Doc: PCD 09 (27924) WC
April 2025

BUREAU OF INDIAN STANDARDS
DRAFT FOR COMMENTS ONLY
(*Not to be reproduced without permission of BIS*
or used as an Indian Standard)

भारतीय मानक मसौदा

इथाइलीन ऑक्साइड — विशिष्टि

(IS 5573 *का दूसरा पुनरीक्षण*)

Draft *Indian Standard*

ETHYLENE OXIDE — SPECIFICATION

(*Second Revision of* IS 5573)
(ICS 71.080.60)

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09	Last date for Comments: **24 June 2025**

FOREWORD

(*Formal clauses to be added later*)

Ethylene oxide is used in organic synthesis, especially in the production of ethylene glycol. It forms starting material for the manufacture of acrylonitrile and non-ionic surfactants. It is also used as a fumigant for foodstuffs and in textiles and pharmaceutical industry.

This standard was first published in 1969 and subsequently revised in 1984. In first revision requirement of colour was included. The requirements of aldehydes content and non-volatile matter were modified. The requirement of total chlorides were modified to ionizable chlorides content.

In this revision, the changes are as follows:

a) Modified the description of the material;
b) Modified the requirements of the following parameters, keeping in mind the quality of product used and manufactured: aldehyde (*as* acetaldehyde content), moisture content, acidity (as CH_3COOH), non-volatile matter and ethylene oxide;
c) Modified the test method for determination of colour and aldehyde (*as* acetaldehyde content);
d) Incorporated alternate test method for determination of moisture content, acidity (as CH_3COOH), ionizable chlorides and non-volatile matter;
e) Considering toxicity and carcinogen nature of the material and colour being not an important parameter, colour requirement is made optional parameter to be tested; and

f) Modified the formula for determining the ethylene oxide content (purity).

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, the methods of sampling and test for ethylene oxide.

2 REFERENCES

The standards given below 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 possibilities of applying the most recent edition of these standards:

IS No.	*Title*
IS 265 : 2021	Hydrochloric acid — Specification (*fifth revision*)
IS 915 : 2012 / ISO 1042 : 1998	Laboratory glassware — One-mark volumetric flasks (*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 2362 : 1993	Determination of water by Karl Fischer method — Test method (*second revision*)
IS 6269 : 1971	Code of safety for ethylene oxide

3 REQUIREMENTS

3.1 Description

The material shall be transparent, colourless gas or liquid, free from any suspended or entrained matter.

3.2 The material shall comply with the requirements prescribed in Table 1, when tested according to the methods given in col (4) of Table 1.

Table 1 Requirements for Ethylene Oxide
(*Clause* 3.2)

Sl No.	Characteristic	Requirement	Method of test, Ref to Annex

(1)	(2)	(3)	(4)
i)	Colour, Pt-Co scale, *Max*[1]	10	A
ii)	Aldehydes (as acetaldehyde content), ppm, *Max*	50	B
iii)	Moisture content, ppm, *Max*	300	C
iv)	Acidity (as CH_3COOH), ppm, *Max*	20	D
v)	Ionisable chlorides, mg/l, *Max*	10	E
vi)	Non-volatile matter[2], ppm, *Max*	50	F
vii)	Ethylene oxide content (by difference), percent by mass, *Min*	99.95	G

[1] Optional parameter
[2] The non- volatile matter content may increase during storage with time, but the limit specified shall be adhered to at the time of supply by the manufacturer.

3.3 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.

4 PRECAUTIONS IN HANDLING

4.1 Owing to the flammability and toxicity of ethylene oxide in its liquid and vapour states and also owing to the dangers inherent in the combustion or explosive decomposition of ethylene oxide, every reasonable precaution shall be observed in its handling, use and storage (*see* IS 6269).

5 PACKING AND MARKING

5.1 Packing

5.1.1 The material shall be supplied in cylinders or in rail tank-cars or road tankers of approved design and of suitable capacity.

5.1.2 The cylinders shall conform to the requirements prescribed in *Gas Cylinder Rules*, 2016 of the Government of India, with such modifications as may be ordered from time to time by the Chief Inspector of Explosives, Government of India, or other duly constituted authority.

5.2 Marking

5.2.1 The marking, painting, labelling and transport of cylinders, rail tank-cars and road tankers shall be in accordance with the requirements of the *Gas Cylinder Rules*, 2016 of the Government of India, with such modifications as may be ordered from time to time by the Chief Inspector of Explosives, Government of India, or other duly constituted authority.

5.2.2 The cylinders, rail tank-cars and road tankers shall also be suitably labelled as shown in Fig. 1 and 5 of IS 1260 (Part 1).

5.2.3 *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.

