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*Draft Indian Standard*

**PHENYL ETHYL METHYL ETHER — SPECIFICATION**

*(Second Revision of IS 7697)*

ICS No. 71.100.60

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Fragrance and Flavour Sectional Committee  
PCD 18

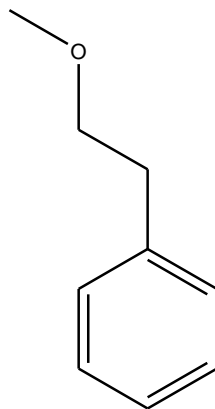
Last date for comments:  
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**FOREWORD**

*(Formal clauses will be added later)*

Phenyl ethyl methyl ether, C<sub>9</sub>H<sub>12</sub>O also known as 2-methoxyethylbenzene or pandanol is prepared by the methylation of phenyl ethyl alcohol. It gives a green top note resembling the top note of *KEWDA* flower in which it occurs. It is represented by the following structural formula:



Molecular Mass 136 (C<sub>9</sub>H<sub>12</sub>O)  
Phenyl Ethyl Methyl Ether

This standard was first published in 1975. At that time due to non-availability of a standardized test procedure for chromatographic analysis of this material, it was decided GLC method would be incorporated at a later date. This standard was first revised in 1991 to include gas chromatographic method of analysis as the main method for determination of purity of phenyl ethyl methyl ether. Besides, two new requirements namely minimum purity and peroxide value were included in the first revision.

In this (second) revision, the requirement of boiling point and an alternative method for determination of relative density using digital density meter have been incorporated. The gas chromatographic analysis for purity determination of phenyl ethyl methyl ether has also been upgraded from Packed Column GC to Capillary Column GC which gives more accurate results.

The Committee responsible for the formulation of this standard noted that as a practice, the fragrance and flavor ingredients are traded at international level quoting the relative density and refractive index at 20°C rather than at 27°C as prescribed in the Indian standards. Therefore, the Committee felt that the standard should have values for these important characteristics at 27°C (to cater to domestic needs) and 20°C (to facilitate international trade).

The composition of the Committee, responsible for the formulation of this standard is given at Annex E (will be added later).

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 of 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 shall be the same as that of the specified value in the standard.

## 1 SCOPE

This draft standard prescribes the requirements and the methods of sampling and test for phenyl ethyl methyl ether.

## 2 REFERENCE

The following Indian Standards are necessary adjuncts to this standard:

IS No.	Title
IS 326	Methods of sampling and test for natural and synthetic perfumery materials
(Part 1): 2022	Sampling ( <i>fourth revision</i> )
(Part 2): 2023	Preliminary examination of perfumery materials and samples( <i>third revision</i> )
(Part 3): 2006	Determination of relative density ( <i>third revision</i> )
(Part 5): 2006	Determination of refractive index ( <i>third revision</i> )
(Part 6): 2005	Evaluation of miscibility in ethanol ( <i>third revision</i> )
(Part 7) : 2006	Determination of acid value ( <i>Third Revision</i> )
Part 9/Sec 2) : 2017	Determination of ester value after acetylation and free alcohols, Sec 2 Estimation of free alcohols content by determination of ester value after acetylation ( <i>third revision</i> )
(Part 26) : 2017	Methods of sampling and test for natural and synthetic perfumery material: Part 26 general guidance on determination of flashpoint
IS 2284 : 1988	Method for olfactory assessment of natural and synthetic perfumery materials ( <i>first revision</i> )
IS 6597 : 2001	Glossary of terms relating to natural and synthetic perfumery materials ( <i>second revision</i> )

## 3 TERMINOLOGY

For the purpose of this standard, the definitions of terms given in IS 6597 shall apply.

## 4 REQUIREMENTS

### 4.1 Description

Phenyl ethyl methyl ether shall be a clear colourless liquid, free from sediment, suspended matter, separated water and adulterants.

### 4.2 Solubility

When tested as prescribed in IS 326 (Part 6), the material shall be clearly soluble in 9 volumes of 70 percent ethyl alcohol or 3 volumes of 80 percent ethyl alcohol.

