IS 7233 (1991): Pesticide - 2, 4-D, Ethyl Ester Technical Specification [FAD 1: Pesticides and Pesticides Residue Analysis]

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IS-7233 : 1991

(Reaffirmed 2004)

भारतीय मानक

कीटनाशक — 2, 4-डी इथाईल एस्टर तकनीकी — विशिष्टि

(दूसरा पुनरीक्षण)

Indian Standard

PESTICIDE — 2,4-D, ETHYL ESTER TECHNICAL SPECIFICATION

(Second Revision)

UDC 632·95

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

October 1991

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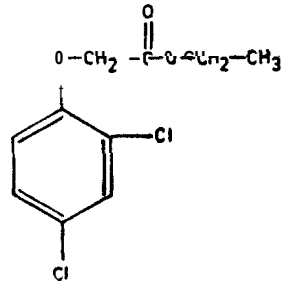
FOREWORD

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

2, 4-D, ethyl ester, technical is the product obtained by the esterification of 2, 4-D and employed in the preparation of herbicidal formulations.

2, 4-D ethyl ester, technical is generally manufactured to contain 90 percent (*m/m*) of 2, 4-D ethyl ester.

The empirical and structural formulae and molecular mass of the product are given below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_{10}H_{10}O_3Cl_2$		248·9

This standard was published in 1974 and revised in 1985. This second revision specifies requirements for 2, 4-D ethyl ester, technical only, which is used for manufacturing formulations like EC, WP and granules.

In the preparation of this standard, due consideration has been given to the provisions of the Insecti-*cides Act, 1968* and the Rules framed thereunder. However, this standard is subjected to the restrictions imposed under the 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

PESTICIDE — 2, 4-D, ETHYL ESTER TECHNICAL SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for 2, 4-D ethyl ester, technical.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1977	Water for general laboratory use (*second* ***revision)***
6940 : 1982	Methods of test for pesticides and their formulations (*first revision)*
8190 (Part '2) 1988	Requirements for packing of pesticides: Part 2 Liquid pesticides (*second revision)*
10946: *1989*	Methods of sampling for technical grade pesticides.

3 REQUIREMENTS

3.1 Description

The material shall be light to dark brown clear liquid free from extraneous impurities or additives.

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

3.3 **Identity Test**

When determined by the method prescribed in Annex G, the retention time of the material shall be the same as that of reference standard 2, 4-D ethyl ester of known purity.

4 PACKING

The material shall be packed according to the requirements prescribed in IS 8190 (Part 2) : 1988.

5 MARKING

The containers shall bear legibly and indelibly the following information in addition to any other information as is necessary under the *Insecticides Act, 1968* and Rules framed thereunder:

a) Name of the material;

b) Indication about the source of manufacture;

c) Date of manufacture and date of expiry;

d) Batch number;

e) 2, 4-D content, percent (***m/m***);

f) Net mass of the contents; and

h) The minimum cautionary notice as worded in the ***Insecticides Act,*** 1968 and Rules,

Table 1 Requirements for 2, 4-D Ethyl Ester, Technical

(Clause 3.2)

Sl NO.	Characteristic	Requirement	Method of Test, Ref to Annex of this Standard
(1)	(2)	(3)	(4)
i)	Total extractable 2,4-D content, percent by mass, ***Min***	**86'0**	**A**
ii)	Free 2, 4-D content, percent by mass, ***Max***	**6'0**	B
iii)	2,4-D ethyl ester content, percent by mass, ***Min***	**90'0**	**C**
iv)	Melting point of extracted acid, °C	**136-140**	**D**
v)	Water content	To pass the test	E
vi)	Suspended solids content, percent by mass, ***Max***	**0'2**	**F**

6 SAMPLING

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

7 TESTS

7.1 Tests shall be carried out by the methods referred to in col 4 of Table 1.

7.2 **Quality of Reagents**

Unless specified otherwise, pure chemicals and distilled water (see IS 1070 : 1977) 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,** *Item* (i)]

DETERMINATION OF TOTAL EXTRACTABLE 2,4-D CONTENT

A-l REAGENTS

A-l.1 Isopropyl Ether or Diethyl Ether

Analytical reagent (AR) grade.

A-l.2 Ethanol

Neutral, AR grade; alternatively, neutral methanol, AR grade may be used.

A-l.3 Standard Lithium Hydroxide Solution

1 N, filter, if necessary during preparation.

A-l.4 Concentrated Hydrochloric Acid

AR grade.

A-l.5 Standard Sodium Hydroxide Solution

0·1 **N.**

A-l.6 Bromothymol Blue Indicator Solution

0·04 **percent solution in alcohol (** *m/m* **).**

A-2 PROCEDURE

A-2.1 Preparation of Sample Solution

Weigh accurately an amount of the sample containing 3·5 to 4·5 g of 2, 4-D into a 250-ml **conical flask fitted** with a ground-glass joint. Add 25 ml of the standard lithium hydroxide and 10 ml of alcohol. Fit a reflux condenser and heat vigorously under reflux for 2 hours. Cool and transfer to a 250-ml volumetric flask and make up the volume to 250 ml with distilled water.

A-2.2 **Volumetric Determination**

A-2.2.1 Take out with a pipette 50 ml aliquot of the aqueous extract in a 250-ml separating funnel, add 3 ml of hydrochloric acid and extract with three successive 25 ml portions of ether, washed previously with dilute alkali to remove impurities. Combine the ether layers, washing in with a small quantity of ether, and wash the combined layers with successive portions of distilled water till free from chlorides. Wash the combined water washings with 5 ml of ether and add this to the combined ether extracts. Wash the total ether solution into a conical flask, evaporate bulk of ether and add 60 ml of ethanol. Titrate with the standard sodium hydroxide solution using bromothymol blue indicator.

