1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for azoxystrobin and tebuconazole suspension 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*
8190 (Part 2) : 1988	Requirements for packing of pesticides Part 2 liquid pesticides (*second revision*)
1070 : 1992	Reagent grade water - Specification (*third revision*)
6940 : 1982	Methods of test for pesticide and their formulations (*first revision*)
10627 : 1983	Methods for sampling of pesticidal formulation

3 REQUIREMENTS

3.1 Constituents

The material shall consist of azoxystrobin and tebuconazole technical, together with suitable carrier(s) stabilizer(s) and other formulant(s).

3.1.2 Azoxystrobin and Tebuconazole technical employed in the manufacture of the material shall conform to specification for Azoxystrobin and Tebuconazole Technical as and when published.

3.2 Description

The material shall be in the form of a tight brown viscous liquid.

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

3.3.1 *Azoxystrobin and Tebuconazole Content* – When determined by the method annexed, the observed Azoxystrobin and Tebuconazole content, percent [m/m), of any of the samples shall not differ from the declared nominal value by more than tolerance limits indicated below:

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

TABLE 1 REQUIREMENTS FOR AZOXYSTROBIN + TEBUCONAZOLE SC

(*Clause 3.3.2)*

Sl. No.	**Characteristic**	**Requirement**	**Method of test, Refer to**
(1)	(2)	(3)	(4)
i.	Azoxystrobin content, percent by mass, *Min*	11.0 (+/- 5%)	Annex A
ii.	Tebuconazole content, percent by mass, *Min*	18.3 (+/- 5%)	Annex B
iii.	Suspensibility, percent by mass, *Min*	90.0	Annex C
iv.	*p*H of 1% aq. solution	6.5-7.0	Annex D
v.	Persistent foam (ml after 1 min), Max	5 ml	Annex E
vi.	Spontaneity of Dispersion , percent by mass, Min	80.0	Annex F
vii.	Density(g/ml) at 20°C	1.09	IS 6940

4 PACKING

The container shall conform to the general requirements specified in of 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 azoxystrobin and tebuconazole 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

Representative samples of the material shall be drawn as prescribed in IS 10627, Methods for Sampling of Pesticidal Formulations.

7 TESTS

7.1 Tests shall be earned out by the methods referred to in 3.2 & 3.3 of this specification.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

ANNEX A

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

DETERMINATION OF AZOXYSTROBIN AND TEBUCONAZOLE

A-1 PRINCIPLE

To determine azoxystrobin and tebuconazole content in a combinational fungicide azoxystrobin 11% and tebuconazole 18.3% m/m SC.

A-2 APPARATUS

A-2.1 Syringe - 25µl capacity

A-2.2.1 *Chromatographic parameters for azoxystrobin*

Instrument	Gas Chromatograph Model GC-17A equipped with Flame Ionization Detector, Auto injector and interfaced with GG-solution software
Column	Fused silica, (25m length x 0.32mm ID. x 0,2um film thickness) CP-Sil 13CB Phase (Cross linked 14% phenyl 86% dimethyl polysiloxane)
Detector	Flame Ionization Detector
Temperature conditions :	
Oven	240°C
Injector	275°C
Detector	325°C
Oven Programme:	
Temp 1	240°C hold 0 min, ramp rate 3°C/min

Temp 2	270°C hold 0 min, ramp rate 30°C/min
Temp 3	320°C hold 5 min
Gas Flow Rate:	
Nitrogen (make up)	30 ml/min
Hydrogen	75 cm/sec Typically psi at 240°C (run at constant pressure)
Air	400 ml/min
Hydrogen (Carrier)	30 ml/min
Volume injected	1 µl
Injection mode split ratio :	
Split	50 : 1
Retention time (Approximate)	
3-(2-pyridyl)-5,6-diphenyl-1,2,4 triazine (Internal standard)	7.1 min
Azoxystrobin	8.1 min

A-2.2.1 *Chromatographic parameters for tebuconazole*

Instrument	Gas Chromatograph Model GC-17A equipped with Flame Ionization Detector, Auto injector and interfaced with GG-solution software
Column	13 CB (25m length x 0.32mm I.D x 0.2 µm film thickness)
Detector	Flame Ionization Detector
Temperature conditions :	
Oven	250°C
Injector	275°C
Detector	290°C
Gas Flow Rate:	
Nitrogen (Column flow)	2.65 ml/min
Make up (Nitrogen)	30 ml/min
Hydrogen	40 ml/min
Air	400 ml/min
Volume injected	0.1 µl
Injection mode split ratio :	
Split	25 : 1
Purge	3.0 ml/min
Retention time (Approximate)	
Tebuconazole	2.6 min
Dioctyl phthalate (Internal standard)	3.4 min

