FOREWORD

(*Formal clause would be added later)*

Thiophanate Methyl 450 g/L + Pyraclostrobin 50 g/L Flowable Suspension Concentrate is used as a Fungicide in Agriculture.

Thiophanate Methyl+ Pyraclostrobin Flowable Suspension Concentrate is generally manufactured to contain Thiophanate Methyl 45% and Pyraclostrobin 5% 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

1.1 This standard prescribes the requirements and the methods of sampling and test for Thiophanate Methyl+ Pyraclostrobin Flowable 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*)
10946 : 1984	Method of sampling for technical grade pesticides
10627 : 1983	Methods for sampling of pesticidal formulations

3 REQUIREMENTS

3.1 Constituents

The material shall consist of Thiophanate Methyl technical and Pyraclostrobin technical together with suitable ingredients.

3.2 Physical

3.2.1 *Description* **—** The material shall be green coloured, flowable liquid, free from external impurities, which on dilution with water readily form a suspension.

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

TABLE 1 REQUIREMENTS FOR THIOPHANATE METHYL 450 g/l + PYRACLOSTROBIN 50 g/l

(*Clause* **3.2.1** and **7.1**)

Sl. No.	Characteristics	Requirement	Annex of this standard
(1)	**(2)**	**(3)**	**(4)**
i.	Thiophanate Methyl content , percent by mass, *Min*		A
ii.	Pyraclostrobin, percent by mass, *Min*		A
iii.	Pourability, percent by mass, *Max*	3	B
iv.	Suspensibility , percent by mass, *Min*	90	IS 6940
v.	Persistent foam (ml), *Max*	60 sec	C
vi.	Wet sieve test (maximum % Passed through 75µm test sieve)	95	IS 6940
vii.	Acidity (as H_2SO_4), percent by mass, *Max*	0.15	IS 6940

3.3 Chemical

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

3.3.1 *Thiophanate Methyl and Pyraclostrobin content*

When determined by the method prescribed (enclosed), the observed Thiophanate Methyl and Pyraclostrobin content (w/v), of any of the sample shall not differ from the declared nominal value by more than the percent tolerance limits indicated below :

Nominal Value, Percent	*Tolerance, Percent*	
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 Thiophanate Methyl and Pyraclostrobin 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**.

3.3.1.2 The average Thiophanate Methyl and Pyraclostrobin content of all samples taken shall not be less than the declared nominal content.

4 PACKING

4.1 The product shall be packed in 80 ml, 250 ml, 400 ml, 1000 ml, 2500 ml and 5000 ml HDPE containers with minimum 1 mm thickness, which shall be further packed in corrugated fiber board boxes as transport packing. The specifications for the containers shall be as agreed between the supplier and the manufacturer.

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 Thiophanate Methyl and Pyraclostrobin content, percent (*m/m*);
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 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 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 THIOPHANATE-METHYL AND PYRACLOSTROBIN CONTENT

A-1 PRINCIPLE

Thiophanate-methyl and Pyraclostrobin content in Thiophanate-methyl 450 g/l + Pyraclostrobin 50 g/l FS formulation was determined by a HPLC-UV method. The identity of the active ingredient was established by comparison with the equivalent authentic standard and quantified the active contents by external standardization method.

A-2 REAGENT

Acetonitrile - HPLC grade Millipore water
Formic acid
Thiophanate-methyl Analytical Standard
Pyraclostrobin Analytical Standard

A-3 PROCEDURE

A-3.1 Preparation of Calibration solution (C2)

Accurately weighed 39.70 mg of Thiophanate methyl (purity 99.78%) and 4.31 mg of Pyraclostrobin (purity 99.9%) reference standards into a 50 mL capacity volumetric flask. Initially the content of the flask was dissolved in an aliquot of acetonitrile and the volume was brought up to the mark with acetonitrile**.**

