भारतीय मानक
Indian Standard

IS 869 : 2020

एथिलीन डाईक्लोराइड की विशिष्टि

(तीसरा पुनरीक्षण)

Specification for Ethylene Dichloride

(Third Revision)

ICS 71.080.20

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

August 2020 **Price Group 4**

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals, Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first issued in 1956, and subsequently revised in 1969 and 1976. In the first revision, two grades of the material were prescribed. In the second revision the two grades were amalgamated and the limits for acidity and residue on evaporation were modified to meet the current needs of the industry. Sampling procedure was modified.

Considering International scenario, this revision has been undertaken. During this revision, requirement for distillation range has been removed and purity has been added, requirement for relative density has been modified and free chlorine has been removed.

Ethylene dichloride is used as solvent for fats, oils, waxes, gums, resins and rubber; in the manufacture of vinyl chloride, and as a constituent of anti-knock fluid. It is blended with about one-third of its volume of carbon tetrachloride yielding a non-flammable mixture, used as grain fumigant.

Ethylene dichloride is flammable having a low flash point. Its vapour produces irritation of respiratory tract and conjunctiva, corneal clouding, equilibrium disturbances, narcosis, and abdominal cramps. Deaths due to liver and kidney injury following ingestion of large amounts of ethylene dichloride have been reported. Hence, care shall be taken while handling this material (*see* also **4.1** and **4.2.2**).

In this revision, considerable assistance has been derived from ASTM D5960-03 Standard Specification for Technical Grade Ethylene Dichloride.

The composition of the Committee responsible for the formulation of this standard is given at Annex C.

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

SPECIFICATION FOR ETHYLENE DICHLORIDE

(Third Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for ethylene dichloride, also known as dichloroethane, used mainly as a solvent, a constituent of fumigant formulations and a base material for vinyl chloride manufacture.

2 REFERENCE

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 agreement based on standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No./Other Publication	*Title*
265 : 1993	Hydrochloric acid — Specification (*fourth revision*)
323 : 2009	Rectified spirit for industrial use — Specification (*second revision*)
517 : 2020	Specification for methanol (methyl alcohol)
915 : 2012	Laboratory glassware — One-mark volumetric flasks (*third revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods (*first revision*)
2362 : 1993	Determination of water by Karl fischer method — Test method (*second revision*)
4161 : 1967	Specification for Nessler cylinders
4905 : 2015	Random sampling and randomization procedures (*first revision*)
8768 : 2000	Method of measurement of colour in liquid chemical products platinum — Cobalt scale (*second revision*)
ASTM D 3401	Standard test methods for water in halogenated organic solvents and their admixtures
ASTM D 4052	Standard test method for density, relative density, and API gravity of liquids by digital density meter
ASTM D 6806	Standard practice for analysis of halogenated organic solvents and their admixtures by gas chromatography

3 REQUIREMENTS

3.1 Description

The material shall consist essentially of ethylene dichloride (1, 2-dichloroethane) and shall be clear and free from sediment or suspended matter.

3.1.1 *Solubility*

The material shall be completely soluble in rectified spirit (*see* IS 323) or methanol (*see* IS 517) in all proportions.

3.2 The material shall also comply with the requirements given in Table 1 when tested as prescribed in col 4 of Table 1.

4 PACKING, STORING AND MARKING

4.1 Packing and Storing

The material shall be packed in mild steel drums, which shall be securely closed. They shall be stored in a cool place away from fire and flames and provided with adequate ventilation (*see* also foreword). Material in bulk can be stored in MS tanks and can be transported through MS tank lorries or as agreed between the purchaser and the supplier.

4.2 Marking

4.2.1 The containers shall be suitably marked with the following information:

a) Name of the material;

b) Net mass of the material;

c) Name of the manufacturer; and

d) Lot or batch number.

For supplies of material in bulk, a test report containing the following information shall be provided for each container:

1) Name of the material
2) Name of the manufacturer
3) Supply date
4) Tanker number
5) Quantity

The test report shall be certified by authorized person of the manufacturer's organization.

4.2.2 All containers in which the material is stored or transported shall also be prominently and clearly marked with the legend FLAMMABLE along with the symbol given in Fig. 5 of IS 1260 (Part 1) and the legend AVOID PROLONGED BREATHING OF THE VAPOUR (***see*** also foreword).

