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भारतीय मानक मसौदा

आइसोप्रोटुरोन, तकनीकी — विशिष्टि

(*आइ एस 12004 का पहला पुनरीक्षण*)

Draft Indian Standard

ISOPROTURON, TECHNICAL — SPECIFICATION

(*first Revision of IS 12004*)

ICS 65.100.20

Pesticides Sectional Committee, FAD 01 **Last date of comments:** 26 December 2024

FOREWORD

(*Adoption clause will be added later*)

Isoproturon, technical is used for making formulations meant for weed control in agricultural crops.

Isoproturon is the accepted common name by the International Organisation for Standardization (ISO) for N, N-dimethyl-N-4-isopropyl- phenyl urea. The empirical and structural formulae, and molecular mass are as given below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_{12}H_{18}N_2O$		206.29

This standard was published in 1987. In this revision, the HPLC method used for determination of active ingredient has been made as the referee method. Also, the standard has been brought out in the latest style and format of the Indian Standards, and references to Indian Standards wherever applicable have been updated. It also incorporates one amendment issued to the previous version of this standard.

In the preparation of this standard, due consideration has been given to the provisions of the

Insecticides Act, 1968, and Rules framed there under. However, this standard is subject 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 : 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 isoproturon, 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*
IS 1070 : 2023	Reagent grade water – Specification (*fourth revision*)
IS 6940 : 202X	Pesticides and their formulations – Test methods (*second revision*) [*Under preparation Doc: FAD 01(25870)F*]
IS 8190 (Part 1) :1988	Requirements for packing of pesticides: Part 1 Solid pesticides (*second revision*)
IS 10946 : 1996	Methods of sampling for technical grade pesticides (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of white to greyish white or yellowish crystalline powder free from extraneous impurities or hard lumps.

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

Table 1 Requirements for Isoproturon, Technical
(*Clause* 3.2)

SI. No.	**Characteristic**	**Requirement**	**Method of Test, Ref to**
(1)	**(2)**	**(3)**	**(4)**
i)	Isoproturon content, percent by mass, *Min*	95.0	Annex A
ii)	Water content, percent by mass, *Max*	0.5	IS 6940
iii)	Melting point, °C	152-158	IS 6940
iv)	Acidity (as H_2SO_4), percent by mass, *Max* Or Alkalinity (as NaOH), percent by mass, *Max*	0.1 0.4	IS 6940 IS 6940

4 PACKING

The material shall be packed according to the requirements given in IS 8190 (Part 1).

5 MARKING

5.1 The container shall bear legibly and indelibly the following information:

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 isoproturon 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

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

7 TESTS

7.1 Tests shall be carried out as referred to in col 4 of Table 1.

7.2 Quality of Reagents

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, *SI. No.* (i)]

DETERMINATION OF ISOPROTURON CONTENTS

A-1 GENERAL

For the determination of isoproturon content, three methods namely UV spectrophotometric method (*see* **A-2**), HPLC method (*see* **A-3**) and basic hydrolysis method (*see* **A-4**) have been specified. The HPLC method shall be the referee method in case of dispute.

A-2 UV SPECTROPHOTOMETRIC METHOD

A-2.1 Principle

The absorbance of a methanolic solution of technical material is measured against the solvent at 242 nm. The isoproturon content of the sample is then computed making use of the absorbance value of a solution of standard isoproturon.

A-2.2 Apparatus

A-2.2.1 *Ultraviolet Spectrophotometer*

A-2.2.2 *Quartz Cells* — Matched pair with path length equal to 1.000 cm.

A-2.2.3 *Volumetric Flasks* — 250 ml, 200 ml and 100 ml capacity.

A-2.2.4 Pipettes — 10 ml and 5 ml capacity (graduated).

A-2.3 Reagents

A-2.3.1 *Methanol* — Spectroscopic grade.

NOTE — The absorbance (1.000 cm cell) of methanol should not, in any case, exceed 0.150 at 240 cm. The spectral absorbance curve should be smooth throughout the 210-300 nm range and should not show any extraneous impurity peaks.