4.3 The material shall also conform to the requirements given in Table 1.

NOTE — In the event of any dispute, method prescribed in IS 326 (Part 3) shall be treated as referee method.

**Table 1 Requirements for Phenyl Ethyl Methyl Ether**  
(Clause 4.3)

SI No.	Characteristic	Requirement	Method of Test, ref to Annex /IS
(1)	(2)	(3)	(4)
i)	Colour and appearance	Clear colourless liquid	Visual Observation
ii)	Odour	Typical strong green note resembling KEWDA flower	IS 2284
iii)	Relative Density		Annex A /IS 326 (Part 3) <sup>(1)</sup>
	at 20 °C	0.946 to 0.954	
	at 27 °C	0.938 to 0.941	
iv)	Refractive index		IS 326 (Part 5)
	at 20 °C	1.497 to 1.509	
	at 27 °C	1.496 to 1.500	
v)	Acid value, <i>Max</i>	0.5	IS 326 (Part 7)
vi)	Boiling point, °C	186 to 192	Annex B
vii)	Peroxide value	10	Annex C
viii)	Purity, percent by mass, <i>Min</i>	99	Annex D
ix)	Flash point, °C Whether Normative or Informative Requirement	(+) 61.5 °C <sup>(2)</sup>	IS 326 (Part 26)

NOTES—

(1) In the event of any dispute, method prescribed in IS 326 (Part 3) shall be treated as referee method.

(2) Value recorded in continuous closed cup Flash point apparatus

## 5 PACKING AND MARKING

### 5.1 Packing

The material shall be supplied in well closed containers, preferably glass, tin-lined, stainless steel or aluminum, as agreed to between the purchaser and the supplier. The material shall be protected from light and stored in a cool and dry place.

## 5.2 Marking

Each container so filled shall bear legibly and indelibly the following information:

- a) Name of the material
- b) Indication of the source of manufacture
- c) Batch number and date of manufacture
- d) Net and gross mass
- e) Net Volume of the material
- f) BIS Certification Marking

The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations made there under. Details of conditions under which a licence for the use of Standard Mark may be granted to manufactures or producers, may be obtained from the Bureau of Indian Standards.

## 6 SAMPLING

6.1 Representative samples of phenyl ethyl methyl ether shall be drawn as prescribed in IS 326 (Part 1).

6.2 Tests for all the requirements shall be carried out on a composite sample.

6.3 *p*-phenyl ethyl methyl ether shall be taken to have conformed to this standard if the composite sample passes all the tests.

## 7 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 result of analysis.

## ANNEX A

[Clause 4.3, Table 1, Sl No. (iii)]

### DETERMINATION OF RELATIVE DENSITY USING DIGITAL DENSITY METER

#### A-1 PRINCIPLE

Digital Density meter operates on U-tube oscillation technology. The extremely fine capillaries are made to oscillate by a piezoelectric or magnetic transducer with a characteristic frequency. The

resulting resonant frequency of the U-tube will depend on the mass of the filled sample. This frequency can be measured very accurately and used to calculate the density of the sample.

## **A-2 APPARTUS**

**A-2.1 Density Meter**– It should have Oscillating U-tube with full range viscosity correction and reference oscillator allows long term calibration stability and measurement at all temperatures with a single calibration

### **A-2.2 Sensitivity**

#### **A-2.2.1 Accuracy**

Density — 0.0001 g/cm<sup>3</sup>  
Temperature — 0.05°C

#### **A-2.2.2 Repeatability**

Density— 0.00005 g/cm<sup>3</sup>  
Temperature— 0.02°C

#### **A-2.2.3 Resolution**

Density — 0.0001 g/cm<sup>3</sup>  
Temperature— 0.01°C

## **A-3 REAGENTS**

**A-3.1 Acetone**, AR Grade for cleaning U tube after use.

## **A-4 CALIBRATION**

Calibration of density meter shall be done on weekly basis with Ultra-pure water at 25°C. Acceptance criteria shall be 0.99704±0.0010.

