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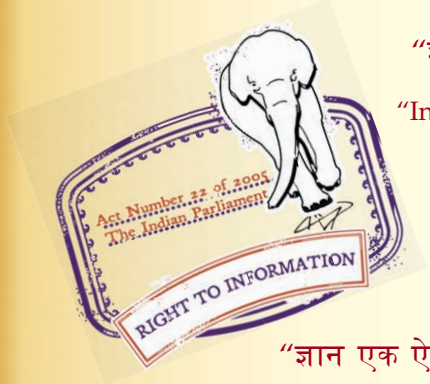
IS 4929 (1978): Dichlorvos, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]



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IS : 4929 - 1978
[Reaffirmed 2012]

Indian Standard
SPECIFICATION FOR DICHLORVOS, TECHNICAL
(First Revision)

Second Reprint JANUARY 1996

UDC 632.951 DICHLORVOS

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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Gr 3 *August* 1978

Indian Standard

SPECIFICATION FOR DICHLORVOS, TECHNICAL

(First Revision)

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(*Continued on page* 10)

Indian Standard

SPECIFICATION FOR DICHLORVOS, TECHNICAL

(First Revision)

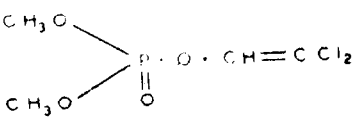
0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 15 February 1978, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 This standard was first published in 1968. In this revision the requirement pertaining to the cautionary notice has been aligned to the Insecticides Act and the Rules framed thereunder and Amendment No. 1 has been incorporated. Other modifications include packaging and sampling requirements. Opportunity has been taken to give reference to IS : 6940-1973* to bring uniformity in testing procedure for the various requirements given in this standard.

0.3 Dichlorvos, technical is used in the preparation of formulations for controlling pests of agricultural importance. This pesticide also finds use in public health and fumigating foodgrains.

0.4 Dichlorvos is the accepted common name by the International Organization for Standardization (ISO) for 2,2-dichlorovinyl dimethyl phosphate. The empirical and structural formulae and molecular mass are as given below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_4H_7O_4Cl_2P$		221

0.5 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.

*Methods of tests for pesticides and their formulation.

0.6 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*. 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 dichlorvos, technical.

2. REQUIREMENT

2.1 Description — The material shall be in the form of clear, mobile and amber coloured liquid free from extraneous impurities or added modifying agents.

2.2 The material shall also comply with the requirements specified in Table 1.

TABLE 1 REQUIREMENT FOR DICHLORVOS, TECHNICAL

(*Clauses* 2.2 *and* 5.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Appendix of this Standard	Cl No. of IS : 6940-1973*
(1)	(2)	(3)	(4)	(5)
i)	Dichlorvos content, percent by mass, *Min*	92·0	A	—
ii)	Moisture content, percent by mass, *Max*	0·02	—	4.1
iii)	Material insoluble in acetone, percent by mass, *Max*	0·5	—	9.1
iv)	Acidity (as H_2SO_4), percent by mass, *Max*†	1·0	—	11.3.2

*Methods of tests for pesticides and their formulation.

†For determination of acidity, the end point can also be determined by a potentiometric titration to *p*H 5·0.

*Rules for rounding off numerical values (*revised*).

3. PACKING AND MARKING

3.1 Packing — The material shall be packed according to the requirements given in IS : 8190 (Part II)-1976*.

3.2 Marking — The containers shall bear legibly and indelibly the following information and any other additional information as is necessary under the Insecticides Act and Rules:

a) Name of the material;
b) Name of the manufacturer;
c) Date of manufacture;
d) Batch number;
e) Net mass of contents;
f) Dichlorvos content percent (*m/m*); and
g) The cautionary notice as worded in the Insecticides Act and Rules.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 'Indian Standard methods for sampling of pesticides and their formulations (*under preparation*)'.

NOTE — Till such time the standard under preparation i published, the matter sshall be as agreed to between the concerned parties.

5. TESTS

5.1 Tests thall be carried out by the methods specified in col 4 and 5 of Table 1.

5.2 Quality of Reagent — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Requirements for packing of pesticides : Part II Liquid pesticides.

†Specification for water for general laboratory use (*second revision*).

APPENDIX A

[*Table* 1, *Item* (i)]

DETERMINATION OF DICHLORVOS CONTENT

A-0. GENERAL

A-0.1 For determining the active ingredient in dichlorvos, technical, two methods, namely, the iodometric method and infra-red spectrophotometric method have been prescribed. Either of these methods may be used for the determination of the active ingredient; however, in the event of dispute, the infra-red spectrophotometric method shall be used as the reference method.

A-1. IODOMETRIC METHOD

A-1.1 Principle — Dichlorvos reacts in alkaline solution with two equivalents of iodine and this reaction, as distinct from impurities, does not proceed with iodine in presence of sodium carbonate solution. Thereby it is possible to estimate it by this kind of differential method.

A-1.2 Reagents

A-1.2.1 *Iodine Solution* — 0·1 N.