6 SAMPLING

6.1 The method of drawing representative samples shall be as prescribed in Annex H.

ANNEX A

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

DETERMINATION OF COLOUR

A-1 APPARATUS

A-1.1 Spectrophotometer, equipped for liquid sample

A-1.1.1 *Spectrophotometer Cells*, 10 mm light path

A-1.1.2 *Colour Comparison Tubes,* 100 ml Nessler tube, provided with ground-on

A-1.1.3 *Colour Comparator*

A-1.2 One-mark Graduated Flasks, 250 ml and 500 ml capacities (*see* IS 915)

A-2 REAGENTS

A-2.1 Cobalt Chloride ($CoCl_2.6H_2O$)

A-2.2 Potassium Chloroplatinate (K_2PtCl_6)

A-2.3 Concentrated Hydrochloric Acid, specific gravity of 1.19 (*see* IS 265)

A-2.4 Preparation of Platinum-Cobalt Stock Solution

Dissolve 1.245 g of potassium chloroplatinate (K_2PtCl_6) and 1.00 g of cobalt chloride

($CoCl_2.6H_2O$) in water. Add 100 ml of hydrochloric acid (HCl) and dilute it to 1 litre with distilled water. This solution has a colour of 500 platinum-cobalt scale units. The absorbance of this solution in cell having 10 mm light path, with distilled water as reference solution, shall lie within the limits as given in Table 2.

Table 2 Absorbance Tolerance Limits for 500 Platinum-Cobalt Stock Solution
(*Clause* A-2.4)

Wavelength, nm	*Absorbance*
430	0.110 to 0.120
455	0.130 to 0.145
480	0.105 to 0.120
510	0.055 to 0.065

A-2.5 Platinum-Cobalt Standards

Prepare Pt-Co standard solutions as given in Table 3 from Pt-Co stock solution by diluting required volume to 100 ml with distilled water in Nessler tube. For more precise measurement of light colour below 15 Pt-Co, prepare solution as given in Table 4, by diluting required volume to 100 ml with distilled water in Nessler tube.

Table 3 Pt-Co Colour Standards Solution
(*Clause* A-2.5)

Colour Standard Number	**Stock Solution, ml**	**Colour Standard Number**	**Stock Solution, ml**
5	1	70	14
10	2	100	20
15	3	150	30
20	4	200	40
25	5	250	50
30	6	300	60
35	7	350	70
40	8	400	80
50	10	450	90
60	12	500	100

Table 4 Pt-Co Colour Standards for Very Light Colours
(*Clause* A-2.5)

Colour Standard Number	**Stock Solution, ml**	**Colour Standard Number**	**Stock Solution, ml**
1	0.20	9	1.80
2	0.40	10	2.00
3	0.60	11	2.20

4	0.80	12	2.40
5	1.00	13	2.60
6	1.20	14	2.80
7	1.40	15	3.00
8	1.60	-	-

A-3 SAMPLE PREPARATION

Take 200 g distilled water in 1 litre conical flask and maintain temperature by keeping it on ice bath. Carefully, take 200 g of ethylene oxide and add in 1 litre flask, with gentle mixing. Loosely place stopper on the flask and keep on the ice bath for cooling.

A-4 PROCEDURE

Filter/centrifuge the sample if visible turbidity/suspended matter is noticed. Take 100 ml sample as prepared into nessler tube. Similarly, pour the standard platinum-cobalt colour matching solution, which appears to have similar intensity of colour, into the other Nessler tube up to the mark. Place the two tubes in a comparator and compare the colour. Repeat, if necessary, with other standard platinum-cobalt colour matching solutions until the closest match is obtained.

NOTE — Colorimeter may also be used.

A-5 REPORT

Express the colour of the sample as the number of platinum-cobalt colour units corresponding to the standard platinum-cobalt colour matching solution having the closest match to the sample.

ANNEX B

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

DETERMINATION OF ALDEHYDES (AS ACETALDEHYDE) CONTENT

B-1 OUTLINE OF THE METHOD

This test method is used to determine aldehydes, based on a colour formation in the presence of ferric chloride and excess reagent. The sample is treated 3-Methyl-2-Benzothiazolone hydrazone hydrochloride (MBTH reagent), which is, which reacts quantitatively with free aldehydes to form the azine derivative and water. The excess MBTH is oxidized with acidic ferric chloride to a reactive cation, which then forms a blue-green complex with the azine, whose colour intensity corresponds to the aldehyde content and is measured Photometrically at 655 nm.

B-2 APPARATUS

B-2.1 UV/Vis Spectrophotometer, with absorption cell 10 mm path length

B-2.2 Graduated Cylinder, 5 ml

B-2.3 Volumetric Flasks, 50 ml, 100 ml and 1 litre

B-2.4 Volumetric Pipettes, 0.5 ml, 1 ml, 2 ml, 3 ml, 4 ml, 5 ml, and 10 ml

B-3 REAGENTS

B-3.1 3-Methyl-2-Benzothiazolone Hydrazone Hydrochloride (MBTH) Solution, 0.2 percent

Weigh 0.2 g of MBTH to the nearest 0.1 mg and dissolve in distilled water in a 100 ml volumetric flask. Dilute it upto the mark and mix well. Filter this solution, if necessary. Store the solution in a refrigerator and prepare it fresh every week. The colourless solution becomes pale yellow in this length of time.

B-3.2 Ferric Chloride Solution

Weigh 2.0 g of ferric chloride hexahydrate and dissolve it in about 90 ml distilled water in a beaker. Adjust the *p*H of the solution to between 1.6 and 1.8 by dropwise addition of concentrated hydrochloric acid. This *p*H range is critical for proper colour development. Transfer the solution in to 100 ml volumetric flask and dilute to the mark with distilled water.

