

**BUREAU OF INDIAN STANDARDS**

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*भारतीय मानक मसौदा*

**बेंजीन - विशिष्ट**

*(IS 534 का छठा पुनरीक्षण)*

*Draft Indian Standard*

**BENZENE - SPECIFICATION**

*(Sixth Revision of IS 534)*

(ICS 71.080.15)

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Petroleum and their Related Products of  
Synthesis or Biological Origin, PCD 3

Last date for comments  
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**FOREWORD**

*(Formal clauses to be added later)*

Benzene is derived by suitable fractionation and refining by washing with acid or hydro refining of crude benzole recovered from the gas produced during carbonization of coal in coke ovens and retorts or recovered as byproduct in petroleum refining or petrochemical operations involving solvent extraction.

Earlier benzene was essentially a coal base product being made available as by-product from coke ovens of steel plants. The requirements and methods of test were also stipulated on the basis of the publications by National Benzole and Allied Products Association (NBA) and the Standardization of Tar Products Test Committee (STPTC), UK, in order to suit the prevailing quality of the product. However, cognizance was taken of the fact that consequent upon exploration of oil fields in the recent past specially the Bombay High, substantial quantities of indigenous crude was being made available, which has completely changed the scenario. On a quantum basis there is a distinct shift in production of various aromatic hydrocarbons from coal base to petroleum base as a result of which benzene is currently being made available. The Committee, therefore, decided to update the standard in accordance with the latest developments in the field, both in production and usage and in the light of the experience gained in the past.

This standard was first published in 1955. In view of the growing demand for benzene for organic synthesis it was considered necessary to include the material for this end use also. It was also felt that pure and ordinary grades of the material should be brought under a composite standard on the subject. The standard was, therefore, revised for the first time in 1965, amalgamating with IS 535 : 1955 'Specification for benzene, pure, nitration grade'.

In the second revision in 1974, the method of test for total sulphur was replaced by Raney nickel method, as the latter was found more convenient and reliable. Various changes covered under Amendment No. 1 issued in March 1973 were also incorporated in the second revision.

In the third revision, in 1992, keeping in view various end uses and the source of crude base, benzene was classified into three types. In view of stringent quality of benzene required for the manufacture of caprolactam, it was decided to incorporate additional requirement of aliphatic and aromatic impurities and bromine index.

In the fourth revision, in 2007, Type C has been deleted in order to align with the international practices. Additional requirements like purity of benzene by GC method including percent of toluene, non-aromatic hydrocarbons, 1,4 dioxane, carbon disulphide, N-formyl morphine as nitrogen, residue on evaporation and n-heptane have been included, in addition to making the requirements of other characteristics more stringent. Requirements like copper corrosion and neutrality have been deleted.

'IS 1840 : 1961 Benzene, reagent grade' was withdrawn, as it was observed by the technical committee, that this standard would cover the requirements of reagent grade benzene.

In the fifth revision, in 2021, requirement of carbon disulphide and residue on evaporation have been deleted, as these parameters are no longer part of the benzene specifications globally. References have been updated. Test methods have been revised including significance, expression of results and precision.

This sixth revision has been brought out to keep pace with the latest technological developments and international practices. In this revision following major changes have been made:

- a) Requirement of n-heptane, higher aliphatic and acyclic compounds (cyclohexane and methyl hexane) removed from Table 1 as requirement for non-aromatic hydrocarbons already prescribed;
- b) Requirement of N-formylmorphine as nitrogen removed from Table 1; and
- c) Reference test methods updated.

The following alternate test methods are also available for the characteristics stated and in case of dispute the referee test method as given in Table 1 shall be used.

<b>Characteristic</b>	<b>Methods of Test</b>
Benzene, Non-aromatic hydrocarbons, Toluene, 1,4, Dioxane	ASTM D7504, ASTM D5713
Colour, Pt-Co scale	ASTM D1209
Relative density, 15.56 / 15.56 °C OR Density at 20 °C	ASTM D4052
Sulphur	ASTM D4045
Distillation	ASTM D850
Bromine index	ASTM D1492, ASTM D2710

Solidification point, anhydrous basis	ASTM D852
Thiophene	ASTM D4735, ASTM D5623
Acid wash colour	ASTM D848

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 specified value in this standard.

## 1 SCOPE

This standard prescribes the requirements and the methods of sampling and tests for benzene.

## 2 REFERENCES

The following standards contain provisions which, through reference in the 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 the standards indicated below:

<i>IS No./International Standard</i>	<i>Title</i>
IS 82 : 1973	Method of sampling and test for thinners and solvents for paints ( <i>first revision</i> )
IS 1070 : 2023	Reagent grade water ( <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 1448	Methods of Test for Petroleum and its Products
(Part 167) : 2018 / ISO 12185 : 1996	Determination of density - Oscillating u - Tube method
(Part 178) : 2020 / ISO 6271 : 2015	Clear Liquids - Estimation of Colour by the Platinum Cobalt Colour Scale
(Part 180) : 2020 / ISO 16591 : 2010	Petroleum Products - Determination of Sulphur Content - Oxidative Microcoulometry Method
IS 1446 : 2002	Classification of dangerous goods ( <i>second revision</i> )
IS 4644 : 1968	Code of safety for benzene, toluene and xylene
IS 4905 : 2015 / ISO 24153 : 2009	Random sampling and randomization procedure ( <i>first revision</i> )
ASTM D5453	Standard test method for determination of total sulfur in light hydrocarbons, spark ignition engine fuel, diesel engine fuel, and engine oil by ultraviolet fluorescence
ASTM D5776	Standard test method for bromine index of aromatic hydrocarbons by electrometric titration
ASTM D7011	Standard test method for determination of trace thiophene in refined benzene by gas chromatography and sulfur selective detection

## 3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 82 and the following shall apply.

**3.1 Bromine Index** — The number of milligrams of bromine consumed by 100 g of sample under given conditions.

**3.2 Solidification Point** — An empirical constant defined as the temperature at which the liquid phase of a substance is in approximate equilibrium with a relatively small portion of the solid phase.

#### **4 TYPES**

There shall be two types of material, namely:

- a) Type A — Suitable for manufacture of caprolactam; and
- b) Type B — Suitable for manufacture of other products.

#### **5 REQUIREMENTS**

##### **5.1 Description**

The material shall be a clear liquid, free of sediments and haze when observed at 18 to 26 °C.

**5.2** The material shall also comply with the requirements given in Table 1 when tested according to the methods given in col 5 of Table 1.

#### **6 PACKING, STORAGE AND MARKING**

##### **6.1 Packing**

**6.1.1** Material shall be packed in suitable containers of appropriate size as agreed to between the purchaser and the supplier.

**6.1.2** The containers shall be securely closed, protected from light, and shall be stored in a cool place.

**6.1.3** All the containers in which the material is packed shall be dry, clean, and free from substances soluble in benzene and leak proof.

##### **6.2 Storage**

The containers for storage and transport of the material, since classified as flammable and dangerous goods, shall, in addition comply with the provisions of *Railways Red Tariff Rules* and *Red Tariff No. 20* issued by the Indian Railways Conference Association along with any future amendments and the requirements as laid down from time to time by the Petroleum and Explosives Safety Organization (PESO), Government of India, for packing, storage and transit of flammable liquids and the Board of Trade Regulations, as applicable thereon, for transport by steamers.

**6.2.1** Necessary safeguards against the risk arising from the storage and handling of large volumes of flammable liquids (*see* IS 1446) shall be provided and all due precautions shall be taken [*see* IS 1260 (Part 1)] at all times to prevent accidents by fire or explosion.

**6.2.2** Except when they are opened for the purpose of cleaning and rendering them free from benzene vapour, all empty tanks or other containers shall be kept securely closed unless they have been thoroughly cleaned and freed from benzene vapour.

### **6.3 Marking**

**6.3.1** Material shall be marked with the following information:

- a) Name and type of material;
- b) Manufacturer's name, initials or trade-mark, if any;
- c) Net mass of material;
- d) Identification in batch number or code number to enable traceability of consignment to date of manufacture; and
- e) Any other statutory requirements.

**6.3.2** Each container shall have the caution label 'FLAMMABLE' together with the corresponding symbol for labelling of dangerous goods as given in IS 1260 (Part 1).

#### **6.3.3 *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 *BIS Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.

## **7 HANDLING**

Benzene is highly toxic and therefore it shall be handled carefully (*see* IS 4644). Exposure of benzene in atmosphere should be monitored regularly. Persons exposed to benzene shall be periodically checked according to State Factory Rules and Local State Regulations.

## **8 SAMPLING**

Representative samples of the material shall be prepared as prescribed in Annex G.

## **9 TEST METHODS**

**9.1** Tests shall be conducted according to the methods referred to in col 5 of Table 1.

### **9.2 Quality of Reagents**

Unless otherwise specified, pure chemicals and reagent grade 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.