## **A-5 GENERAL PROCEDURE**

**A-5.1** Carefully fill the syringe with a representative sample. Try to fill the syringe without any bubbles. If bubbles are present, hold the syringe with the tip upright and allow bubbles to float to the top and then push them out using the plunger.

**A-5.2** Insert the syringe in the inlet nozzle and push the plunger slowly till the liquid just comes out of the outlet nozzle. Let the syringe there, till the complete analysis.

**A-5.3** Wait for stabilization and till reading get display. Note down the reading.

**A-5.4** Wash the U tube twice with solvent at the end of test.

## **ANNEX B**

[Clause 4.3 Table 1, SI No. (vii)]

## DETERMINATION OF BOILING POINT/DISTILLATION RANGE

### B-1 OUTLINE OF THE METHOD

The range of temperature between which a liquid boils or the percentage of the material that distills between two specified temperatures is determined. The lower of the two temperatures is the corrected thermometer reading when the first five drops of distillate have been collected, and the upper temperature is the corrected reading when the percentage specified has been collected.

### B-2 APPARATUS

**B-2.1 Distillation Flask** — Having a bulb of 50 to 60 ml capacity and a neck 10 to 12 mm long and 14 to 16 mm in internal diameter.

**B-2.2 Straight Glass Condenser** — With a water jacket 40 to 60 cm long, the distance from the upper end of the jacket to the neck of the flask being 18 to 25 cm.

**B-2.3 Asbestos Board** — 12 to 15 cm<sup>2</sup> and 3 to 5 mm thick and having a circular perforation, located centrally, to hold the flask. The edge of the perforation shall fit the flask closely when the latter is set into it. The size of the perforation shall be such that when the flask is set into it, the portion of the flask below the upper surface of the asbestos has a capacity of 3 to 4 ml.

### B-2.4 Thermometer

Thermometer conforming to the following requirements is recommended. When placed in position, the top of the bulb of the thermometer is levelled with the centre of the opening of the outlet tube:

Range	-2°C to + 300°C
Graduation	1°C
Immersion	Total
Overall length	380 ± 10 mm
Stem diameter	5.5 to 8.0 mm
Bulb shape	Cylindrical
Bulb length	8 to 16 mm
Bulb diameter	Not less than 5.5 and not greater than stem
Length of graduated portion	223 to 254 mm
Distance from bottom of bulb to 0 <sup>0</sup> C	100 to 110 mm
Longer lines at each	5°C
Figured at each	10°C
Expansion Chamber	Required
Top finish	Ring
Scale error not to exceed	±0.5°C up to 150°C ±1°C above 150°C

Any other thermometer of similar range and accuracy may be used.

### B-3 PROCEDURE

**B-3.1** Place the asbestos board on a tripod or other suitable support. Transfer to the distillation flask 25 ml of the liquid to be tested, insert the thermometer and place the flask in an up-right

position in the perforation of the asbestos board. Connect the distillation flask with the source of heat and distill at the rate of 4 to 5 ml per minute, noting the temperature as soon as 5 drops of the liquid have distilled into the receiver, and when the specified percentage has distilled over.

## ANNEX C

[Clause 4.3, Table 1, SI No. (vi)]

### DETERMINATION OF PEROXIDE VALUE

#### C-1 REAGENTS

**C-1.1 Acetic Acid**—Analytical reagent grade.

**C-1.2 Chloroform**

**C-1.3 Sodium Thiosulphate Solution**— 0.01 N approximately.

**C-1.4 Potassium Iodide**— Saturated solution.

**C-1.5 Starch**— Freshly prepared saturated solution.

#### C-2 PROCEDURE

**C-2.1** Weigh accurately about 2 g of the material into a conical flask (250 ml capacity) with provision for stoppering. Add 25 ml of acetic acid chloroform mixture solution (3:2 v/v). Swirl to dissolve the material completely, Add 2 ml of freshly prepared saturated aqueous solution of potassium iodide. Allow the solution to stand with occasional shaking for 1 min and then add 35 ml of distilled water. Add 2 ml of freshly prepared starch solution. Titrate against sodium thiosulphate (0.01 N). End point being the disappearance of the blue colour. A blank titration using all the reagents but without sample should be carried out.

