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भारतीय मानक मसौदा

एथिलीन डाईक्लोराइड — विशिष्टि

(IS 869 *का चौथा पुनरीक्षण*)

Draft *Indian Standard*

ETHYLENE DICHLORIDE — SPECIFICATION

(*Fourth Revision of* IS 869)

(ICS 71.080.20)

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09	Last date for comments: **20 April 2025**

FOREWORD

(*Formal clauses to be added later*)

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**).

This standard was first published in 1956, and subsequently revised in 1969, 1976 and 2020. 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 needs of the industry. Sampling procedure was also modified.

Considering International scenario, the third revision was taken up. During the third revision, the requirements for distillation range was removed, and purity was added. The requirements for

relative density was modified, and free chlorine was removed. Considerable assistance was derived from ASTM D5960-03 ‘Standard specification for technical grade ethylene dichloride’.

In this revision, indigenous test method based on the following ASTM have been incorporated for parameters like determination of relative density, purity and moisture content, instead of directly referring ASTM:

- ASTM D3401-97 Standard test methods for water in halogenated organic solvents and their admixtures
- ASTM D4052-22 Standard test method for density, relative density, and API gravity of liquids by digital density meter
- ASTM D6806-02 Standard practice for analysis of halogenated organic solvents and their admixtures by gas chromatography

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 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 REFERENCES

The standards listed in Annex A 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 these standards.

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.

3.2 Solubility

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

3.3 The material shall also comply with the requirements given in Table 1, when tested as prescribed in col (4) and col (5) of Table 1.

Table 1 Requirements for Ethylene Dichloride
(*Clause* 3.3)

Sl No.	Characteristics	Requirement	Method of Test, Ref to	
			Annex	IS
(1)	(2)	(3)	(4)	(5)
i)	Relative density at 15 °C/15 °C	1.258 to 1.268	B	—
ii)	1,2-Dichloroethane (EDC), percent by mass, *Min*	99	C	—
iii)	Residue on evaporation, percent by mass, *Max*	0.01	D	—
iv)	Acidity (as HCl), percent by mass, *Max*	0.005	E	—
v)	Colour, Pt-Co, *Max*	20	—	IS 8768

vi)	Moisture content, percent by mass, *Max*	0.08	F	IS 2362[1)]
[1)] In case of dispute IS 2362 shall be the referee method for determination of moisture content.				

3.4 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.

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 mild steel (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) Name of the manufacturer and his recognized trade-mark, if any;
c) Month and year of manufacture;
d) Net mass of the material in the container;
e) Lot or batch number; and
f) Any other statutory requirements.

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

a) Name of the material;
b) Name of the manufacturer;
c) Supply date;
d) Tanker number; and
e) Quantity.

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

4.2.3 All containers (including containers for bulk transport/storing) in which the material is stored or transported shall also be prominently and clearly marked with the word '**FLAMMABLE'** along

with the symbol given in Fig. 5 of IS 1260 (Part 1). The label shall also bear the following clear and legible instruction:

AVOID PROLONGED BREATHING OF THE VAPOUR

4.2.4 *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

The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Annex G.

ANNEX A
(*Clause* 2)
LIST OF REFERRED STANDARDS

IS No.	*Title*
IS 323 : 2009	Rectified spirit for industrial use — Specification (*second revision*)
IS 517 : 2020	Specification for methanol (methyl alcohol) (*third revision*)
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 1260 (Part 1) : 1973	Pictorial markings for handling and labelling of goods: Part 1 Dangerous goods (*first revision*)
IS 1447 (Part 1) : 2021	Methods of sampling of petroleum and its products: Part 1 Manual sampling (*second revision*)
IS 2362 : 1993	Determination of water by Karl Fischer method — Test method (*second revision*)
IS 4905 : 2015/ISO 24153 : 2009	Random sampling and randomization procedures (*first revision*)
IS 8768 : 2000	Method of measurement of colour in liquid chemical products platinum-cobalt scale (*second revision*)

