Doc: FAD 01 (20125) WC
January 2024

BUREAU OF INDIAN STANDARDS

DRAFT FOR COMMENTS ONLY

भारतीय मानक मसौदा

फ्लक्सापायरोक्सैड + पायराक्लोस्ट्रोबिन सस्पेंशन कॉन्सन्ट्रेट - विशिष्टि

Draft Indian Standard

FLUXAPYROXAD + PYRACLOSTROBIN SUSPENSION CONCENTRATE – SPECIFICATION

ICS No. 65.100.30

Pesticides Sectional Committee, FAD 01 Last Date of Comments: **14 March 2024**

FOREWORD

(*Formal clause would be added later)*

Fluxapyroxad + Pyraclostrobin suspension concentrate is used as a fungicide in agriculture.

Fluxapyroxad + Pyraclostrobin suspension concentrate is generally manufactured to contain 167 g/l of fluxapyroxad and 333 g/l of pyraclostrobin or 250 g/l of fluxapyroxad and 250 g/l of pyraclostrobin.

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 Fluxapyroxad + Pyraclostrobin 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*
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 formulations

3 REQUIREMENTS

3.1 Constituents

The material shall consist of Fluxapyroxad technical and Pyraclostrobin technical, together with suitable ingredients.

3.2 Physical

3.2.1 *Description* **—** The material shall be off-white liquid, free from extraneous matters, which on dilution with water readily forms a suspension, which is suitable for spray.

3.2.2 *p*H – When determined by the method prescribed in Annex A, the *p*H of 1% aqueous solution of the material shall be in the range 5.5 – 8.5.

3.2.3 *Pourability* – When determined by the method prescribed in Annex B, the pourability shall not be more than 5.

3.2.4 *Spontaneity of Dispersion* – When determined by the method prescribed in Annex C, the spontaneity of dispersion shall not be less than 70 percent (*m/m*).

3.2.5 *Suspensibility* – When determined by the method prescribed in Annex D, the suspensibility shall be minimum 70 percent (*m*/*m*).

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

3.2.7 *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 Annex F.

3.3 Chemical

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

3.3.1 *Fluxapyroxad and Pyraclostrobin content*

When determined by the method prescribed in Annex G, the observed fluxapyroxad and pyraclostrobin content (*m*/*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	
Above 9 and below 50	±5	of the nominal
50 and above	+5	value
	-3	

3.3.1.1 The actual value of fluxapyroxad 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 fluxapyroxad and pyraclostrobin content of all samples taken shall not be less than the declared nominal content.

4 PACKAGING

The product shall be packed in HDPE containers with minimum 1 mm thickness, which shall be further packed in conjugated fiber board boxes as transport packing. 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 fluxapyroxad 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

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 **3.2.1** to **3.2.7** and **3.3.1.**

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

[*Clause* 3.2.2]

DETERMINATION OF *p*H

A-1 PRINCIPAL

Determine the *p*H value of a mixture of a sample with a water or of an undiluted aqueous formulation by means of a *p*H meter.

A-2 CHEMICALS

A-2.1 *p*H 4.0 Standard Buffer Solution

A-2.2 *p*H 9.0 Standard Buffer Solution

A-2.3 Distilled Water (CO_2 free).

A-3 APPARATUS

A-3.1 Standard Measuring Flask, 100 ml capacity

A-3.2 Beaker, 100 ml capacity

A-3.3 Glass Rod

A-3.4 Weighing Balance

A-3.5 *p*H Meter

A-4 PROCEDURE

A-4.1 Preparation of 1% Sample Solution

Weigh accurately 1 g of homogenized sample into 100 ml standard measuring flask and make up to the mark using distilled water (CO_2 free).

A-4.2 Calibration of *p*H Meter

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

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

Add the 1.0 g of test sample into a100 ml volumetric flask containing 50 ml of distilled water. Bring the volume of the flask up to 100 ml mark using distilled water and shake vigorously for 1 minute, allow the suspension to settle for 1 minute and then transfer the supernatant liquid into a beaker. Measure the *p*H of supernatant liquid at one minute and two minutes. The change in *p*H was not more than 0.1 unit after the initial immersion of the electrode and record data.

ANNEX B
[*Clause* 3.2.3]
DETERMINATION OF POURABILITY

B-1 OUTLINE OF THE METHOD

The suspension concentrate is allowed to stand for a definite time and the amount remaining in the container after a standardized pouring procedure is determined. The container is rinsed and the amount then remaining is determined.