#### C-4 CALCULATION

**C-4.1** Calculate the peroxide value in the material as follows:

$$\text{Peroxide value, milliequivalents of peroxy oxygen/kg} = \frac{(V_1 - V_2) \times N \times 1000}{M}$$

where

$V_1$  = volume, in ml, of sodium thiosulphate solution used for the test sample;  
 $V_2$  = volume, in ml, of sodium thiosulphate solution used for blank titration;  
 $N$  = normality of sodium thiosulphate; and  
 $M$  = mass in g, of the test sample.

## ANNEX D

[Clause 4.3, Table 1, SI No. (viii)]

### GAS CHROMATOGRAPHIC ANALYSIS OF PARA-CRESYL METHYL ETHER

**D-1**The chromatographic conditions given here are for guidance only.

## D-2 Outline of the Method

**D-2.1** Gas Chromatography (GC) is an analytical instrument used for separating and analyzing volatile substances in a gas phase. A sample is rapidly vaporized at the injection port. The sample is carried through the heated column by a chemically inert mobile phase. The sample components are separated based on the boiling points and relative affinity to the stationary phase within the column. The sample components are detected at the detector and presented as peaks on a chromatogram.

**D-2.2** This method describes open-tubular, capillary column gas chromatography procedures for the analysis of Phenyl Ethyl Methyl Ether, using either single-column or dual-column/dual-detector. A neat sample of the material shall be injected into the gas chromatograph, from where it is carried by the carrier gas from one end of the column to the other.

**D-2.3** Equal weight of samples (phenyl ethyl methyl ether; 5mg) and internal standard (nonane; 5mg) were dissolved in dichloromethane (1 mL) and injected in a gas chromatograph where it was carried by the carrier gas (nitrogen) from one end of the column to the other. During its movement, the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column. The detector signals on transmission to the recorder plots the chromatogram.

## D-2 APPARATUS

**D-2.1 GAS CHROMATOGRAPH** — A gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. The typical chromatogram for phenyl ethyl methyl ether using a chromatograph with the following chromatographic conditions is shown in Fig. 1. This is a general user guide for any typical gas chromatography equipped with Flame Ionization Detector (FID).

NOTE — Gas chromatograph should have features of accurate temperature controls and precise injection systems with suitable inlet port for capillary column installation.



## D-2.2 GC CONDITIONS FOR NON-POLAR CAPILLARY COLUMN

Sl No.	Description	Parameter			
1	<i>Column Conditions</i>				
i)	Material	5% diphenyl non-polar fused silica capillary column			
ii)	Size	30 m (length) × 0.25 mm (ID)			
iii)	thickness	0.25 µm			
2	Sample size	0.2 µl (Neat)			
3	<i>Gas Conditions</i>				
i)	Carrier gas	Hydrogen (99.999 percent pure)			
ii)	Carrier flow	1.0 ml/ min (Constant flow)			
iii)	Injection port temperature	250 °C			
iv)	Detector port temperature	280 °C			
v)	Hydrogen flow	30 mL/min			
vi)	Zero air flow	300 mL/min			
vii)	Make up flow	29 mL/min			
4	Split Ratio	100:1			
5	<i>Oven Conditions</i>	Rate (°C/min)	Temperature (°C)	Time (min)	Total (min)
i)	Initial		60	0.00	0.00
a)	Ramp # 1	3.0	240	2.00	62.00
b)	Ramp # 2	10.0	310	1.00	70.00

NOTE — The above gas chromatographic conditions are suggestive /typical. However, any GC with equivalent column may be used provided standardization/calibration is done after setting up chromatographic conditions for the desired/required resolution.

