FOREWORD

(*Formal clause would be added later)*

Fluxapyroxad 333 g/l Flowable Suspension Concentrate used as a seed treatment Fungicide in Agriculture.

Fluxapyroxad 333 g/l Flowable Suspension Concentrate is generally manufactured to contain Fluxapyroxad 333 g/l 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 Fluxapyroxad 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*)
10627 : 1983	Methods for sampling of pesticidal formulation

3 REQUIREMENTS

3.1 Constituents

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

3.2 Physical

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

3.2.1 *Description*

The material shall be magenta to red coloured, flowable liquid, free from external impurities, which on dilution with water readily forms a suspension.

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

TABLE 1 REQUIREMENTS FOR FLUXAPYROXAD FLOWABLE SUSPENSION CONCENTRATE

(*Clause* ***3.2.2*** and ***7.1)***

Sl. No.	**Characteristic**	**Requirement**	**Annex of this Standard**
(1)	**(2)**	**(3)**	**(4)**
i)	Fluxapyroxad content, percent by mass, *Min*		A
ii)	Pourability, percentage by mass, *Max*	3	B
iii)	Suspensibility, percent by mass, *Min*	90	IS 6940
iv)	Wet Sieve Test, percent by mass, *Min*	95	C
v)	Persistent Foam, *Max*	60 sec	D
vi)	Ph	5 to 9	E

3.3 Chemical

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

3.3.1 *Fluxapyroxad content*

When determined by the method prescribed (enclosed as Annex-I), the observed Fluxapyroxad 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	
10 and below 50	±5	of the nominal
50 and above	+5	value
	-3	

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

4 PACKING

4.1 The product shall be packed in 40 ml, 100 ml, 200 ml, 400 ml, 800 ml and 1000 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 Fluxapyroxad 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 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. **3.3.1** 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 3.3.1 of this standard.

7 TESTS

7.1 Tests shall be carried out by the appropriate methods referred to in **3.2.2.**

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 FLUXAPYROXAD CONTENT

A-1 PRINCIPLE

Fluxapyroxad content in Fluxapyroxad 333 g/l FS (BAS 700 05F) formulation samples were determined by a HPLC method. The identity of the active ingredient was established by comparison with the equivalent authentic standard.

A-2 REAGENT

Acetonitrile – HPLC grade

Millipore water

Acetophenone – Internal standard

Fluxapyroxad Analytical standard

A-3 PROCEDURE

A-3.1 Preparation of internal standard solution

About 200 mg of acetophenone was weighed into 100 ml volumetric flask. The content was dissolved in acetonitrile and the volume was brought up to the mark with acetonitrile.

A-3.2 Preparation of standard solution

Accurately (98.82 and 99.02 mg) of Fluxapyroxad reference standard (CA and CB) of purity 99.7% was weighed into 100 ml volumetric flask. The content was dissolved in acetonitrile and added 10 ml of internal standard solution and then volume was brought upto mark using acetonitrile.

A-3.3 Preparation of sample solution

Accurately 310.60 mg and 334.13 mg of BAS 700 05F sample in a 100 ml volumetric flask. The content was dissolved in acetonitrile and added 10 ml of internal standard solution and then volume was brought upto the mark using acetonitrile.

A-3.4 Sample Analysis

Injected in the sequence CA, S1R1, S1R2, CB, S2R1, S2R2 and analyzed for Fluxapyroxad content.

A-3.5 Chromotographic separation parameter

Instrument		HPLC 1200 series
Column length, mm		C18 (50 mm length × 4.6 mm I. D × 3.5 µ particle size)
Mobile Phase		Acetonitrile (HPLC grade) : MilliQ water (35 : 65 *v/v*)
Detecter		230 nm
Column temperature		40 °C
Injection Volume		5 µL
Total Run Time		15 min
Flow Rate		1.0 ml/min
Retention time (approx)	Acetophenone	5.9 mins
	Fluxapyroxad	7.8 mins

A-4 CALCULATION

Fluxapyroxad content, percent by mass = $\frac{H_w \times M \times P \times I_r}{H_s \times w \times I_q}$