ANNEX B
[*Table* 1, *Sl No.* (i)]
DETERMINATION OF RELATIVE DENSITY

B-1 METHOD A – PYKNOMETER METHOD

B-1.1 Apparatus

B-1.1.1 *Pyknometer or Relative Density Bottle* — 25 ml capacity

B-1.1.2 *Water Bath* — Maintained at (15.0 ± 0.2) °C

B-1.2 Procedure

Clean and dry the pyknometer or relative density bottle. Weigh it, fill it with freshly boiled distilled water, place it in the bath maintained at (15.0 ± 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 ± 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 pyknometer or the relative density bottle, clean and dry it, and repeat the operation with the material at (15.0 ± 0.2) °C.

B-1.3 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 + 0.001 5 for each degree celsius fall in temperature and − 0.001 5 for each degree celsius rise in temperature.

B-2 METHOD B – DIGITAL DENSITY METER

B-2.1 Outline of the Method

Approximately 1 ml to 2 ml of sample is injected into an oscillating sample tube. The change in oscillating frequency, resulting from the shift in the tube's mass, is used in conjunction with calibration data to determine the relative density of the sample.

B-2.2 Apparatus

B-2.2.1 *Digital Density Analyzer*

It consist of a U-shaped, oscillating sample tube and a system for electronic excitation, frequency counting, and display.

B-2.2.2 *Circulating Constant Temperature Bath*

It is capable of maintaining the temperature of the circulating liquid constant to 60.05 °C in desired range.

B-2.2.3 *Syringes (For Manual Injection)* — 2 ml

NOTE — Syringe tip or an adapter tip shall fit the opening of the oscillating tube.

B-2.2.4 *Autosampler (For Automated Injection)*

B-2.2.5 *Thermometer* — Calibrated and graduated to 0.1 °C

NOTES

1 The thermometer shall bear a valid calibration certificate from any institution authorized to issue calibration certificate traceable to international or national measurement standards.
2 Temperature sensing device (TSD), capable of monitoring the observed test temperature to within an accuracy of ± 0.05 °C may also be used to monitor the temperature.

B-2.3 Reagents

B-2.3.1 *Cleaning Solvent* — Such as petroleum naphtha or other solvents capable of removing samples entirely from flushing tube.

WARNING — Petroleum naphtha is extremely flammable.

B-2.3.2 *Acetone* — For flushing and drying the sample tube

B-2.3.3 *Dry Air* — For drying the oscillator tube

B-2.4 Preparation of Apparatus

Set up density analyzer (including the constant temperature bath and related attachments, if necessary) following the manufacturer's manual. Adjust the bath or internal temperature control to establish and sustain the desired test temperature within the analyzer's sample compartment. Perform instrument calibration at the specific temperature at which the relative density of the sample is measured by procedure given at **B-2.5**.

B-2.5 Calibration

NOTE — Calibration of the instrument is to be done as first step, whenever there is a change in the test temperature.

B-2.5.1 For calibration of instrument, whenever required, calculate constants *A* and *B* from periods of oscillation (*T*) observed when the sample cell contains air and freshly boiled reagent water.

B-2.5.1.1 While monitoring the oscillator period, *T*, flush the sample tube with a cleaning solvent, followed by an acetone flush, and dry with dry air. Contaminated or humid air can impact calibration. If required, pass the air used for calibration through a suitable purification and drying train. To prevent ingress of moist air, the inlet and outlet ports for the U-tube must be plugged during the measurement of calibration air.

B-2.5.1.2 The dry air in the U-tube is allowed to reach thermal equilibrium with the test temperature and *T*-value for air is recorded.

B-2.5.1.3 Introduce about 1 ml to 2 ml of freshly boiled reagent water into the sample tube using a suitable syringe. Ensure the test portion is homogeneous and free of even the smallest air or gas bubbles. Allow the display to stabilize and record the *T*-value for water.