B-3.3 Standard Formaldehyde Solution, 1 ml = 8 µg

B-3.3.1 Weigh approximately 2.0 g to 2.2 g (m_1) of the formaldehyde solution with known concentration (C_1) to the nearest 0.1 mg, into a 100 ml volumetric flask. Make it up to the mark with distilled water and mix thoroughly. Pipette 1.0 ml of this solution into a 1 l volumetric flask. Make it up to the mark with distilled water and mix thoroughly.

B-3.3.2 Calculate the exact formaldehyde content of this solution (C_2), in µg/ml:

$$C_2,\ \mu\text{g/ml} = \frac{m_1 \times C_1}{10}$$

where

C_1 = concentration of concentrated formaldehyde solution used, in g/100 ml; and
m_1 = mass of concentrated formaldehyde solution taken, in g.

B-4 CALIBRATION

B-4.1 Take six 50 ml volumetric flask and prepare solutions by adding reagents as mentioned below:

Calibration Solution No.	*MBTH Solution, ml*	*Standard HCHO Solution, ml*
Blank 1	10.0	0.0
1	10.0	0.5
2	10.0	1.0
3	10.0	2.0

4	10.0	3.0
5	10.0	4.0

B-4.2 After adding the reagents, allow the flasks to stand in the dark for 30 min. Remove the flasks and immediately add 5 ml ferric chloride solution to each, and dilute each to 50 ml with distilled water. Mix these solutions and allow them to stand for 30 min. Measure the absorption directly against the “blank” at 655 nm, by setting zero absorbance with “blank” solution. Measure and plot the concentration (µg) versus absorption reading to obtain a calibration curve.

B-5 PROCEDURE

Take 10 ml chilled MBTH solution into each of two 50 ml volumetric flasks. Label one of the flask as blank and in another flask add adequate 0.5 g to 2 g of ethylene oxide sample. Mix the solutions by swirling and loosely stopper these flasks. Allow the flasks to stand in the dark for 30 min in chilled condition. Now remove the flasks and immediately add 5 ml ferric chloride solution to each flask and dilute each to 50 ml with distilled water. Mix these solutions and allow them to stand in the dark for 30 min. Measure the absorption directly against the “blank” at 655 nm, by setting zero absorbance with “blank” solution. Obtain the micrograms of formaldehyde from the net absorbance from the calibration curve as plotted.

B-6 CALCULATION

$$\text{Acetaldehyde (}\textit{as}\text{ } CH_3CHO\text{), mg/kg} = \frac{\text{Amount of formaldehyde (µg)}}{Weight\ of\ Sample\ (g)} \times \frac{44.05}{30.03}$$

B-7 REPORT

Report the aldehyde (*as* acetaldehyde) content in percent by mass.

ANNEX C

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

DETERMINATION OF MOISTURE CONTENT

C-1 GENERAL

Two methods namely, Method A determination of water by IS 2362 and Method B determination of water by volumetric Karl Fischer titration have been prescribed. In case of dispute, Method A shall be the referee method.

C-2 METHOD A

C-2.1 Take 20 ml of the material and determine its moisture content by the method given in IS 2362.

C-3 METHOD B

C-3.1 Outline of the Method

This method is used to determine water content by volumetric Karl Fischer titration. The method use automatic titration system capable of determining the Karl Fischer titration end point amperometrically. The sample is dissolved in a suitable solvent and titrated with Karl Fischer reagent, which consists of iodine, sulfur dioxide, organic base, and a solvent (typically an alcohol, such as methanol, ethylene glycol, or 2-methoxyethanol). The titration end point is determined amperometrically with a platinum electrode which senses a sharp change in cell resistance when the iodine is reduced by sulfur dioxide in the presence of water.

C-3.2 Apparatus

C-3.2.1 *Karl Fischer Volumetric Titrator*

C-3.3 Reagents

C-3.3.1 *Karl Fischer Reagents*, both pyridine and pyridine-free Karl Fischer reagents are available. However, use of pyridine-free reagents is recommended as it is less toxic, less odour and more stable than those containing pyridine. Ready-made standardized Karl Fischer reagents may also be used.

C-3.3.2 *Solvents*, any solvent given below may be used.

C-3.3.2.1 *Acetic acid*, glacial

C-3.3.2.2 *Ethylene glycol mono-methyl ether*, 0.1 percent moisture, maximum

C-3.3.2.3 *Methanol*, 0.15 percent moisture, maximum

C-3.3.2.4 *Pyridine*, 0.1 percent moisture, maximum

C-3.3.3 *Mixed Solvents*, in addition to single solvents the following mixed solvents may also be used.

C-3.3.3.1 *Methanol-chloroform* (1 : 3)

Mix 1 volume of methanol with 3 volumes of chloroform.

C-3.3.3.2 *Methanol-salicylic acid*

Dissolve 150 g of salicylic acid in 1 l of methanol.