**Table 1 Requirements for Benzene**  
(Clauses 5.2, and 9.1)

SI No.	Characteristic	Requirement		Methods of Test
		Type A	Type B	
(1)	(2)	(3)	(4)	(5)
i)	Benzene, percent by mass, <i>Min</i>	99.90	99.80	Annex A <sup>1)</sup>
ii)	Non-aromatic hydrocarbons, percent by mass, <i>Max</i>	0.10	0.20	Annex A <sup>1)</sup>
iii)	Toluene, percent by mass, <i>Max</i>	0.01	0.05	Annex A <sup>1)</sup>
iv)	1,4 Dioxane, percent by mass, <i>Max</i>	0.01	-	Annex A <sup>1)</sup>
v)	Colour, Pt-Co scale, <i>Max</i>	10	10	IS 1448 (Part 178) <sup>1)</sup> / Annex B
vi)	Relative density, 15.56 / 15.56°C, kg/m <sup>3</sup>	882 to 886	882 to 886	IS 1448 (Part 167) <sup>1)</sup>
	( <i>or</i> )			
	Density at 20°C, kg/m <sup>3</sup>	878 to 882	878 to 882	IS 1448 (Part 167) <sup>1)</sup>
vii)	Sulphur, mg/kg, <i>Max</i>	1	As agreed to between the purchaser and the supplier	ASTM D5453 <sup>1)</sup> / IS 1448 (Part 180)
viii)	Distillation range (1 to 96 percent) including the temperature 80.1 °C at 101.31 kPa (760 mm Hg) pressure, °C, <i>Max</i> <sup>2)</sup>	1.0	1.0	Annex C <sup>1)</sup>
ix)	Bromine index, mg/100 g, <i>Max</i>	10	20	ASTM D5776 <sup>1)</sup> / Annex D
x)	Solidification point, anhydrous basis, °C, <i>Min</i> <sup>2)</sup>	5.45	5.35	Annex E <sup>1)</sup>
xi)	Thiophene, mg/kg, <i>Max</i>	0.6	1.0	ASTM D7011 <sup>1)</sup>

xii)	Acid wash colour, <i>Max</i>	Pass with 1	Pass with 1	Annex F <sup>1)</sup>
NOTES				
<sup>1)</sup> Referee method in case of dispute				
<sup>2)</sup> If purity by GC method [Sl no. (i)] is carried out, distillation test and solidification point test are optional.				

## ANNEX A

[Table 1, Sl No. (i), (ii), (iii) and (iv)]

### DETERMINATION OF PURITY OF BENZENE BY GC

#### A-1 OUTLINE OF THE METHOD

**A-1.1** A known amount of an internal standard is added to the specimen. A small volume of this mixture is injected into a gas chromatograph equipped with a flame ionization detector (FID) and a capillary column.

**A-1.2** The peak area of each impurity and the internal standard is measured by an electronic integrator. The concentration of each impurity is calculated from the ratio of the peak area of the internal standard versus the peak area of the impurity. Purity is calculated by subtracting the sum of the impurities found from 100.00 mass, percent. Results are reported in mass, percent.

#### A-2 SIGNIFICANCE

Paraffins, olefins, naphthenes and aromatic contents of petroleum products are determined by GC. The total composition of a liquid or gas with its various components are used by analysts for studying the performance of the plant and its equipment. Sometimes these compositions also indicate the quality of the product. It is also used to check the mass balance and the reactions of various catalysts.

#### A-3 INTERFERENCES

**A-3.1** Benzene is typically resolved from naturally occurring components with boiling points less than 138 °C. Naturally occurring components include non-aromatic hydrocarbons, toluene, C<sub>8</sub>, aromatics and 1,4-dioxane. An adequate separation of known impurities from benzene should be evaluated for the column selected.



**A-3.2** The internal standard chosen shall be sufficiently resolved from any impurity and the benzene peak.

#### A-4 APPARATUS

**A-4.1 Gas Chromatograph** — Any chromatograph having a flame ionization detector that can be operated at the conditions given in Table 2 shall be used. The system shall have sufficient sensitivity to obtain a minimum peak height response for a 0.000 5 mass, percent impurity twice the height of the signal background noise.

**A-4.2 Electronic Integrator** — Computer based capable of handling internal standard calculations and peak grouping is recommended.

**A-4.3 Column** — Fused silica capillary column with crosslinked polyethylene glycol stationary phase is recommended. Alternate stationary phases may be used, if they produce at least the same aromatic separation and elute C<sub>9</sub> non-aromatic impurities before benzene.

**A-4.4 Microsyringes** — 10 and 100 microlitre (μl) capacity.

#### A-5 REAGENTS AND MATERIALS

**A-5.1 Carrier Gas** — chromatographic grade helium is recommended.

**Table 2 Typical Instrumental Parameters**  
(Clauses A-4.1 and A-6)

SI No	Characteristic	Requirement
(1)	(2)	(3)
i)	Detector	Flame ionization
ii)	Column:	Fused silica
	a) Length, m	50
	b) Inside diameter, mm	0.32
	c) Stationary phase	Crosslinked polyethylene glycol
	d) Film thickness, μm	0.25
iii)	Temperatures:	
	a) Injector, °C	200
	b) Detector, °C	250
	c) Column, °C	70 isothermal
iv)	Carrier gas	Helium
v)	Linear velocity, cm/s	22
vi)	Split ratio	200 : 1
vii)	Sample size, μl	0.5
viii)	Recorder	Electronic integration required

**A-5.2 High Purity Benzene** — 99.99 percent by mass, minimum, prepared by multiple step recrystallization of commercially available benzene of not less than 99 percent purity.

**A-5.3 Internal Standard, n-Nonane, (nC<sub>9</sub>)** — with a purity of 99 percent by mass, minimum is recommended. Other compounds may be acceptable provided, they can be obtained in high purity and meet the requirements of **A-3.2**.

**A-5.4** Pure compounds for calibration should include toluene, benzene, ethyl benzene, cyclohexane and 1,4- dioxane of a purity not less than 99 percent. If the purity of the calibration compounds is less than 99 percent, the concentration and identification of impurities must be known so that the composition of the final weighed blends can be adjusted for the presence of the impurities.

## A-6 PREPARATION OF APPARATUS

Follow manufacturer's instructions for mounting the column into the chromatograph and adjusting the instrument to the conditions described in Table 2. Allow sufficient time for the equipment to reach equilibrium.

## A-7 CALIBRATION

**A-7.1** Prepare a synthetic mixture of high purity benzene and representative impurities by direct weighing. Weigh each impurity to the nearest 0.1 mg. Table 3 contains a typical calibration blend. Cyclohexane is used for the non-aromatic portion and ethyl benzene for the C<sub>8</sub> aromatic portion.

**Table 3 Typical Calibration Blend**  
(Clause A-7.1)

SI No.	Compound	Weight, Percent
(1)	(2)	(3)
i)	Benzene	99.0000
ii)	Toluene	0.0500
iii)	Cyclohexane	0.0500
iv)	Ethyl benzene	0.0500
v)	1,4 Dioxane	0.0200

**A-7.2** Using the exact mass for each impurity, calculate the percent by mass, concentration of the calibration blend.

**A-7.3** Into a 50 ml volumetric flask, add 50 ml of nC<sub>9</sub> to 49.95 ml of the calibration blend and mix well. Using a density of 0.874 g/ml for the calibration blend and a density of 0.718 g/ml for the nC<sub>9</sub>, the resulting nC<sub>9</sub>, concentration will be 0.0825 percent by mass.

**A-7.4** Inject 0.5 µl of the blend with internal standard into the chromatograph and integrate the area under each peak, excluding benzene.

**A-7.5** Calculate the relative response factors (*RRF*) as follows:

$$RRF_i = \frac{(A_s)(C_i)}{(C_s)(A_i)}$$

where

$RRF_i$  = impurity, i;

$A_s$  = peak area of internal standard;

$A_i$  = peak area of impurity, i;

$C_i$  = percent by mass, impurity, i, from **A-7.2**; and

$C_s$  = concentration of internal standard, percent by mass from **A-7.3**.

## **A-8 PROCEDURE**

**A-8.1** Into a 50 ml volumetric flask, add 50 µl of *n*C<sub>9</sub> internal standard and dilute to the mark with sample. Mix well.

**A-8.2** Inject 0.5 µl of mixture into the chromatograph.

**A-8.3** Integrate the area under all peaks except for benzene. Sum the non-aromatic fraction up to *n*C<sub>9</sub>, for reporting as a single component

## **A-9 CALCULATION**

**A-9.1** Calculate the amounts of each individual impurity as required. Sum the areas of all the non-aromatic peaks.

**A-9.2** Calculate the mass, percent concentration of each impurity as follows:

$$C_i = \frac{(A_i)(RRF_i)(C_s)}{(A_s)}$$

**A-9.3** Calculate the benzene purity as follows:

$$\text{Benzene, percent by mass} = 100.00 - C_i$$

where

$C_i$  = total concentration of all impurities, percent by mass.