4.2.3 *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.

5 SAMPLING

Representative samples of the material shall be drawn and their conformity to the requirements judged as prescribed in Annex B.

Table 1 Requirements for Ethylene Dichloride

(*Clause* 3.2)

Sl No.	Characteristic	Requirement	Method of Test Ref to Clause No. in Annex A/IS/Other publication
(1)	(2)	(3)	(4)
i)	Relative density at 15°C/15°C	1.258 to 1.268	A-2/ASTM D 4052
ii)	1,2 Dichloroethane (EDC), weight percent, *Min.*	99	ASTM D 6806[1)]
iii)	Residue on evaporation, percent by mass, Max	0.01	A-3
iv)	Acidity (as HCl), percent by mass, *Max*	0.005	A-4
v)	Colour, *Max*	20 APHA	A-6/IS 8768
vi)	Moisture content, percent by mass, *Max*	0.08	A-5/ASTM D 3401

[1)] The Gas Chromatographic (GC) conditions as given in ASTM D 6806 are suggestive. However, any GC method having difference in Detector, Column packing material and type (like packed/capillary, diameter, length, film thickness etc), Calibration technique (internal std, external std, area normalization, percent area etc), Carrier Gas (He, H_2, N_2) may be used with applicable GC operating parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard.

ANNEX A

(*Table* 1)

METHODS OF TEST FOR ETHYLENE DICHLORIDE

A-1 QUALITY OF REAGENTS

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

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

A-2 DETERMINATION OF RELATIVE DENSITY

A-2.1 Apparatus

A-2.1.1 *Pyknometer or Relative Density Bottle* — 25 ml capacity.

A-2.1.2 *Water-Bath* — maintained at 15.0 ± 0.2°C.

A-2.2 Procedure

Clean and dry the pyknometcr or relative density bottle. Weigh it, fill it with freshly boiled distilled water, place it in the bath maintained at 15 ± 0.2°C, and allow sufficient time (about 45 min) to attain the temperature of the bath. Then insert the capillary stopper which has also been brought to 15 ± 0.2°C. Wipe the excess liquid from the stopper, remove the pyknometer or the relative density bottle from the bath, bring to room temperature and weigh. Empty the pyknometcr or the relative density bottle, clean and dry it, and repeat the operation with the material at 15 ± 0.2°C.

A-2.3 Calculation

$$\text{Relative density at } 15°C/15°C = \frac{A-B}{C-B}$$

Where,

A = mass in g, of the pyknometer or relative density bottle filled with the material;

B = mass in g, of the dry pyknometer or relative density bottle; and

C = mass in g, of the pyknometer or relative density bottle filled with water.

NOTE — The correction factor for relative density is +0.0015 for each degree Celsius fall in temperature and − 0.001 5 for each degree Celsius rise in temperature.

A-3 DETERMINATION OF RESIDUE ON EVAPORATION

A-3.1 Outline of the Method

A known amount of the material is evaporated to dryness, the residue cooled and weighed.

A-3.2 Procedure

Accurately weigh about 100 g of the material in a tared silica basin. Gently evaporate it to dryness on a water bath under a fume hood. Dry the residue, for one hour in an oven at 105 ± 2°C and cool in a desiccator and weigh again.

A-3.3 Calculation

Difference in mass of the silica basin gives the mass of the residue on evaporation. Express the mass of the residue as percentage of the mass of the material taken for the test.

A-4 DETERMINATION OF ACIDITY

A-4.1 Apparatus

A-4.1.1 *Conical Flask* — 300 ml capacity, glass-stoppered.

A-4.2 Reagents

A-4.2.1 *Phenolphthalein Indicator* — Dissolve 0.5 g of phenolphthalein in 100 ml of rectified spirit (*see* IS 323) or Methanol (***see*** IS 517) and make the solution faintly pink by adding dilute sodium hydroxide solution.

A-4.2.2 *Standard Sodium Hydroxide Solution* — 0.01 N.

