भारतीय मानक

Indian Standard

IS/IEC 60867 : 2022

इन्सुलेट तरल पदार्थ — सिंथेटिक सुगंधित हाइड्रोकार्बन पर आधारित अप्रयुक्त तरल पदार्थ — विशिष्टि

Insulating Liquids — Unused Liquids Based on Synthetic Aromatic Hydrocarbons — Specification

ICS 29.040.10

भारतीय मानक ब्यूरो
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NATIONAL FOREWORD

This Indian Standard which is identical to IEC 60867 : 2022 'Insulating liquids — Specifications for unused liquids based on synthetic aromatic hydrocarbons' issued by the International Electrotechnical Commission (IEC) was adopted by the Bureau of Indian Standards on the recommendation of the Fluids for Electrotechnical Applications Sectional Committee and approval of the Electrotechnical Division Council.

This Indian Standard covers specifications and test methods for unused synthetic aromatic hydrocarbons intended for use as insulating liquid in cables and capacitors.

The text of IEC standard has been approved as suitable for publication as an Indian Standard without deviations. Certain terminologies and conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'; and

b) Comma (,) has been used as a decimal marker, while in Indian Standards the current practice is to use a point (.) as the decimal marker.

In this standard, reference appears to International Standards for which Indian Standards also exists. The corresponding Indian Standards, which are to be substituted, are listed below along with their degree of equivalence for the editions indicated:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 2592 Petroleum and related products — Determination of flash point — Cleveland open cup method (PMOC)	IS 1448 (Part 69) : 2019/ISO 2592 : 2017 Methods of test for petroleum and its products: Part 69 Determination of flash and fire points — Cleveland open cup method (*second revision*)	Identical
ISO 3016 Petroleum and related products from natural or synthetic sources – Determination of pour point	IS 1448 (Part 10/Sec 2) : 2021/ISO 3016 : 2019 Methods of test for petroleum and its products: Part 10 Petroleum and related products from natural or synthetic sources, Section 2 Determination of pour point (*third revision*)	Identical
ISO 3104 Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity	IS 1448 (Part 25/Sec 1) : 2018/ISO 3104 : 1994 Methods of test for petroleum and its products: Part 25 Transparent and opaque liquids, Section 1 Determination of kinematic viscosity and calculation of dynamic viscosity (*second revision*)	Identical

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 3675 Crude petroleum and liquid petroleum products — Laboratory determination of density — Hydrometer method	IS 1448 (Part 16) : 2014/ISO 3675 : 1998 Methods of test for petroleum and its products: Part 16 Crude petroleum and liquid petroleum products — Laboratory determination of density — Hydrometer method (*fourth revision*)	Identical
ISO 9562 Water quality — Determination of adsorbable organically bound halogens (AOX)	IS 3025 (Part 70) : 2018/ISO 9562 : 2004 Methods of sampling and test (Physical and Chemical) for water and waste water: Part 70 Adsorbable organically bound halogens (AOX)	Identical
ISO 12185 Crude petroleum and petroleum products — Determination of density — Oscillating U-tube method	IS 1448 (Part 167) : 2018/ISO 12185 : 1996 Methods of test for petroleum and its products: Part 167 Determination of density — Oscillating U-tube method (*third revision*)	Identical
IEC 60156 Insulating liquids — Determination of the breakdown voltage at power frequency — Test method	IS 6792 : 2023/IEC 60156 : 2018 Insulating liquids — Determination of the breakdown voltage at power frequency — Test method (*third revision*)	Identical
IEC 60247 Insulating liquids — Measurement of relative permittivity, dielectric dissipation factor (tanδ) and d.c. resistivity	IS 16840 : 2018/IEC 60247 : 2004 Insulating liquids — Measurement of relative permittivity, dielectric dissipation factor (Tan δ) and d.c. resistivity	Identical
IEC 60475 Method of sampling insulating liquids	IS 6855 : 2023/IEC 60475 : 2022 Method of sampling insulating liquids	Identical
IEC 60628 Gassing ofi nsulating liquids under electrical stress and ionization	IS 12475 (Part 1 & 2) : 1988 Specification for gassing of insulating liquid under electric stress and ionization	Modified/Technically Equivalent
IEC 60666 Detection and determination of specified additives in mineral insulating oils	IS 13631 : 2017/IEC 60666 : 2010 Detection and determination of specified additives in mineral insulating oils (*first revision*)	Identical
IEC 60814 Insulating liquids — Oil-impregnated paper and pressboard — Determination of water by automatic coulometric karl fischer titration	IS 13567 : 2018/IEC 60814 : 1997 Insulating liquids — Oil-impregnated paper and pressboard Determination of water by automatic coulometric karl fischer titration (*first revision)*	Identical