## **A-10 EXPRESSION OF RESULTS**

Report benzene and the total impurities to the nearest 0.01 percent, and individual impurities to the nearest 0.001 percent.

**A-11 PRECISION**

<b>Component</b>	<b>Concentration (mg/kg)</b>		
	<i>Average Concentration</i>	<i>Repeatability</i>	<i>Reproducibility</i>
Non- aromatics	22	19	20
	737	70	184
Toluene	14	2	6
	116	4	54
Ethylbenzene	14	3	7
	121	7	14
1, 4 Dioxane	10	2.3	4.8
	5	1.9	2.5
Benzene (weight percent)	99.87	0.01	0.027
	99.99	0.002	0.004

**ANNEX B**

[Table 1, Sl No. (v)]

**DETERMINATION OF COLOUR (PLATINUM-COBALT SCALE)****B-1 OUTLINE OF THE METHOD**

This method includes visual comparison of the colour of a sample with that of colour standard, and expression of the result in terms of Hazen (platinum-cobalt) colour units. For routine control purposes an instrument such as a comparator, colorimeter or spectrophotometer may be used, provided that it has first been established that the results so obtained are identical with those obtained by visual comparison.

**B-2 SIGNIFICANCE**

Also termed as Hazen Colour Unit, it is the colour of a solution containing 1 mg of platinum per litre in the form of chloroplatinic acid, in the presence of 2 mg of cobalt (II) chloride hexahydrate per litre. Colour does not have much significance as an identification aid or quality of product. The colour indicates whether there is intergrade mixing up with dark oil. It is an indication of the degree of refining of the products.

**B-3 CHEMICALS****B-3.1 Cobalt (II) Chloride Hexahydrate (CoCl<sub>2</sub>.6H<sub>2</sub>O)**

**B-3.2 Hydrochloric Acid** — approximately 1.19 g/ ml. About 38 percent (m/m) solution, or approximately 12 N solution.

**B-3.3 Chloroplatinic Acid** — Dissolve 1.00 g platinum in a sufficient quantity of aqua regia in a glass or porcelain dish by heating on a boiling water bath. When the metal has dissolved, evaporate the solution to dryness. Add 4 ml of the hydrochloric acid solution (**B-3.2**) and again evaporate to dryness. Repeat this operation twice more. 2.10 g of chloroplatinic acid (H<sub>2</sub>PtCl<sub>6</sub>) is obtained.

(or)

**B-3.4 Potassium Chloroplatinate (K<sub>2</sub>PtCl<sub>6</sub>)****B-4 APPARATUS**

**B-4.1 Two Colorimetric Tubes** — flat based, with a graduation mark at least 100 mm above the base and matched especially with respect to colour of glass and height of graduation mark above the base. Suitable tubes are available commercially as 50 ml or 100 ml Nessler cylinders.

NOTE — For the measurement of low colorations (less than 50 Hazen units), the height of the graduation mark above the base must be greater than that for the measurement of deeper colours.

The height must be such that, on looking through this liquid, a clear distinction between the standard Hazen matching solution and the sample can be clearly identified.

## B-5 PREPARATION OF STANDARD COLORIMETRIC SOLUTIONS

**B-5.1 Standard Colorimetric Solution, 500 Hazen Units** — Dissolve 2.00 g of the cobalt chloride (**B-3.1**), and the equivalent of 1.00 g of platinum, that is either 2.10 g of the chloroplatinic acid (**B-3.3**), or 2.49 g of the potassium chloroplatinate (**B-3.4**), in water in a 2000 ml one mark volumetric flask, add 200 ml of the hydrochloric acid solution (**B-3.2**), dilute to the mark and mix. This solution has a colour of 500 Hazen colour units.

**B-5.2 Standard Hazen Matching Solution (Diluted Solution)** — Into two series of ten 500 ml and fourteen 250 ml one mark volumetric flasks, place the volumes of standard colorimetric solution (*see* **B-5.1**) shown in Table 4, dilute to the mark and mix.

### B-5.3 Storage

Store these solutions (**B-5.1** and **B-5.2**) in the dark in stoppered glass bottles. Under these conditions the colour standard solution (**B-5.1**) is stable for at least one year. The standard Hazen matching solution (**B-5.2**), although stable for at least one month, shall preferably be prepared fresh.

## B-6 PROCEDURE

**B-6.1** First check visually that the sample has colour characteristics close to those of the standard Hazen matching solution (**B-5.2**). If not, follow the instructions given in **B-7.2**.

**B-6.2** Pour into one of the colorimetric tubes (**B-4.1**) a quantity of the sample sufficient to fill it to the graduation mark. Similarly pour the standard Hazen matching solution (**B-5.2**) which appears to have a similar colour into the other tube to the mark.

**B-6.3** Compare the colour of the sample with that of the standard, by looking down the tubes from top to bottom against a white background strongly illuminated by daylight or by an electric 'daylight' lamp, taking care to avoid any side illumination.

**B-6.4** Repeat, if necessary, with other standard Hazen matching solutions until the closest match is obtained.

**Table 4 Standard Hazen Matching Solutions**

(Clause B-5.2)

500 ml Volumetric Flasks		250 ml Volumetric Flasks	
Volume of Standard Colorimetric Solution ( <i>see</i> <b>B-5.1</b> )	Corresponding Colour	Volume of Standard Colorimetric Solution ( <i>see</i> <b>B-5.1</b> )	Corresponding Colour

ml	Hazen Units	ml	Hazen Units
0	0	30	60
5	5	35	70
10	10	40	80
15	15	45	90
20	20	50	100
25	25	62.5	125
30	30	75	150
35	35	87.5	175
40	40	100	200
50	50	125	250
-	-	150	300
-	-	175	350
-	-	200	400
-	-	225	450

## NOTES

**1.** Instruments are available which permit visual comparison of a liquid of a given depth, with a moving tinted glass disc corresponding to the different standard Hazen matching solutions. The use of such instruments, whose standards are very stable, is permissible provided that it has been established that using these tinted glass discs give the same results as do the corresponding standard Hazen matching solutions.

**2.** For routine control purposes, a colorimeter or spectrophotometer may be used, the instrument being standardized by means of the standard calorimetric solutions (**B-5.1** and **B-5.2**), provided that it has been confirmed that the use of that instrument gives the same results as does visual comparison.

**B-7 EXPRESSION OF RESULTS**

**B-7.1** Express the colour of the sample as the number of Hazen colour units corresponding to the standard Hazen matching solution having the closest match to the sample.

**B-7.2** If the colour of the sample does not correspond with that of any of the standard Hazen matching solutions (brownish-yellow), give, if possible, an estimate of the colour and a description of the observed colour.

**B-8 PRECISION****B-8.1 Repeatability**

Two results, each the mean of duplicates, obtained by the same operator on different days should be considered suspect, if they differ by more than two platinum-cobalt units.

### **B-8.2 Reproducibility**

Two results, each the mean of duplicates, obtained by operators in different laboratories, should be considered suspect, if they differ by more than seven platinum-cobalt units.



## ANNEX C

[Table 1, Sl No. (viii)]

**DETERMINATION OF DISTILLATION RANGE****C-1 OUTLINE OF THE METHOD**

The distillation is carried out via a carefully controlled distillation wherein temperature readings are noted for the first drop of distillate and at the dry point.

**C-2 SIGNIFICANCE**

Distillation indicates volatility of the product. This is an important characteristic not only for the identification of the product, but also for its application. It is also used as an internal quality control tool, and in R&D work on hydrocarbons and related materials. It gives a broad indication of general purity of the product.

**C-3 APPARATUS**

**C-3.1 Flask** — A standard 200 ml side-tube, heat-resistant glass distillation flask conforming to the following dimensions (*see* Fig. 1):

Diameter of bulb, outside, mm	$76 \pm 1.5$
Diameter of neck, inside, mm	$21 \pm 1$
Height of flask, outside, mm	$179 \pm 3$
Vertical distance from bottom of bulb outside to bottom of vapour- tube opening in neck, mm	$120 \pm 3$
Length of side tube, mm	$100 \pm 3$
Diameter of side tube, outside, mm	$7 \pm 0.5$
Angle of wide tube with vertical axis of bulb and neck	$75 \pm 3$

**C-3.2 Thermometer (Manual)** — A suitable thermometer having 0.2 °C subdivisions and covering the entire range may be used.

(*or*)

**C-3.3 Thermometer sensor (Automatic)** — Thermocouples or Resistance Temperature Detectors (RTDs) must have the same temperature lag and accuracy as the mercury glass thermometer. Confirmation of the calibration of these temperature sensors is to be done at regular intervals. One method is to distil pure toluene and compare the temperature indicated by the thermocouple or RTD with that as shown by the thermometer.

