भारतीय मानक

Indian Standard

IS 7696 : 2023

पैरा-क्रिसाईल मिथाईल ईथर — विशिष्टि

(दूसरा पुनरीक्षण)

Para-Cresyl Methyl Ether — Specification

(Second Revision)

ICS 71.100.60

भारतीय मानक ब्यूरो
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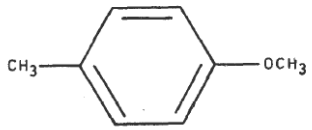
April 2023 **Price Group 7**

Fragrance and Flavour Sectional Committee, PCD 18

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Fragrance and Flavour Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Para-cresyl methyl ether ($C_8H_{10}O$), also known as methyl para-cresol is usually prepared by the methylation of para-cresol. It is also present in oil of ylang ylang, cananga and others. Its structural formula is as follows:



PARA-CRESYL METHYL ETHER
(Molecular Mass 122·17)

It is of considerable importance in perfumery and is used in many floral compositions. It is also used to a limited extent in flavours.

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 para-cresyl methyl ether. Besides, two new requirements namely minimum purity and peroxide value were included in this revision.

In this (*second revision*), an alternative method for determination of relative density using digital density meter has been incorporated. The gas chromatographic analysis for purity determination of *p*-cresyl methyl ether has also up-graded from Packed Column GC to Capillary Column GC which gives more accurate results. Also, the maximum limit for peroxide value has been updated to 3 ppm. in this revision.

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 ºC rather than at 27ºC as prescribed in the Indian Standards. Therefore, the committee felt that the standard should have values for these important characteristics at 27 ºC (to cater to domestic needs) and 20 ºC (to facilitate international trade).

The composition of the committee, responsible for the formulation of this standard is listed in Annex F.

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.

Indian Standard

PARA-CRESYL METHYL ETHER — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for para-cresyl methyl ether.

2 REFERENCES

The standards listed below contain provisions which, through reference in text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All the 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 listed below:

IS No.	*Title*
IS 326	Methods of sampling and test for natural and synthetic perfumery materials
(Part 1) : 2022	Sampling (*fourth revision*)
(Part 2) : 1980	Preliminary examination of perfumery materials and samples (*second revision*)
(Part 3) : 2006	Determination of relative density (*third revision*)
(Part 5) : 2006	Determination of refractive index (*third revision*)
(Part 6) : 2005	Evaluation of miscibility in ethanol (*third revision*)
(Part 9/Sec 2) : 2017	Determination of ester value after acetylation and free alcohols, Section 2 Estimation of free alcohols content by determination of ester value after acetylation (*third revision*)
IS 2284 : 1988	Method for olfactory assessment of natural and synthetic perfumery materials (*first revision*)
IS 6597 : 2001	Glossary of terms relating to natural and synthetic perfumery materials (*second revision*)

3 TERMINOLOGY

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

4 REQUIREMENTS

4.1 Description

Para-cresyl 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.

5 PACKING AND MARKING

5.1 Packing

The material shall be supplied in well closed containers, preferably glass, tin-lined, stainless steel or aluminium, 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; and
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 thereunder. 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 para-cresyl 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*-Cresyl 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 : 1992 'Reagent grade water — Specification (*third revision*)' shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

Table 1 Requirements for Para-Cresyl Methyl Ether
(*Clause* 4.3)

Sl No.	Characteristic	Requirement	Method of Test, ref to
(1)	(2)	(3)	(4)
i)	Colour and appearance	Colourless liquid	Visual Observation
ii)	Odour	Characteristic pungent odour suggestive of ylang ylang, free from objectionable by odours such as cresylic, etc	IS 2284
iii)	Relative Density		Annex A/ IS 326 (Part 3)
	at 20 ºC	0.963 0 to 0.973 0	
	at 27 ºC	0.964 7 to 0.9687	
iv)	Refractive index		IS 326 (Part 5)
	at 20 ºC	1.510 0 to 1.513 0	
	at 27 ºC	1.509 0 to 1.512 0	
v)	Free *p*-cresol, percent by mass, *Max*	2.0	Annex B
vi)	Peroxide value, *Max*	3.0	Annex C
vii)	Boiling point, ºC	175 to 176	Annex D
viii)	Purity, percent by mass, *Min*	98	Annex E

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

ANNEX A
[*Clause* 4.3, *Table* 1, and *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 — 0.000 1 g/cm³
Temperature — 0.05 °C

A-2.2.2 *Repeatability*

Density — 0.000 05 g/cm³
Temperature — 0.02°C

A-2.2.3 *Resolution*

Density — 0.000 1 g/cm³
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 0.997 04 ± 0.001 0.