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
IEC 61619 Insulating liquids — Contamination by polychlorinated biphenyls (PCBs) — Method of determination by capillary column gas chromatography	IS 16082 : 2013/IEC 61619 : 1997 Insulating liquids — Contamination by polychlorinated biphenyls (PCBs) — Method of determination by capillary column gas chromatography	Identical
IEC 61620 Insulating liquids — Determination of the dielectric dissipation factor by measurement of the conductance and capacitance — Test method	IS 16086 : 2013/IEC 61620 : 1998 Insulating liquids — Determination of the dielectric dissipation factor by measurement of the conductance and capacitance — Test method	Identical
IEC 62021-1 Insulating liquids — Determination of acidity — Part 1: Automatic potentiometric titration	IS 16863 (Part 1) : 2018/IEC 62021-1 : 2003 Insulating liquids — Determination of acidity: Part 1 Automatic potentiometric titration	Identical
IEC 62021-2 Insulating liquids — Determination of acidity — Part 2: Colourimetric titration	IS 16863 (Part 2) : 2018/ IEC 62021-2 : 2007 Insulating liquids — Determination of acidity: Part 2 Colourimetric titration	Identical
IEC 62021-3 Insulating liquids — Determination of acidity — Part 3: Test methods for non-mineral insulating oils	IS 16863 (Part 3) : 2018/ IEC 62021-3 : 2014 Insulating liquids — Determination of acidity: Part 3 Test methods for non-mineral insulating oils	Identical
IEC 62535 Insulating liquids — Test method for detection potentially corrosive sulphur in used and unused insulating oil	IS 16310 : 2017/IEC 62535 : 2008 Insulating liquids — Test method for detection of potentially corrosive sulphur in used and unused insulating oil	Identical

The Committee has reviewed the provisions of the following International Standards referred in this adopted standard and decided that they are acceptable for use in conjunction with this standard:

International Standard	*Title*
ASTM D1275	Standard test method for corrosive sulfur in electrical insulatingliquids
ASTM D4929	Standard test method for determination of organic chloride content in crude oil
ASTM D7042	Standard test method for dynamic viscosity and density of liquids by stabinger viscometer (and the calculation of kinematic viscosity)
ASTM D7536	Standard test method for chlorine in aromatics by monochromatic Wavelength Dispersive X-ray fluorescence spectrometry
DIN 51353	Testing of insulating oils; detection of corrosive sulfur; Silver strip test

Only the English language text has been retained while adopting it in this Indian Standard, and as such, the page numbers given here are not the same as in the IEC Publication.

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 roundedoff in accordance with IS 2 : 2022 ‘Rules for rounding of numerical values (*second revision*)’.The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

CONTENTS

INTRODUCTION

WARNING – Health and safety

This document does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

The synthetic aromatic hydrocarbon insulating liquids which are the subject of this document should be handled with due regard to personal hygiene. Direct contact with the eyes can cause irritation. In the case of eye contact, irrigation with copious quantities of clean running water should be carried out and medical advice sought. Some of the tests specified in this document involve the use of processes that could lead to a hazardous situation. Attention is drawn to the relevant standard for guidance.

WARNING – Environment

This document is applicable to synthetic aromatic hydrocarbon insulating liquids, chemicals and used sample containers. The disposal of these items can be subject to regulatory requirements with regard to their impact on the environment. Every precaution should be taken to prevent release of insulating liquids into the environment.