**C-3.4 Condenser (Manual)** — The condenser tube may consist of a straight glass tube 600 to 610 mm in length and 12 mm in inside diameter, of standard wall thickness (about 1.25 mm) with the exit end cut off square and ground flat. It shall be set in a cooling trough so that at least 380 mm of

the tube is in contact with the water. Clearance between the condenser tube and any parallel side of the trough shall be not less than 19 mm. The water in the cooling trough shall be maintained at 100 °C. This may be done by adding ice to the water or by circulating chilled water through the trough. The trough shall be so mounted that the condenser tube is set at an angle of 75° with the vertical.

(or)

**C-3.5 Condenser (Automatic)** — Condenser of the automatic apparatus shall be in accordance with the instrument manufacturer and conform to the physical specifications described in **C-3.4**.

**C-3.6 Crow Receiver (Manual)** — A graduate of the cylindrical type, of uniform diameter, with a pressed or moulded base and a lipped top. It shall be graduated to contain 100 ml, and the graduated portion shall be neither less than 178 mm nor more than 203 mm in length. It shall be graduated in single millilitres and each fifth mark shall be distinguished by a longer line. It shall be numbered from the bottom up at intervals of 10 ml. The overall height of the graduate shall not be less than 248 mm nor more than 260 mm. The graduations shall not be in error by more than 1 ml at any point on the scale. The bottom 1 ml graduation may be omitted.

**C-3.7 Receiver / Level Follower (Automatic)** — A receiver to be used with measurements in accordance with the instrument manufacturer and conform to the physical specifications described in **C-3.6**. For automatic apparatus, the level follower of the apparatus will have a resolution of 0.1 ml with an accuracy of  $\pm 1$  ml. The calibration of the assembly shall be carried out as given in manufacturer's instructions which involves verifying the output with the receiver containing 5 and 100 ml of material, respectively.

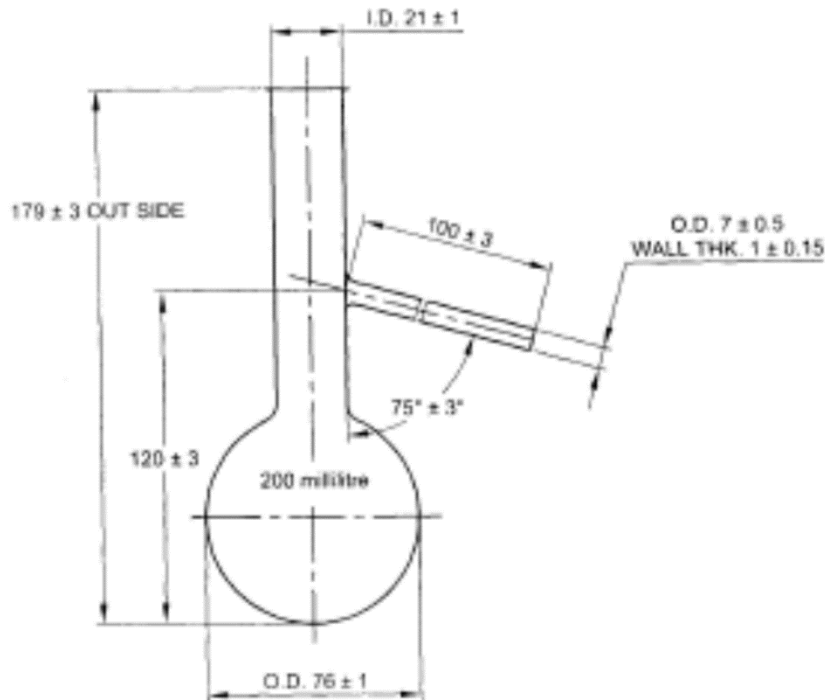
**C-3.8 Support for Flask** — A sheet of 3 to 6 mm hard insulation board 152 mm<sup>2</sup> with a circular hole in the centre, supported on a circular metal shield enclosing the bunsen burner, and approximately 50 mm higher than the top of the burner. The hole shall be 25 mm in diameter.

**C-3.9 Heater (Manual)** — An electric heater or a Bunsen burner, fully adjustable and capable of giving sufficient heat to distill the product at the required rate is used. When a Bunsen burner is used, the burner shall be adjusted so as to produce an entirely blue flame. In case of dispute concerning results obtained with gas heat versus electric heat, gas heat shall be accepted as the standard.

(or)

**C-3.10 Heater (Automatic)** — An electric heater of the automatic apparatus shall be in accordance with the instrument manufacturer and conform to the physical specifications described in **C-3.9**.

**CAUTION** — Superheating of the flask can cause erroneous results and is more likely to occur with electric heaters than with bunsen burners as heat source.



All dimensions are in millimeters

**FIG. 1 DISTILLATION FLASK**

#### **C-4 ASSEMBLY OF THE APPARATUS**

**C-4.1** Assemble the apparatus as shown in Fig. 2. Mount the flask on the insulation board of appropriate dimensions, with the side tube extending through a tightly fitting cork stopper about 50 mm into the condenser tube.

**C-4.2** Support the distillation thermometer in the neck of the flask by means of a cork stopper with the thermometer vertical and centred in the neck of the flask and in such a position that the top of the bulb (or top of contraction bulb, if present) is level with the lowest point of juncture between the side tube and the neck of the flask (*see* Fig. 3).

**C-4.3** Place the burner directly under the centre of the hole in the insulation board.

#### **C-5 PROCEDURE**

##### **C-5.1 Manual Distillation Procedure**

Carefully measure a 100 ml sample of the material to be tested in the 100 ml graduated cylinder at room temperature and transfer to the distillation flask, draining the cylinder at least 15 s. This is preferably done before mounting the flask in position, in order to prevent liquid from entering the side arm. Connect the flask to condenser and apparatus, assembled as described in C-4. Do not rinse

out the graduated cylinder used to measure the sample for distillation, but place under the lower end of the condenser tube to receive the distillate. Heat the flask slowly, especially after ebullition has begun, so as to allow the mercury column of the thermometer to become fully expanded before the first drop distils over. Regulate the rate of heating so that the ring of condensing vapour on the wall of the flask reaches the lower edge of the side arm in not less than 90 s, and preferably approximately 120 s, from the start of the rise of the vapour ring. The total time from the start of the heating until the first drop falls into the receiver should be not less than 5 min or more than 10 min. Avoid major changes in heating rate. Even operation is best gained through experience with the method. When distillation starts, adjust the receiver to allow condensation to flow down its inner wall to prevent loss of spattering; then adjust the heater to continue the distillation at the rate of 5 to 7 ml/min (about 2 drops/s). Maintain this rate, and continue the distillation to dryness. The total yield of distillate shall not be less than 97 percent.

### C-5.2 Automatic Distillation Procedure

Connect the distillation flask to the automatic distillation equipment as described in C-5.1. Fit the temperature sensor to the flask for automatic distillation equipment according to the manufacturer's instructions.