## D-3 REAGENTS

**D-3.1** During the course of analysis, unless specified otherwise, use only reagent of recognized analytical grade.

**D-3.2** All suitable gases of high purity, depending on the type of detector shall be used.

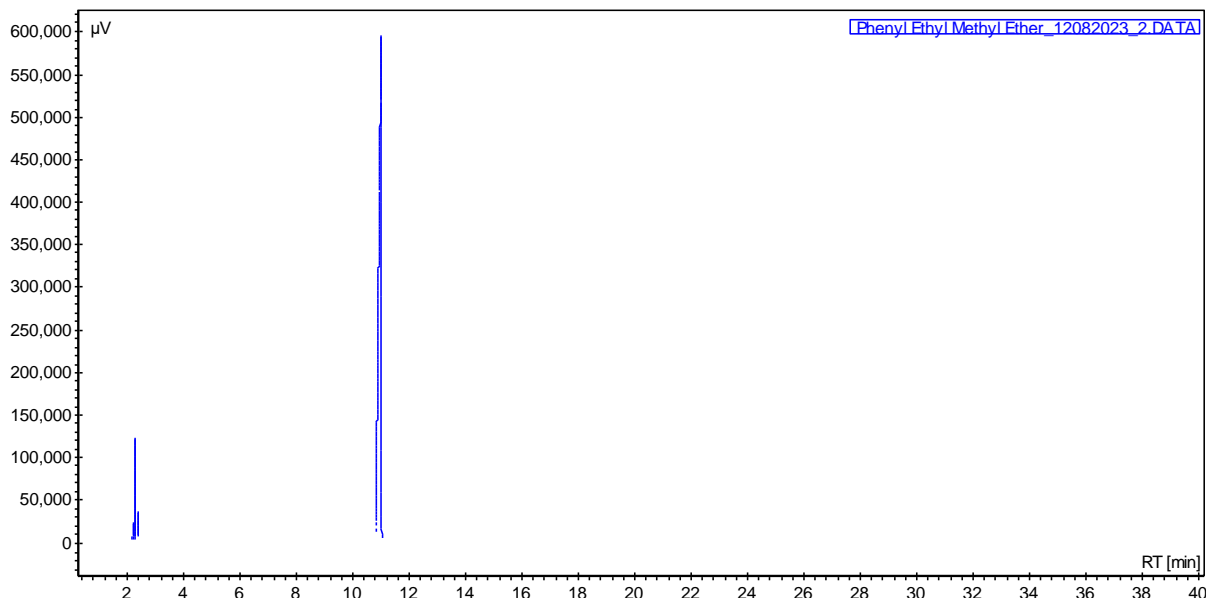


Fig. 1 A typical Chromatogram of phenyl ethyl methyl ether

#### D-4 CALCULATION

##### D-4.1 Area Measurement (*see* Note 1)

Since normal peaks approximate a triangle, the area is measured by multiplying the peak height with the width of half-height. Normal peak base is not taken since large deviations may be observed due to tailing or adsorption. This technique is rapid, simple and fairly accurate when peaks are symmetrical and of reasonable width.

##### D-4.2 Area Normalization (*see* Note 2)

By normalizing, it is meant, calculating the percentage composition by measuring the area of each and dividing the individual areas by total area, for example,

$$\text{Percentage of A, peak} = \frac{\text{Area of A}}{\text{Total Area}} \times 100$$

where,  $A$  = Area count of phenyl ethyl methyl ether

NOTES —

1 Other methods of area measurements, namely, triangulation, discintegrator and electronic digitalintegrator, if fixed with GC machine, would be of great advantage.

2 Internal standardization method may be used if pure appropriate internal standard is available. This method is known as relative or indirect calibration.

Percentage weight for component was calculated from the predicted RRF (relative response factor) using *n*-nonane as reference standard.