A-1.2.2 *Sodium Hydroxide Solution* — 2 N.

A-1.2.3 *Hydrochloric Acid Solution* — 5 N.

A-1.2.4 *Sodium Thiosulphate Solution* — 0·1 N.

A-1.2.5 *Sodium Carbonate Solution* — 2 N.

A-1.2.6 *Starch Indicator Solution*

A-1.3 Procedure — Accurately weigh 1 g of the material and transfer into a 250-ml volumetric flask and add distilled water to 250-ml mark of the flask. Transfer 25 ml of this solution into an iodine flask, add 20 ml of the standard iodine solution and then add 20 ml of the standard sodium hydroxide solution. Allow the solution to stand for 5 minutes at 20° to 30°C and acidify with 20 ml of standard hydrochloric acid and titrate with the sodium thiosulphate using starch solution as the indicator. Carry out a second titration adding 5 ml of the iodine solution to 25 ml of the freshly prepared sample solution. Further, add 10 ml of the standard sodium carbonate solution and allow to stand for 5 to 10 minutes at 20° to 30° C; acidify with 20 ml hydrochloric acid and titrate with the sodium thiosulphate solution using starch solution as indicator.

A-1.4 Calculation

A-1.4.1 Dichlorvos content, percent by mass $= \dfrac{(A - B) \times N \times 11{\cdot}05}{m}$

where

A = volume, in ml, of iodine solution used in first titration,

B = volume, in ml, of iodine solution used in second titration,

N = normality of the standard iodine solution, and

m = mass, in g, of the sample taken.

A-2. INFRA-RED SPECTROPHOTOMETRIC METHOD

A-2.1 Method — The method consists in dissolving the material in chloroform, and measurement of the infra-red absorbance at about 10·2 micron. This net absorbance is used to obtain the concentration of dichlorvos from previously prepared calibration curve relating to net absorbance to concentration of dichlorvos.

A-2.2 Apparatus

A-2.2.1 *Infra-Red Spectrophotometer* — capable of recording in the region of 2 to 15 microns, with the slit width, gain and response time and scanning speed adjustable to produce a satisfactory signal-to-noise ratio and adequate resolution under the conditions of the test (in general, the minimum slit width giving a signal-to-noise ratio of about 100 to 1 is chosen).

A-2.2.2 *Absorption Cells* — sealed absorption cells with sodium chloride windows, having a path length of about 0·2 mm.

A-2.2.3 *Hypodermic Syringe* — of 1·0 ml capacity with an 18-gauge (stubbs) slip-on-type needle.

A-2.3 Reagents

A-2.3.1 *Standard Dichlorvos* — recrystallized, of known dichlorvos content.

A-2.3.2 *Chloroform* — analytical reagent grade.

A-2.4 Procedure

A-2.4.1 *Preparation Calibration Graph* — Prepare the calibration graph for samples in chloroform as given in **A-2.4.1.1** to **A-2.4.1.4**.

A-2.4.1.1 Weigh accurately into each of five 10-ml volumetric flasks 25, 75, 100, 150 and 200 mg of the standard dichlorvos (*see* **A-2.3.1**); dissolve in chloroform and dilute to the mark (the concentrations of these solutions will be 2·5, 7·5, 10, 15 and 20 g per litre).

A-2.4.1.2 Fill the absorption cell with chloroform by means of the hypodermic syringe. Adjust the spectrophotometer to the optimum instrument settings with respect to gain, slit width, response, chart speed and wave-length scanning speed. Make a scan with chloroform in the cell over the wave-length region of 9·9 to 10·6 microns.

A-2.4.1.3 Without changing the instrument settings, fill the cell, in turn, with each of the calibration solutions starting with the most dilute. Scan each of these solutions over the 9·9 to 10·6 microns.

A-2.4.1.4 For each of the scans obtained, draw perpendiculars to the zero radiation line through the absorption peak of the calibration solution at about 10·2 micron and the reference minima at about 10·0 micron and 10·4 micron. Measure the radiant power P_o and P as shown in Fig. 1. The distances may be measured in any convenient units, provided the same units are used throughout the determination. Calculate the absorbance as the logarithm of the ratio of the incident power (P_o) to the transmitted radiant power (P). Repeat the calculation of the absorbances of the calibration solution using the reference minimum at about 10·4 micron. Subtract the absorbance of the cell plus chloroform from the absorbances of the cell plus calibration solutions. Make a plot of the net absorbances as ordinate against the corresponding concentrations of dichlorvos in g/l as abscissa for each of the reference points used that is the absorption minima at about 10·0 micron and 10·4 micron respectively.