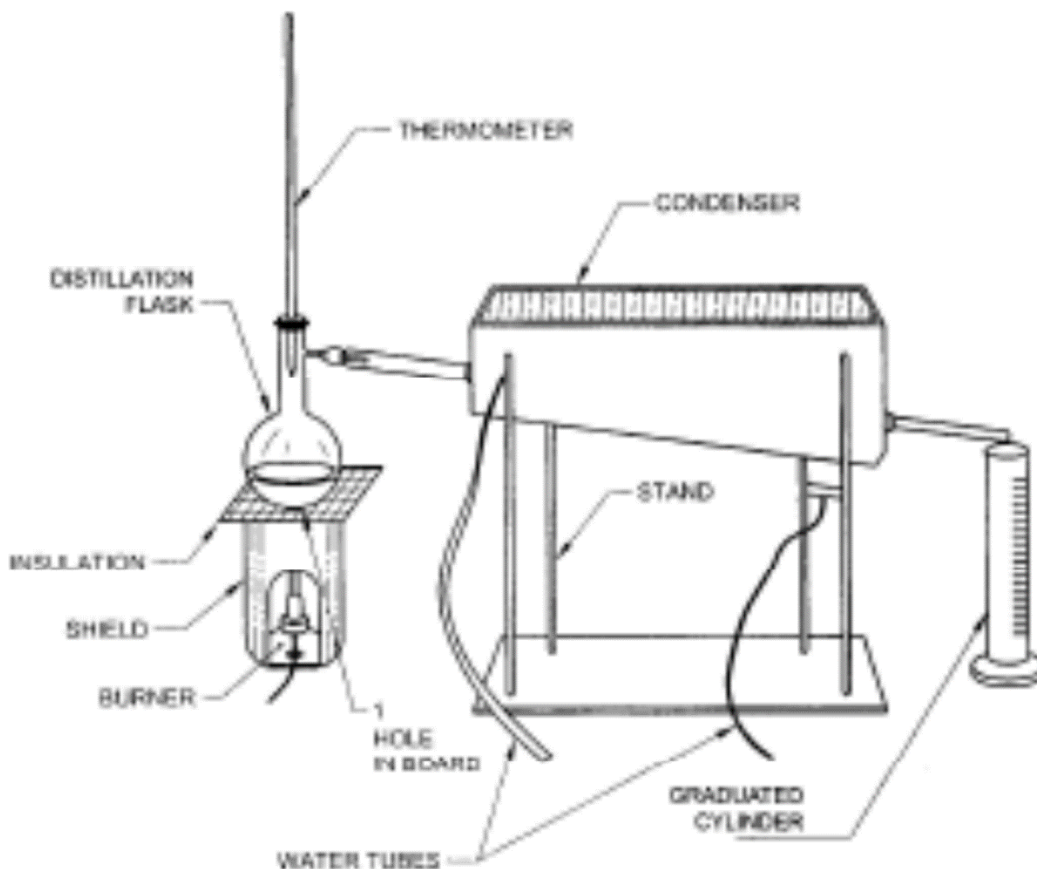
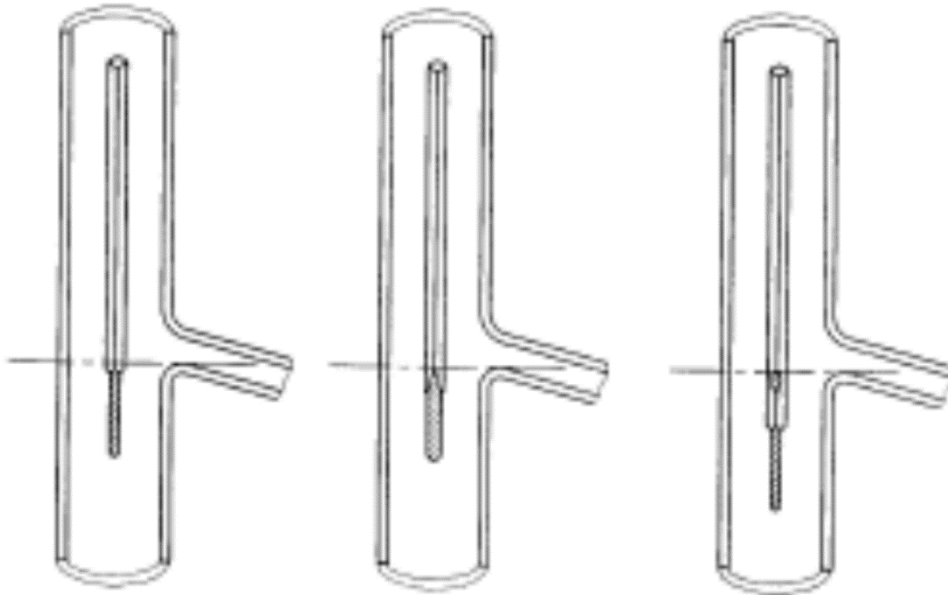


FIG. 2 APPARATUS ASSEMBLY FOR DISTILLATION FLASK



**FIG. 3 POSITION OF THERMOMETER IN DISTILLATION FLASK**

**C-5.3** Take the temperature reading when the first drop of distillate falls into the receiving cylinder and report as the initial boiling point (IBP). Take a final reading when the liquid just disappears from the flask, and report this reading as the dry point temperature (DPT).

**C-5.4** Observe and record the following additional data at the time and place of the distillation test:

- Correction for inaccuracy of the thermometer, and
- Barometer reading and temperature of the barometer. The observed barometric pressure shall be corrected by reference to standard tables and reported in terms of ml of mercury at 0 °C.

## **C-6 TEMPERATURE CORRECTIONS**

### **C-6.1 Inaccuracy of Thermometer**

This correction shall be obtained by calibration of the thermometer used in the test and applied to the observed thermometer reading.

### **C-6.2 Variation from Standard Barometric Pressure**

This correction shall be applied to the observed temperature after correcting for accuracy of the thermometer and is determined by the following equation:

$$C = [0.0427 + \{0.000025 \times (760 - P)\}] \times (760 - P)$$

where

C = the correction, in °C; and

P = the measured barometric pressure, in ml of mercury, corrected to 0°C.

### **C-6.3 Combined Corrections**

If the overall distillation range of the sample does not exceed 2 °C, a combined correction for thermometer inaccuracy and barometric pressure may be made on the basis of the difference between the observed 50 percent boiling point and the true boiling point at 760 mm of benzene, that is, 80.1 °C.

## **C-7 EXPRESSION OF RESULTS**

Report observed temperatures to the nearest 0.1°C, in a manner conforming to the specifications of the material tested.

## **C-8 PRECISISON**

### **C-8.1 Automatic or Manual Distillation Method**

**C-8.1.1 Repeatability** — Duplicate results by the same operator should be considered suspect, if they differ by more than 0.16.

**C-8.1.2 Reproducibility** — Results submitted by each of two laboratories should be considered suspect, if the two results differ by more than 0.42.

## ANNEX D

[Table 1, Sl No. (ix)]

### DETERMINATION OF BROMINE INDEX

#### D-1 OUTLINE OF THE METHOD

The sample is added to a solvent and titrated with electrolytically generated bromine at room temperature. The end point is determined by a dead stop method. The time of titration is proportional to the bromine added to the specimen.

#### D-2 SIGNIFICANCE

It is the number of milligrams of bromine that will react with 100 g of sample under the conditions of the test. This test method provides a measure of trace amounts of unsaturated hydrocarbons in petroleum distillates an estimate of the quantity of these materials is useful in assessing the suitability of the lighter fractions for use as reaction solvents. This test also gives a broad indication of olefinic content.

#### D-3 APPARATUS

**D-3.1 Amperometric Coulometric Apparatus** — It is an automatic apparatus, suitable for bromine index titrations with variable generator current and timer. A typical circuit diagram of suitable equipment is shown in Fig. 4.

**D-3.2 Syringe** — 2 ml with needle and rubber cap seal.

**D-3.3 Stirrer** — magnetic.

#### D-4 REAGENTS

**D-4.1 Electrolyte** — To make 1 litre, mix 600 ml of glacial acetic acid, 260 ml of absolute methanol, and 140 ml of potassium bromide solution (119 g/l) (**D-4.2**). Dissolve 2 g of mercuric acetate in this mixture.

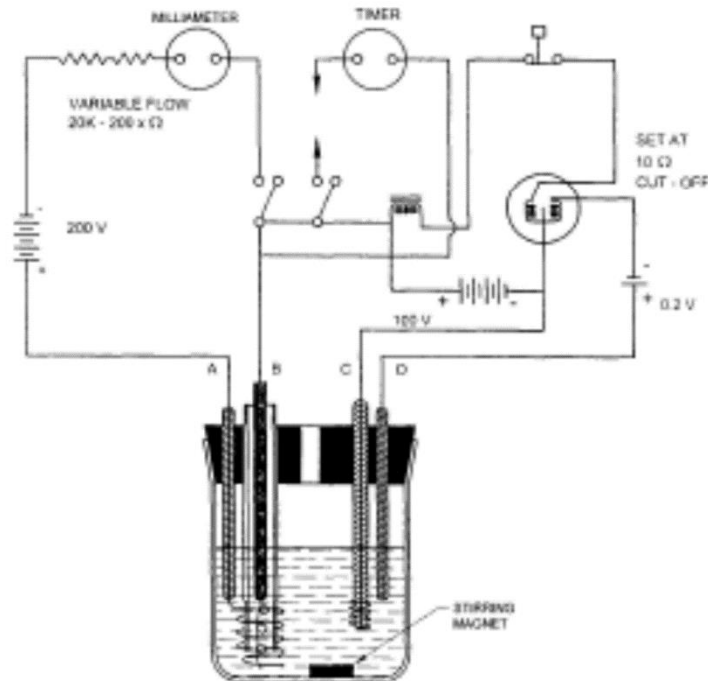
**D-4.2 Potassium Bromide Solution (119 g/l)** — Dissolve 119 g of potassium bromide (KBr) in water and dilute to one litre.

#### D-5 PROCEDURE

**D-5.1** Place 50 ml of electrolyte in a clean, dry titration cell, insert the electrodes, and begin stirring. Apply the generation current in accordance with Table 6.

**Table 6 Sample Size and Generation Current**  
(Clause F-5.1 and F-5.3)

Sl No.	Estimated Bromine Index	Specimen Weight, g	Generation Current, mA
(1)	(2)	(3)	(4)
i)	0-20	1.000	1.0
ii)	20-200	0.600	5.0
iii)	200-2 000	0.060	5.0



**FIG. 4 AUTOMATIC AMPEROMETRIC – COULOMETRIC TITRATION CIRCUIT**

**D-5.2** Before introducing the sample and immediately before each determination, bring the coulometer to equilibrium.

**D-5.3** Draw into the syringe the amount of sample prescribed in Table 6 corresponding to the estimated bromine index. Wipe the needle with a clean cloth, attach a rubber cap seal to the needle, and weigh on the analytical balance. Remove the seal, add the sample to the electrolyte, and set the timer to zero. Replace the seal, reweigh the syringe, and calculate the sample weight.