Indian Standard

INSULATING LIQUIDS — UNUSED LIQUIDS BASED ON SYNTHETIC AROMATIC HYDROCARBONS — SPECIFICATION

1 Scope

This document covers specifications and test methods for unused synthetic aromatic hydrocarbons intended for use as insulating liquid in cables and capacitors.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60156, *Insulating liquids – Determination of the breakdown voltage at power frequency – Test method*

IEC 60247, *Insulating liquids – Measurement of relative permittivity, dielectric dissipation factor (tanδ) and d.c. resistivity*

IEC 60475, *Method of sampling insulating liquids*

IEC 60628, *Gassing of insulating liquids under electrical stress and ionization*

IEC 60666, *Detection and determination of specified additives in mineral insulating oils*

IEC 60814, *Insulating liquids – Oil-impregnated paper and pressboard – Determination of water by automatic coulometric Karl Fischer titration*

IEC 61619, *Insulating liquids – Contamination by polychlorinated biphenyls (PCBs) – Method of determination by capillary column gas chromatography*

IEC 61620, *Insulating liquids – Determination of the dielectric dissipation factor by measurement of the conductance and capacitance – Test method*

IEC 62021 (all parts), *Insulating liquids – Determination of acidity*

IEC 62535, *Insulating liquids – Test method for detection potentially corrosive sulphur in used and unused insulating oil*

ISO 2592, *Petroleum and related products – Determination of flash point – Cleveland open cup method (PMOC)*

ISO 3016, *Petroleum and related products from natural or synthetic sources – Determination of pour point*

ISO 3104, *Petroleum products – Transparent and opaque liquids – Determination of kinematic viscosity and calculation of dynamic viscosity*

ISO 3675, *Crude petroleum and liquid petroleum products – Laboratory determination of density – Hydrometer method*

ISO 9562, *Water quality – Determination of adsorbable organically bound halogens (AOX)*

ISO 12185, *Crude petroleum and petroleum products – Determination of density – Oscillating U-tube method*

ASTM D1275, *Standard test method for corrosive sulfur in electrical insulating liquids*

ASTM D4929, *Standard Test Method for Determination of Organic Chloride Content in Crude Oil*

ASTM D7042, *Standard Test Method for Dynamic Viscosity and Density of Liquids by Stabinger Viscometer (and the Calculation of Kinematic Viscosity)*

ASTM D7536, *Standard Test Method for Chlorine in Aromatics by Monochromatic Wavelength Dispersive X-ray Fluorescence Spectrometry*

DIN 51353, *Testing of insulating oils; detection of corrosive sulfur; Silver strip test*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- IEC Electropedia: available at https://www.electropedia.org/
- ISO Online browsing platform: available at https://www.iso.org/obp

3.1
alkylbenzenes
insulating liquids consisting of a benzene ring and an alkyl group where the alkyl group can be a straight chain or a branched-chain type

Note 1 to entry: Examples of IR spectra are given in Annex B.

3.2
alkyldiphenylethanes
insulating liquids consisting of diphenylethanes derivatives where the two aryl groups normally carry short alkyl groups

Note 1 to entry: Examples of IR spectra are given in Annex B.

3.3
alkylnaphthalenes
insulating liquids consisting of a naphthalene structure with substituent alkyl groups

Note 1 to entry: Examples of IR spectra are given in Annex B.

3.4
methylpolyarylmethanes
insulating liquids consisting of methylpolyarylm ethanes derivatives mainly based on a blend of mono/di-benzyl toluene (M/DBT)

Note 1 to entry: Examples of IR spectra are given in Annex B.

4 Identification and general delivery requirements

Identification and general delivery requirements are as follows:

a) The synthetic aromatic hydrocarbons type of liquid is normally delivered in bulk, rail-tank cars, tank containers or packed in drums or intermediate bulk containers (IBC). These shall be clean and suitable for this purpose to avoid any contamination. The supplier shall take all the precautions to ensure the delivery product will be in accordance with the requirements of this document.

b) Drums and sample containers shall carry at least the following markings:
   – supplier's designation;
   – labelling in accordance with the Globally Harmonized System of Classification and Labelling of Chemicals (GHS);
   – liquid quantity.

c) As agreed between the supplier and purchaser each liquid delivery shall be accompanied by a document specifying the supplier’s designation, labelling and compliance certificate. A delivery shall be traceable to a manufactured batch.

d) The supplier shall declare the generic type of all additives, and the concentrations in the case of antioxidants and passivators.

5 Sampling

Sampling shall be carried out in accordance with the procedure described in IEC 60475.

6 Test methods

6.1 Appearance

Appearance shall be evaluated by examining, in transmitted light, a representative sample of the liquid having a thickness of approximately 10 cm, at ambient temperature.