B-2.5.1.4 Calculate the density of air at the test temperature using the following equation:

$$d_a = 0.001\ 293 \text{ x } \frac{273.15}{T} \text{ x } \frac{P}{101.325}$$

where

d_a = density, in g/ml, of air at test temperature;
T = temperature, in K; and
P = barometric pressure, in kPa.

B-2.5.1.5 Using the observed *T*-values and the reference values for air and density of water d_w = 1.000 g/ml and, calculate the values of the constants *A* and *B* using the following:

$$A = \frac{(T_w^2 - T_a^2)}{(1.000 - d_a)}$$

$$B = T_a^2 - (A \text{ x } d_a)$$

where

T_w= observed period of oscillation, in µs, for the cell containing water;
T_a= observed period of oscillation, in µs, for the cell containing air; and
d_a = density of air, in g/ml, at the test temperature.

NOTE — Alternatively, use the *T* and *d* values for the other reference liquid, if one is used.

B-2.5.1.6 If the instrument is equipped to calculate density from the constants *A* and *B* and the observed *T*-value from the sample, then enter the constants into the instrument memory as per the manufacturer's instructions. Alternatively, if the instrument is designed for it, allow it to make the necessary corrections in the calibration or adjustment constants as part of the built-in calibration or adjustment procedure.

B-2.5.1.7 Verify the calibration and make adjustments, if necessary, by performing the routine calibration check.

B-2.5.2 Some density meter analyzers may require weekly calibration adjustments to constants *A* and *B*, which can be made if needed without repeating the calculation procedure.

NOTE — The need for a change in calibration is generally due to deposits in the sample tube that are not removed by the routine flushing procedure. Although this condition can be compensated for by adjusting *A* and *B*. It is good practice to clean the tube with a strong oxidizing acid.

B-2.5.2.1 Flush and dry the sample tube as described in **B-2.5.1.1,** allowing the display reading to stabilize. If the display does not show the correct density for air at the test temperature, repeat the cleaning procedure or adjust the value of constant *B*, starting with the last decimal place until the correct density is displayed.

B-2.5.2.2 If adjustment to constant *B* was necessary in **B-2.5.2.1**, continue the recalibration by introducing freshly boiled reagent water into the sample tube and allow the display reading to stabilize. If the instrument has been calibrated to display the density, adjust the reading to the correct value for water at the test temperature by changing the value of constant *A*, starting with the last decimal place. If the instrument has been calibrated to display the relative density, adjust the reading to the value 1.000 0.

B-2.5.3 Some analyzer models are designed to display only the measured period of oscillation (*T*-values), and their calibration requires the determination of an instrument constant *K*, which must be used to calculate relative density from the observed data.

B-2.5.3.1 Flush and dry the sample tube as described in **B-2.5.1.1** and allow the display reading to stabilize. Record the *T*-value for air.

B-2.5.3.2 Introduce freshly boiled reagent water into the sample tube, allow the display reading to stabilize, and record the *T*-value for water.

B-2.5.3.3 Using the observed *T*-values and density for water and air, calculate the instrument constant *K* using the following:

$$K = \frac{1.000\,0 - d_a}{(T_w^2 - T_a^2)}$$

where

T_w= observed period of oscillation, in µs, for the cell containing water;
T_a= observed period of oscillation, in µs, for the cell containing air; and
d_a = density of air, in g/ml, at the test temperature.

B-2.6 Procedure

B-2.6.1 *Manual Injection*

B-2.6.1.1 Take 1 ml to 2 ml of the sample in a clean, dry tube of the instrument using a syringe or by siphoning, ensuring that the sample tube is properly filled, homogeneous and free of gas bubbles. Check the integrity of the filled sample by using optical or physical methods to verify absence of gas bubbles. If gas bubbles are detected, empty and refill the sample tube, and recheck for gas bubbles.

B-2.6.1.2 Once the instrument displays a stable reading to four significant figures for relative density and five for *T*-values, indicating temperature equilibrium, record the appropriate values.

B-2.6.1.3 If two determinations by manual injection is done, the difference between the two values recorded should not differ by 0.000 2 for relative density. The value to be reported shall be average of both the determinations.