B-2 APPARATUS / EQUIPMENT

A 500 ml stoppered measuring cylinder as per following requirements:

a) Volume equivalent to 1 subdivision of the scale	: 5 ml
b) Capacity corresponding to lowest graduation mark	: 50 ml
c) Capacity corresponding to highest graduation mark	: 500 ml
d) Length of scale	: 250 mm
e) Overall height	: 39 cm
f) Diameter of base	: 10 cm
g) Stopper	: B 34

NOTE — High density polyethylene bottles, 1 000 ml volume and Kilner jars, 700 ml volume, can be used but this must be recorded with the result.

B-3 PROCEDURE

Weigh the empty container along with stopper (W_0) and add enough of the test item taken from a recently mixed bulk sample to leave approximately 20 percent of the volume of the container as ullage. Replace the stopper and reweigh the container (W_1). Allow the container to stand undisturbed for 24 h and then pour out the suspension concentrate (*see* Note 1) for 60 s at an angle of 45° and then finally invert the container for 60 s (*see* Note 2). Re-weigh the container and stopper (W_2).

Add distilled water at 20°C (a volume of 80 percent of that of the container) and replace the stopper. Invert the container 10 times (*see* Note 3) and empty the container as before and reweigh the container and stopper (W_3). Calculate the residue (R) and the rinsed residue (R_1).

B-4 CALCULATION

$$\text{Residue (R), percent by mass} = \frac{W_2 - W_0}{W_1 - W_0} \times 100$$

$$\text{Rinsed residue } (R_1), \text{ percent by mass} = \frac{W_3 - W_0}{W_1 - W_0} \times 100$$

where,

W_0 = weight of the empty container, in g;

W_1 = weight of the container with the sample, in g;

W_2 = weight of the container after the pouring out of the suspension concentrate, in g;

W_3 = weight of the container after removal of distilled water, in g;

NOTES

1 The length of the standing period and the temperature should be agreed previously.
2 A square sided container should be held so that a flat side is underneath.
3 The term invert the container means that the container's vertical axis is turned through 180°C and then brought back to its original position, the whole operation taking about 2s.

ANNEX C
[*Clause* 3.2.4]
DETERMINATION OF SPONTANEITY OF DISPERSION

C-1 PRINCIPLE

The method is broadly similar to that used to determine the suspensibility of concentrates, except that it employs only one inversion and 5 min standing time. It involves preparing 250 ml of a mixture formulation and water, mixed with only one inversion of the measuring cylinder. After standing under defined condition the top nine-tenth is removed, and the remaining tenth assayed chemically, gravimetrically or by solvent extraction. The spontaneity of dispersion is calculated.

C-2 REAGENT

C-2.1 Standard Water

C-3 APPARATUS

C-3.1 Constant Temperature Bath

C-3.2 Graduated Cylinders

C-3.3 Glass Suction Tubes

C-3.4 Centrifuge

C-3.5 Filtration Assembly

C-4 PROCEDURE

The standard water, measuring cylinder and sample to be used in the determination is equilibrated at 25 ± 5 °C before starting the test.

Determine the density of the formulation to calculate the mass of formulation equivalent to 12.5 ml (w g). Pour the standard water (237.5 ml) into the graduated cylinder which is kept on a top–pan balance, and add the calculated mass of homogenized formulation from a small beaker held so that the tip is 1 cm above the top of the cylinder. Complete the addition within the time limit of 15 seconds.

As soon as the formulation is added, close the cylinder with stopper and invert it once. Allow the cylinder to stand in an upright position on a bench free from vibration or direct sources of heat for 5 min ± 10 s. At the end of this time, carefully remove the stopper withdrawn the top 225 ml of suspension by means of the suction tube connected to reservoir and suitable pump. It will be ensured that the tip of the tube will be always only few millimeters below the surface of the suspension. Assay the 25 ± 1 ml of dilute suspension remaining in the cylinder for its active ingredient.

C-5 CALCULATION

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

where,

a = percentage by mass in formulation;

w = mass of formulation added to the cylinder, in g;

C = mass in the whole cylinder, in g = $\frac{w \times a}{100}$;