C-3.3.3.3 *Pyridine-ethylene glycol* (1: 4)

Mix 1 volume of pyridine with 4 volumes of ethylene glycol.

C-3.3.3.4 *Pyridine-methanol* (1: 4)

Mix 1 volume of pyridine with 4 volumes of methanol.

C-3.4 Procedure

C-3.4.1 Pipette 25 ml to 50 ml of the selected solvent into the titration cell. Follow the instrument manufacturer's instructions and titrate the water in the solvent with Karl Fischer reagent. Karl Fischer reagent that is used should be of appropriate titer as determined by the amount of water expected in the sample.

C-3.4.2 Pipette appropriate amount of sample as suggested in Karl Fischer instrument operation manual, containing an anticipated moisture content that will give a fast and accurate titration and again titrate the water in the solvent with Karl Fischer reagent.

C-3.5 Calculation

C-3.5.1 Calculate the water content of the sample as follows:

$$\text{Moisture content, percent by mass} = \frac{(A-B) \times F \times 0.001 \times 100}{W}$$

where

A = reagent required for titration of the sample, in ml;
B = reagent required to titrate solvent blank, in ml;
F = water equivalent of water per ml of Karl Fisher reagent, in mg and;
W = sample taken, in g.

ANNEX D

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

DETERMINATION OF ACIDITY (AS CH_3COOH)

D-1 GENERAL

Two methods namely, Method A determination of acidity by titrating sample with barium hydroxide solution and Method B determination of acidity by titrating sample with sodium hydroxide have been prescribed. In case of dispute, Method B shall be the referee method.

D-2 METHOD A

D-2.1 Outline of the Method

The material is treated with barium hydroxide solution under specified conditions and the excess of barium hydroxide solution titrated with hydrochloric acid. Acidity is then determined from the barium hydroxide solution used up.

D-2.2 Reagents

D-2.2.1 *Barium Hydroxide Solution*

Take 3.2 g of barium hydroxide in a 1 000 ml volumetric flask and dissolve it in distilled water (previously boiled and cooled to expel carbon dioxide). Now dilute it to 1 000 ml with distilled water. Use absorbent tube to prevent carbon dioxide from the atmosphere to come in contact with barium hydroxide solution. When the precipitate of barium carbonate is produced, it shall be filtered off.

D-2.2.2 *Standard Hydrochloric Acid*, 0.02 N

D-2.2.3 *Phenolphthalein Indicator Solution*

Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent (*v/v*) rectified spirit.

D-2.3 Procedure

D-2.3.1 Pipette 25 ml of barium hydroxide solution into a 500 ml conical flask (*see* Fig. 3) in nitrogen atmosphere and stopper at once. Introduce nitrogen gas through the rubber tube A (*see* Fig. 3) to substitute air in the flask by nitrogen and then clamp the free ends of tubes A and B with pinch-cocks.

D-2.3.2 Weigh the flask containing barium hydroxide solution with the stopper and tubes, cool to 0 °C to 5 °C in an ice-water bath and connect the rubber tube A or B to the small bomb containing the samples of the material which has been cooled to 0 °C to 5 °C. Release both pinch-cocks at A and B to introduce slowly about 100 g of the material into the flask, clamp again the pinch-cocks and weigh the flask immediately to obtain the mass of the sample introduced. Then the material is taken from the sample bottle or measuring cylinder, weigh the flask on a rough balance, cool to 0 °C to 5 °C in the ice-water bath, open the rubber stopper just a little to put about 100 g of the sample cooled to 0 °C to 5 °C into the flask, stopper and weigh the flask immediately to obtain the mass of the sample introduced. Release the pinch-cocks in a draught chamber, swirl the flask and allow to stand at the room temperature for 10 min. Then heat the flask gently in a water bath until the contents of the flask cease to boil, rinse the whole surface of glass tubes with 100 ml of distilled water previously freed from carbon dioxide and titrate the solution with standard hydrochloric acid in nitrogen atmosphere using phenolphthalein indicator solution to the end point when the pink colour disappears. Carry out a blank test as well.

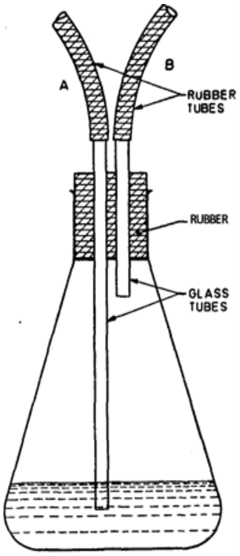

Fig. 3 ASSEMBLY OF APPARATUS

D-2.4 Calculation

$$\text{Acidity (as } CH_3COOH\text{), percent by mass} = \frac{100 \times 0.0605 \times (V_1 - V_2) \times N}{M}$$

where

V_1 = volume of standard hydrochloric acid used in blank test, in ml;
V_2 = volume of standard hydrochloric acid used in test with the material, in ml;
N = normality of standard hydrochloric acid; and
M = mass of the material, in g.

D-3 METHOD B

D-3.1 Outline of the Method

This method is used to determine the acidity as acetic acid by titrating with sodium hydroxide, after removing carbon dioxide by partial evaporation on the sample.

