

**BUREAU OF INDIAN STANDARDS**

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<p>भारतीय मानक मसौदा ( मिथायल क्लोराइड — विशिष्टि ) ( दूसरा पुनरीक्षण )  <i>Draft Indian Standard</i> Methyl chloride – Specification ( <i>Second Revision</i> )  (ICS 71.100.20)</p>	
Industrial Gases Sectional Committee, CHD 06	Last Date for Comments: 30 November 2023

FOREWORD

*(Formal clause shall be added later)*

Methyl chloride finds use as a refrigerant and as a methylating agent in organic synthesis. It is used as a propellant in high pressure aerosols. Other important applications of methyl chloride include production of weedicides, silicones, butyl rubber, gasoline antiknock compounds, etc.

Methyl chloride is moderately flammable and hence normal precautions should be observed during its handling. The vapour is harmful if inhaled or observed by the skin. The containers should be opened slowly and used with adequate ventilation.

This standard was originally issued in 1974. First revision of this standard was brought out in 1993 and methods of test for determination of non-volatile matter, moisture and free chlorine had been modified. In this revision the committee felt a need to revise the standard with a view to update the standard based on the experience of last three decades and on the currently available data.

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 requirements and methods of sampling and test for methyl chloride.

## 2 REFERENCES

The Indian standards listed 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 Indian standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No</i>	<i>Title</i>
IS 5 : 2007	Colours for ready mixed paints and enamels ( <i>sixth revision</i> )
IS 1070 : 2023	Reagent Grade Water Specification ( <i>fourth revision</i> )
IS 1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods Part 1 Dangerous goods ( <i>first revision</i> ).
IS 2362 : 1993	Determination of water by karl fischer method - Test method ( <i>second revision</i> )
IS 3224 : 2021	Valve for compressed gas cylinders excluding liquefied petroleum gas (LPG) cylinders - Specification ( <i>fourth revision</i> )
4825 : 1982	Liquid-in-glass solid-stem reference thermometers ( <i>first revision</i> )
7062 : 1973	Glossary of terms used in gas industry

## 3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 7062 shall apply.

## 4 REQUIREMENTS

### 4.1 Description

The material shall consist essentially of methyl chloride (CH<sub>3</sub>Cl), also known as monochloro methane. It is a colourless liquifiable gas or liquid, under pressure, with a faintly sweet odour.

**4.2** This material shall comply with the requirements specified in Table 1 when tested in accordance with the methods of test prescribed in **Annex A**.

**Table 1 Requirements for Methyl Chloride**

(*Clause 4.2*)

SI No.	Characteristic	Requirement	Methods of Test, Ref to CI No. in Annex A
(1)	(2)	(3)	(4)
i)	Non-volatile matter, parts per million, Max	100	A-2
ii)	Acidity (as HCl), parts per million, Max	15	A-3
iii)	Moisture, parts per million, Max	100	A-4
iv)	Boiling range (760 mm Hg):		
	a) Initial boiling point, °C, Min	– 24.6	
	b) Boiling point at 95 percent distillation stage, °C, Ma	– 23.6	A-5

## 5 PACKING AND MARKING

### 5.1 Packing

5.1.1 The material shall be packed in suitable steel cylinders or large cylindrical steel containers.

5.1.2 The valve outlet for cylinders or containers shall conform to outlet No. 6 of IS 3224.

### 5.2 Marking

The cylinder or container shall be legibly and indelibly marked with the following information:

- a) Name of the material;
- b) Gross and net mass of contents;
- c) Indication of the source of manufacturer;
- d) Lot number to enable the batch of manufacture to be traced from records; and
- e) The minimum caution words as under:

WARNING — Flammable Liquid and Gas under Pressure-Vapour. Harmful. Keep away from heat, spark and open flame. Use only with adequate ventilation. Avoid breathing vapour. Avoid contact with skin.

5.2.1 The cylinders shall be painted with a coat of light brunswick green paint (IS Colour No 225) over the body and signal red paint (IS Colour No. 537) at the neck (*see* IS 5).

5.2.2 The cylinders and containers shall also be marked with an appropriate symbol in accordance with IS 1260 (Part 1)

#### 5.2.3 BIS Certification Marking

The containers may also be marked with the BIS Certification Mark.

5.2.3.1 The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.

## 6 SAMPLING

Representative samples of the material shall be drawn and adjudged as prescribed in **Annex B**.

## ANNEX A

(Clause 4.2)

### METHODS OF TEST FOR METHYL CHLORIDE

#### A-1 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.

#### A-2 DETERMINATION OF NON-VOLATILE MATTER

##### A-2.1 General

A known quantity of the material is evaporated and the residue is weighed.

