IS 8963 (2006): Chlorpyrifos, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]

BLANK PAGE

भारतीय मानक

क्लोरपाइरीफॉस, तकनीकी — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

CHLORPYRIFOS, TECHNICAL — SPECIFICATION

(First Revision)

ICS 65.100.10

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

February 2006

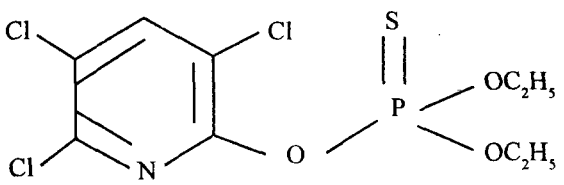
Price Group 2

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides and Pesticides Residue Analysis Sectional Committee had been approved by the Food and Agriculture Division Council.

Chlorpyrifos is the common name accepted by the International Organization for Standardization (ISO) for O, O-diethyl-O-(3, 5, 6-trichloro-2-pyridinyl) phosphorothioate.

The molecular formula, structural formula and the molecular mass are as indicated below:

Molecular Formula	*Structural Formula*	*Molecular Mass*
$C_9H_{11}Cl_3NO_3PS$		350.5

Chlorpyrifos, technical is used for formulations for the control of insect pests.

In 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 the *Insecticides 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 : 1960 '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.

Indian Standard

CHLORPYRIFOS, TECHNICAL — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for chlorpyrifos, technical.

2 REFERENCES

The following standards 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 indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
6940 : 1982	Methods of test for pesticides and their formulations *(first revision)*
8190 (Part 1) : 1988	Requirement for packaging of pesticides: Part 1 solid pesticides (*second revision*)
10946 : 1996	Methods of sampling of technical grade pesticides (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of crystalline solid, white to pale yellow, insoluble in water. Some liquid fraction may also be present in the container, particularly in hot weather.

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

4 PACKING

The material shall be packed in clean and dry containers made of mild steel, properly and suitably lacquered from inside, or having a polyethylene liner of thickness not less than 0.125 mm. The general requirements given in IS 8190 (Part 1) shall also be followed.

5 MARKING

5.1 The containers shall bear legibly and indelibly the following information and other additional information, as is 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 contents;
g) Nominal chlorpyrifos content, percent (*m/m*);
h) A cautionary notice as worded in the *Insecticides Act,* 1968, and Rules framed thereunder; and
j) Any other information required under the *Standards of Weights and Measures (Packaging Commodities) Rules*, 1977.

5.2 BIS Certification Marking

The product may also be marked with the Standard Mark.

5.2.1 The use of the standard mark is governed by the

Table 1 Requirements for Chlorpyrifos, Technical

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex of this Standard	Clause No. of IS 6940
(1)	(2)	(3)	(4)	(5)
i)	Chlorpyrifos content, percent by mass, *Min*	94	A	—
ii)	Water content, percent by mass, *Max*	0.1	—	4.1
iii)	Acidity (as H_2SO_4), percent by mass, *Max*	0.1	—	11.3.2
iv)	Material insoluble in acetone, percent by mass, *Max*	0.5	—	9

provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

Representative samples of the material shall be drawn according to IS 10946.

7 TESTS

7.1 Tests shall be carried out by the appropriate methods referred in col 4 and 5 of the Table 1, as specified under **3.2**.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and reagent grade water (*see* IS 1070) shall be employed in test.

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 CHLORPYRIFOS CONTENT

A-0 METHODS

Two methods Method I and Method II are prescribed for the determination of chlorpyrifos content. Method I shall, however, be used in case of dispute.

A-1 METHOD I

A-1.1 Principle

Chlorpyrifos is determined by temperature programmed gas chromatography, using the internal standard technique.