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

ANNEX D
[*Clause* 3.2.5]
DETERMINATION OF SUSPENSIBILITY

D-1 PRINICPLE

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

D-2 REAGENT

D-2.1 Standard Water

D-3 APPARATUS

D-3.1 Constant Temperature Bath

D-3.2 Graduated Cylinders

D-3.3 Glass Suction Tubes

D-3.4 Centrifuge

D-3.5 Filtration Assembly

D-1 PROCEDURE

D-1.1 Weigh approximately 5 g (±0.2 g) of material into a 100 ml beaker and added a 10 ml of the standard hard water at 30 ± 1 °C. Allow to stand for 30 seconds and then stir by hand for 30 seconds with a glass rod 4-6 mm in diameter at not more than, 4 rev/s. Transfer the slurry into a 250 ml graduated measuring cylinder. Wash the beaker with small quantities of standard hard water at 30 ± 1 °C and transfer the washings to the cylinder. The cylinder volume is brought upto the 250 ml mark with standard water at 30 ± 1 °C. Close the cylinder with the stopper and invert it sharply through 30 complete cycles within one minute. Allow the cylinder to stand at rest for 30 minutes in a water bath free from vibrations maintained 30 ± 1 °C. At the end of the settling period, dip the nozzle of a glass tube into the supernatant liquid contained in the cylinder and withdrawn nine-tenths (225 ml) of the suspension with minimum disturbance within 10 to 15 seconds, using vacuum pump maintaining the nozzle of the glass tube just below the surface of the suspension in the Cylinder. Discard the suspension so withdrawn and the sediment at the bottom of the cylinder not exceeding 25 ml one-tenth of the suspension is analyzed for the amount of active ingredient by HPLC (*see* Annex A).

D-1.2 Sample Preparation Method:

Take the remaining suspension (25ml) in 100 ml volumetric flask. Rinse the measuring cylinder with 25 ml distilled water and transfer the content in to a 100 ml volumetric flask. Repeat the rinse process again to ensure 100 percent transfer of suspension from measuring cylinder to volumetric flask. Make up to the mark with distilled water. Analyze for the amount of active ingredient by HPLC as per the procedure above.

D-2 CALCULATION

Suspensibility, percent by mass = $\frac{1000\ (M-m)}{9 \times M}$

where,

M = mass in g of pesticide (a.i) present in the material taken for the preparation of the suspension;

m = mass in g of pesticide (a.i) found in the suspension including the sediment remaining in the bottom of the graduated cylinder

ANNEX E
[*Clause* 3.2.6]
DETERMINATION OF PERSISTENT FOAM

E-1 OUTLINE OF THE 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 time is measured.

E-2 APPARATUS

E-2.1 Graduated Cylinder

Glass stoppered of 250 ml capacity, with 2 ml graduations. The distance between the 0 mark and the 250 ml mark should be 20 to 21.5 cm and between the 250 ml mark and the bottom of the stoppered, 6-8 cm (cylinder should be clean and free from grease).

E-2.2 Stopwatch

E-3 REAGENT

Standard water

E-4 PROCEDURE

The mass of sample to be taken is that mass required to make 200 ml of a diluted formulation with a concentration recommended in the directions for use supplied with the product. When 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 the sample. Fill to 200 ml with standard hard water and record the temperature (*see* Note 1). Stopper the cylinder and then invert it 30 times by hand through 180 degrees and back to its original position, the whole operation being completed in approximately 2 s. Place the stoppered cylinder upright on the bench and immediately start the stopwatch. Read the volume of foam produced and remaining after 1 min (*see* Note 2).

NOTES
1 The recommended temperature is 25 ± 5°C.
2 A few bubbles round the periphery are not significant. Any volumes above the 250 ml mark should be marked on the outside and the volume of foam thus determined.

ANNEX F
[*Clause* 3.2.7]
DETERMINATION OF WET SIEVE

F-1 PRINCIPLE

The method is suitable for the determination of the amount of the non-dispersible material in Fluxapyroxad + Pyraclostrobin SC.

F-2 APPARATUS

F-2.1 Test Sieve – specified IS Sieve [*see* IS 460 (Part 1)] prepared for the test by removing any film, grease or any other water repellant material and then by drying

F-2.2 Rubber Hose – 10 mm internal diameter

F-3 PROCEDURE

Wet Sieving – Transfer 10 g of the sample to the sieve, rinsing with tap water. Wash the material on the sieve with an oscillating jet of tap water. Using a rubber hose of 10 mm internal diameter. delivering 4-5 litres of water per min. Continue the washing for 10 min, directing the water from the circumference of the sieve towards the center and keeping the end of the hose at a distance of between 2-5 cm from the surface of sieve. Transfer the residue to a pre-weighed watch glass with a jet of distilled water from a wash bottle. Dry to constant weight and record the weight of the sample to the nearest 0.01 g.

F-4 CALCULATION

Material passing through the IS sieve, percent by mass = $100\ (1-m/M)$

where,

m = mass in gm of the dry residue obtained; and
M = mass in g of the material taken for test.