A-2.2.2 Carry out a blank determination with 60 ml of ethanol and 90 ml of ether.

A-3 CALCULATION

Total extractable 2, 4-D content, percent by mass

$$= (V_1 - V_2) \times N \times 0.221 \times \frac{250}{50} \times \frac{100}{m}$$

where

V_1 = volume, in ml, of standard sodium hydroxide solution required for the test;

V_2 = volume, in ml, of standard sodium hydroxide solution required for the blank determination;

N = normality of the standard sodium hydroxide solution; and

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

ANNEX B

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

DETERMINATION OF FREE 2,4-D CONTENT

B-l REAGENTS

B-l.1 Ethanol

Neutral, alternatively methanol, neutral may be used.

B-1.2 Standard Sodium Hydroxide Solution

0·1 N.

B-l.3 Bromothymol Blue Indicator Solution

0·04 percent solution in alcohol (*m/v*).

B-2 PROCEDURE

B-2.1 Weigh accurately about 10 g of the sample into a 500-ml conical flask and dissolve in 200 ml of ethanol. Cool to 40°C, if required, by keeping the flask on a ice-water-bath and titrate

with the standard sodium hydroxide solution, using 1 ml of bromothymol blue as indicator. The titration should be carried out rapidly to the first appearance of green colour.

B-2.2 Carry out a blank determination with 200 ml of ethanol, 5 ml of freshly boiled distilled water, and 1 ml of bromothymol blue indicator.

B-3 CALCULATION

$$\text{Free 2, 4-D content percent by mass} = \frac{(V_1 - V_2) \times N \times 22{\cdot}1}{m}$$

where

V_1 = volume, in ml, of standard sodium hydroxide solution required for the test;

V_2 = volume, in ml, of standard sodium hydroxide solution required for blank determination;

N = normality of standard sodium hydroxide solution; and

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

ANNEX C

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

DETERMINATION OF 2, 4-D ETHYL ESTER CONTENT

C-1 CALCULATION

$$\text{2, 4-D ethyl ester content} = (A - B) \times \frac{249}{221}$$

where

A = total extractable 2, 4-D content (see A-3), and

B = free 2, 4-D content (see B-3).

ANNEX D

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

DETERMINATION OF MELTING POINT OF EXTRACTED 2,4-D

D-l GENERAL

2, 4-D is extracted with ether from 2,4-D ethyl ester technical, then after drying, melting point is determined.

D-2 REAGENTS

D-2.1 Ether

D-2.2 Acetone **or Benzene**

D-2.3 Concentrated Hydrochloric Acid

D-3 PROCEDURE

D-3.1 Transfer a 25 ml aliquot of the sample solution (see A-2.1) to a 100-ml separating funnel and extract with three successive 25 ml portions of ether. Discard the ether layers and add 3 ml of hydrochloric acid to the aqueous layer. Extract with three successive 25 ml portions of ether and combine the ether layers in another separating funnel. Wash with three successive 25 ml portions of distilled water and discard the water washings. Shake the ether extract with about 5 g of anhydrous sodium sulphate and filter into a 250-ml conical flask. Distil off the solvent. Add 3 ml of acetone or benzene and evaporate it off. Dry the residue for one hour at 105°C.

D-3.2 Determine the melting point of the finely powdered residue as per the method prescribed in 6 of IS 6940 : 1982.

ANNEX E

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

DETERMINATION OF WATER CONTENT

E-l PROCEDURE

Weigh accurately about 25 g of the sample in a conical flask and dissolve in 75 ml of toluene. The solution should be free from visible water.

ANNEX F

[*Table* 1, Item (vi)]

DETERMINATION OF SUSPENDED SOLIDS CONTENT

F-1 PROCEDURE

Filter the solution prepared as described in **F-1** through a tared sintered-glass crucible, washing the flask with toluene. Wash the residue in the crucible with small quantities of toluene. Dry the crucible to constant mass at 105°C. Express the mass of residue as a percentage of the mass of the initial sample.

F-2 CALCULATION

Suspended solids content, percent by mass $= \frac{m \times 104}{M}$

where

m = *mass*, in g, of residue left in the crucible; and

M = mass, in g, of the sample taken for the test (see **F-1**).

ANNEX G

(*Clause* **3.3**)

IDENTITY TEST

G-1 GENERAL

The retention time of 2, 4-D ethyl ester, technical is compared with that of reference standard 2, 4-D ethyl ester on a gas liquid chromatograph equipped with a flame ionization detector (FID).

G-2 APPARATUS

G-2.1 Gas Liquid **Chromatograph** (GLC)

Equipped with a FID and may be coupled to a printer-plotter *cum* integrator. The operative parameters are suggested below, which can be changed, in any other GLC equipment employed, provided standardization is done:

Column — Glass, 200 cm length and 6 mm ID, packed with 3 percent OV 17 on Chromosorb W-HP (80-100 mesh)

Temperature:

Column oven	— 180°C
Injection port	— 250°C
Detector	— 260°C

Gases and corresponding flow rates:

Nitrogen (carrier)	— 30 ml/min
Hydrogen (fuel)	— 25 ml/min
Air	— 300 ml/min

G-2.2 Microlitre Syringe

5-10 μl capacity.

G-3 PROCEDURE

Inject 2·0 μl of 0·2 percent (*m/v*) 2, 4-D ethyl ester reference standard solution in acetone into the chromatograph and note down the retention time of the peak in the chromatogram. Similarly, inject 2·0 μl of 0·2 percent (*m/v*) solution of 2, 4-D ethyl ester technical sample and note down the retention time. The retention time for the sample shall be identical with that of the reference standard 2, 4-D ethyl ester.

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Doc : No. FADC 1 (4001)

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Printed at New India Printing Press. Khurja, India