B-2.6.2 *Automated Injection*

B-2.6.2.1 An autosampler is used when analyzing samples through automated injection. Follow appropriate operating instructions to ensure the integrity of the test specimen before analysis and for transferring a representative test specimen into the instrument for analysis.

B-2.6.2.2 Record the relative density determined by the analyses.

B-2.7 Calculation

B-2.7.1 *Calculating Density Analyzers*

The recorded value is the final result when a single determination is conducted (or taking the average of two determinations as the final result), expressed as relative density.

B-2.7.2 *Non-calculating Density Analyzers*

Using the observed *T*-value for the sample and the *T*-value for water and appropriate instrument constant, *K*, calculate relative density by the following:

$$\text{Relative Density, t/t} = 1 + [K_2 \text{ x } (T_s^2\text{-}T_w^2)]$$

where

T_w= observed period of oscillation, in µs, for the cell containing water;
T_s= observed period of oscillation, in µs, for the cell containing air;
K_2 = instrument constant for relative density; and
t = temperature of test, °C.

NOTE — Digital density meter having inbuilt calculation of variables may also be used and shall be calibrated as equipment manual.

ANNEX C
[*Table* 1, *Sl No.* (ii)]
DETERMINATION OF ETHYLENE DICHLORIDE CONTENT (PURITY)

C-1 GENERAL

This method is used to determine ethylene dichloride content (purity), its impurities and stabilizers present by gas chromatographic method. Purity will, unless otherwise stated, be determined by subtracting from 100, the sum of impurities (as determined by gas chromatography) and stabilizers only. Table 2 shows the indicative list of possible impurities and stabilizers for ethylene dichloride.

Table 2 Indicative Impurities and Stabilizers
(*Clause* C-1)

Sl No.	Indicative Impurities and Stabilizers
(1)	(2)
i)	Ethylene
ii)	Vinyl chloride
iii)	Ethyl chloride
iv)	2-Chloropropane
v)	1-Chloropropane
vi)	*trans*-1,2-Dichloroethylene
vii)	1,1-Dichloroethane
viii)	Chloroprene
ix)	*cis*-1,2-Dichloroethylene
x)	Chloroform
xi)	Carbon tetrachloride

xii)	Benzene
xiii)	Trichloroethylene
xiv)	2,3-Dichlorobutane
xv)	1,1,2-Trichloroethane
xvi)	Tetrachloroethylene
xvii)	Chlorobenzene
xviii)	Tetrachloroethane
xix)	2,2-Dichlorodiethyl ether
xx)	*para*-Dichlorobenzene
xxi)	*ortho*-Dichlorobenzene
xxii)	1,2,4-Trichlorobenzene
xxiii)	Any other organic impurities as per licensor's process / technology

C-2 APPARATUS

C-2.1 Gas Chromatograph

C-2.1.1 Any gas chromatograph available with a flame ionization detector, a split/splitless injector and a suitable electronic integrator/software may be used with following accessories and operating conditions:

Column	:	Non-polar capillary columns of about 0.32 mm by 30 m or 60 m or equivalent
Carrier gas	:	Helium or hydrogen or nitrogen
Carrier gas flow rate, ml/min	:	1 to 3
Sample size, µl	:	0.15 to 1
Injector temperature	:	200 °C
Detector temperature	:	200 °C to 240 °C
Oven temperature for isothermal	:	40 °C to 80 °C
Programmed oven temperature	:	50 °C to 200 °C

NOTES

1 The above gas chromatographic (GC) conditions 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 standard, external standard, 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.

2 GC-Mass Spectrometry (GC-MS) is an alternate method to measure the required components.

C-2.2 Syringe — 1 µl

NOTE — Auto sampler may also be used to inject the sample into the column.

C-3 PROCEDURE

C-3.1 The separation is determined to be adequate by preparing standards of known amounts of the impurities and stabilizers in concentrations near enough to the expected concentrations in the sample for the instrument response to be linear over the concentration range of interest. Minimum three injections of this standard are made and the average shall be considered for evaluating the results obtained for the concentrations of the individual impurities and stabilizers with the standard/known values.