A-2.4 Procedure

A-2.4.1 Weigh out accurately about 150 mg of the standard isoproturon material into a dry 250 ml flask, dissolve and dilute to volume with methanol. Pipette out 10 ml of this solution into a 100 ml flask and dilute to volume with methanol. Pipette out 10 ml of this solution into a third flask of 100 ml capacity and dilute to volume. This final solution is taken for absorbance measurement. Weigh out accurately about 150 mg of the isoproturon, technical sample to be analyzed, into a dry 250 ml flask and proceed exactly as suggested above for the standard preparation.

A-2.4.2 *Spectrophotometric Determination* — Measure the absorbance of the standard as well as sample solutions described under **A-2.4.1** at 242 nm using the 1.000 cm cuvette and methanol as blank.

A-2.5 Calculations

Isoproturon content, percent by mass = $\frac{m_1 \times A_1 \times P}{A_1 \times m_2}$

where

m_1 = mass, in mg, of the standard taken;

A_2 = absorbance of the sample;

P = percentage purity of standard isoproturon;

A_1= absorbance of the standard isoproturon; and

m_2 = mass, in mg, of the sample taken.

A-3 HPLC METHOD

A-3.1 Principle — A HPLC unit with a UV detector is used for this assay. Using a solution containing known amounts of the standard isoproturon sample and the internal standard, the response factor, *RF*, for isoproturon in the internal approach is arrived at. A solution containing a known mass of the isoproturon sample and internal standard is injected subsequently into the HPLC unit. The percentage of isoproturon in the sample is then computed by the standard relationship.

A-3.2 Apparatus

A-3.2.1 High performance liquid chromatograph equipped with a printer-plotter-cum-integrator and UV Detector. The suggestive HPLC operating conditions are given below. However, these operating conditions are likely to change with change in HPLC equipment employed and are allowed provided standardization is done:

Column	Silica, Stainless Steel 10 μm internal diameter (i.d.) 25 cm x 4.6 mm
Solvent system	a) Cyclohexane 90 percent (*v*/*v*) b) Isopropanol 10 percent (*v*/*v*)
Detector	UV (at 254 nm)
Solvent flow rate	1.5 ml/min
Chart speed	0.2 cm/min
Sample size	10 μl

A-3.2.2 *Volumetric Flask* — 50 ml and 100 ml capacity.

A-3.2.3 *Pipettes (graduated)* — 2 ml, 5 ml and 10 ml capacity.

A-3.3 Reagents

A-3.3.1 *Internal Standard* — acetanilide, A.R. or equivalent grade.

A-3.3.2 *Cyclohexane* — spectroscopic grade.

A-3.3.3 *Isopropanol* — spectroscopic grade.

A-3.3.4 *Isoproturon* — of known purity.

A-3.4 Preparation of the Standard and Sample Solutions

A-3.4.1 Weigh out accurately 0.25 g of acetanilide into a 100 ml volumetric flask and make up to volume using the cyclohexane isopropanol mixture (90:10, *v*/*v*). This will give a solution containing 2.5 mg/ ml of the internal standard.

A-3.4.2 Weigh out 0.5 g of standard isoproturon into a 100 ml volumetric flask and dissolve it in 25 ml isopropanol. Make up to volume using the cyclohexane. This will give a stock solution containing 5 mg/ml of the standard isoproturon. Pipette out 5 ml of this standard solution into a 50 ml volumetric flask. Then pipette out 5 ml of the internal standard solution into the same flask. Mix well. Make up to the mark using the solvent mixture. Call this, Solution A. Weigh out 0.5 g of the isoproturon sample and proceed exactly as in the case of the standard sample. Call this, Solution B.

A-3.5 Procedure

Introduce 10 μl of the Solution A and Solution B into the HPLC unit. From the integrator print out and note down the peak areas of the isoproturon and acetanilide peaks in both the cases. Adjust the attenuation in such a way that the isoproturon and acetanilide peaks are obtained within the scale in both the cases. (This attenuation may change from equipment to equipment). Compute the percentage of isoproturon content in the sample as indicated in **A-3.6**.