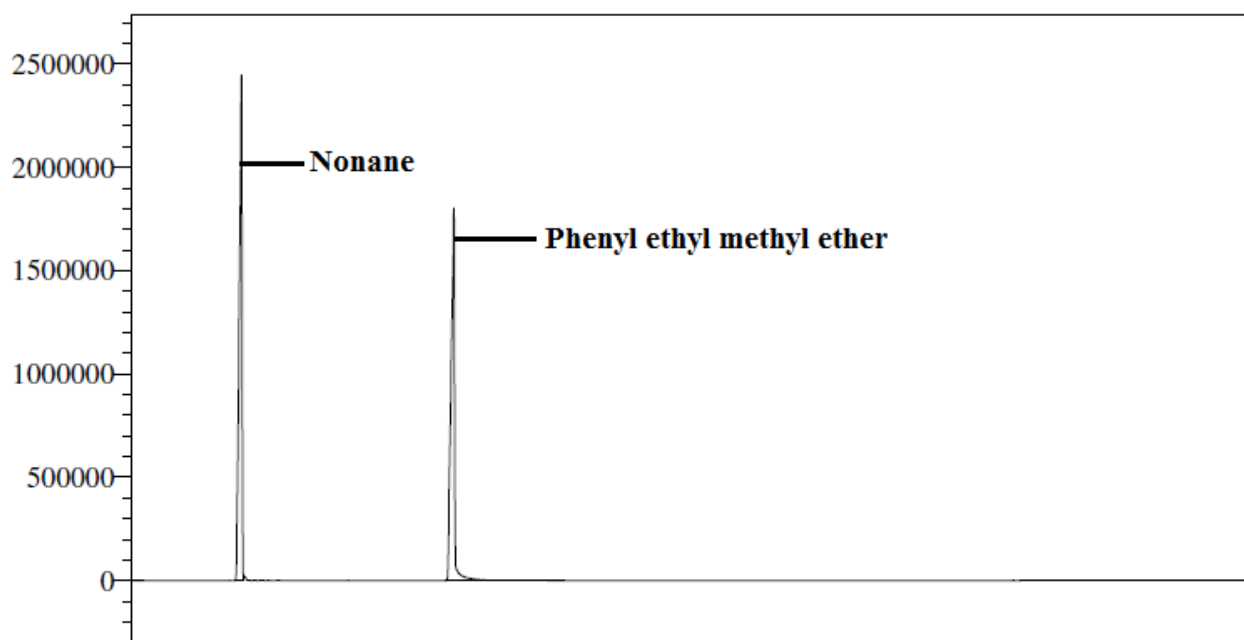


FIG. 1 A typical chromatogram of Phenyl ethyl methyl ether

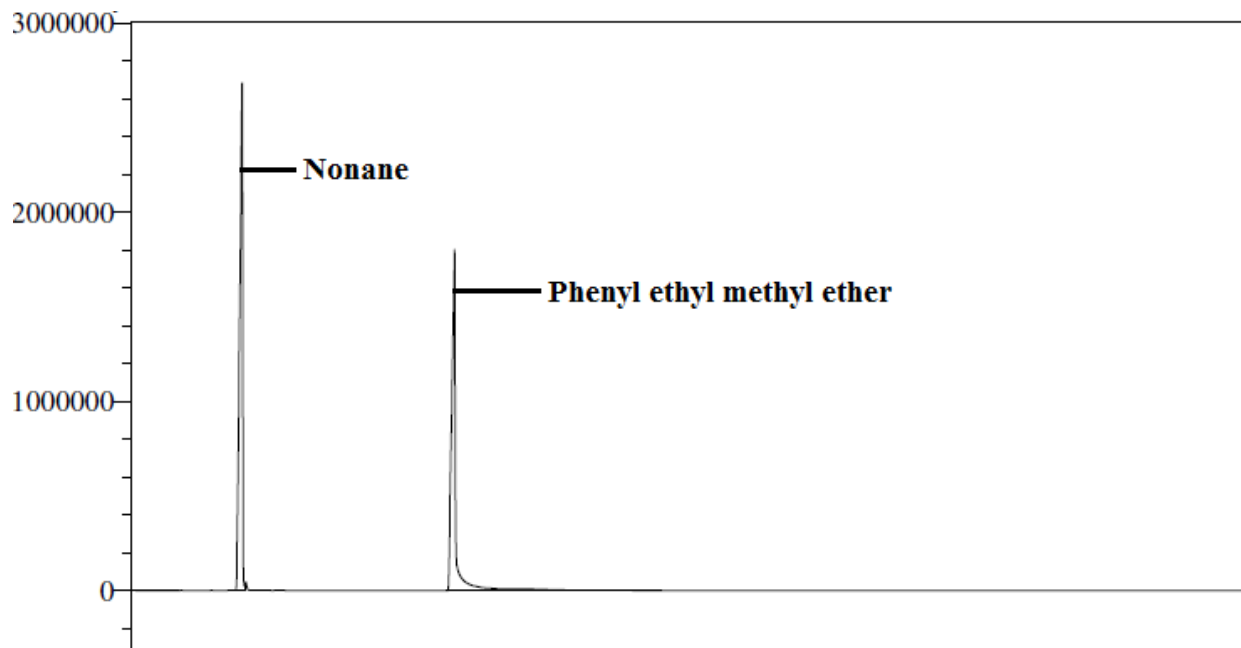