D-3.2 Apparatus

D-3.2.1 *Conical Flasks*, 250 ml

D-3.2.2 *Graduated Cylinder*, 50 ml and 100 ml

D-3.2.3 *Burette*, 10 ml (with 0.05 ml graduations) and 50 ml

D-3.2.4 *Balance*, capable of weighing nearest to 0.1 mg

D-3.2.5 *Oven*

D-3.2.6 *Desiccator*, with drying agent

D-3.3 Reagents

D-3.3.1 *Standard Sodium Hydroxide Solution*, 0.02 N

D-3.3.2 *Phenolphthalein Indicator*

Dissolve 0.5 g phenolphthalein in 100 ml alcohol.

D-3.3.3 *Potassium Hydrogen Phthalate*, AR grade, dried at 105 °C for 2 h and then cool in a desiccator.

D-3.4 Procedure

Take (70 ± 5) ml of ethylene oxide sample in 100 ml cylinder, keep aside in fuming chamber and allow evaporating to about 50 ml. Now transfer it to a 250 ml Erlenmeyer flask, add few drops of solution of phenolphthalein indicator and titrate with 0.02 N sodium hydroxide until a faint pink colour persists for at least 15 s.

D-3.5 Calculation

$$\text{Acidity (as } CH_3COOH\text{), mg/kg} = \frac{10^6 \times 0.060\,5 \times V_1 \times N}{V \times d}$$

where

V_1 = volume, in ml, of standard sodium hydroxide used in test with the material,;
V = volume, in ml, of sample;
N = normality of standard sodium hydroxide; and
d = density, in g, of the material, (typically 0.897 g/ml).

ANNEX E

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

DETERMINATION OF IONISABLE CHLORIDES

E-1 GENERAL

Three methods namely, Method A determination of ionizable chloride by titrating with standard mercuric nitrate solution, Method B determination of ionizable chloride by titrating with silver nitrate and Method C determination of ionizable chloride by spectrophotometer have been prescribed. In case of dispute, Method C shall be the referee method.

E-2 METHOD A

E-2.1 Outline of the Method

A measured amount of cooled material is introduced in a beaker containing chloride-free demineralized water. The chloride content of the sample is estimated by titrating the contents of the beaker with standard mercuric nitrate solution using diphenyl carbazone in presence of bromophenol blue indicator.

E-2.2 Apparatus

E-2.2.1 *Micro-burette,* 5 ml, with 0.0l ml graduation intervals

E-2.2.2 *Graduated Cylinder,* 100 ml

E-2.2.3 *Beakers,* 250 ml and 500 ml

E-2.2.4 *Conical Flask,* 250 ml

E-2.2.5 *Volumetric Flasks,* 250 ml and 1 litre

E-2.3 Reagents

E-2.3.1 *Mercuric Nitrate Solution* [$Hg(NO_3)_2.H_2O$], 0.014 1 N

Dissolve 2.42 g of mercuric nitrate in 25 ml of water acidified with 0.25 ml of concentrated nitric acid (HNO_3, relative density 1.42). Dilute the acidified $Hg(NO_3)_2$ solution with demineralized water to 1 l. Filter, if necessary, and standardize against the standard sodium chloride solution.

E-2.3.2 *Mixed Indicator Solution*

Dissolve 0.5 g of crystalline diphenyl-carbazone and 0.05 g of bromophenol blue powder in 75 ml of ethyl alcohol (95 percent) and dilute to 100 ml with the alcohol. Store in a brown bottle and discard after five months. Methanol or isopropanol may be used if pure ethyl alcohol is not available.

E-2.3.3 *Nitric Acid* (HNO_3)

Mix 3 volumes of concentrated nitric acid (HNO_3, relative density 1. 42) with 997 volumes of demineralized water.

E-2.3.4 *Sodium Hydroxide Solution,* 10 g/l aqueous

E-2.3.5 *Sodium Chloride Solution,* 0.025 N

Dry about 2 g of sodium chloride (analytical reagent) for 1 h at 600 °C. Dissolve (1.461 3 ± 0.000 2) g of the dry salt in water and dilute to l litre in a volumetric flask.

E-2.4 Procedure

Take a beaker of 500 ml capacity and add to it 250 ml chloride-free water. With a cooled graduated cylinder (**E-2.2.2**) add 100 ml of ethylene oxide to the above beaker containing water. Allow ethylene oxide in the beaker to evaporate at room temperature in a fume cupboard. When the ethylene oxide is completely evaporated, add 5 to 10 drops of mixed indicator solution (**E-2.3.2**) and shake or swirl the flask. If a blue violet or red colour develops, add nitric acid (HNO_3) (**E-2.3.3**) dropwise until the colour changes to yellow. Add 1 ml of excess acid. If a yellow or orange colour forms immediately on addition of the mixed indicator, add sodium hydroxide (NaOH) solution (**E-2.3.4**) dropwise until colour changes to blue violet, then add nitric acid (HNO_3) (**E-2.3.3**) dropwise until the colour changes to yellow and further add 1 ml excess of acid. Titrate the solution with 0.014 1 N $Hg(NO_3)_2$ solution (**E-2.3.1**) until a blue violet colour persists. Record the millilitres of $Hg(NO_3)_2$ solution added. Do the blank titration without the sample.