A-3 REAGENTS

A-3.1 Acetonitrile

A-3.2 Azoxystrobin Analytical Standard

A-3.3 Tebuconazole Analytical standard

A-4 PROCEDURE

A-4.1 Outline of the method

Azoxystrobin and Tebuconazole content in azoxystrobin 11% and tebuconazole 18.3% SC *m*/*m* sample was determined by a GC method. The identity of the active ingredient to established by comparison until the equivalent authentic standard and quantified the active contents by external standardisation method.

A-4.2 Preparation of Internal Standard Solution

A-4.2.2 *Azoxystrobin*

Accurately 0.25g of 3-(2-pyridyl)-5,6-diphenyl-1,2,4 triazine was weighed in a 100 ml volumetric flask. The contents is dissolved and the flask was brought up to the mark with acetone

A-4.2.1 *Tebuconazole*

Accurately 0.5g of dioctyl phthalate was weighed in a 100 ml of volumetric flask. The contents are dissolved and the flask is brought up to the mark with acetone

A-4.3 Preparation of Standard Solution Azoxystrobin

A-4.3.1 *Azoxystrobin*

About 5.20 mg of azoxystrobin reference standard (C2) of purity 99.7% weighed into a 15 ml capacity bottle and 10 ml of internal standard is added by using pipette and sonicated above 1 minute to dissolve the content.

A-4.3.2 *Tebuconazole*

Accurately 20.20 mg of tebuconazole reference standard (C2) of purity 98.2% is weighed into a 25 ml volumetric flask and 4 ml of internal standard is added by using pipette and sonicated above 1 minute to dissolve the content.

A-4.4 Preparation of sample solution:

A-4.4.1 *Azoxystrobin*

Approximately 45 (± 3) mg of formulation sample was weighed into two different (R1 and R2) 15 ml capacity bottles, to each bottle 10 ml of internal standard was added by using pipette. Sonicated for about 1 minute to dissolve the content in the bottle.

A-4.4.2 *Tebuconazole*

Approximately 100 (± 6) mg of formulation sample was weighed in to two different (R1 and R2) 25 ml of volumetric flask and 4 ml of internal standard is added by using pipette. The contents are dissolved and the flask was brought up to the mark with acetone.

A-4.5 Sample analysis:

Injected in the sequence C2, S1R1, S1R2, C2, S2R1, S2R2 and analysed for Azoxystrobin and Tebuconazole content.

A-5 ESTIMATION

The sample as well as reference standard solution of both azoxystrobin and tebuconazole was injected in GC and flame ionisation detector. The peak integration and peak area calculation is carried out using GC Solution software.

A-6 CALCULATION

Azoxystrobin/Tebuconazole content (% *m*/*m*) = $\frac{H_w \times I_r \times M \times P}{H_S \times I_q \times w}$

Where

H_s = Peak area of azoxystrobin/tebuconazole in the standard solution (µV-sec);

H_w = Peak area of azoxystrobin/tebuconazole in the sample solution (µV-sec);

I_r = Peak area of internal in the standard solution (µV-sec);

I_s = Peak area of internal in the sample solution (µV-sec);

M = Mass of azoxystrobin/tebuconazole in the standard solution (mg)

w = Mass of sample taken (mg)

P = Purity of azoxystrobin/tebuconazole reference standard (%)