Fig. 2 A typical chromatogram of Phenyl ethyl methyl ether (Ultra 2)

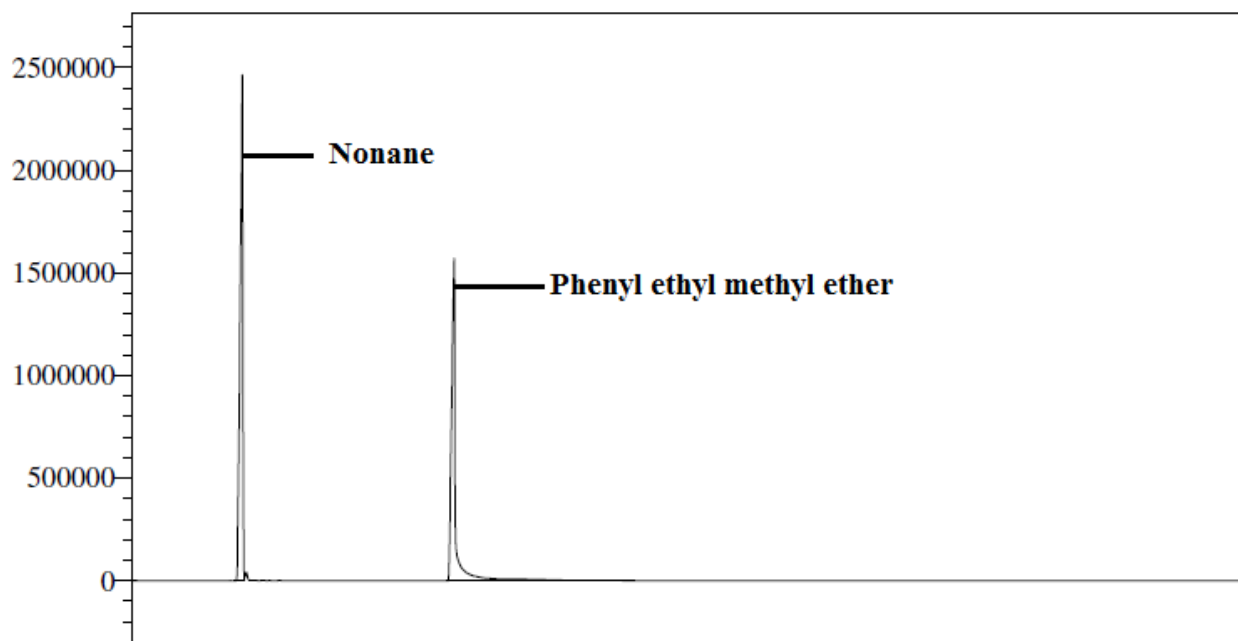


Fig. 3 A typical chromatogram of Phenyl ethyl methyl ether (Kelkar)