A.4.3 Procedure

Weigh accurately about 100 g of the material into the glass-stoppered conical flask. Add 100 ml of freshly boiled and cooled distilled water which has been previously neutralized to phenolphthalein or Bromo thymol blue indicator and shake vigorously. Allow the layers to separate. Separate the aqueous layer, add to it 0.5 ml of phenolphthalein or Bromo thymol blue indicator and titrate with standard sodium hydroxide solution using a micro-burette.

A-4.4 Calculation

$$\text{Acidity (as HCl), percent by mass} = \frac{0.365\,VN \times 100}{M}$$

Where,

V = volume in ml, of standard sodium hydroxide solution;

N = normality of standard sodium hydroxide solution, and

M = mass in g, of the material taken for the test.

A-5 DETERMINATION OF MOISTURE CONTENT

The moisture content of the material shall be determined in accordance with the method prescribed in IS 2362 or ASTM D 3401.

A-6 TEST FOR COLOUR

A-6.1 Outline of the Method

The colour of the material is compared with that of the colour standard and expressed in terms of platinum cobalt units. The platinum cobalt unit is defined as the colour of an aqueous solution containing 1 part per million of platinum in the form of chloroplatinic acid and 2 parts per million of cobaltous chloride ($CoC1_2.6H_2O$).

A-6.2 Apparatus

A-6.2.1 *Nessler Cylinders* — 100 ml capacity (*see* IS 4161).

A-6.2.2 *One-Mark Volumetric Flasks* —250 and 500 ml capacities (***see*** IS 915).

A-6.3 Reagents

A-6.3.1 *Cobaltous Chloride Hexahydrate* — Solid.

A-6.3.2 *Hydrochloric Acid* — Relative density 1.16 (*see* IS 265).

A-6.3.3 *Chloroplatinic Acid* — Dissolve 250 mg of platinum in small quantity of *aqua regia* contained in a glass or porcelain basin by heating on a water-bath. When the metal has dissolved, evaporate the solution to dryness. Add 1 ml of hydrochloric acid and again evaporate to dryness. Carry out this operation twice again.

A-6.4 Preparation of Colour Standards

A-6.4.1 Dissolve 0.50 g of cobaltous chloride hexahydrate and whole of the chloroplatinic acid in 50 ml of hydrochloric acid. Warm, if necessary, to obtain a clear solution and after cooling, pour into 500-m1 one-mark volumetric flask. Dilute with water to the mark.

A-6.4.2 Pipette 10, 20, 25, 30, 40 and 50 ml of the solution into 250 m1 one-mark volumetric flasks and dilute the contents of each flask with water to the mark. These diluted solutions correspond to a colour of 20, 40, 50, 60, 80 and 100 Hazen units respectively. These shall be prepared freshly.

A-6.5 Procedure

Fill one of the Nessler cylinders to the mark with the material to be tested and the others with the colour standards. Compare the colour of the material with colour standards using a white background.

A-6.6 Report the colour, in Hazen units, of the colour standard which matches the sample.

ANNEX B

(*Clause* 5)

SAMPLING OF ETHYLENE DICHLORIDE

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.1 The sampling instrument shall be clean and dry.

B-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.4 The samples shall be placed in suitable, clean, dry, airtight, dark or amber glass or metal containers on which the material has no action.

B-1.5 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.6 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling, and the month and year of manufacture of the material.

B-1.7 Samples shall be stored in the dark.

B-2 SAMPLING INSTRUMENT

B-2.1 The following forms of sampling instrument may be used:

a) Sampling bottle or can, for taking samples from tanks or drums;
b) Sampling tube, for taking samples from bottles or small containers or from storage tanks; and
c) Sample point from pump discharge material after thorough re-circulation of the material in tank/ storage tank (wherever possible).

B-2.1.1 *Sampling Bottle or Can* — Consists of a weighted glass or metal container with removable stopper or top to which is attached a light chain (*see* Fig. 1). The bottle or the can is fastened to a suitable pole. For taking a sample, the bottle or the can is lowered into the tank to the required depth and the stopper is then removed by means of the chain.

B-2.1.2 *Sampling Tube* — Made of metal or thick glass, is 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 2). The ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end.

B-2.1.2.1 For small containers, the size of the sampling tube may be altered suitably.