E-2.5 Calculation

$$\text{Chloride, mg/l} = \frac{(V_1 - V_2) \text{ x } N \text{ x } 35\,500}{V}$$

where

V_1 = volume, in ml, of standard $Hg(NO_3)_2$ solution required for titration of the sample;
V_2 = volume, in ml, of standard $Hg(NO_3)_2$ solution required for titration of the blank;
V = volume, in ml, of ethylene oxide taken; and
N = normality of $Hg(NO_3)_2$ solution.

E-3 METHOD B

E-3.1 Outline of the Method

This method is used to determine chloride in ethylene oxide. Any chloride present are made to react with a measured amount of silver nitrate and the excess is determined with thiocyanate solution. The silver nitrate consumed is a measure of the chloride content.

E-3.2 Apparatus

E-3.2.1 *Erlenmeyer Flask*, 250 ml

E-3.2.2 *Graduated Cylinder*, 50 ml

E-3.2.3 *Volumetric Flask*, 100 ml

E-3.2.4 *Graduated Pipette*, 5 ml

E-3.2.5 *Burette*, 10 ml (with 0.05 ml graduations)

E-3.2.6 *Balance*, capable of weighing nearest to 0.1 mg

E-3.3 Reagents

E-3.3.1 *Concentrated Nitric Acid*, 70 percent; specific gravity 1.42

E-3.3.2 *Nitrobenzene*

E-3.3.3 *Ferric Alum Indicator* [$FeNH_4(SO_4)_2.12H_2O$]

Dissolve 25 ml of ferric alum in 50 ml of distilled water. Add 1 ml of concentrated nitric acid and dilute to 100 ml.

E-3.3.4 *Standardized Ammonium Thiocyanate* (NH_4SCN), 0.1 N

Dissolve 7.7 g of ammonium thiocyanate (NH_4SCN) in 100 ml of distilled water and dilute to 1 l. Standardize this solution against 0.1 N silver nitrate solution.

E-3.3.5 *Standardized Silver Nitrate*, 0.1 N

Dissolve 17 g of pure silver nitrate in 100 ml of distilled water and dilute to 1 l. Standardize this solution.

E-3.4 Procedure

E-3.4.1 Take 10 ml of the chilled ethylene oxide sample into 50 ml of water in an Erlenmeyer flask using chilled 10 ml graduated cylinder. Now add 3 ml of nitric acid and 5 ml of 0.1 N silver nitrate to the flask. If a precipitate is formed, add 3 ml of nitrobenzene and 3 ml of ferric alum indicator, shake the flask vigorously and titrate the excess silver nitrate with 0.1 N ammonium thiocyanate.

E-3.4.2 The end point is indicated by change from white or pale yellow to a permanent tinge of reddish brown. The further addition of a few drops of thiocyanate beyond the end point should give a darker solution.

E-3.5 Calculation

$$\text{Ionisable chlorides, weight percent (mg/kg)} = \frac{(S - T) \times 0.035\,5 \times 10^{6}}{\text{Volume of sample (in ml)} \times \text{density of material}}$$

where,

S = (volume, in ml, of silver nitrate) x (normality of silver nitrate)
T = (volume, in ml, of ammonium thiocyanate) x (normality of ammonium thiocyanate)

E-3.6 Report

Report chloride up to two decimal places as mg/l.

E-4 METHOD C

This method is used to determine chloride ions in ethylene oxide. Trace chlorides are precipitated with silver nitrate solution in an acidic aqueous solution of the sample and the turbidity is determined on a spectrophotometer as a direct measure of the chloride concentration.

E-4.1 Apparatus

E-4.1.1 *Spectrophotometer*

E-4.1.2 *Volumetric Flask*, 250 ml

E-4.1.3 *Measuring Cylinder*, 25 ml

E-4.1.4 *Cooling System or Ice Bath*, to maintain temperature of about 5 °C

E-4.2 Reagents

E-4.2.1 *Nitric Acid*, 7.5 N

E-4.2.2 *Nitrobenzene*

E-4.2.3 *Silver Nitrate Crystals*

E-4.2.4 *Standard Sodium Chloride Solution*

Take 0.500 g sodium chloride dried crystal in 1 l volumetric flask and dissolve it with distilled water. Now make it up the mark with distilled water. 1 ml of this solution contains 0.303 4 mg chloride.

E-4.3 Calibration

Take 0.5 ml, 1.0 ml, 2.0 ml, 5.0 ml, and 10.0 ml of standard sodium chloride solution in 250 ml volumetric flask and proceed as mentioned in procedure. Measure and plot the calibration graph against concentration (mg/l) and spectrophotometer reading at 410 nm.

E-4.4 Procedure

Carefully, take 25 ml of ethylene oxide sample in 250 ml flask and to it add 100 ml cooled distilled water. To another 250 ml flask, labeled as blank, take 100 ml distilled water. To each of these volumetric flasks, add 2 ml of 7.5 N nitric acid and about 0.20 g silver nitrate crystals. Gently mix the solution and dilute each solution to mark with cooled distilled water and mix again. Allow the solutions to stand exactly for 30 min. Mix the solution and read absorbance at 410 nm. Obtain the equivalent milligrams of chloride from the plotted calibration curve.