A-3.2 Preparation of sample solution

Weighed about 100 mg of the formulation in two different (S1 and S2) 50 ml volumetric flasks. Exactly 5 mL of water was added to each volumetric flask to suspend the formulation. The volumes of the flasks were brought up to the mark with acetonitrile. A portion of the solution was filtered through 0.2 micron PTFE nylon syringe filter and analysed in duplicate by HPLC-UV method

A-3.3 Chromotographic Separation Parameter

Instrument		HPLC 1200 Series
Column used		C18 (250 x 4.60 mm i.d. x 5.0 µm particle size)
Mobile Phase		70% A= Acetonitrile (HPLC grade) 30% B = 0. 1 % Formic acid
Detector		Diode Array Detector (DAD)
Wave Length		270 nm
Column oven temperature		30 °C
Injection Volume		5 µL
Flow rate		1 ml/min
Retention time (approximately)	Thiophanate-methyl	3.5 min
	Pyraclostrobin	8.3 min

A-4 CALCULATION

A-4.1 Thiophanate-methyl

Thiophanate-methyl Content, percent by mass = $\frac{H_W \times M \times P}{H_s \times w}$

where

*H*s = Peak area of Thiophanate-methyl in Standard solution(mAU)

*H*w = Peak area of Thiophanate-methyl in the sample solution(mAU)

M = Weight of Thiophanate-methyl in the Standard solution(mg)

w = Weight of sample taken (mg)

P = Purity of Thiophanate-methyl reference standard (%)

Thiophanate-methyl content(% *w/v*)= Thiophanate-methyl content(% *m/m*) X Density

A-4.2 Pyraclostrobin

Pyraclostrobin Content, percent by mass = $\frac{H_W \times M \times P}{H_s \times w}$

where

*H*s = Peak area of Pyraclostrobin in Standard solution (mAU)

*H*w = Peak area of Pyraclostrobin in the sample solution (mAU)

M = Weight of Pyraclostrobin in the Standard solution (mg)

w = Weight of sample taken (mg)

P = Purity of Pyraclostrobin reference standard (%)

Pyraclostrobin content(% *w/v*)= Pyraclostrobin content(% *m/m*) X Density

Density of Thiophanate Methyl 450 g/l + Pyraclostrobin 50 g/l formulation = 1.223 g/mL

ANNEX B

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

DETERMINATION OF POURABILITY IN THIOPHANATE-METHYL AND PYRACLOSTROBIN CONTENT

C-1 PROCEDURE

Weighed the empty container and stopper (w_0 g) and add enough of the suspension concentrate taken from mixed bulk sample to leave approximately 20% of the volume of the container as ullage. Replaced the stopper and reweigh the container (w_1 g). Allowed the container to stand undisturbed for 24 hrs and then poured out the suspension concentrate for 60 s at an angle of 45° and then finally inverted the container for 60 s. Reweighed the container and stopper (w_2 g).

Added distilled water at 20 °C (a volume of 80% of that of the container) and replaced the stopper. Inverted the container 10 times and made empty the container as before and reweighed the container and stopper (w_3 g). Calculated the residue (R) and the rinsed residue (R').

C-4 CALCULATIONS

$$R = \frac{(w_2 - w_0)}{(w_1 - w_0)} \times 100$$

$$R' = \frac{(w_3 - w_0)}{(w_1 - w_0)} \times 100$$

ANNEX C

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

DETERMINATION OF PERSISTANT FOAM IN THIOPHANATE-METHYL AND PYRACLOSTROBIN CONTENT

C-1 PROCEDURE

The mass of the sample taken is the mass required to make 200 ml of a suspension with a concentration recommended in the directions for use supplied with the product (2ml).

Standard water (Approx 180 ml) was added to a 250 ml measuring cylinder standing on a top pan balance and the test item of maximum concentration recommended was weighed (2 ml). Standard water was added until the distance between the suspension surface and the bottom of the ground glass joint is 9 ± 0.1 cm. The cylinder was closed with the stopper and inverted 30 times, placed upright on the bench and the stopwatch was started immediately. The volume of foam produced and remaining after 10±1 sec, 1, 3, and 12 min± 10 sec noted