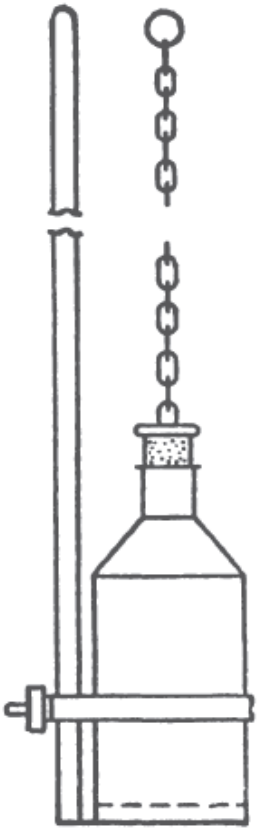

FIG. 1 SAMPLING BOTTLE OR CAN

B-3 SCALE OF SAMPLING

B-3.1 For Tanks and Drums — Each tank or drum shall be sampled separately.

B-3.2 For Bottles and Small Containers — Each lot (*see* **B-3.2.1**) shall be sampled separately.

B-3.2.1 *Lot*

In any consignment, all containers/storage tank from a single batch of manufacture shall constitute a lot. In case of continuous production, material produced in a day under similar condition shall be constitute a lot. If a consignment is known to consist of different batches of manufacture or of different sizes of containers, the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

B-3.2.2 The number of containers to be selected from a lot shall depend on the size of the lot and shall be in accordance with Table 2.

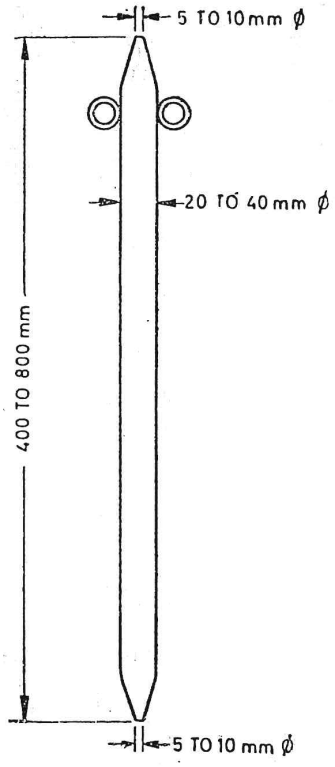

FIG. 2 SAMPLING TUBE

Table 2 Number of Containers to be Selected from a Lot

(*Clause* B-3.2.2)

Lot Size	No. of Containers To be Selected
(1)	(2)
Up to 15	3
16 to 40	4
41 to 65	5
66 to 110	7
111 and above	10

B-3.2.3 The containers shall be selected at random from the lot and in order to ensure randomness of selection, random sampling procedures given in IS 4905 may be followed.

B-4 COMPOSITE SAMPLE

B-4.1 From Tanks, Tank Lorries and Drums — As far as possible, samples from tank, tank lorry or drum should be drawn during the operation of filling. In that case equal amounts of the material shall be collected at regular intervals so as to get a total of about 1 500 ml. Where it is not possible to take a sample during filling, the material shall be drawn from different positions and depths with the sampling bottle or can after thoroughly agitating the material so as to ensure a fair amount of homogeneity.

In case of continuous activity pertaining to production/ loading/unloading etc, samples to be taken through suction pump discharge or other suitable means at regular intervals after transfer of each 20 percent quantity starting from zero. Finally, the composite sample will be prepared by thorough mixing of all the samples.

The composite sample of the material thus prepared shall be divided into three equal portions, one for the purchaser, another for the supplier and the third for the referee.

B-4.2 From Bottles and Small Containers

From each of the bottles or containers selected according to **B-3.2.3**, a small representative portion of the material shall be drawn with the help of the sampling tube. Equal quantities of the material so drawn from the various containers shall be thoroughly mixed to form a test sample of about 1 500 ml. This shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

B-4.3 All the test samples shall be transferred to separate containers, sealed and labelled with full identification particulars. The referee test sample bearing the seal of both the purchaser and the supplier shall be kept at a place agreed to between the two and shall be used in case of a dispute.

B-4.4 Tests for the determination of all the requirements given in this specification shall be performed on the test sample obtained as in **B-4.1** or **B-4.2.**

B-5 CRITERIA FOR CONFORMITY

B-5.1 The lot shall be declared as conforming to this specification if all the test results satisfy the requirements prescribed under **2**.