A-3.6 Calculation

$$\text{Isoproturon content, percent by mass} = \frac{m_1 \times A_2 \times P \times A_3}{A_1 \times m_2 \times A_4}$$

where

m_1 = mass of standard isoproturon in Solution A;

A_2 = area of isoproturon peak in Solution B;

P = percentage purity of standard isoproturon;

A_3 = area of internal standard peak in Solution A;

A_1 = area of standard isoproturon peak in Solution A;

m_2 = mass of the isoproturon sample in Solution B; and

A_4 = area of internal standard peak in Solution B.

A-3.7 Precision — Data obtained by this method indicate a standard deviation of 0.5 for isoproturon at 96 percent level.

A-4 BASIC HYDROLYSIS METHOD

A-4.1 Principle

Isoproturon is hydrolysed using potassium hydroxide in aqueous diethylene glycol (1:1 by volume). Dimethylamine, the volatile product of hydrolysis, is distilled off and absorbed in standard hydrochloric acid. From the quantity of acid consumed by dimethylamine, the percentage of isoproturon present in the original sample is computed.

A-4.2 Apparatus

A-4.2.1 An all glass distillation assembly of a suitable type. A suggestive assembly is shown in Fig. 1.

A-4.3 Reagents

A-4.3.1 *Hydrochloric Acid Solution* — 0.2 N.

A-4.3.2 *Sodium Hydroxide Solution* — 0.2 N.

A-4.3.3 *Potassium Hydroxide Solution* — 35 percent aqueous.

A-4.3.4 *Diethylene Glycol*

A-4.3.5 *Silicon Defoamer*

A-4.4 Procedure

Accurately weigh 1.0 ± 0.1 g of the isoproturon, technical sample and transfer it quantitatively to a clean dry reaction flask of the hydrolysis-cum-distillation apparatus. Add 50 ml of diethylene glycol and one or two boiling chips. One ml of silicon defoamer may be added to check foaming. Attach the reaction flask to the other parts of the assembly as shown in Fig. 1. 50 ml of hydrochloric acid is taken in a beaker in which the delivery tube end dips as shown in Fig. 1. Add 50 ml of potassium hydroxide solution to the flask through a dropping funnel. Close the stop-cock of the dropping funnel as soon as the addition of alkali is complete. Heat the contents of the reaction flask to boiling. Continue the distillation up to about 24 h. The distillation can be stopped when the contents of the flask become orange brown in colour. The distillate will cease to be alkaline by that time. This can further be confirmed using a multi range *p*H paper to test the distillate. The *p*H of the distillate, as indicated by the colour of the *p*H paper, must be 7. Take care to disconnect the delivery tube from the lower end of the condenser before switching off the heating source. If this is not done, the distillate will be sucked back into the distilling flask, when it starts cooling down, after the removal of the heating source. If, however, arrangements can be made to maintain a flow of nitrogen gas through the distillation assembly, then the back suction problem will not be experienced. Rinse the connecting tube with distilled water and add the rinsings to the receiver beaker. Titrate the contents of the beaker with 0.2 N sodium hydroxide solution using phenolphthalein as indicator. At the end point, colourless solution will turn pink.

A-4.5 Calculations

$$\text{Isoproturon content, percent by mass} = \frac{[(V_1 \times N_1) - (V_2 \times N_2)]}{M} \times 20.6$$