##### A-2.2 Apparatus

###### A-2.2.1 Conical Flask

250 ml capacity with a side tube and provided with ground glass stopper.

#### **A-2.2.2 Gas Washing Bottles**

250 ml capacity, tall form with fitted tube and ground glass stopper.

#### **A-2.2.3 Graduated Cylinder**

100 ml capacity.

### **A-2.3 Reagents**

#### **A-2.3.1 Bromothymol Blue Indicator**

Dissolve 0.1 g of bromothymol blue in 100 ml of 50 percent rectified spirit

### **A-2.4 Procedure**

Measure 100 ml of neutralized water into each of two 250 ml gas washing bottles and add 6 drops of bromothymol blue indicator. Connect the two gas washing bottles in series. Weigh accurately a stoppered 250 ml conical flask, with two boiling chips, which has been previously dried in an air oven maintained at  $(105 \pm 2)^\circ\text{C}$  for 1 h and subsequently cooled to room temperature by keeping it, in a desiccator for 1 h. Chill the flask in dry-ice bath. Remove the flask from the bath and connect the side tube of the flask to the gas washing bottles. With the help of a 100 ml graduated cylinder, measure 100-ml of the liquefied sample into the flask, and close the stopper. Evaporate the sample to dryness. After evaporation is complete, rinse out the conical flask with dry air, dry it in oven and cool in a desiccator, as done before. Reweigh the flask and its content. The increase in mass is calculated as the non-volatile matter on evaporation.

### **A-2.5 Calculation**

$$\text{Non - volatile matter, parts per million} = \frac{M_1}{M} \times 100$$

Where

$M_1$  = mass, in g, of residue obtained; and

$M$  = mass, in g, of the sample taken for the test calculated as (volume  $\times$  relative density)

NOTE — Relative density of liquefied methyl chloride is 1.

## **A-3 DETERMINATION OF ACIDITY**

### **A-3.1 General**

Acidity is determined by titrating the water in which the acid is absorbed by evaporating a known mass of the material.

**A-3.2 Apparatus** — Same as in **A-2.2**.

### **A-3.3 Reagents**

**A-3.3.1 Standard Sodium Hydroxide Solution** — 0.1 N

**A-3.3.2 Bromothymol Blue Indicator** Same as in **A-2.3.1**.

### **A-3.4 Procedure**

Pour the water from the gas washing bottles into the conical flask used in **A-2.4**. Add bromothymol blue indicator solution and titrate with standard sodium hydroxide solution. Carry out a blank test for the quantity of water used and deduct from the titer value.

### **A-3.5 Calculation**

$$\text{Acidity ( as HC1 ), parts per million} = \frac{V \times N \times 36.5 \times 10^3}{M}$$

Where

$V$  = volume in ml of standard sodium hydroxide solution, after deducting blank;

$N$  = normality of standard sodium hydroxide solution; and

$M$  = mass in g of the sample taken for the test

## **A-4 DETERMINATION OF MOISTURE**

### **A-4.1 General**

Moisture is determined by titrating a known volume of liquified sample against standard Karl-Fischer reagent.

**A-4.2** Take 100 ml (100 g) of the liquified methyl chloride sample slowly in 50 ml of methanol (whose water content has been previously determined). Determine the water content in the sample by the Karl-Fischer method as prescribed in IS 2362

## **A-5 DETERMINATION OF BOILING RANGE**

### **A-5.1 Apparatus**

#### **A-5.1.1 Thermometer**

Range — 40°C to + 10°C, 80 mm immersion (*see* IS 4825).

**A-5.1.2 Graduated Cylinder** — 100 ml.

#### **A-5.1.3 Boiling Chips**

**A-5.1.4 Barometer**

#### **A-5.1.5 Dry Ice-Acetone Bath**

### **A-5.2 Procedure**

Take 100 ml of liquified methyl chloride in a graduated cylinder containing boiling chips. Immediately immerse the bulb of thermometer in the methyl chloride. As soon as the temperature of the thermometer stops dropping rapidly, raise the bulb of the thermometer 1 mm above the surface of the methyl chloride and read the initial boiling temperature. If this temperature after correction is below  $-24.6^{\circ}\text{C}$ , immediately immerse the graduated cylinder in a dry ice-acetone bath until boiling has stopped. Remove the cylinder, note the volume and determine the temperature at which boiling begins as before. Repeat this operation until a boiling point not lower than  $-24.6^{\circ}\text{C}$  corrected is obtained. Record the corrected temperature (A) and the volume at this point.

