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मानक

IS 14159 (1994): Cartap Hydrochloride, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]



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, Indian Standard

CARTAP HYDROCHLORIDE, TECHNICAL — SPECIFICATION

UDC 632.951 CAR

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

Price Group 2

FOREWORD

This Indian Standard 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.

Cartap hydrochloride, technical is employed in the preparation of insecticidal formulations.

Cartap hydrochloride is the accepted common name by the International Organization for Standardization (ISO) for S, S'-2-dimethylaminotrimethylene bis (thiocarbamate) hydrochloride. The empirical and structural formulae and molecular mass of cartap hydrochloride are given below:

Empirical FormulaStructural FormulaMolecular Mass $C_7H_{16}ClN_3O_2S_2$ CH_3
 CH_3 CH_2
 CH_2 SCONH2.HCl
 CH_2 273.8

In the preparation of this standard, due consideration has been given to the provisions of the Insecticides Act, 1968 and the Rules framed thereunder and the Standards of Weights and Measures (Packaged Commodities), Rules, 1977. 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 : 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

CARTAP HYDROCHLORIDE, TECHNICAL — SPECIFICATION

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1 SCOPE

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

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard :

IS No.			Title
4070	4000	-	-

1070:1992	revision)
5741 : 1970	Method for determination of pH
6940 : 1982	Methods of test for pesticide and their formulations (first revision)
8190 (Part 1) : 1988	Requirements for packing of pes- ticides : Part 1 Solid pesticides (second revision)
10946 : 1984	Methods of sampling of technical grade pesticides

3 REQUIREMENTS

3.1 Description

The material shall be in the form of white crystalline powder, free from extraneous impurities or added modifying agents and having a slight odour.

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

4 PACKING

The material shall be packed in fibre board drum or fibre board box provided with LDPE liner of the thickness not less than 0.062 mm. The material shall also conform to 2 of IS 8190 (Part 1) : 1988.

5 MARKING

5.1 The following information shall be marked legibly and indelibly on each container in addition to any other information as required under the *Insecticides Act*, 1968 and *Rules* framed thereunder:

- a) Name of the material;
- b) Name of the manufacturer;
- c) Date of manufacture;
- d) Date of expiry;
- e) Batch number;
- f) Cartap hydrochloride content, percent (m/m);
- g) Net mass of contents;
- h) Cautionary notice as worded in the *Insec*ticides Act, 1968 and Rules framed thereunder; and
- j) Any other requirements as given under the Standards of Weights and Measures (Packaged Commodities) Rules, 1977.

5.2 BIS Certification Marking

The product may also be marked with the Standard Mark.

Table 1	Requirements for the Cartap Hydrochloride, Technical
	(<i>Clause</i> 3.2)

SI	Characteristic	Requirement	Method of Test, Ref to	
NO.			Annex of This Standard	Cl No. of IS 6940 : 1982
(1)	(2)	(3)	(4)	(5)
i)	Cartap hydrochloride content, percent by mass (m/m), Min	98.0	A	<u></u>
ii)	Moisture content, percent by mass (m/m), Max	1.0		4
iii)	pH of 10 percent (m/m) aqueous extract (see NOTE)	3-4.5	В	

NOTE — Cartap hydrochloride is very acidic and remains stable in acidic medium. It is hydrolysed in neutral or alkaline medium. Therefore *p*H instead of acidity has been specified.

IS 14159 : 1994

5.2.1 The use of the Standard Mark is governed by the provisions of 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 Standared 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 as prescribed in IS 10946 : 1984.

7 TESTS

7.1 Tests shall be carried out in accordance with the method referred in col 4 and 5 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

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

ANNEX A

[Table 1 Item (i)]

DETERMINATION OF CARTAP HYDROCHLORIDE CONTENT

A-1 GENERAL

Two methods, namely spectrophotometric method (A-2) and iodimetric method (A-3) have been prescribed. Any one of these methods may be used. Spectrophotometric method shall be the referee method.

A-2 SPECTROPHOTOMETRIC METHOD

A-2.1 Principle

Cartap hydrochloride is dissolved in methanol and reacted with 5,5-dithiobis (2-nitrobenzoic acid) to form 2-nitro-5-thiobenzoic acid, a yellow coloured compound, which is quantitatively determined with the help of a spectrophotometer at 4.12 nm.

A-2.2 Apparatus

A-2.2.1 Spectrophotometer

A-2.2.2 Mechanical Shaker

A-2.3 Reagent

A-2.3.1 Cartap Hydrochloride Reference Standard — of known purity.

A-2.3.2 Cartap Hydrochloride Standard Solution

Weigh accurately into a 100-ml volumetric flask, a quantity of cartap hydrochloride reference standard so as to contain 100 mg of cartap hydrochloride. Dissolve and make up the volume to the mark with methanol.

A-2.3.3 5,5-dithiobis [2-nitrobenzoic acid (DTNB)] Analytical — Reagent grade.

A-2.3.4 DTNB Solution

Weigh accurately 50 mg DTNB into a 100-ml volumetric flask. Dissolve and make up the volume to the mark with methanol.

A-2.3.5 Buffer Solution

Take 80 ml each of phosphoric acid, boric acid and acctic acid into a 1000-ml volumetric flask and dilute with distilled water to the mark. Add sodium hydroxide solution and adjust the pH of the solution at 9.0 with pH-meter.