**D-5.4** Begin titration of the sample. As the titration proceeds, keep the generation current at the selected value. The generation of bromine will continue as long as it is consumed by the sample. At the end point an incremental increase in bromine concentration causes the titrator and timer to stop automatically. Forty seconds after the titrator has shut off, continue the titration. If the titrator cuts off immediately, the end point has been reached and the titration may be considered complete. Otherwise, it may be necessary to continue the titration in steps, waiting about 40 s between steps, until the titration time increment is 4 s or less. Note the total titration time and generation current.

## D-6 CALCULATION

Calculate the bromine index, B, as follows:

$$B = \frac{T \times I \times 79.9}{965 \times M}$$

where

T = titration time, in s;

I = generation current, in mA; and

M = mass of sample, in g.

## D-7 EXPRESSION OF RESULTS

Bromine index to be reported to the nearest unit.

## D-8 PRECISION

**D-8.1 Repeatability** — Duplicate results by the same operator should be considered suspect, if results differ by more than 1.15.

**D-8.2 Reproducibility** —The results submitted by two laboratories should be considered suspect, if they differ by more than 4.1.

**ANNEX E**  
 [Table 1, Sl No, (x)]  
**DETERMINATION OF SOLIDIFICATION POINT**

**E-1 OUTLINE OF THE METHOD**

Solidification point is measured by noting the maximum temperature reached during a controlled cooling cycle after the appearance of a solid phase.

**E-2 SIGNIFICANCE**

Solidification point is the temperature at which the liquid phase of a substance is in approximate equilibrium with a relatively small portion of the solid phase. It is used as criteria for determining the purity of benzene. The closer the solidification point reaches that of pure benzene, the purer the sample.

**E-3 APPARATUS**

**E-3.1 Benzene Container** — air jacketed.

**E-3.1.1 Inner Container** — a test tube 15 mm in outside diameter and 125 mm in length,

**E-3.1.2 Air Jacket** — a standard test tube 25 mm in outside diameter and 150 mm in length.

**E-3.1.3 Insulation** — with dry absorbent cotton or glass wool.

**E-3.2 Benzene Container (Thick Walled)** — a glass test tube 18 mm in outside diameter, 14 mm in inside diameter and 150 mm in length.

**E-3.3 Ice Bath** — a one litre beaker or similar suitable container having an effective depth of at least 127 mm and filled with chipped or shaved ice.

**E-3.4 Stirrer** — consisting of a 1 mm wire (copper or stainless steel) or a 2 mm glass rod with one end bent into a circular form at right angles to the shaft so that it will move freely in the annular space between the thermometer stem and the wall of the smaller test tube.

**E-3.5 Temperature Measurement Device** — either device described below has been found satisfactory.

**E-3.5.1 Thermometer** — Benzene freezing point thermometer having a range from 4.0 to 6.0°C and least count of 0.01°C and conforming to the following requirements:

Name	Solidification Point of Benzene
------	---------------------------------

Reference Fig. No.	10
Range, °C	4 to 6
Immersion	Total
a) Graduations, °C:	
i) Subdivisions	0.02
ii) Long lines at each	0.1
iii) Numbers at each	0.02
iv) Scale error, Max	0.04
b) Expansion chamber:	
i) Permit heating to	50 °C
ii) Total length, mm	210 to 220
iii) Stem OD, mm	6.0 to 7.0
iv) Bulb length, mm	25 to 35
v) Bulb OD, mm	6.0 to greater than stem
c) Scale location:	
i) Bottom of the bulb to line at	4 °C
ii) Distance, mm	110 to 115
iii) Length of graduated portion, mm	45 to 75

**E-3.5.2 Thermistor** — Type CSP, with accuracy of 0.01°C with a nominal resistance at 2500 Ω at 25°C. The thermistor shall be equipped with an ohmmeter capable of reading resistance to the nearest 0.1 Ω.

**E-3.6 Stirring Apparatus (Optional)** — The apparatus may be an acceptable replacement for manually stirring the benzene solution.

#### E-4 PREPARATION OF APPARATUS

**E-4.1** Fit the benzene container with a two-hole cork stopper. Through one hole insert the temperature measurement device. The thermometer should be inserted up to the 4.0°C mark. The thermistor should be inserted so as to contact the benzene solution. Through the other hole insert shaft of the stirrer.

**E-4.2** If using the benzene container (air jacketed), place a 3.2 mm layer of dry absorbent cotton or glass wool in the bottom of the larger test tube and insert the inner container up to the lip into a cork stopper or annular ring of cork that just fits into the mouth of the air jacket. Fig 5 shows the typical setup for this method.

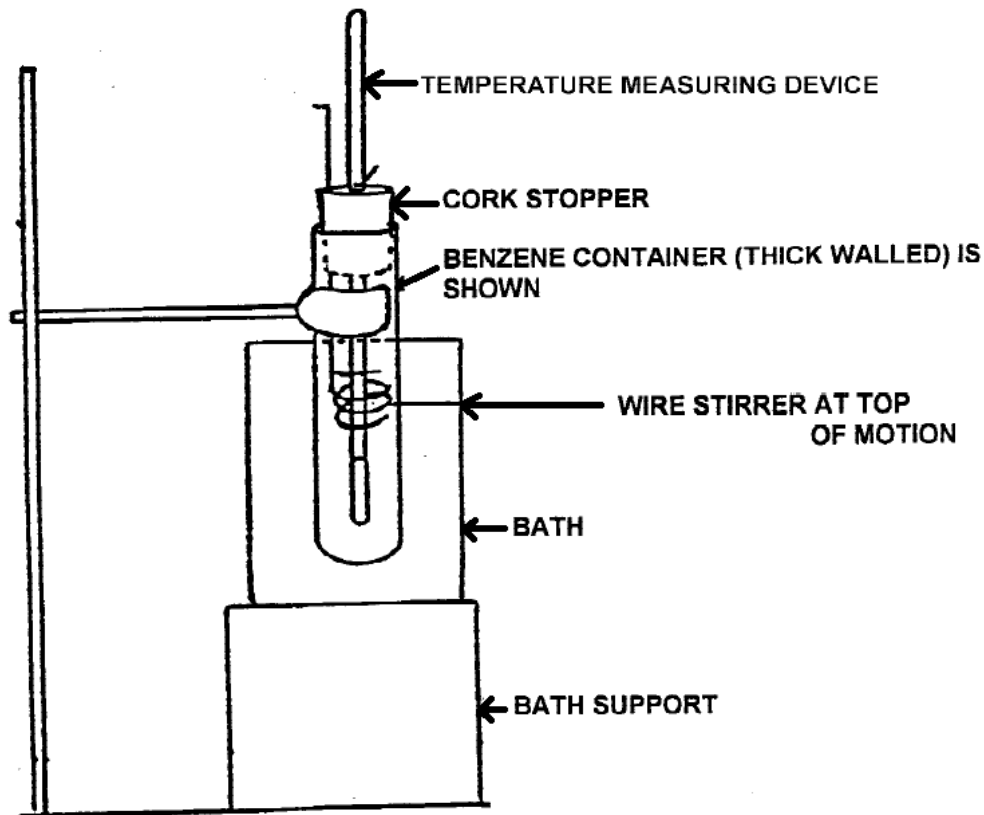


FIG. 5 BENZENE SOLIDIFICATION POINT APPARATUS

### E-5 PROCEDURE

**E-5.1** Saturate the sample of benzene with water by placing 7 to 8 ml of the sample in the benzene container, add one drop of water, and shake the tube and contents vigorously.

**E-5.2** Place the cork stopper onto the benzene container and onto the stirring apparatus, if available.

**E-5.3** When using the benzene container (air jacket), the operator may cool the smaller test tube and contents rapidly to about 6°C in the ice bath, while stirring. Wipe dry the outside of the smaller test tube and insert it into the larger test tube. Place the assembled test tubes in the ice bath.

**E-5.4** Stir the benzene continuously and observe the thermometer reading closely. The temperature will fall to a minimum, then rise to a maximum, remain constant at this maximum for approximately 15 s, and then fall again (*see Note*). The minimum temperature is due to super cooling before solidification starts and shall not be more than 0.7 °C below the maximum. Record the maximum constant temperature observed to the nearest 0.01 °C and designate it as 'wet' (*see Note*).