A-1.2 Apparatus

A-1.2.1 *Gas Liquid Chromatograph* — A gas liquid chromatograph equipped with flame ionization detector and a printer-plotter-cum-integrator is used. The suggested operative parameters are as follows, but can be changed, if necessary, provided standardization is done:

Column	: Glass, 1 m length, 2 mm I.D., 3 percent OV–101 on gaschrome Q or chromosorb WHP (80-100 mesh)
Temperature	: 170°C for 1 min
Oven	: Temperature programming 10°C/min up to 260°C. Hold for 3 min
Injection port	: 270°C
Detector	: 290°C
Carrier gas	: Nitrogen
Carrier gas flow	: 30 ml/min
Hydrogen gas flow	: 30 ml/min
Air (FID)	: 300 ml/min
Injection volume	: 2 microlitres

A-1.2.2 *Injection Volume*, 2 ml capacity.

A-1.2.3 *Standard Glassware*

A-1.2.4 *Microsyringe*, of 5 microlitre capacity.

A-1.3 Reagents

A-1.3.1 *Ethyl Acetate*, A.R. grade or equivalent.

A-1.3.2 *Chlorpyrifos*, reference standard of known purity.

A-1.3.3 *Dioctyl Phthalate*, A.R. grade or equivalent (internal standard).

A-1.4 Procedure

A-1.4.1 *Preparation of Internal Standard Solution*

Weigh (to the nearest of 0.1 mg) 0.5 g of dioctyl phthalate into a 250 ml volumetric flask. Dissolve it, and make up to the volume with ethyl acetate. Shake well to homogenize.

A-1.4.2 *Preparation of Standard Solution*

Weigh (to the nearest of 0.1 mg) 0.15 g of chlorpyrifos reference standard into a 50 ml volumetric flask. Add 25 ml of internal standard solution, and make up to 50 ml mark with ethyl acetate. Shake well to homogenize.

A-1.4.3 *Preparation of Sample Solution*

Weigh a sample (to the nearest of 0.1 mg) quantity so as to contain about 0.15 g of chlorpyrifos, into a 50 ml volumetric flask. Add 25 ml of internal standard

solution and make up to 50 ml mark with ethyl acetate. Shake well to homogenize.

A-1.5 Estimation

Inject the standard solution repeatedly, until the area ratio of reference standard and internal standard of two successive injections do not deviate from each other by more than 2 percent. Then inject the sample solution. The sequence of injection to be followed is as follows:

$$\ldots C; S_1; C; S_2, C \ldots$$

where

C = standard solution, and

S = sample solution.

A-1.6 Guide Values

Retention times	:	Chlorpyrifos 4.2 min (approximately), internal standard dioctyl phthalate 9.0 min (approximately)
Total run time	:	13 min

A-1.7 Calculation

From the chromatograms of standard solution and sample solution, measure the peak areas of chlorpyrifos and internal standard peak, and calculate the percentage of chlorpyrifos as follows:

$$\text{Chlorpyrifos, percent by mass} = \frac{M_1 \times A_3 \times A_2 \times P_1}{M_2 \times A_4 \times A_1}$$

where

A_1 = area of chlorpyrifos peak in the chromatogram of the standard solution;

A_2 = area of chlorpyrifos peak in the chromatogram of the sample solution;

A_3 = area of internal standard peak in the chromatogram of the standard solution;

A_4 = area of internal standard peak in the chromatogram of the sample solution;

M_1 = mass of chlorpyrifos in the standard solution, in g;

M_2 = mass of the sample taken for test, in g; and

P_1 = percentage purity of chlorpyrifos reference standard.

A-2 METHOD II

A-2.1 Principle

Chlorpyrifos is determined by isothermal Gas Chromatography using the internal standard technique.

A-2.2 Apparatus

A-2.2.1 *Gas Liquid Chromatograph* — A gas liquid chromatograph equipped with flame ionization detector and a printer-plotter-cum-integrator is used. The suggested operative parameters are as follows, but can be changed, if necessary, provided standardization is done.

Column	:	Stainless steel, 180 cm length, 4 mm I.D, packed with 3 percent OV-17 on chromosorb WHP (80-100 mesh)
Temperature:		
Oven	:	220°C
Injection	:	260°C
Detector	:	270°C
Carrier Gas	:	Nitrogen
Carrier Gas Flow	:	30 ml/min
Hydrogen Gas Flow	:	40 ml/min
Air (FID)	:	400 ml/min

A-2.2.2 *Injection*, volume, 2 µl.