C-4 CALCULATION

C-4.1 Purity, percent by mass =

$$100 - (Impurity\ content, percent\ by\ mass + Stabilizer\ content, percent\ by\ mass)$$

ANNEX D

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

DETERMINATION OF RESIDUE ON EVAPORATION

D-1 OUTLINE OF THE METHOD

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

D-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 1 h in an oven at (105 ± 2) °C and cool in a desiccator and weigh again.

D-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.

ANNEX E

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

DETERMINATION OF ACIDITY

E-1 APPARATUS

E-1.1 Conical Flask — 300 ml capacity, glass stoppered

E-2 REAGENTS

E-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.

E-2.2 Standard Sodium Hydroxide Solution — 0.01 N

E-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 bromothymol blue indicator and shake vigorously. Allow the layers to separate. Separate the aqueous layer, add to it 0.5 ml of phenolphthalein or bromothymol blue indicator and titrate with standard sodium hydroxide solution using a micro-burette.

E-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.

NOTE — Auto-titrator using *p*H electrode can also be used to carry out titration.

ANNEX F

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

DETERMINATION OF MOISTURE CONTENT

F-1 GENERAL

Two methods, namely, Method A Determination of moisture content by using coulometric karl fischer titrator and Method B Determination of moisture content by using volumetric Karl Fischer titrator have been prescribed.

F-2 OUTLINE OF TEST METHODS

F-2.1 The determination of water is based on the reaction of water and iodine in the presence of sulfur dioxide, alcohol, and an organic base as per the equation given below:

$$H_2O + I_2 + SO_2 + CH_3OH + 3RN \rightarrow (RNH)SO_4CH_3 + 2(RNH)I$$

where

RN = organic base

F-2.2 In the volumetric titration test method, the sample is added to Karl Fischer solvent containing sulfur dioxide and an amine dissolved in anhydrous methanol. This solution is then titrated with an anhydrous solvent containing standardized iodine.

F-2.3 In the coulometric titration test method, there is no need for iodine standardization. In this approach, the sample is injected into an electrolytic cell where the iodine required for the reaction with water is generated through the anodic oxidation of iodide.

F-2.4 In both methods, the end point is determined amperometrically using a platinum electrode. This electrode can detect a sharp change in cell resistance when all the water content in the sample has completely reacted with iodine.

F-3 METHOD A — COULOMETRIC KARL FISCHER TITRATION METHOD

F-3.1 Apparatus

F-3.1.1 *Coulometric Titrator* — With a single or dual bath electrolytic cell, dual platinum electrode, magnetic stirrer, and control unit

F-3.1.2 *Syringes* — 2 ml, 5 ml, 10 ml and 20 ml

F-3.1.3 *Micro-syringe* — 5 µl

F-3.1.4 *Silicone Rubber Block or Silicone Rubber Septa*

F-3.1.5 *Drying Oven with Air Circulation*

F-3.1.6 *Desiccator*

F-3.1.7 *Analytical Balance*

F-3.2 Reagents

F-3.2.1 *Anode Reagent* — For dual bath titration

F-3.2.2 *Cathode Reagent* — For dual bath titration

F-3.2.3 *Single Bath Reagent*

F-3.3 Procedure

F-3.3.1 Set the Karl Fischer coulometric titrator in accordance with procedure as mentioned in instrument manufacturers manual. After setting the instrument add the appropriate amount of coulomat reagents to the anode and cathode compartments of the titration cell. The cell solutions must be made anhydrous before introducing the sample. This can be achieved by either pre-titrating the cell contents or by adding a small amount of iodine/methanol solution until a faint brownish coloration develops.

F-3.3.2 The amount of sample injected into the cell depends on the moisture content present. Table 3 provides the suggested sample volumes corresponding to expected water concentrations.