A-2.3.6 Methanol-analytical reagent grade.

A-2.3.7 Phosphoric Acid-0.5 M (49.0 g/1).

A-2.3.8 Boric Acid — 0.5 M (30.915 g/1).

A-2.3.9 Acetic Acid—0.5 M (30.025 g/1).

A-2.3.10 Sodium Hydroxide Solution—0.2 N.

A-2.4 Procedure

A-2.4.1 Preparation of Standard Curve

Take out with a pipette 8 ml cartap hydrochloride reference standard solution into a 100-ml volumetric flask and make up the volume to the mark with methanol. Take out with a pipette 0, 1, 2, 3 and 4 ml of this solution into five separate 50-ml volumetric flasks and add 4, 3, 2, 1 and 0 ml methanol into the respective flasks to get a constant volume of 4 ml. Add with a pipette 2 ml of DTNB solution into each flask, mix well. Make up the volume with the buffer solution and again mix thoroughly. Keep the volumetric flasks for a specific period of reaction time depending on the prevailing conditions, as given below:

Room Temperature	Reaction Time	
(^{o}C)	(minutes)	
20-26	60	
27	55	
28	50	
29	45	
30	40	
31	35	
32	30	
33	25	
34	21	
35	18	

Immediately measure the absorbance at 412 nm keeping the buffer solution in the reference cell and prepare a calibration curve by plotting cartap hydrochloride (mg) as abscissa and absorbance as the ordinate. Solution to be used in reference cell shall be a mixture of 4 ml methanol and 2 ml DTNB solution diluted to 50 ml with buffer solution.

A-2.4.2 Preparation of Sample Solution

Weigh accurately a quantity of the sample so as to contain 100 mg of cartap hydrochloride into a 100ml volumertic flask and make the volume to the mark with methanol. Take out with a pipette 2ml of this solution into a 50ml volumetic flask and make up the volume to the mark with methanol.

A-2.4.3 Estimation

Take 1 ml of the solution (see A-2.4.2) into a 50-ml volumetric flask and add with a pipette exactly 3 ml of methanol followed by exactly 2 ml of DTNB solution. Mix thoroughly and make up the volume to the mark with the buffer solution. Keep the flasks for the same reaction time as in the preparation of standard curve (A-2.4.1). Measure the absorbance as described in A-2.4.1. Determine the quantity of cartap hydrochloride in the sample from the calibration curve and calculate as follows:

A-2.5 Calculation

Cartap hydrochloride content,

$$=\frac{A\times100\times50}{M\times2}\times100$$

percent by mass where

- A = quantity of cartap hydrochloride, in mg, obtained from the calibration curve.
- M = mass, in mg, of the sample taken for the test.

A-3 IODIMETRIC METHOD

A-3.1 Reagents

A-3.1.1 Potassium Hydroxide Solution-1 N.

Take 56.7 g of potassium hydroxide into a 1 000-ml volumetric flask and make up the volume to the mark with water. Mix well.

A-3.1.2 Sulphuric Acid — 5 N.

Add 150 ml concentrated sulphuric acid drop by drop to 1 000 ml of ice cold water while mixing well.

A-3.1.3 Starch Reagent Solution

Take about 1 g of starch and mix well with 10 ml of water. Slowly add 200 ml hot water while mixing well. Boil till the solution becomes semitransparent and then cool. Prepare the solution when required.

A-3.1.4 Standard Iodine Solution — 1 N.

Weigh accurately 12.7 g of iodine (analytical reagent grade) into a 1 000-ml volumetric flask. Add 100 ml potassium iodide solution and mix well to dissolve. Add 1 ml of dilute hydrochloric acid. Stir well to get a homogeneous solution and filter. Make up the volume to the mark with water.

NOTE- Activity factor of iodine solution is 1.00.

A-3.1.5 Potassium Iodide Solution—40 percent (m/v).

A-3.1.6 Disodium or Tetrasodium Salt of EDTA Solution—0.01 M. Take 3.72 g of disodium or tetrasodium salt of EDTA into a 1 000-ml volumetric flask and make up the volume to the mark with water.

A-3.1.7 Methanol-20 percent (v/v).

A-3.1.8 Dilute Hydrochloric Acid

A-3.2 Procedure

A-3.2.1 Weigh accurately a portion of the sample equivalent to about 250 mg of cartap hydrochloride and transfer in a 300-ml Erlenmeyer flask with a glass stopper.

A-3.2.2 Add about 100 ml methanol, shake for 5 minutes and further add about 20 ml disodium or tetrasodium salt of EDTA solution.

A-3.2.3 Add about 30 ml potassium hydroxide solution immediately and about 10 ml sulphuric acid after slight shaking.

A-3.2.4 Add 2 ml starch reagent solution to the flask (see A-3.2.3) and titrate against standard iodine solution.

A-3.3 Calculation

Cartap hydrochloride content,

percent by mass
$$= \frac{A \times 13.69}{M} \times 100$$

where

- A = volume, in ml, of standard iodine solution consumed in titration; and
- M = mass, in mg, of sample taken for test.

ANNEX B

[Table 1, Item (iii)]

DETERMINATION OF pH

B-1 PROCEDURE

Dissolve 10 g of the material in 100 ml freshly boiled and cooled water and mix the material thoroughly with the help of glass rod and determine the pH value of the solution with a pH meter as prescribed in IS 5741 : 1970.

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