A-2.2.3 *Standard Glassware*

A-2.2.4 *Microsyringe*, of 5 microlitre capacity.

A-2.3 Reagents

A-2.3.1 *Acetone*, A.R. grade or equivalent.

A-2.3.2 *Chlorpyrifos*, reference standard of known purity.

A-2.3.3 *Dibutyl Phthalate*, A.R. grade or equivalent.

A-2.4 Procedure

A-2.4.1 *Preparation of Internal Standard Solution*

Weigh (to the nearest of 0.1 mg) 0.9 g of dibutyl phthalate into a 250 ml volumetric flask. Dissolve it, and make up to the volume with acetone. Shake well to homogenize.

A-2.4.2 *Preparation of Standard Solution*

Weigh (to the nearest of 0.1 mg) 0.11 g of chlorpyrifos reference standard into a 25 ml volumetric flask. Add 10 ml of internal standard solution, and make up to 25 ml mark with acetone. Shake well to homogenize.

A-2.4.3 *Preparation of Sample Solution*

Weigh a sample (to the nearest of 0.1 mg) quantity so as to contain about 0.11 g of chlorpyrifos into a 25 ml volumetric flask. Add 10 ml of internal standard solution, and make up to 25 ml mark with acetone and shake well to homogenize.

A-2.5 Estimation

Inject the standard solution repeatedly, until the area

ratio of reference substance and internal standard of two successive injections do not deviate from each other by more than 2 percent. Then inject the sample solution. The sequence of injection to be followed is as follows:

$$\ldots C; S_1; C; S_2, C, \ldots$$

where

C = standard solution, and

S = sample solution.

A-2.6 Guide Values

Retention times : Chlorpyrifos 6 min (approximately), dibutyl phthalate 7 min (approximately)

A-2.7 Calculation

From the chromatograms of standard solution and sample solution, measure the peak areas of chlorpyrifos and internal standard peak, and calculate the percentage of chlorpyrifos as follows:

$$\text{Chlorpyrifos, percent by mass} = \frac{M_1 \times A_3 \times A_2 \times P_1}{M_2 \times A_4 \times A_1}$$

where

A_1 = area of chlorpyrifos peak in the chromatogram of the standard solution;

A_2 = area of chlorpyrifos peak in the chromatogram of the sample solution;

A_3 = area of internal standard peak in the chromatogram of the standard solution;

A_4 = area of internal standard peak in the chromatogram of the sample solution;

M_1 = mass of chlorpyrifos in standard solution, in g;

M_2 = mass of the sample taken for test, in g; and

P_1 = percentage purity of chlorpyrifos reference standard.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act,* 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc : No. FAD 1 (1381).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110 002
Telephones : 2323 01 31, 2323 33 75, 2323 94 02

Telegrams : Manaksanstha
(Common to all offices)

Regional Offices :		Telephone
Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110 002	2323 76 17, 2323 38 41
Eastern	: 1/14 C.I.T. Scheme VII M, V. I. P. Road, Kankurgachi, KOLKATA 700 054	2337 84 99, 2337 85 61, 2337 86 26, 2337 91 20
Northern	: SCO 335-336, Sector 34-A, CHANDIGARH 160 022	260 38 43, 260 92 85
Southern	: C.I.T. Campus, IV Cross Road, CHENNAI 600 113	2254 12 16, 2254 14 42, 2254 25 19, 2254 23 15
Western	: Manakalaya, E9 MIDC, Marol, Andheri (East), MUMBAI 400 093	2832 92 95, 2832 78 58, 2832 78 91, 2832 78 92

Branches : AHMEDABAD. BANGALORE. BHOPAL. BHUBANESHWAR. COIMBATORE. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. KANPUR. LUCKNOW. NAGPUR. NALAGARH. PATNA. PUNE. RAJKOT. THIRUVANANTHAPURAM. VISAKHAPATNAM.

Printed at Prabhat Offset Press, New Delhi-2