A-7 CHROMATOGRAM

Model chromatogram enclosed

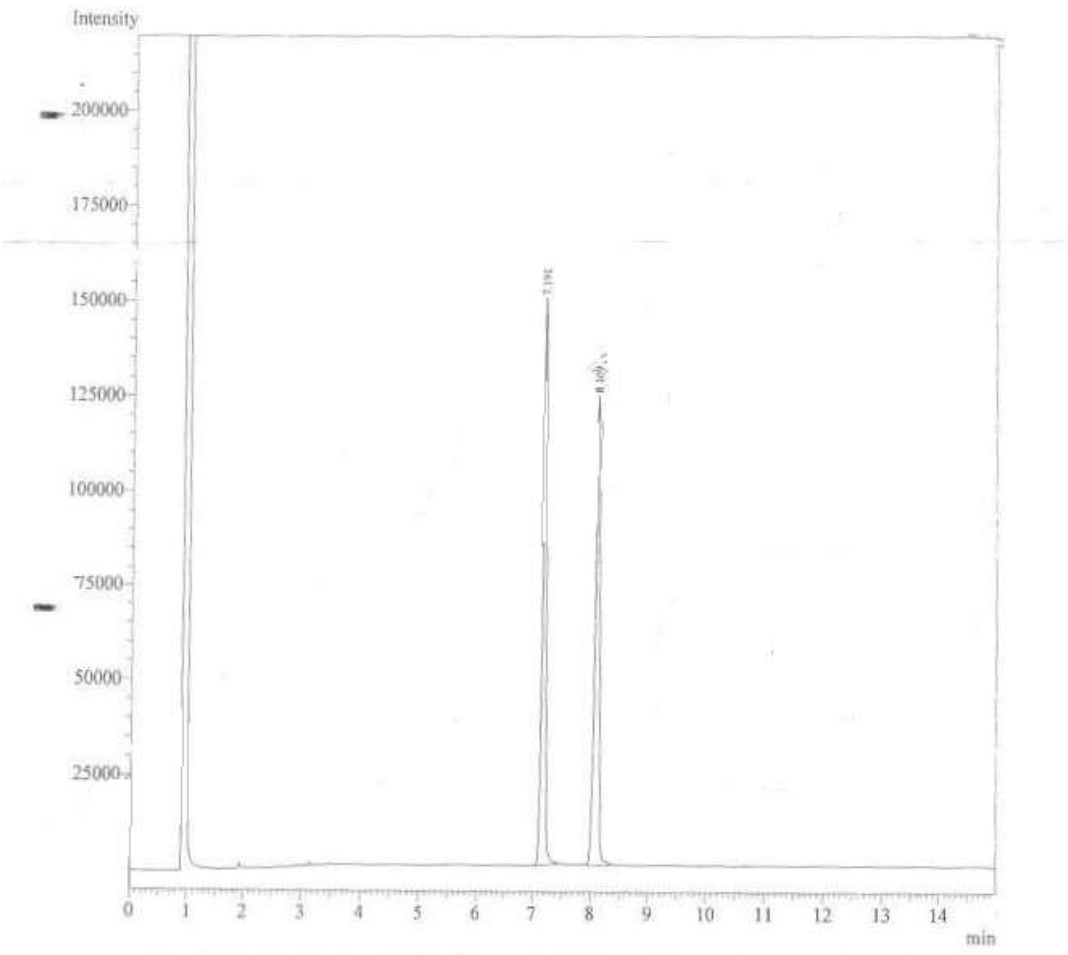

Fig 1. Azoxystrobin

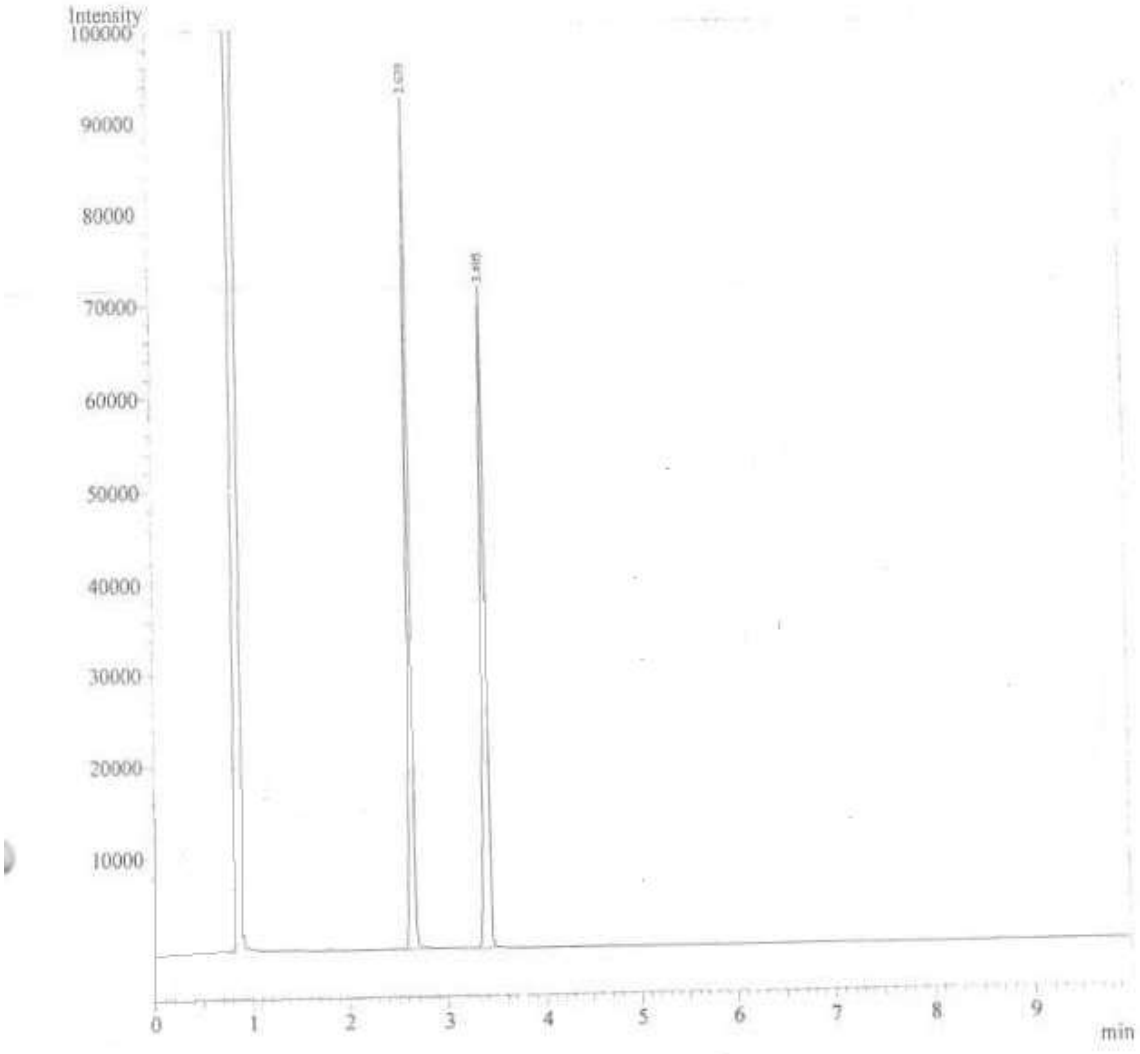

Fig 2. Tebuconazole

ANNEX B

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

DETERMINATION OF PERSISTENT FOAM

B-1 REAGENT

Calcium carbonate

Hydrochloric acid

Ammonia solution

Magnesium oxide

B-2 APPARATUS

Glassware – Stoppered measuring glass cylinder (100 ml capacity), Weighing bottle

Beaker (500 ml), Standard flask (100 and 1000 ml capacity)

Stop watch

Weighing balance

B-3 PREPARATION

B-3.1 Preparation of Solution - I

Accurately 4 g of calcium carbonate was taken in a 500 ml glass beaker containing 50 ml of distilled water. Then 82 ml of 1 M hydrochloric acid was added into die same beaker. pH of this solution was adjusted to 7.0 by using 1 M hydrochloric acid and 1 M ammonia solution. Then it was transferred into 1000 ml standard flask and diluted upto the mark by using distilled water.

B-3.2 Preparation of Solution — II

Accurately 1,613 g of Magnesium oxide was taken in 500 ml beaker containing 50 ml distilled water. Then 82 ml of 1 M hydrochloric acid was added into the same beaker. pH of this solution was adjusted to 7.0 by using 1 M hydrochloric acid and 1 M ammonia solution. Then it was transferred into 1000 ml standard flask and diluted up to the mark by using distilled water.

B-3.3 Preparation of Standard water

Accurately 100 ml of solution -I and 25 ml of solution -II was taken in 1000 ml standard flask. pH of this solution was adjusted to 7.14 by using IM hydrochloric acid and IM Ammonia solution. Then it was diluted up to the mark by using deionized water.

B-3.4 Determination of Persistence foam

Accurately 180 ml of standard water taken in a 250 ml stopper measuring cylinder. Then 0.6 ml of test item was added into the same cylinder. Standard water was added until the distance between the suspension surface and the bottom of the ground glass joint is 9±0.1 cm. It was made up to the mark by using standard water. This cylinder was inverted at 30 times. Then it was placed upright on the bench and volume of foam was measured after 1 minute by using stop watch. This study was conducted twice and the results were reported.