6.2 Density

Density shall be measured in accordance with ISO 12185 (reference method). ISO 3675 and ASTM D7042 are acceptable.

6.3 Kinematic viscosity

Kinematic viscosity shall be measured according to ISO 3104 (reference method) or ASTM D7042.

6.4 Flash point

Flash point shall be determined according to ISO 2592.

6.5 Pour point

Pour point shall be determined according to ISO 3016.

6.6 Acidity

Acidity shall be measured according to the IEC 62021 series. Any part of the IEC 62021 series (IEC 62021-1 or IEC 62021-2 or IEC 62021-3) can be used.

6.7 Chlorine content

Chlorine content shall be determined using a test method according to ISO 9562 for the determination of adsorbable organically bound halogens (AOX) or ASTM D4929 for the determination of organic chloride content in crude oil or ASTM D7536 for the determination of chlorine in aromatics by monochromatic wavelength dispersive X-ray fluorescence spectrometry. The method described in Annex A can also be used.

6.8 Water content

Water content shall be determined according to IEC 60814.

6.9 Corrosive and potentially corrosive sulphur

Corrosive and potentially corrosive sulphur shall be determined according to DIN 51353 and ASTM D1275 and IEC 62535.

6.10 Additives

The chemical family of all additives shall be declared in product data sheets and certificates of compliance. For antioxidant additives and passivators (IEC 60666), their concentrations shall also be stated.

6.11 Breakdown voltage

Breakdown voltage shall be determined according to IEC 60156.

6.12 Dielectric dissipation factor and volume resistivity

The properties shall be determined according to IEC 60247 (reference method) or IEC 61620.

6.13 Gassing tendency

The gassing shall be determined by Method A of IEC 60628.

NOTE Synthetic aromatic hydrocarbon compositions have been used as additive to improve the gassing tendency of insulating liquids.

6.14 Polychlorinated biphenyl (PCBs) content

Synthetic aromatic hydrocarbons shall be free from PCBs. The reference test method is IEC 61619.

NOTE Acceptable limits of total or individual PCBs are specified in national and local regulations. Further European specifications are described in Directive 96/59/EC and UN Guidelines for the identification of PCBs and materials containing PCBs.

7 Specifications for capacitor and cable alkylbenzenes

When tested in accordance with the methods specified in Clause 6, the properties of insulating liquids based on alkylbenzenes which are intended for use as impregnant in capacitors and hollow-core cables shall meet the requirements and specifications given in Table 1.

8 Specifications for capacitor alkyldiphenylethanes

When tested in accordance with the methods specified in Clause 6, the properties of insulating liquids based on alkyldiphenylethanes used as impregnant in capacitors shall meet the requirements and specifications given in Table 2.

9 Specifications for capacitor alkylnaphthalenes

When tested in accordance with the methods specified in Clause 6 the properties of insulating liquids based on alkylnaphthalenes used as impregnant in capacitors shall meet the requirements and specifications given in Table 2.

10 Specifications for capacitor methylpolyarylmethanes

When tested in accordance with the methods specified in Clause 6, the properties of insulating liquids based on methylpolyarylmethanes used as impregnant in capacitors shall meet the requirements and specifications given in Table 3.