ANNEX C

(*Foreword*)

COMMITTEE COMPOSITION

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

Organization	*Representative(s)*
Chemical Engineering and Process Development Division, NCL, Pune	Dr C. V. Rode (***Chairman***)
Alkyl Amines Chemicals Limited, Mumbai	Shri S. V. Nikumbhe Shri Sameer Katdare (*Alternate*)
All India Alcohol-Based Industries Development Association (AABIDA), Mumbai	Shri K. L. Raphael Shri Kirti Gajjar (*Alternate*)
All India Distillers Association (AIDA), New Delhi	Shri V. N. Raina
BASF India Limited, Mumbai	Shri Kiran Bhat Shri Hemal (*Alternate*)
CSIR-Central Drug Research Institute (CDRI), Lucknow	Dr Sanjeev Kanojiya
Central Revenue Control Laboratory (CRCL), New Delhi	Dr T. A. Sreenivasa Rao
Chemical And Petrochemicals Manufacturers Association (CPMA), New Delhi	Shri Kamal Nanavaty
Deepak Fertilizer	Dr Satish Chand Saini Shri Suresh Amle (*Alternate*)
Deepak Phenolics Limited	Dr S. Samal Shri Sandipkumar Pancha (*Alternate*)
Dow Corning India Limited, Mumbai	Shri Abraham Barretto Shri Ritesh Gulabani (*Alternate*)
Godavari Biorefineries, Mumbai	Shri Shanul Laxmanrao Pagar Shri Wani A. J. (*Alternate*)
Gujarat Narmada Valley Fertilizers Company Limited, Gujarat	Dr M. J. Kapadia Shri P. R. Desai (*Alternate*)
Hindustan Organic Chemicals Limited (HOCL), Mumbai	Shri Deleep Kumar K. Shri V. Mohan (*Alternate*)
India Glycols Limited, Uttarakhand	Shri S. R. Soni Shri Alok Singhal (*Alternate*)
Indian Chemical Council (ICC), Mumbai	Dr Mritunjay Chaubey Shri J. I. Sevak (*Alternate*)
Jubilant Life Sciences Limited, Uttar Pradesh	Shri Hari Mohan Lohani
Laxmi Organic Indusrties, Mumbai	Shri J. P. Suryavanshi Dr Vijay S. Mishra (*Alternate*)
Department of Chemicals & Petrochemicals, Ministry of Chemicals & Fertilizers, New Delhi	Shri O. P. Sharma Shri Varun Singh Poonia (*Alternate*)
National Chemical Laboratory (NCL), Pune	Dr Udaya Kiran Marelli
National Test House (NTH), Ghaziabad	Shri Debashis Saha Dr Gopal Krishan (*Alternate*)
Reliance India Limited (RIL), Mumbai	Shri K. K. Sreeramachandran Shri Vasant Warke (*Alternate*)

Organization	*Representative(s)*
United Phosphorus Limited (UPL), Mumbai	SHRI M. D. VACHHANI
Director General, BIS	SHRI N. K. BANSAL, SCIENTIST 'F' AND HEAD (PCD) [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary

SHRI VIJAY KUMAR GUPTA
SCIENTIST 'C' (PCD), BIS

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc No.: PCD 09 (15702).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	2323 7617, 2323 3841
Eastern	: 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi, KOLKATA 700054	2337 8499, 2337 8561, 2337 8626, 2337 9120
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg, CHANDIGARH 160019	265 0206, 265 0290
Southern	: C.I.T. Campus, IV Cross Road, CHENNAI 600113	2254 1216, 2254 1442, 2254 2519, 2254 2315
Western	: Manakalaya, E9 MIDC, Marol, Andheri (East), MUMBAI 400093	2832 9295, 2832 7858, 2832 7891, 2832 7892

Branches : AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. COIMBATORE. DEHRADUN. DURGAPUR. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. JAMMU. JAMSHEDPUR. KOCHI. LUCKNOW. NAGPUR. PARWANOO. PATNA. PUNE. RAIPUR. RAJKOT. VISAKHAPATNAM.

Published by BIS, New Delhi