**A-5.2.1** Place the cylinder in a 2 litre beaker containing about 800 ml of water at  $35^{\circ}\text{C}$ . Add hot water, as required, to maintain the temperature at  $35^{\circ}\text{C}$ . Allow the methyl chloride to evaporate until the volume boiled off is 95 ml plus the volume recorded when the temperature not lower than  $-24.6^{\circ}\text{C}$  was obtained. Determine the temperature (B) at this point by the same procedure as in **A-5.2**.

### **A-5.3 Calculation**

The corrected temperature, A and B represent the boiling range for 95 percent of the material. Mark temperature corrections by applying the thermometer corrections and barometric pressure correction. The latter is equal to  $+0.032^{\circ}\text{C}$  ( $760 - P$ ), where P is the barometric pressure in mm Hg.

## **A-6 DETERMINATION OF FREE CHLORIDE**

### **A-6.1 General**

A known quantity of liquified sample is scrubbed through gas washing bottles containing potassium iodide-starch solution and the liberated iodine is titrated against standard sodium Thiosulphate.

### **A-6.2 Apparatus**

#### **A-6.2.1 Conical Flask**

250 ml capacity, titted with a side tube and provided with a stopper.

**A-6.2.2 Graduated Cylinder** — 100 ml capacity

**A-6.2.3 Gas Wash Bottles** — 250 ml capacity.

### **A-6.3 Reagents**

#### **A-6.3.1 Potassium Iodide (10 Percent)**

Dissolve 25 g of potassium iodide in distilled water and make to 250 ml.

#### **A-6.3.2 Starch (1 Percent Solution)**

Mix 1 g of soluble starch with 10 ml of water and add this mixture, without stirring, to 90 ml of boiling water. Boil for further 5 minutes and cool.

#### **A-6.3.3 Standard Sodium Thiosulphate Solution (0.2 N)**

Dissolve 4 963.6 g of sodium thiosulphate pentahydrate in distilled water along with 0.1 g of sodium carbonate and make up to 1 litre with water. Standardize the solution using 0.02 N potassium dichromate with dilute sulphuric acid and potassium iodide-starch solution.

### **A-6.4 Procedure**

Take 50 ml of the liquified sample into the conical flask and connect it to the scrubbing gas wash bottles containing 100 ml of potassium iodide-starch solution. After evaporation is complete, collect the scrubbed water from the gas wash bottles and titrate against standard sodium thiosulphate solution.

### **A-6.5 Calculation**

$$\text{Free chlorine, ppm} = \frac{35460 \times V \times N}{M}$$

Where

$V$  = volume, in ml, of standard sodium thiosulphate solution;

$N$  = normality of standard sodium thiosulphate solution; and

$M$  = mass, in g, of the sample taken for the test.'

## **ANNEX B**

(Clause 6)

### **SAMPLING OF METHYL CHLORIDE**

#### **B-1 GENERAL**

**B-1.1** Samples shall be drawn and stored in a protected but well-ventilated place.

**B-1.2** The sampling flask and other equipment used in the collection of sample shall be free from any type of contamination.

**B-1.3** In drawing and handling the samples, the precautions described in 5.2 (c) shall be observed.

#### **B-2 SCALE OF SAMPLING**

##### **B-2.1 Lot**

**B-2.1.1** If the material is supplied in tanks (1 000 kg or more), each tank shall constitute a lot.

**B-2.1.2** In case of cylinders, a lot shall consist of all cylinders charged from one source.

**B-2.2** For ascertaining conformity of the material to the requirements of the specification, samples shall be drawn from each lot and separately tested.

**B-2.2.1** In the case of cylinders, the following will be the sample size for different lot sizes

Lot Size	Sample Size
( 1 )	( 2 )
Up to 25	3
26 to 50	4
51 to 100	5
101 and above	6

**B-2.3** The cylinder shall be selected at random with the help of random number tables (*see* IS 4905). In the absence of random number table, systematic sampling may be adopted by arranging the cylinder in the lot in one order as 1, 2, 3, up to  $r$  ( where  $r$  is the integral part of  $N/n$  ) and selecting every  $r$ th cylinder till the required sample size is obtained

**B-2.4** From each cylinder in the sample, sufficient quantity of methyl chloride shall be collected in a sample flask to conduct all the tests prescribed in Table 1.

**B-2.5** In the case of large tanks, three individual samples shall be collected from different parts of the tank.

### **B-3 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY**

**B-3.1** Test for all the characteristics shall be conducted on individual samples (numbering 3 or more).

**B-3.2** All the test results shall comply with the corresponding requirements given in Table 1 for acceptance of the lot.