Where

H_s = Peak area of Fluxapyroxad in the standard solution (mAU*sec)

H_w = Peak area Fluxapyroxad in the sample solution (mAU*sec)

M = Mass of Fluxapyroxad in the standard solution (mg)

w = Mass of sample taken (mg)

P = Purity of Fluxapyroxad reference standard (%)

I_r = Peak area of internal standard in the Standard solution (mAU*sec)

I_q = Peak area of internal standard in the Sample solution (mAU*sec)

Fluxapyroxad content (% *w/v)* = Fluxapyroxad percent by mass × Density

Density of Fluxapyroxad 333 g/l FS formulation = 1.16 g/ml

ANNEX B

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

DETERMINATION OF POURABILITY IN FLUXAPYROXAD CONTENT

B-1 PRINCIPAL

The suspension concentrate is allowed to stand for 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

Container a 500 ml stoppered measuring Cylinder.

B-3 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 at 20±2 °C 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).

B-4 CALCULATIONS

Calculated the residue (R) and the rinsed residue (R').

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

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

ANNEX C

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

DETERMINATION OF WET SIEVE TEST IN FLUXAPYROXAD CONTENT

C-1 PRINCIPLE

A sample of the formulation is dispersed in water and the suspension formed is transferred to a sieve and washed. The amount of the material retained on the sieve is determined by drying and weighing.

C-2 APPARATUS

75 micron sieve

4-6 mm Diameter glass rod

250 ml beaker

Oven

Dessicator

Mettler Toledo balance with an accuracy of 0.01 mg

C-3 PROCEDURE

A 10g of the material was weighed into a 250 ml beaker and dissolved in 100 ml of tap water by stirring using a magnetic stirrer bar and allowed to stand for 60 sec. The slurry was transferred to 75 µm sieve which was thoroughly wetted by immersing in water and the material on the sieve was washed with a gentle stream of tap water till the amount of residues on the sieve appeared to remain constant. The retained residue on the sieve was determined by decanting the residue to a tared glass dish using a jet of deionized water. It was dried to constant weight and weight of the sample was recorded.

C-4 CALCULATION

Percentage of material retained in 75µm sieve = $\frac{W_3 - W_2}{W_1} \times 100$

Where,

W_1 = Weight of test substance

W_2 = Weight of empty glass dish

W_3 = Weight of glass dish + quantity of dried residue of test substance retained in 75 µm sieve

ANNEX D

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

DETERMINATION OF PERSISTENT FOAM IN FLUXAPYROXAD CONTENT

D-1 PRINCIPLE

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 a definite time is measured.

D-2 REAGENT

Standard water D

D-3 APPARATUS

Graduated cylinder, glass stoppered, 250 ml. 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.

D-4 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.

ANNEX E

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

DETERMINATION OF *p*H IN FLUXAPYROXAD CONTENT

E-1 PRINCIPAL

The *p*H meter was given digital display of *p*H of solution over the range 1-14. Before analysis *p*H meter was calibrated by using standard buffer solutions.

E-2 CHEMICALS

*p*H 4.0 standard buffer solution

*p*H 9.0 standard buffer solution

Distilled water (CO_2 free).

E-3 APPARATUS

Standard measuring flask 100 ml

Beaker 100 ml

Glass rod

Weighing balance

*p*H meter

E-4 PROCEDURE

E-4.1 Preparation of 1% sample solution

Accurately 1g of homogenized sample was weighed into 100 ml standard measuring flask and made upto the mark using distilled water (CO_2 free).

E-4.2 Calibration of *p*H meter

The *p*H meter was calibrated 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.

E-4.3 *p*H determination of the sample at 25 ± 1 °C

The test item (~1g) was added into separate 100 ml volumetric flask containing 50 ml of standard heavy water. The volume of the flask was brought up to 100 ml mark using distilled water and shaken vigorously for 1 minute, the suspension was allowed to settle for 1 minute and then the supernatant liquid was transferred to a beaker. The *p*H of supernatant liquid was measured 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 data was recorded.