A-2.5 Estimation of Dichlorvos — Weigh accurately into a 50-ml volumetric flask an amount of sample sufficient to give a 1 percent (m/v) solution of dichlorvos and dilute to the mark with chloroform. Mix thoroughly and fill the calibrated liquid absorption cell with the sample solution. Using the same instrument settings that were used for the

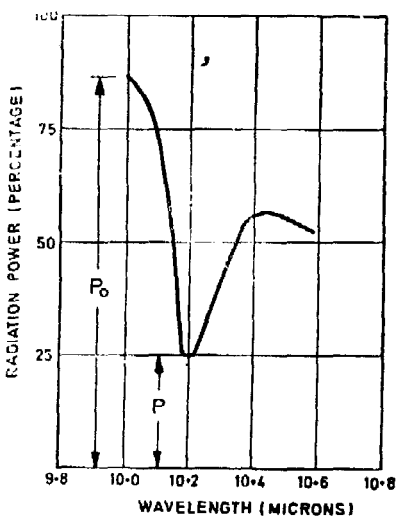


FIG. 1 DICHLORVOS INFRA-RED ABSORPTION SPECTRUM — REFERENCE-POINT TECHNIQUE

calibration, obtain a scan of the sample solution over the 9·9 to 10·6 micron region. Calculate the absorbances of the sample solution for the two reference minima as described in **A-2.4.1.1**.

A-2.6 Calculation — From the computed absorbances (*see* **A-2.5**) read the concentrations of dichlorvos from the calibration graph (*see* **A-2.4.1.1**).

$$\text{Dichlorvos content, percent by mass} = \frac{A \times V}{M \times 10}$$

where

A = the mean value in, g/l, determined from the use of two reference minima;

V = volume, in ml, of the sample solution; and

M = mass, in g, of the sample.

(*Continued from page* 2)

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AMENDMENT NO 1 MAY 1982

TO

IS:4929-1978 SPECIFICATION FOR DICHLORVOS, TECHNICAL

(First Revision)

Alterations

(*Pages 6 and 7, clauses A-1.3 to A-1.4.1*) - Substitute the following for the existing matter:

'A-1.3 Procedure - Accurately weigh one g of material and transfer into a 250-ml volumetric flask and add distilled water to 250-ml mark of the flask. Transfer 25 ml of this solution into an iodine flask, and 20 ml of the standard iodine solution and then add 20 ml of the standard sodium hydroxide solution. Allow the solution to stand for 5 minutes at 20 to 30°C and acidify with 20 ml standard hydrochloric acid and titrate with the sodium thiosulphate using starch solution as the indicator. Note the titre (= A ml). Carry out a blank with the same quantities of the reagents. Note the titre (= V ml). For the second titration, pipette out 25 ml of the stock solution into another 250-ml iodine flask, add 5 ml of standard iodine solution and then add 10 ml sodium carbonate solution. Allow the solution to stand for 5 minutes at 20 to 30 °C, acidify with 20 ml hydrochloric acid and titrate with sodium thiosulphate solution using starch solution as indicator. Note the titre (= B ml). Carry out a blank with the same quantities of reagents. Note the titre (= U ml).

A-1.4 Calculation

A-1.4.1 Dichlorvos content, percent by

$$\text{mass} = \frac{[(V - A) - (U - B)] \times N \times 110.5}{M}$$

where

N = normality of the standard sodium thiosulphate solution, and

M = mass in g of the sample taken for the test.'

(AFCDC 6)

AMENDMENT NO. 2 DECEMBER 1995
TO
IS 4929 : 1978 SPECIFICATION FOR DICHLORVOS, TECHNICAL

(First Revision)

(*Page* 3, *clause* 0.2) — Substitute 'IS : 6940 - 1982*' *for* 'IS : 6940 - 1973*'.

(*Page* 3, *foot-note marked* '*') — Insert '(*first revision*)' at the end of text.

(*Page* 4, *Table* 1, *col* 5) — Substitute 'IS : 6940 - 1982*' *for* 'IS : 6940 - 1973*'.

(*Page* 4, *Table* 1, *foot-note marked* '*') — Insert '(*first revision*)' at the end of text.

(*Page* 5, *clause* 3.1) — Substitute 'IS : 8190 (Part 2) - 1988*' *for* 'IS : 8190 (Part II) : 1976*'.

(*Page* 5, *foot-note marked* '*') — Insert '(*second revision*)' at the end of text.

(*Page* 5, *clause* 4.1) — Substitute the following for the existing:

'4.1 Representative samples of the material shall be drawn as prescribed in IS 10946 : 1984‡'.

(*Page* 5, *foot-note with* '‡' *mark*) — Insert the following at the bottom of the page:

'‡Methods of sampling for technical grades pesticides.'

(*Page* 5, *clause* 5.2) — Substitute 'IS 1070 : 1992†' *for* 'IS : 1070 - 1977†'.

(*Page* 5, *foot-note with* '†' *mark*) — Substitute 'Reagent grade water (*third revision*)' for the existing title.

(FAD 1)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 3 MAY 2002
TO
IS 4929 : 1978 SPECIFICATION FOR DICHLORVOS, TECHNICAL

(First Revision)

(*Page* 7, *clause* **A-2.2.2**, *line* 1) — Substitute' sodium chloride or potassium bromide' *for* 'sodium chloride'

(FAD 1)

Reprography Unit, BIS, New Delhi, India