### NOTES

1. If distinct minimum and maximum points are not evident, or if the temperature does not remain constant at the maximum for at least 15 s, the determination shall be repeated.
2. The precision can be increased to  $\pm 0.01^{\circ}\text{C}$  by using a magnifying glass that ensures a reading perpendicular to the stem of the thermometer. In such cases it may be necessary to correct for stem exposure, that under ordinary conditions this correction will be less than  $0.01^{\circ}\text{C}$ .

## **E-6 EXPRESSION OF RESULTS**

Results shall be reported on the anhydrous basis. Since the determination is actually made on water-saturated benzene, the solidification point shall be corrected to the anhydrous basis by adding  $0.09^{\circ}\text{C}$  to the observed maximum temperature following the minimum corrections for the accuracy of the thermometer shall be made.

## **E-7 PRECISION**

Duplicate determinations on the same sample shall not differ by more than  $0.02^{\circ}\text{C}$  (*see E-5.4, Note*).

**E-7.1 Repeatability** — Results in the same laboratory should not be considered suspect unless they differ more than  $0.04^{\circ}\text{C}$ .

**E-7.2 Reproducibility** — Results submitted by each of two laboratories should not be considered suspect unless they differ by more than  $0.05^{\circ}\text{C}$ .

**ANNEX F**  
[Table 1, Sl No. (xii)]  
**DETERMINATION OF ACID WASH COLOUR**

**F-1 OUTLINE OF THE METHOD**

A mixture of the aromatic hydrocarbon and sulphuric acid is vigorously shaken and the colour of the acid layer is compared with that of colour standards prepared from  $\text{CoCl}_2$  and  $\text{FeCl}_3$ .

**F-2 SIGNIFICANCE**

It is used for setting specifications for benzene, toluene, xylene and other aromatic hydrocarbons. It may also be used as a quality control tool and in R&D work. The colour developed in the acid layer gives an indication of impurities which if sulfonated would cause the material to be discoloured.

**F-3 APPARATUS**

**F-3.1 Containers for Colour Standards** — Clear and unblemished, clean, French square, flint glass, flat-bottom, glass-stoppered, bottles holding 31 to 33 ml when filled to the neck. The bottles shall be numbered consecutively from 0 to 14.

**F-3.2 Test Containers** — Containers exactly like described in **F-3.1** except that each French square bottle shall be marked by etching to show when the bottle contains the volume of 7 and 28 ml, respectively. Coloured crayons and similar markers shall not be used for marking the bottles.

**F-4 REAGENTS**

**F-4.1 Hydrochloric Acid (1 : 39)** — mix 25 ml of hydrochloric acid (31 percent by mass, HCl) with 975 ml of water.

**F-4.2 Cobalt Chloride ( $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ )**

**F-4.3 Ferric Chloride ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ )**

**F-4.4 Potassium Chromate ( $\text{K}_2\text{CrO}_4$ )**

**F-4.5 Potassium Dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ )**

**F-4.6 Sulphuric Acid** (96 weight percent)

**F-5 PREPARATION OF REFERENCE COLOUR STANDARDS****F-5.1 Stock Solutions**

Prepare the following basic reagent solutions for use in preparing the reference colour standard.

**F-5.1.1 Solution A** — Dissolve 59.50 g of  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$  in HCl and make up to one litre in a volumetric flask with HCl.

**F-5.1.2 Solution B** — Dissolve 45.054 g of  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  in HCl and make up to one litre in a volumetric flask with HCl.

**F-5.1.3 Solution C** — Mix 3.5 percent volumes of Solution A with 36.5 volumes of Solution B and dilute with 90 volumes of water.

**F-5.1.4 Solution D** — Mix 3.5 volumes of Solution A with 36.5 volumes of Solution B.

**F-5.1.5 Solution E** — Prepare an aqueous solution of  $\text{K}_2\text{Cr}_2\text{O}_4$ , saturated at 21°C.

**F-5.1.6 Solution F** — Prepare an aqueous solution of  $\text{K}_2\text{Cr}_2\text{O}_7$ , saturated at 21°C and dilute with an equal volume of water.

**F-5.2** Prepare reference colour standard solutions having the following compositions and numbered from 0 to 14.

No. 0 — Distilled water.

No. 1 — 1 volume of Solution C plus 1 volume of water.

No. 2 — 5.5 volumes of Solution C plus 2 volumes of water.

No. 3 — Solution C.

No. 4 — 1 volume of Solution D plus 1 volume of water.

No. 5 — 5.5 volumes of Solution D plus 2 volumes of water.

No. 6 — Solution D.

No. 7 — 5 volumes of Solution E plus 2 volumes of water.

No. 8 — Solution E.

No. 9 — 7 volumes of Solution E plus 0.5 volume of Solution F.

No. 10 — 6.5 volumes of Solution E plus 1 volume of Solution F.

No. 11 — 5.5 volumes of Solution E plus 2 volumes of Solution F.

No. 12 — 1 volume of Solution E plus 1 volume of Solution F.

No. 13 — 2 volumes of Solution E plus 5 volumes of Solution F.

No. 14 — Solution F.

**F-5.3** Rinse the No. 0 container (*see F-3.1*) and its glass stopper three times with water, fill with water, and stopper. Rinse the No.1 container and its stopper three times with reference colour standard solution No.1 (*see F-5.2*), fill with this solution, and stopper. In this way prepare the set of containers of colour standards from 0 to 14 having the compositions shown for the corresponding colour solution standards in **F-5.2**. When filling the French square bottles, leave 6 mm of vapour space below the neck of the bottle. Seal each container with paraffin to prevent loss by evaporation or seepage.

NOTE— It is recommended to prepare the color standards from fresh solutions, every year.

**F-6 PROCEDURE**

**F-6.1** Rinse a test container (*see F-3.1*) twice with acid of the strength specified in Table 8 for the type of sample to be tested (*see Note 1*). Drain the rinsings and fill with the acid up to the 7 ml mark. Add sufficient sample to bring the total volume to the 28 ml mark (*see Note 2*). Insert the stopper, hold a finger over the stopper, and give vigorous shakes with a stroke of 13 to 25 cm, shaking for a total of 150 cycles over a period of 40 to 50 s that is at a rate of 3 to 3.75 cycles/s.

## NOTES

1. Concentrated sulphuric acid will cause severe burns on contact with the skin. As a precaution the test container should be wrapped in a towel or enclosed in a plastic bag during the shaking period.
2. If the room temperature is above 29°C, maintain the acid sample and reference colour standards at a temperature between 25°C and 29°C through the test, and insulate the test container in some convenient way, such as wrapping with a cloth, during the shaking period.

**F-6.2** Allow the container to stand protected from direct sunlight, for the period of time shown in Table 8. Without further delay invert the container gently once or twice to obtain a uniform colour in the acid layer, and compare the colour of the acid layer with that of the standards (*see F-5.3*). Make the comparison against a white background or against daylight, using transmitted light (*see Note*). When testing samples in Group 1 (*see Table 8*), observe the colour of the oil layer as well as that of the acid layer.

NOTE — Agreement of results may be improved by using a colour comparator of a suitable type for observing the colour of the acid layer in comparison with the reference standard colour solution.

**F-6.3** Designate the colour of the acid layer by the number of the nearest matching standard, following the number with a plus or minus sign if the sample is darker or lighter, respectively, than the standard. Disregard any difference in hue and determine only whether the colour of the acid layer is darker or lighter than the colour of the reference standard to which the sample most nearly corresponds. If the hue of the acid colour is different from the hue of the reference colour standard, record the colour number followed by (X). Thus 'No. 4 — (X)' means that the acid wash test colour is slightly lighter than No. 4 colour standard and that the hue of the No. 4 colour standard is not the same as the hue of the acid layer.

**F-7 INTERPRETATION OF RESULTS**

**F-7.1** Report sample (*see Table 8*) as passing the test only when the oil layer shows no change in colour and when the acid layer is not darker than the specified colour standard.

**Table 8 Acid Strength and Standing Times**

(Clauses F-6.1, F-6.2 and F-7.1)

Sample	Acid Strength, Percent	Standing Time, min
--------	---------------------------	-----------------------



(1)	(2)	(3)
Benzene, Toluene, Xylene (except Xylene, industrial grade), Any other most highly refined products	96	15

**F-7.2** A cloudiness of haze in the oil layer should not be interpreted as a change in colour.

### **F-8 PRECISION**

Precision data have not been established for all types of samples on which this test method is used.

