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भारतीय मानक मसौदा

मोनोक्रोटोफॉस, तकनीकी — विशिष्टि

(*आइ एस 8025 का तीसरा पुनरीक्षण*)

Draft Indian Standard

MONOCROTOPHOS, TECHNICAL — SPECIFICATION

(*Third Revision of IS 8025*)

ICS No. 65.100.10

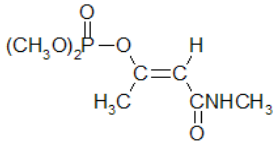
Pesticides Sectional Committee, FAD 01 **Last Date of Comments: 3 July 2024**

FOREWORD

(*Formal clauses will be added later*)

Monocrotophos, technical, exists in two isomeric forms, namely, *cis* isomer and *trans* isomer. Of these two isomeric forms, only the *cis* isomer is effective. This is a systemic as well as contact insecticide and employed in the preparation of pesticidal formulations for the control of insect and acarine pests of agricultural crops.

Monocrotophos is the name accepted by the International Organization for Standardization (ISO) for the insecticidal chemical dimethyl phosphate ester with (E)-3-hydroxy-*N*-methylcrotonamide or 3-hydroxy-*N*-methylcrotonamide dimethyl phosphate or dimethyl cis-1 -methyl-2-(methyl carbamoyl) vinyl phosphate or 3-(dimethoxy-phosphinyloxy)-*N*-methylisocrotonamide. The empirical and structural formulae and molecular mass of monocrotophos are as indicated below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_7H_{14}NO_5P$		223.17

This standard was published in 1976 and first revised in 1983. The second revision was issued in 1990 to update the various requirements in the light of the experience gained.

In this revision, 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.

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*)' This 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 requirements and the methods of sampling and test for monocrotophos, technical.

2 REFERENCES

The following Indian Standards contain provisions which through reference in this text, constitute provision 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 : 1982	Methods of test for pesticides and their formulations (*first revision*)
IS 8190 (Part 2) : 1988	Requirements for packing of pesticides: Part 2 Liquid 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 brown viscous liquid or crystalline semi-solid.

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

Table 1 Requirements of Monocrotophos, Technical

(*Clause* 3.2)

Sl. No.	**Characteristic**	**Requirements**	**Method of Test, Ref to**
(1)	(2)	(3)	(4)
i)	Monocrotophos content, percent by mass, *Min*	68.0	Annex A
ii)	Free acetic acid mono-methylamide (MMA), percent by mass, *Max*	4.0	Annex B
iii)	Acidity (as H_2SO_4), percent by mass, *Max*	3.0	IS 6940

4 PACKING

The material shall be packed as per requirements given in IS 8190 (Part 2).

5 MARKING

5.1 The containers shall be securely closed and 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 monocrotophos 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 sample of this material shall be drawn in accordance with IS 10946.

7 TESTS

Tests shall be carried out by appropriate methods as referred in col (4) of Table 1.

8 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, *Sl No.* (i)]

DETERMINATION OF MONOCROTOPHOS CONTENT

A-1 PRINCIPLE

Monocrotophos is hydrolyzed in an alkaline medium, and as a result, aceto-acetic acid monomethylamide (MMA) forms, which is quantitatively determined colorimetrically as Iron (III) complex. Free MMA is determined separately and subtracted.

A-2 REAGENTS

A-2.1 Methanol

A-2.2 Standard Sodium Hydroxide Solution - 5 N.

A-2.3 Nitric Acid - 1 N.

A-2.4 Glacial Acetic Acid

A-2.5 Acetic Acid in Methanol Solution - 5 percent (*v*/*v*) prepared by diluting 10 ml of glacial acetic acid with methanol to a volume of 200 ml.

A-2.6 Reference Standard of Aceto-Acetic Acid Monomethylamide (MMA) - of purity not less than 98 percent.

NOTE - Anhydrous MMA standard samples are susceptible to degradation by ingress of moisture and air during storage. Store in a cool, dry and dark place. Avoid repeated opening of bottle containing the reference MMA.

A-2.6.1 *Preparation of Pure MMA from 50 Percent Aqueous Solution*

A-2.6.1.1 Take 200 g of the material (50 percent MMA) in the flask and attach it to vacuum rotatory pump. Evaporate water from the flask maintaining temperature of the bath at 60 °C, till the moisture is 1 to 2 percent only. This can be verified on weighing the receiver flask, which should contain at least 98 g of the distilled off material. Take out the flask containing the material and dissolve it in minimum amount of warm benzene. Filter this solution through Buchner funnel and collect the filtrate. Adjust the volume of benzene in the filtrate to three times the mass of the material and leave it overnight in the freezer. Next day the crystals appear in the solution which are filtered through Buchner funnel and the filtrate is kept in the freezer for further crystallisation.

A-2.6.1.2 Two crops of crystals are obtained from the mother liquor. The crystals are pooled and kept overnight in vacuum at room temperature to dry. On the following day determine the purity by taking 0.2 g of the material, adding to it 100 ml of reagent mixture (10 parts of acetic acid and 1 part of hydrochloric acid) and 10 ml of saturated concentrated solution of potassium chloride. Titrate potentiometrically standard sodium nitrate solution at polarisation current of 5 μA and potential drop of 650 mV.

1 ml of 0.1 N $NaNO_2$ = 11.51 mg of MMA

A-2.7 Ferric Chloride Solution

Prepared by dissolving 40.5 g of ferric chloride ($FeCl_3.6H_2O$) in methanol and made up to 1 litre.

A-2.8 Phenolphthalein Indicator Solution

Prepared by dissolving 0.2 g of phenolphthalein in 95 percent ethanol and made up to 100 ml.