Table 1 – Specifications for capacitor and cable alkylbenzenes

Property	Test method	Permissible value			Permissible value
		Cables type[a]			Capacitors
		Type I	Type II	Type III	
Physical					
– Appearance		Clear, no suspended matter or sediments			Clear, no suspended matter or sediments
– Density at 20 °C (kg/dm^3)	ISO 12185[b], ISO 3675 or ASTM D7042	Max. 0,880	Max. 0,880	Max. 0,885	Max. 0,880
– Kinematic viscosity at 40 °C (mm^2/s)	ISO 3104[b] or ASTM D7042	< 6	6 to 11	11 to 50	Max. 5
– Pour point (°C)	ISO 3016	Max. −45	Max. −45	Max. −30	Max. −45
Chemical					
– Acidity (mg KOH/g)	IEC 62021	Max. 0,03			Max. 0,03
– Chlorine content (mg/kg)	ISO 9562 or ASTM D4929 or ASTM D7536. Method described in Annex A can also be used.	Max. 30			Max. 30
– Water content (mg/kg)	IEC 60814	Max. 75[c]			Max. 75[c]
– Corrosive sulphur	DIN 51353	Not corrosive			Not corrosive
– Corrosive sulphur	ASTM D1275	Not corrosive			Not corrosive
– Potentially corrosive sulphur	IEC 62535	Not corrosive			Not corrosive
– All Additives[d] (%)	IEC 60666	≤ 1,0			≤ 1,0
Electrical					
– Breakdown voltage (kV)	IEC 60156	Min. 35[e] /70[e]			Min. 35[e] /70[e]
– Volume resistivity at 90 °C (GΩ · m)	IEC 60247 or IEC 61620	Min. 500[c]			Min. 500[c]
– Dielectric dissipation factor, tanδ at 90 °C and 40 Hz to 60 Hz	IEC 60247 or IEC 61620	Max. 0,002[c]			Max. 0,002[c]
– Gassing tendency[f] (mm^3/min)	IEC 60628/Method A	Max. −20			Max. −20
– Permittivity[g]	IEC 60247 or IEC 61620	2,20 to 2,40			2,20 to 2,40
Health, safety and environment (HSE)					
– Flash point (°C)	ISO 2592	Min. 110	Min. 130	Min. 150	Min. 110
– PCB content (mg/kg)	IEC 61619	Not-detected (ND) (< 2)			Not detected (ND) (< 2)

[a] Alkylbenzenes can be produced at different density and viscosity levels with their own level of characteristics to be used in different applications (Type I, II and III).

[b] Reference method.

[c] These values relate to liquids as received.

[d] The supplier shall declare the function and chemical family of all additives, and the concentrations in the case of inhibitors, antioxidants and passivators.

[e] The value of 35 kV is for the liquid, as received. The value of 70 kV is for the liquid after laboratory treatment consisting of filtration of liquid at 60 °C by vacuum pressure below 2,5 kPa through sintered glass filtering (with maximum pore size of 2,5 µm). This test shall be conducted in accordance with IEC 60156.

[f] Specification requires the gassing limits as measured by method A.

[g] Shall be reported for information.

Table 2 – Specifications for capacitor alkyldiphenylethanes and alkylnaphthalenes

Property	Test method	Permissible value capacitors only
Physical		
– Appearance		Clear, no suspended matter or sediments
– Density at 20 °C (kg/dm^3)	ISO 12185[a], ISO 3675 or ASTM D7042	Max. 1,000
– Kinematic viscosity at 40 °C (mm^2/s)	ISO 3104[a] or ASTM D7042	Max. 8[b]
– Pour point (°C)	ISO 3016	Max. −40
Chemical		
– Acidity (mg KOH/g)	IEC 62021	Max. 0,03
– Chlorine content (mg/kg)	ISO 9562 or ASTM D4929 or ASTM D7536. Method described in Annex A can also be used.	Max. 30
– Water content (mg/kg)	IEC 60814	Max. 100
– Corrosive sulphur	DIN 51353	Not corrosive
– Corrosive sulphur	ASTM D1275	Not corrosive
– Potentially corrosive sulphur	IEC 62535	Not corrosive
– All Additives[d] (%)	IEC 60666	Max. 1 %
Electrical		
– Breakdown voltage (kV)	IEC 60156	Min. 35[e]/70[e]
– Volume resistivity at 90 °C (GΩ · m)	IEC 60247	Min. 500[c]
– Dielectric dissipation factor, tan δ at 90 °C and 40 Hz to 60 Hz	IEC 60247	Max. 0,002[c]
– Gassing tendency[f] (mm^3/min)	IEC 60628/Method A	Max. −100
– Permittivity[g]	IEC 60247[a] or IEC 61620	2,30 to 2,80
Health, safety and environment (HSE)		
– Flash point (°C)	ISO 2592	Min. 130
– PCB (mg/kg)	IEC 61619	Not detected (ND) (< 2)

[a] Reference method.

[b] Maximum value for alkyldiphenylethanes is 7.

[c] These values relate to liquids as received.

[d] The supplier shall declare the function and chemical family of all additives, and the concentrations in the case of inhibitors, antioxidants and passivators.

[e] The value of 35 kV is for the liquid, as received. The value of 70 kV is for the liquid after laboratory treatment consisting of filtration of liquid at 60 °C by vacuum pressure below 2,5 kPa through sintered glass filtering (with maximum pore size of 2,5 µm). This test shall be conducted in accordance with IEC 60156.