Precision estimates are as follows:

<b>Aromatic hydrocarbon</b>	<b>Average Acid Wash Color</b>	<b>95 percent Repeatability</b>	<b>95 percent Reproducibility</b>
Benzene	1.4	0.75	2.34
	6.1	1.85	4.47
Xylene	4.7	0.4	1.39
	10.2	1.14	3.52

**ANNEX G**  
(*Clause 8*)  
**SAMPLING OF BENZENE**

**G-1 GENERAL REQUIREMENTS OF SAMPLING**

**G-1.1** In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed:

- a) Samples shall not be taken in an exposed place;
- b) The sampling instrument shall be clean and dry and shall be made of low or reduced spark generating material;
- c) The samples, the material being sampled, the sampling instrument and the containers for samples shall be protected from adventitious contamination;
- d) To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or stirring or both, or by rolling, so as to bring all portions into uniform distribution;
- e) The sample containers shall be of such a size that they are almost, but not completely, filled by the sample;
- f) The samples shall be placed in suitable, clean, dry and air-tight glass containers preferably of amber or blue colour;
- g) The sample container shall be sealed air tight with a suitable stopper after filling and marked with full details of sampling, such as the type of benzene, the date of sampling, the year of manufacture of material, the batch number, the name of the sampler etc. Particular care shall be taken to ensure that sealing methods do not contaminate the sample; and
- h) Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

**G-1.2 Additional Precautions**

The following additional precautions shall be observed:

- a) Rubber stoppers or composition corks shall not be used for closing the sample bottles;
- b) Sealing wax or other plastic material, if used, shall be applied in such a way that it does not contaminate the sample when the bottles are opened; and
- c) Each sample container shall be protected by covers of oil proof paper, metal foil, viscose or other suitable impervious material over the stopper to keep away moisture and dust from the mouth of the bottle and to protect it while being handled.

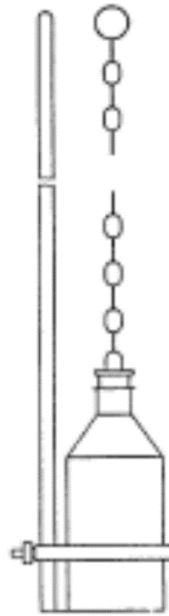
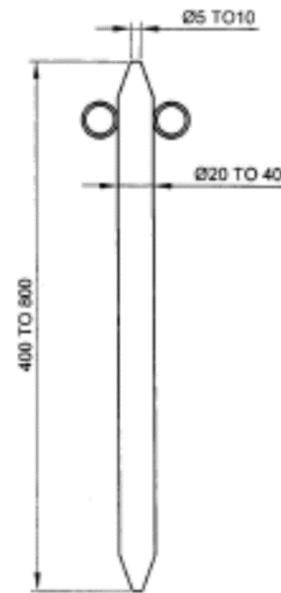


FIG. 7 SAMPLING BOTTLE OR CAN



All dimensions are in millimeters

FIG. 8 SAMPLING TUBE

## G-2 SAMPLING INSTRUMENT

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

- Sampling bottle or can for taking samples from various depths in large tanks, and
- Sampling tube.

**G-2.1.1** *Sampling Bottle or Can* — It consists of a weighed bottle or metal container with removable stopper or top, to which is attached a light chain (*see* Fig. 7). The bottle or can is fastened to a suitable pole. For taking a sample, it is lowered in the tank to the required depth, and the stopper or top is removed by means of the chain for filling the container.

**G-2.1.2** *Sampling Tube* — It is made of metal or thick glass and is about 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 8). The upper and lower ends are conical and reach 5 to 10 mm internal diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For taking a sample, the apparatus is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn. For small containers, the size of the sampling tube may be altered suitably

## G-3 SCALE OF SAMPLING

### G-3.1 Lot

In a single consignment, all the containers of the same size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of containers of different sizes or of different batches of manufacture, then the containers belonging to the same size and batch of manufacture shall be grouped together and each such group shall constitute a separate lot. In case the consignment is in large tanks or vessels, the tanks or vessels belonging to the same batch of manufacture shall constitute a lot. For ascertaining the conformity of the lot to the requirements of the specification, tests shall be carried out for each lot separately.

### G-3.2 Sampling from Containers

**G-3.2.1** The number of containers  $n$  to be selected for sampling shall depend on the size  $N$  of the lot and shall be in accordance with Table 10.

**Table 10 Scale of Sampling for Containers**  
(Clause G-3.2.1)

Sl No.	Lot Size (n)	No. of Containers to be Selected (N)
(1)	(2)	(3)
i)	Up to 100	5
ii)	101 to 200	6
iii)	201 to 300	7
iv)	301 to 400	8
v)	401 to 500	9
vi)	501 and above	10

NOTE — In the case of very small lots where the selection of the five containers may be uneconomical, all the containers shall be selected.

**G-3.2.2** The containers shall be selected at random. In order to ensure the randomness of selection, random sampling procedure given in IS 4905 may be adopted.

**G-3.3 Sampling from Tanks or Vessels** — Each of the tanks/vessels in the lot shall be sampled separately for determining the conformity of the lot to the requirements of the specification.

## G-4 PREPARATION OF THE TEST SAMPLES

### G-4.1 Test Samples from Containers

To ensure that the sample taken from each container is fairly representative, the contents shall be mixed thoroughly, when possible, by shaking or stirring or rolling. Draw small samples of the material from various depths with the help of the sampling tube (*see* Fig. 8). The approximate quantity of the material to be drawn from a container shall be nearly equal to thrice the quantity required for testing purposes as indicated in **G-5.1**.

**G-4.1.1** Out of the material drawn from individual containers, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample, sufficient for carrying out triplicate determinations for all the characteristics specified under **G-5**. The composite sample shall be divided into three equal parts, one for the purchaser and another for the supplier and the third for the referee.

**G-4.1.2** The remaining portion of the material from each container shall be divided into 3 equal parts, each forming an individual sample. One set of individual samples representing the containers n selected shall be for the purchaser, another for the supplier and the third for the referee.

**G-4.1.3** All the individual and composite samples shall be transferred to separate sample containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in **G-1.1(g)**.

**G-4.1.4** The referee test sample, consisting of a composite sample and a set of n individual samples, shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of any dispute.

#### **G-4.2 Test Samples from Tanks/Vessels**

**G-4.2.1** For drawing a sample from a tank/vessel, lower the closed sampling bottle or can (*see G-2.1*) slowly to the required depth, open and fill it at that depth. Three samples shall be obtained at levels of one-tenth of the depth of the liquid from the top surface (top sample), one half of the depth (middle sample) and nine-tenths of the depth of the liquid from the top surface (lower sample). All the three samples thus obtained from a tank/vessel shall be mixed together in a clean dry container, and shall be divided into three parts, one for the purchaser and another for the supplier and the third for the referee. Each of the tanks/vessels in the lot shall be sampled in the above manner and separate samples obtained for each of the tanks/vessels. The approximate quantity of the material to be drawn from a tank/vessel shall nearly be equal to thrice the quantity required for carrying out tests for all the requirements prescribed in **G-5**.

**G-4.2.2** All the samples thus obtained from the tanks/ vessels in the lot shall be transferred to separate sample containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in **G-1.1(g)**.

**G-4.2.3** The referee test samples consisting of the samples from the tanks/vessels in the lot, shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of any dispute.

#### **G-5 NUMBER OF TESTS**

##### **G-5.1 For Samples from Containers**

**G-5.1.1** Tests for the determination of distillation range, residue on evaporation, solidification point, aromatic impurities, non-aromatic hydrocarbons and aliphatic impurities shall be conducted on each of the individual samples separately (*see G-4.1.2*).

**G-5.1.2** Tests for the determination of all other characteristics given in Table 1 shall be conducted on the composite samples separately (*see G-4.1.1*).

**G-5.2 For Samples from Tanks/Vessels** — Tests for the determination of all the characteristics given in Table 1 shall be conducted on the samples from different tanks or vessels separately.

## **G-6 CRITERIA FOR CONFORMITY**

### **G-6.1 For Containers**

#### **G-6.1.1** *For Distillation Range*

##### **G-6.1.1.1** *For individual samples*

The lot shall be declared as conforming to the requirements of the distillation range if test results for each of the individual samples tested in respect of distillation range satisfy the requirements as given in Table 1, Sl No. (v).

**G-6.1.1.2** For residue on evaporation, solidification point, aromatic impurities and aliphatic impurities which have been tested on the individual samples, the mean ( $X$ ) and the range ( $R$ ) of test results, shall be computed (the range is defined as the difference between the maximum and the minimum values of test results):

- a) The lot shall be declared as conforming to the requirements of residue on evaporation, aromatic impurities and aliphatic impurities if the value of the expression  $(X + 0.6 R)$  as calculated from relevant test results are less than or equal to the respective values for each of the requirement given in Table 1.
- b) The lot shall be declared as conforming to the requirements of solidification point if the value of the expression  $(X - 0.6 R)$  as calculated from relevant test results is equal to or more than the respective values given in Table I.

##### **G-6.1.1.3** *For composite samples*

In respect of all other characteristics, the lot shall be considered as conforming, if the composite sample satisfies each one of these requirements.

### **G-6.2 For Tanks/Vessels**

The lot shall be declared as conforming to the specified requirements for various characteristics, if each of the test results satisfies the relevant requirements specified in the standard individually

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