Table 3 Recommended Volume of Sample for Coulometric Titration
(*Clause* F-3.3.2)

Sl No.	Water Content, ppm	Sample Weight, g
(1)	(2)	(3)
i)	1 000	0.5
ii)	500	0.5
iii)	100	1.0
iv)	50	2.0
v)	10	5.0

F-3.3.3 Before use, clean and thoroughly dry the sampling syringe in a hot air oven, then cool it in a desiccator. Now fill the syringe to the desired level with the sample, draw back the plunger to ensure no sample is left in the needle.

F-3.3.4 Cover the syringe needle with a silicone rubber block or a piece of silicone rubber septa to prevent evaporation or spillage while weighing the sample. Weigh the syringe and contents to the nearest 0.000 1 g.

F-3.3.5 Remove the silicone block, insert the needle into the titration cell septum, and inject the sample slowly, ensuring the needle does not touch the anode solution. Withdraw the needle slowly from the cell and place the silicone block onto the needle's tip. Weigh the empty syringe again.

The difference between the first and second weights represents the amount of sample injected into the titration cell.

F-3.3.6 Start the instrument for titration to take place. After titration, note the moisture content (µg or mg) present from the reading displayed on the instrument.

F-3.4 Calculation

Moisture content in the material is calculated:

$$\text{Moisture content, ppm} = \frac{Water\ content\ (in\ \mu g)}{Sample\ injected\ (in\ g)}$$

F-4 METHOD B — POTENTIOMETRIC OR VOLUMETRIC KARL FISCHER TITRATION METHOD

F-4.1 Apparatus

F-4.1.1 *Volumetric Titrator* — With titration cell, dual platinum electrode, magnetic stirrer, dispensing burette and control unit.

F-4.1.2 *Syringes* — 2 ml, 5 ml, 10 ml and 20 ml

F-4.1.3 *Micro-syringe* — 5 µl

F-4.1.4 *Silicone Rubber Block or Silicone Rubber Septa*

F-4.1.5 *Drying Oven with Air Circulation*

F-4.1.6 *Desiccator*

F-4.1.7 *Analytical Balance*

F-4.2 Reagents

F-4.2.1 *Karl Fischer Volumetric Titrant* — Mixture of an organic amine, sulfur dioxide, and iodine dissolved in a non-hygroscopic solvent.

F-4.2.2 *Karl Fischer Solvent* — Anhydrous methanol or any appropriate solvent

F-4.3 Procedure

F-4.3.1 Set the Karl Fischer volumetric titrator in accordance with procedure as mentioned in instrument manufacturer's manual.

F-4.3.2 Take a known quantity of Karl Fischer solvent, enough to cover the electrode ends and also the titrant burette tip, in a clean and dry titration cell. Set the stirring speed to generate a well-defined vortex within the solvent. Also before introducing the sample, pre-titrate the solvent to eliminate all moisture present in the solvent.

F-4.3.3 The amount of sample injected into the cell depends on the moisture content present. Table 4 provides the suggested sample size corresponding to expected water concentrations and also various titrant concentration.

Table 4 Recommended Volume of Sample for Volumetric Titration
(*Clause* F-4.3.3)

Sl No.	Water Content, ppm	5 mg H_2O/ml Titrant, g	2 mg H_2O/ml Titrant, g	15 mg H_2O/ ml Titrant, g
(1)	(2)	(3)	(4)	(5)
i)	1 000	2.5 to 10	1 to 10	0.5 to 5
ii)	500	5 to 20	2 to 20	1 to 10
iii)	100	20	10 to 20	5 to 20
iv)	50	-	20	10 to 20
v)	10	-	> 20	> 20

NOTE — Generally a titrant with a capacity of 1 mg or 2 mg H_2O/ml is appropriate for determining moisture content in halogenated solvents.

F-4.3.4 Before use, clean and thoroughly dry the sampling syringe in a hot air oven, then cool it in a desiccator. Now fill the syringe to the desired level with the sample, draw back the plunger to ensure no sample is left in the needle.

F-4.3.5 Cover the syringe needle with a silicone rubber block or a piece of silicone rubber septa to prevent evaporation or spillage while weighing the sample. Weigh the syringe and contents to the nearest 0.000 1 g.

