject.

A-4.4 Estimation

Inject 1 µl of reference standard solution until the ISTD Response ratio obtained for two successive chromatograms do not deviate from each other by more than 2 %.

Then use the following injection sequence,

-----C S₁ S₁ C S₂ S₂ C S₃ S₃-----

Where,

C = Standard Solution

S = Sample Solution (1,2,3,--,n)

From the chromatograms of standard solution and sample solution, measure the ISTD response ratio of Azoxystrobin and Difenoconazole and compute the percentage as given in A-5.

A-4.4.1 Retention time (Guide values)

Difenoconazole 6.85 min

Internal standard 7.39 min

Azoxystrobin 9.01 min

A-4.4.2 Total run Approx 15.5 minutes

Retention time can vary depending on the column source & condition

A-5 CALCULATION

A-5.1 Difenoconazole content percent by mass = $\frac{M1 \times A2 \times P}{M2 \times A1}$

Where,

M1 = mass in g of standard difenoconazole in standard solution

M2 = mass in g of the sample taken for test

A1 = ISTD Response ratio of difenoconazole in the chromatogram of standard

A2 = ISTD Response ratio of difenoconazole in the Chromatogram of Sample

p = percentage purity of difenoconazole in reference standard

A-5.2

Azoxystrobin content percent by mass = $\frac{M1 \times A2 \times P}{M2 \times A1}$

Where,

M1 = mass in g of standard azoxystrobin in standard solution

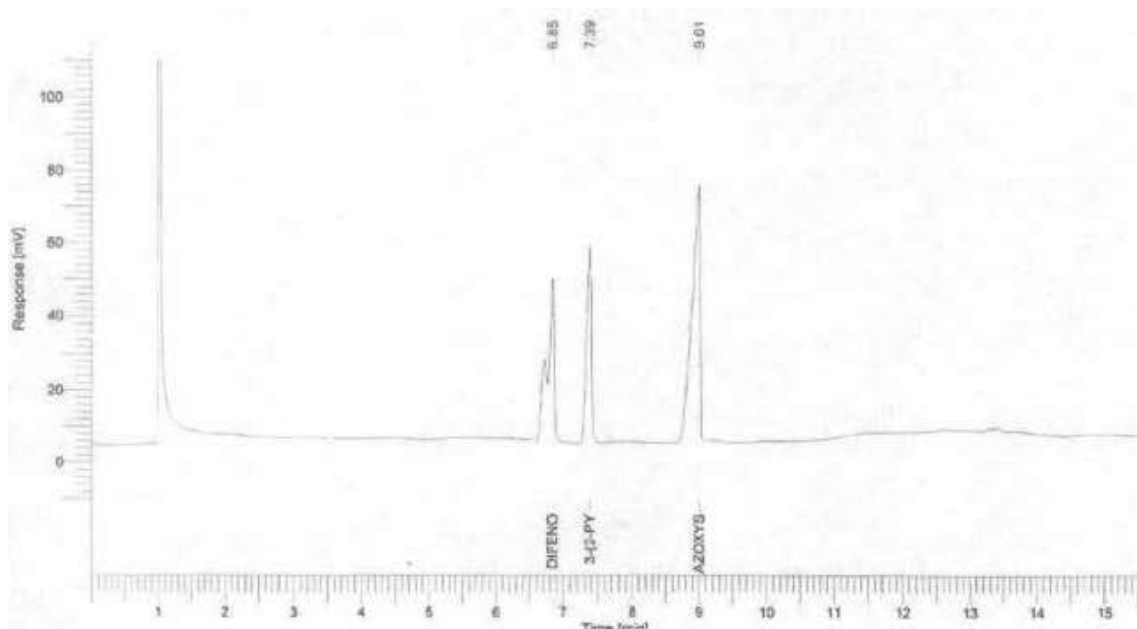
M2 = mass in g of the sample taken for test

A1 = ISTD Response ratio of azoxystrobin e in the chromatogram of standard

A2 = ISTD Response ratio of azoxystrobin in the Chromatogram of Sample

p = percentage purity of azoxystrobin in reference standard

A typical chromatogram is enclosed



ANNEX – B

Table 1, Sl. No. (iii)

DETERMINATION OF pH

B-1 OUTLINE OF METHOD

The pH value of a mixture of a sample with water or of an undiluted aqueous formulation is determined by means of a pH meter and an electrode system.

B-2 REAGENTS

Buffer solution pH 9.2, and pH 4.0

B-3 APPARATUS

- 1 pH meter capable of at least two point calibration
- 2 Electrode system e.g. glass electrode system, conditioned and stored according to the manufacturer's instructions.
- 3 Measuring cylinder stoppered, 100 ml
4. Beaker 200 ml.

B-4 PROCEDURE:

a. Calibration

Operate the pH meter and the electrode system according to the manufacturer's instructions. Calibrate the system (pH meter and electrode) according to the manufacturer's instructions using at least two appropriate buffer solutions.

b. Measurement of pH values

Weigh 1.0 gm (to the nearest 0.01 g) of the sample in to a measuring cylinder containing about 50 ml of water, make up to 100 ml with water and shake vigorously until completely mixed or dispersed. If necessary, transfer the solution or dispersion to a beaker (200 ml) and allow any suspended material to settle for 1 min. ensure that the temperature of the sample/water mixture does not differ from the temperature of the buffer used at the time of calibration. Immerse the electrode into the liquid and measure its pH without stirring. Record the pH value after 1 minute.
Read the pH value with an accuracy of ± 0.1 pH units.

ANNEX- C
Table 1, Sl. No. (iii)

DETERMINATION OF PERSISTENT FOAMING

C-1 OUTLINE OF METHOD

The suspension concentrate is diluted in a measuring cylinder of standard dimensions which is inverted 30 times and the amount of foam created and remaining after certain times is measured.

Reagent

Standard hard water.

Apparatus

1. 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

2. stopwatch

Procedure:

The mass of sample to be taken is that mass required 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 hard 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

hard water up to 200 ml mark. 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 after one minute.