ANNEX G

[*Clause* 3.3.1]

DETERMINATION OF FLUXAPYROXAD AND PYRACLOSTROBIN CONTENT

G-1 PRINCIPLE

Fluxapyroxad and pyraclostrobin content in Fluxapyroxad + Pyraclostrobin SC formulation can be determined by a HPLC-UV method. The identity of the active ingredient can be established by comparison with the equivalent authentic standards and quantified the active contents by external standardization method.

G-2 REAGENT

G-2.1 Acetonitrile – HPLC grade

G-2.2 Water – HPLC grade

G-2.3 Fluxapyroxad Analytical Standard, of known purity

G-2.4 Pyraclostrobin Analytical Standard, of known purity

G-2.5 Orthophosphoric Acid – AR grade

G-2.6 Chromatographic Separation Parameter

Instrument	HPLC equipped with Binary pump, degasser, column oven UV/PDA/DAD detector

Column used		C18 (250 mm length x 4.60 mm internal diameter (i.d.) x 5 µm particle size)
Mobile Phase		HPLC Grade water + (0.1 % Orthophosphoric Acid) 400 ml + Acetonitrile 600 ml. Mix well and filter under vacuum through 0.45micron filter
Wave Length		260 nm
Column oven temperature		40°C (Method is reproducible from 30°C to 45°C)
Injection Volume		20 µl
Flow rate		1.0 ml/min
Run time		20 min
Retention time (approximately)	Fluxapyroxad	8.6 min
	Pyraclostrobin	18.3 min

G-3 PROCEDURE

G-3.1 Preparation of Standard and Sample Solutions for Analysis of SC Formulation Containing Fluxapyroxad 167 g/l and Pyraclostrobin 333 g/l)

G-3.1.1 *Preparation of Standard Solution*

Weigh accurately about 30 mg of fluxapyroxad of known purity and 60 mg of pyraclostrobin of known purity reference standards into a 100 ml volumetric flask. Dissolve the content of the flask and bring the volume up to the mark with acetonitrile.

G-3.1.2 *Preparation of Sample Solution*

Weigh an amount of sample to contain equal quantities of active ingredients present in the standard solution into 100 ml volumetric flasks. Dissolve the content of the flask, bring volume up to the mark with acetonitrile. Pass the sample solution through 0.45 µm syringe filters prior to HPLC analysis.

G-3.2 Preparation of Standard and Sample Solutions for Analysis of SC formulation containing Fluxapyroxad 250 g/l and Pyraclostrobin 250 g/l)

G-3.2.1 *Preparation of Standard Solution*

Weigh accurately about 45 mg of fluxapyroxad of known purity and 45 mg of pyraclostrobin of known purity reference standards into a 100 ml volumetric flask. Dissolve the content of the flask and bring the volume up to the mark with acetonitrile.

G-3.2.2 *Preparation of Sample Solution*

Weigh an amount of sample to contain equal quantities of active ingredients present in the standard solution into a l00 ml volumetric flasks. Dissolve the content of the flask, bring volume up to the mark with acetonitrile. Pass the sample solution through 0.45 µm syringe filters prior to HPLC analysis.

G-3.3 Sample Analysis

Inject standard solution until the area of standards of two successive injections do not deviate from each other by more than 2 percent. Then inject the sample solution. Use the following injection sequence:

…c_1, s_1, c_2, s_2,…..

C = standard solution, and

S = sample solution.

From the chromatograms of the standard solution and sample solution, measure the peak areas and calculate the percentage of the Fluxapyroxad and pyraclostrobin as given in **G-4.**

G-4 CALCULATIONS

G-4.1 Fluxapyroxad

Fluxapyroxad content, percent by mass = $\frac{M_1 \times A_2 \times P_1 \times DF}{M_2 \times A_1}$

where,

A_1= Peak area of fluxapyroxad in the standard solution;

A_2= Peak area of fluxapyroxad in the sample solution;

M_1= Mass of standard fluxapyroxad taken for test, in g;

M_2= Mass of sample taken for test, in g;

P_1 = Percent purity of fluxapyroxad standard;

DF = Density of item = 1.166 g/cm^3

G-4.2 Pyraclostrobin

Pyraclostrobin content, percent by mass = $\frac{M_3 \times A_4 \times P_2 \times DF}{M_2 \times A_3}$

where,

A_3= Peak area of pyraclostrobin in the standard solution;

A_4= Peak area of pyraclostrobin in the sample solution;

M_3= Mass of standard pyraclostrobin taken for test, in g;

M_2= Mass of sample taken for test, in g;

P_2 = Percent purity of pyraclostrobin standard;

DF = Density of item = 1.166 g/cm^3