A-2.9 Standard Solution of MMA or Monocrotophos

Weigh about 0.15 g accurately of reference standard MMA and dissolve in methanol in a 250 ml volumetric flask and make up the volume.

A-2.10 Sodium Nitrate Solution

Measure 10 ml of 5 N sodium hydroxide into a 250 ml volumetric flask, 50 ml of water, 1 ml of the indicator solution, neutralize with 1 N nitric acid and make up to the mark with water.

A-2.11 Blank Solution

In a 100 ml volumetric flask, mix 10 ml of sodium nitrate solution with 10 ml of acetic acid in methanol and 10 ml of ferric chloride solution and make up to the mark with methanol.

A-2.12 Standard Sodium Nitrate Solution - 0.1 N.

A-2.13 Hydrochloric Acid - concentrated.

A-3 PROCEDURE

A-3.1 Weigh accurately a quantity of the sample equivalent to about 1.5 g of monocrotophos as active ingredient, transfer into a 100 ml volumetric flask and make up to the mark with methanol. This stock solution is also used for free MMA determination (*see* **Annex B**). Pipette 10 ml of this dilution into a 250 ml volumetric flask, and 10 ml standard solution hydroxide solution, shake thoroughly and allow to stand at (25 ± 5) °C for 30 min. Then add 1.0 ml of phenolphthalein indicator and titrate with 1 N nitric acid until the violet colour disappears. Temperature. during neutralization should be maintained at 25 °C. Then fill the volumetric flask to the mark with water. Pipette 10 ml of this solution into a l00 ml volumetric flask and successive 10 ml of acetic acid in methanol solution, 50 ml methanol and 10 ml of the ferric chloride solution. Make up the volume with methanol and mix well. This constitutes the test solution.

A-3.2 Pour 10 ml of the standard solution of MMA into a 100 ml volumetric flask. Add 10 ml of sodium nitrate solution, 10 ml acetic acid in methanol solution, 50 ml of methanol and 10 ml of the ferric chloride solution. Fill with methanol to the mark. This constitutes the comparison solution. Allow the sample solution as well as the solution to stand at (25 ± 5) °C for 10 min and measure their absorbances in a spectrophotometer at 544 nm wavelength and a l cm cell.

NOTE- The test solution and the comparison solution should be prepared at the same time. The measurement of the absorbances of both solutions has to be carried out successively at short intervals. From the preparation of the solutions to the measurement, not more than 30 min should elapse.

A-3.3 CALCULATION

Monocrotophos content, percent by mass = $1940 \times \frac{A_t \times m_s \times P}{A_s \times m_t \times 100} - K$

where,

A_t = absorbance of test solution;

A_s = absorbance of the standard reference comparison solution of MMA;

m_s = mass, in g, of the reference standard MMA (100 percent purity basis);

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

P = purity of the reference standard MMA; and

K = correction value of monocrotophos content due to free MMA (*see* **Annex B**)

ANNEX B

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

DETERMINATION OF FREE ACETO-ACETIC ACID MONOMETHYL AMIDE (MMA)

B-1 REAGENTS

B-1.1 Methanol

B-1.2 Standard Sodium Hydroxide Solution - 5 N**.**

B-1.3 Nitric Acid - 1 N**.**

B-1.4 Glacial Acetic Acid

B-1.5 Acetic Acid in Methanol Solution - 5 percent (*see* **A-2.5**)**.**

B-1.6 Preparation of Pure MMA Reference Standard (*see* **A-2.6**)**.**

B-1.7 Ferric Chloride Solution (*see* **A-2.7**)**.**

B-1.8 Phenolphthalein Indicator (*see* **A-2.8**)**.**

B-1.9 Standard Solution of MMA (*see* **A-2.9**)**.**

B-1.10 Blank Solution

Mix in a 100 ml volumetric flask, 10 ml of acetic acid in methanol and 10 ml of ferric solution and make up to the mark with methanol.

B-1.11 Comparison Solution

Pour 10 ml of the standard solution of MMA into a 100 ml volumetric flask, add 10 ml acetic acid in methanol, 50 ml methanol, 10 ml ferric chloride solution, mix well and fill up to the mark with methanol.

B-2 PROCEDURE

Take with a pipette 10 ml of the stock solution (*see* **A-3.1**) into a 100 ml volumetric flask and while keeping at (25 ± 5) °C, add 10.0 ml acetic acid in methanol solution, 50 ml methanol and

10 ml ferric chloride solution. Fill immediately with methanol to the mark and shake the solution thoroughly. This constitutes the test solution. Set the stop watch in motion. Pour the solution into a 1 cm cell and measure the absorbance at 544 nm wavelength in a spectrophotometer. This measurement shall be done 30 sec after the preparation of the solution is completed. The running of the absorbances shall be watched for the next 3 min at 30 sec intervals. The obtained absorbance curve is extrapolated at the time 'zero'.

B-3 CALCULATION

B-3.1 $K = 77.6 \times \frac{A_t \times m_s \times P}{A_s \times m_t \times 100}$

where,

A_t = absorbance of test solution extrapolated at the time 'zero';

A_s = absorbance of the comparison solution;

m_s = mass, in g, of the reference standard (100 percent purity basis);

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

P = purity of the reference standard.

B-3.2 Free aceto-acetic acid monomethyl amide (MMA), percent by mass = $\frac{K}{1.94}$

NOTE - In the determination of free MMA, the absorption of the sample solution must not exceed 0.7. When higher concentrations occur, take 2.0 ml or 90 ml of the stock solution (*see* **A-3.1**). In the calculation, the dilution has to be considered accordingly