F-4.3.6 Remove the silicone block, insert the needle into the titration cell septum, and inject the sample slowly, ensuring the needle does not touch the anode solution. Withdraw the needle slowly from the cell and place the silicone block onto the needle's tip. Weigh the empty syringe again. The difference between the first and second weights represents the amount of sample injected into the titration cell.

F-4.3.7 Start the instrument for titration to take place. After titration, note the moisture content (µg or mg) present, from the reading displayed on the instrument.

F-4.4 Calculation

Moisture content in the material is calculated:

$$\text{Moisture content, ppm} = \frac{Water\ content\ (in\ \mu g)}{Sample\ injected\ (in\ g)}$$

F-5 REPORT

The moisture content is to be reports as percent by mass.

ANNEX G
(*Clause* 5)
SAMPLING OF ETHYLENE DICHLORIDE

G-1 GENERAL REQUIREMENTS OF SAMPLING

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

G-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.

G-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.

G-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.

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

G-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.

G-1.7 Samples shall be stored in the dark.

G-2 SAMPLING INSTRUMENT

G-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).

G-2.1.1 *Sampling Bottle or Can*

It 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.

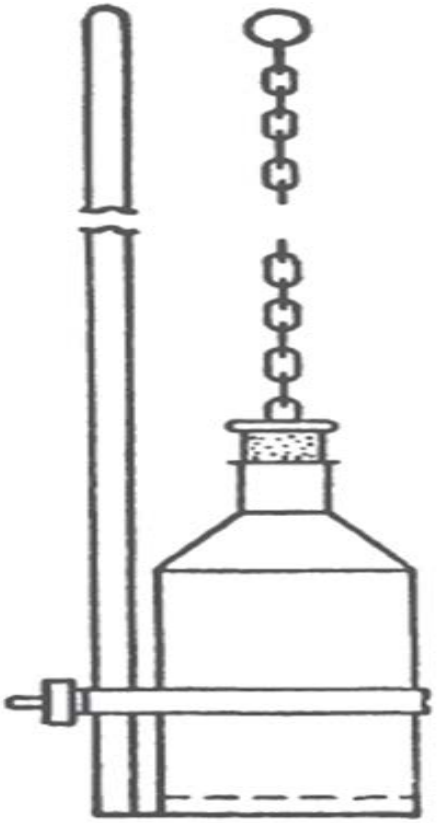

FIG. 1 SAMPLING BOTTLE OR CAN

G-2.1.2 *Sampling Tube*

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

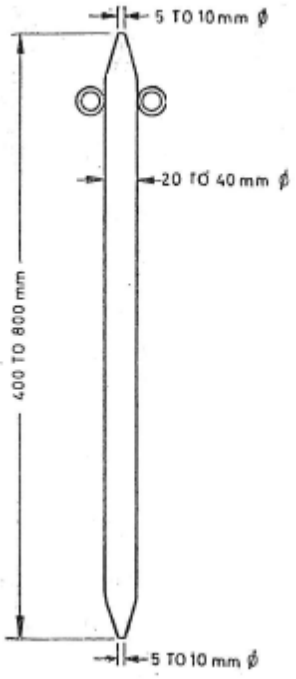

FIG. 2 SAMPLING TUBE

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

G-3 SCALE OF SAMPLING

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

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

G-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.

G-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 5.

G-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.

Table 5 Number of Containers to be Selected from a Lot
(*Clause* G-3.2.2)

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

G-4 COMPOSITE SAMPLE

G-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.

G-4.2 From Bottles and Small Containers

From each of the bottles or containers selected according to **G-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.

G-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.

NOTE — IS 1447 (Part 1) or alternate sampling techniques may also be used complying to integrity of sampling process and incorporating adequate safety precautions.

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

G-5 CRITERIA FOR CONFORMITY

G-5.1 The lot shall be declared as conforming to this specification if all the test results satisfy the requirements prescribed under **3**.