E-4.5 Calculation

$$\text{Concentration of chloride (ppm)} = \frac{mg\ in\ chloride \times 1000}{\text{Volume of sample (in ml) x density of material}}$$

E-4.6 Report

Report chloride up to two decimal places as mg/l.

ANNEX F

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

DETERMINATION OF NON-VOLATILE MATTER

F-1 GENERAL

Two methods namely, Method A determination of non-volatile matter by evaporating sample to dryness under vacuum and Method B determination of non-volatile matter by evaporating and then drying the residue at 110 °C have been prescribed. In case of dispute Method B shall be the referee method.

F-2 METHOD A

F-2.1 Outline of the Method

Ethylene oxide is evaporated to dryness under vacuum in a vacuum desiccator.

F-2.2 Apparatus

F-2.2.1 *Evaporator,* comprising vacuum desiccator which can be heated; infrared lamp with control transformer; mercury manometer; safety bottle; and water jet air pump.

F-2.2.2 *Oven,* attaining temperature of (105 ± 5) °C

F-2.2.3 *Gum Test Dish,* 150 ml capacity

F-2.2.4 *Measuring Cylinder,* 10 ml capacity

F-2.2.5 *Desiccator,* without drying agent

F-2.2.6 *Protective Metal Cage,* for the vacuum desiccator

F-2.2.7 *Refrigerator*

F-2.3 Procedure

Remove clean gum test dish from oven at (105 ± 5) °C and place in the desiccator without drying agent for 30 min and weigh the dish nearest to 0.l mg. Use heating periods of 15 min and cooling periods of 30 min. Continue heating and cooling till the difference between two consecutive weighing is 0.5 mg or less. Take 100 ml of the sample by means of the measuring cylinder in the weighed dish (*see* Note below the calculation). Place the dish in vacuum desiccator; place lid on the desiccator and position a protective metal cage over this equipment. Put water jet air pump into operation. First adjust infrared lamp to 180 V and after 5 min to 200 V (distance to desiccator approximately 20 cm). Evaporate the sample to dryness.

Switch off infrared lamp, open air inlet, switch off water jet air pump and remove dish from desiccator and place in the oven at (105 ± 5) °C for approximately 1 h. Remove dish from oven and place it in desiccator without drying agent (**F-2.2.5**) for 30 min. Weigh dish with residue nearest to 0.1 mg. Redry for 15 min and weigh and continue the drying and weighing of the dish till the loss of mass between two consecutive weighing is 0.5 mg or less.

F-2.4 Calculation

$$\text{Non-volatile matter, percent by mass} = \frac{(M_2 - M_1) \times 100}{V - 0.89}$$

where

M_1 = mass of empty dish, in g;
M_2 = mass of dish and residue, in g; and
V = volume of sample taken for test, in ml.

NOTES

1 The gum test dish to be handled only with crucible tongs and washed with acetone.
2 Leave sample, measuring cylinder and gum test dish after weighing, in refrigerator for at least 30 min before carrying out the determination.

F-3 METHOD B

F-3.1 Outline of the Method

This method describes the procedure for the determination of non-volatile matter in ethylene oxide (EO). The presence of any contaminants boiling over 110 °C is determined by evaporating and then drying the residue at that temperature.

F-3.2 Apparatus

F-3.2.1 *Industrial Hot Air Oven*, capable of maintaining (110 ± 5) °C

F-3.2.2 *Pyrex Evaporating Dish*, 150 ml capacity

F-3.2.3 *Graduated Measuring Cylinder*, 100 ml

F-3.2.4 *Vacuum Desiccators*

F-3.2.5 *Analytical Balance*, capable of weighing up to 0.1 mg

F-3.3 Procedure

Dry a 125 ml evaporating dish in an oven at 110 °C for an hour, cool in a desiccator and weigh to the nearest 0.1 mg. Record the weight of the evaporating dish (W_1). Take 100 ml ethylene oxide sample using a graduated cylinder to the conditioned evaporating dish and allow the sample to evaporate at room temperature, water bath or hot plate in the fume hood. After the liquid has completely evaporated, put the evaporating dish in oven at 110°C for an hour. Cool the dish in desiccator and reweight (W_2) to the nearest 0.1 mg.

F-3.4 Calculation

$$\text{Non-volatile matter, percent by weight} = \frac{(W_2 - W_1) \text{ x } 100}{V \text{ x } 0.897}$$

where

V = volume of sample taken, in ml;
W_1 = weight of evaporating dish, in g and;
W_2 = weight of evaporating dish and residue, in g.