where

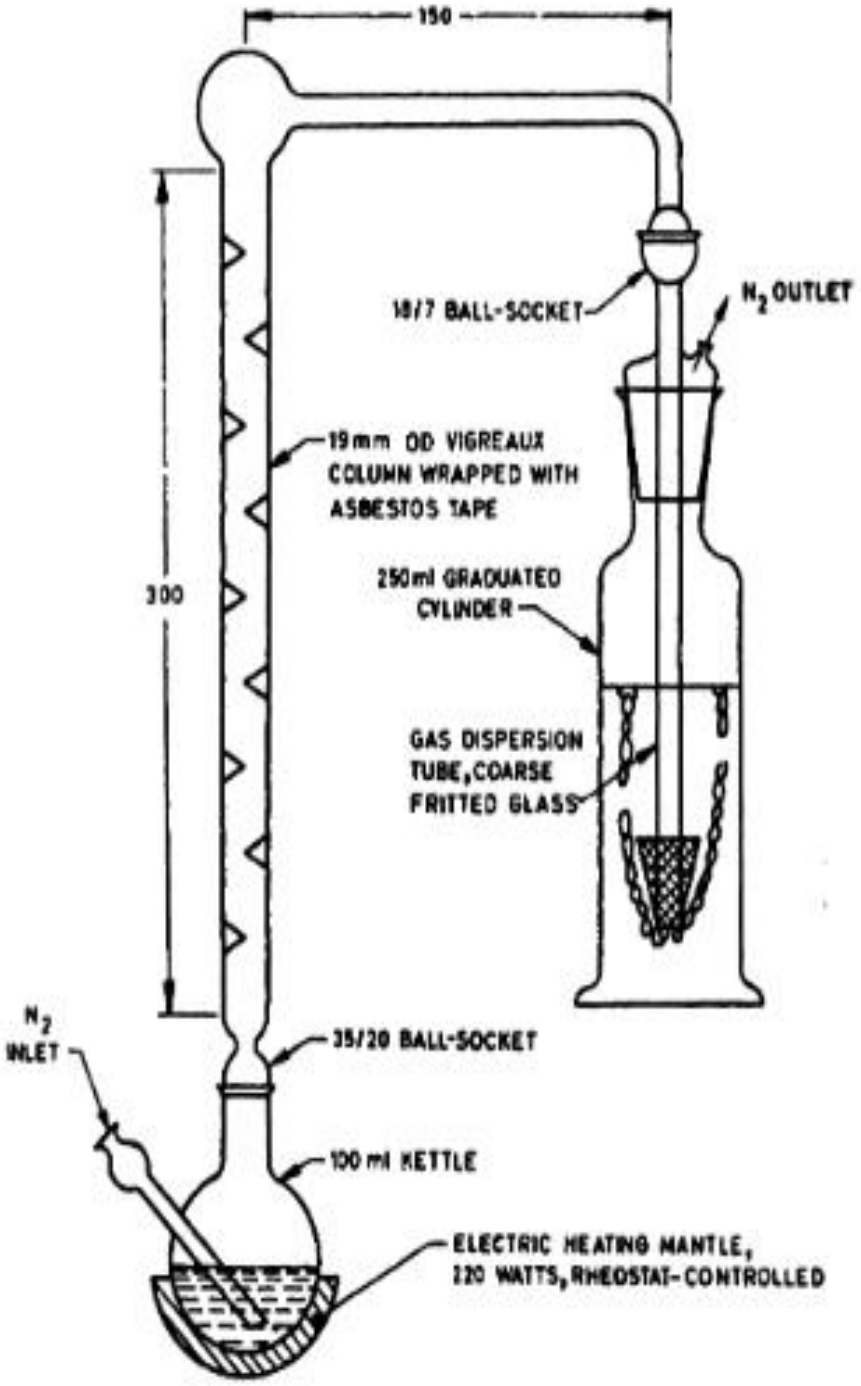
V_1 = volume, in ml, of hydrochloric acid originally taken in the receiver beaker;

N_1 = normality of the hydrochloric acid;

V_2 = volume, in ml, of sodium hydroxide consumed in the titration;

N_2 = normality of sodium hydroxide; and

M = mass, in g, of the sample taken for analysis.



All dimensions in millimetres

FIG. 1 APPARATUS FOR DETERMINATION OF ISOPROTURON CONTENT (ALKALINE HYDROLYSIS METHOD)