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, and *Sl No*. (v)]

DETERMINATION OF FREE PARA-CRESOL

B-1 PROCEDURE

Weight accurately about 5 g of sample into a 150 ml glass-stoppered flask. Pipette 10 ml of 10 percent acetic anhydride solution in anhydrous pyridine into the flask. Using the same pipette, transfer 10 ml of acetic anhydride solution into a second flask containing no sample and treat it as a blank. Connect each flask to a water-cooled condenser and reflux for one hour. A guard tube filled with fused calcium chloride should be fixed to the open end of condenser. Discontinue heating the flasks for a few minutes until the temperature of the mixture is below 100°C and then add 25 ml of 50 percent ethyl alcohol-water mixture down the condenser into each flask. Reflux again for 10 minutes to hydrolyze the acetic anhydride. Allow to cool slightly and disconnect the flasks. When the blank and samples are cool, titrate with 0.5 N potassium hydroxide solution to the end point of phenolphthalein indicator.

B-2 CALCULATION

B-2.1 Free para-cresol, percent by mass

$$\frac{5.407 \times N(V_2 - V_1)}{M}$$

where

N = normality of the potassium hydroxide solution used;

V_1 = volume, in ml, of potassium hydroxide solution used in the blank determination;

V_2 = volume, in ml, of potassium hydroxide solution used in the test sample solution; and

M = mass, in g, of the test sample.

ANNEX C

[*Clause* 4.3, *Table* 1, and *Sl 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 minute 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-3 CALCULATION

C-3.1 Calculate the peroxide value in the material as follows:

Peroxide value, milli equivalents/ kg =

$$\frac{(V_1 - V_2) \times N \times 1\,000}{M}$$

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, and *Sl No*. (vii)]

DETERMINATION OF BOILING POINT/ DISTILLATION RANGE

D-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.

D-2 APPARATUS

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

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

D-2.3 Asbestos Board — 12 cm^2 to 15 cm^2 and 3 mm 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 ml to 4 ml.

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

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

D-3 PROCEDURE

D-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 ml/min to 5 ml/min, 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 E

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

GAS CHROMATOGRAPHIC ANALYSIS OF PARA-CRESYL METHYL ETHER

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

E-2 Outline of the Method

E-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 though 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.

E-2.2 This method describes open-tubular, capillary column gas chromatography procedures for the analysis of Para- Cresyl 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.

E-3 APPARATUS

E-3.1 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 *p*-cresyl 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.

E-3.2 Instrument Specifications

The instrument specification shall be as listed below:

Description	*Parameter*			
Column Conditions				
Material	100 percent Dimethyl polysiloxane Non-polar and low bleed with a high temperature limit			
Size	30 m (length) × 0.25 mm (ID)			
Thickness	0.25 µm			
Sample size	0.2 µl			
Gas Conditions				
Carrier gas	Nitrogen (99.999 percent pure)			
Carrier flow (ml/ min)	1.0, Constant flow			
Injection por temperature ºC	250			
Detector por temperature, ºC	280			
Hydrogen flow, l/min	40			
Zero air flow, ml/min	400			
Make up flow, l/min	30			
Split Ratio	150 : 1			
Oven Conditions	Rate, ºC /min	Temperature, ºC	Hold time, min	Run time, min
Initial	—	70	0	0

Description	*Parameter*			
Ramp # 1	5	150	0	16
Ramp # 2	25	280	0	21.2

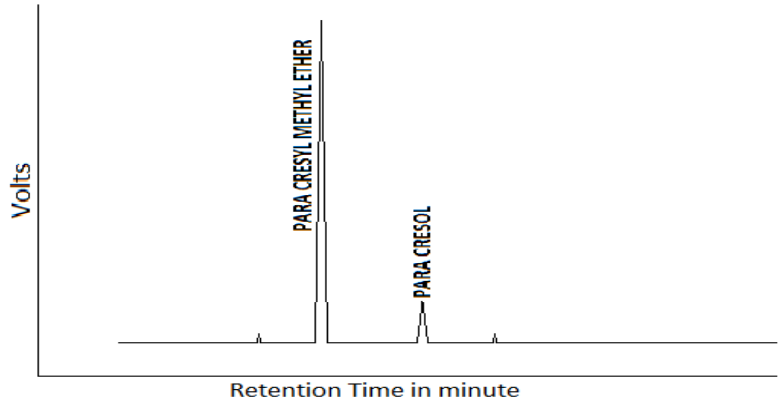


FIG. 1 A TYPICAL CHROMATOGRAM OF PARA-CRESYL METHYL ETHER

E-4 REAGENTS

E-4.1 During the course of analysis, unless specified otherwise, use only reagent of recognized analytical grade.

E-4.2 All suitable gases of high purity, depending on the type of detector shall be used.

E-5 CALCULATION

E-5.1 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. The 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.

E-5.2 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,

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

where

A = Area count of Para-Cresyl methyl ether.

NOTES

1 Other methods of area measurements, namely, triangulation, disc integrator and electronic digital integrator, if fixed with GLC 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.

ANNEX F

(*Foreword*)

COMMITTEE COMPOSITION

Fragrance and Flavour Sectional Committee, PCD 18

Organization	*Representative*(*s*)
CSIR-Central Institute of Medical and Aromatic Plants, Lucknow	DR PRABODH K. TRIVEDI **(*Chairperson*)**
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Member Secretary
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SCIENTIST 'B'/ASSISTANT DIRECTOR
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Bureau of Indian Standards

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Review of Indian Standards

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Amend No.	Date of Issue	Text Affected

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