ANNEX G

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

DETERMINATION OF ETHTLENE OXIDE CONTENT (PURITY)

G-1 The ethylene oxide content shall be determined by difference as given below:

Ethylene oxide, percent by mass = 100 — (moisture content + aldehyde + acidity + non-volatile matter)

ANNEX H

(*Clause* 6.1)

SAMPLING OF ETHYLENE OXIDE

H-1 SAMPLING FROM CYLINDERS OF CAPACITY UP TO 500 KILOGRAMS

H-1.1 In a single consignment of the material, all the cylinders of the same size and drawn from the same batch of manufacture shall constitute a lot. If a consignment of the material is known to consist of different batches of manufacture or of different sizes of cylinders, the cylinders belonging to the same batch and size shall be grouped together and each such group shall constitute separate lots.

H-1.2 The number of cylinders to be selected from each lot shall depend on the lot size and shall be obtained as follows:

Sl No. (1)	*Lot Size* *N* (2)	*Sample Size* *n* (3)
i)	Up to 50	2
ii)	51 to 150	3
iii)	151 to 300	4
iv)	301 to 500	5
v)	501 and above	6

H-1.3 These cylinders shall be selected at random and in order to ensure randomness of selection a random number table as agreed to between the purchaser and the supplier shall be used. In case such a table is not available the following procedure shall be adopted:

Starting from any cylinder in the lot, count them as 1, 2, 3,... up to r and so on in one order where r is the integral part of N/n (see **H-1.2**). Every rth cylinder thus counted shall be withdrawn to constitute the sample.

H-2 SAMPLING FROM CYLINDERS OF CAPACITY OVER 500 KILOGRAMS, ROAD TANKERS AND RAIL TANK-CARS

H-2.1 Each of such containers shall be treated as a separate lot.

H-2.2 Methods of Sampling

H-2.2.1 The sample shall be taken from the container into a small bomb, sample bottle or measuring cylinder as specified below by placing the container in such a position as to discharge liquid content.

H-2.2.2 Sample for moisture content and acidity tests shall be taken in a small bomb. When the sample is intended for immediate use, a sample bottle or measuring cylinder may, however, be used. Samples for all other tests, such as appearance, acetaldehyde content, ionisable chlorides and non-volatile matter can be taken either in a small bomb, sample bottle or a measuring cylinder.

H-2.3 Sampling in the small bomb, sample bottle or measuring cylinder shall be carried out in accordance with the procedures given in **H-2.3.1.**

H-2.3.1 *Sampling in Small Bomb*

The small bomb shall be of steel with a capacity of about 1.5 l and weighing about 5 kg, and shall be furnished with a stainless-steel flexible connecting tube for introducing nitrogen gas, a flexible connecting tube for introducing the material, a mouthpiece for reducing pressure and a mouthpiece for taking out the material. The procedure for taking out the sample from the container of material shall be as follows:

a) Attach the mouthpiece for reducing pressure to the bomb to reduce the pressure in it through a vacuum pump and then introduce nitrogen gas to the pressure of nearly 6 kg/cm^2 through the flexible connecting tube for introducing nitrogen gas which has previously been filled with nitrogen gas to displace air in it.
b) Then expel nitrogen gas in the bomb, introduce again nitrogen gas to the pressure of nearly 6 kg/cm^2 as before and expel nitrogen gas to the extent at which the inner pressure in the bomb remains somewhat higher than the atmospheric pressure.
c) Attach to the bomb the flexible connecting tube for introducing ethylene oxide and the mouthpiece for taking out ethylene oxide to connect the connecting tube with the container of material. In the operation, the connecting tube and the bomb shall be loosely connected at first and tightened firmly after small amount of the material has been expelled into the air by opening the valve of the container for a while.
d) Now open the valve of the bomb, then open the valve of the container to introduce about 500 g of the material into the bomb and close the valves of the container and the bomb in the order described.
e) Detach from the bomb the flexible connecting tube for introducing the material, shake the bomb well to wash the inside thereof with the material and allow the liquid portion to flow out.
f) Again connect the bomb to the container in the same manner as before and introduce the material into bomb until the inner pressures in the container and the bomb come to equilibrium.
g) When the sample is taken by a bomb which has been used for sampling a different lot, drain out the liquid material remaining in the bomb to the last drop, then introduce nitrogen gas into the bomb and repeat the procedure following the washing of the bomb with the material in the same manner as before.

H-2.3.2 *Sampling in Sample Bottle*

The sample bottle shall previously be dried and stoppered, and appropriate amount of the material necessary for the tests shall be introduced into the bottle from the outlet of the container through an ice-water jacket, the bottle itself being ice-cooled. When not in use, the sample shall be stored in the bottle stoppered tightly in a dry cool place.

H-2.3.3 *Sampling in Measuring Cylinder*

The measuring cylinder shall previously be dried and into it the material shall be introduced as in **H-2.3.2** to serve for immediate test.

H-2.4 Test Samples

H-2.4.1 For carrying out all the tests, two different sets of the samples shall be taken from each of the cylinders selected as in **H-1.2** and four different sets of samples shall be taken from each of the containers mentioned in **H-2**. The sets of samples so obtained shall be termed individual test samples.

H-2.5 CRITERIA FOR CONFORMITY

H-2.5.1 All the individual test samples shall be tested separately for all the requirements of this specification.

H-2.5.2 A lot shall be declared as conforming to the requirements of this specification if all the test results on all the individua1 test samples satisfy the corresponding requirements of this specification.