[f] Specification requires the gassing limits as measured by method A.

[g] Shall be reported for information.

Table 3 – Specifications for capacitor methylpolyarylmethanes

Property	Test method	Permissible value for capacitors only
Physical		
– Appearance	-	Clear, no suspended matter or sediments
– Density at 20 °C (kg/dm^3)	ISO12185[a], ISO 3675 or ASTM D7042	Max. 1,020
– Kinematic viscosity at 40 °C (mm^2/s)	ISO 3104[a] or ASTM D7042	Max. 5
– Pour point (°C)		Max. −50
Chemical		
– Neutralization value (mg KOH/g)	IEC 62021	Max. 0,03
– Chlorine content (mg/kg)	ISO 9562 or ASTM D4929 or ASTM D7536. Method described in Annex A can also be used.	Max. 30
– Water content (mg/kg)	IEC 60814	Max. 100
– Corrosive sulphur	DIN 51353	Not corrosive
– Corrosive sulphur	ASTM D1275	Not corrosive
– Potentially corrosive sulphur	IEC 62535	Not corrosive
– All additives[c] (%)	IEC 60666	< 1,0
Electrical		
– Breakdown voltage (kV)	IEC 60156	Min. 35[d]/70[d]
– Volume resistivity at 90 °C (GΩ · m)	IEC 60247[a] or IEC 61620	Min. 250[b]
– Dielectric dissipation factor, tan δ at 90 °C and 40 Hz to 60 Hz	IEC 60247[a] or IEC 61620	Max. 0,004[b]
– Gassing tendency[e] (mm^3/min)	IEC 60628/Method A	Max. −130
– Permittivity[f]	IEC 60247	2,30 to 2,80
Health, safety and environment (HSE)		
– Flash point (°C)	ISO 2592	Min. 130
– PCB (mg/kg)	IEC 61619	Not detected (ND) (< 2)

[a] Reference method.

[b] These values relate to liquids as received.

[c] The supplier shall declare the function and chemical family of all additives, and the concentrations in the case of inhibitors antioxidants and passivators.

[d] The value of 35 kV is for the liquid, as received. The value of 70 kV is for the liquid after laboratory treatment consisting of filtration of liquid at 60 °C by vacuum pressure below 2,5 kPa through sintered glass filtering (with maximum pore size of 2,5 µm). This test shall be conducted in accordance with IEC 60156.

[e] Specification requires the gassing limits as measured by method A.

[f] Shall be reported for information.

Annex A
(informative)

Test method for determination of chlorine content

A.1 Test method for determination of total chloride

A.1.1 Reagents

– standard solution of nitric acid (HNO_3), analytical grade. Dilute 190 g of concentrated nitric acid to 1 dm^3 with distilled water;

– isopropyl alcohol, analytical grade;

– silver nitrate ($AgNO_3$), analytical grade. Standard solution (0,025 mol/dm^3);

  0,4247 g of silver nitrate weighed accurately. Transfer it to a 1 l volumetric flask and add distilled water to dissolve. Add 3 cm^3 of concentrated nitric acid (density 1,42 kg/dm^3) and then add distilled water to the 1 dm^3 mark of the volumetric flask. Standardize this solution against a pure chloride standard. Check the solution at least monthly to ensure a constant reagent. Dry the silver nitrate overnight in a desiccator before making up the solution. Both the solid material and the solution shall be protected from light by storage in brown glassware in the dark.

– sodium diphenyl solution ($C_6H_5C_6H_4Na$). Organic halogen reagent: 30 cm^3 of this reagent are normally needed to give excess reagent. The preparation of sodium diphenyl solution is described in 'McCoy – The Inorganic Analysis of Petroleum, Chemical Publishing Co. Inc., 212 Fifth Avenue, New York'.

NOTE A sodium chloride crystal as used in infrared spectrometer cells is a suitable chloride standard.

A.1.2 Apparatus

– separatory funnel, 250 cm^3;

– potentiometric titration apparatus;

– electrodes: silver and glass electrode combination is preferred. A silver electrode with a mercurous sulphate reference electrode is an acceptable alternative;

– micro-burette, 5 cm^3 with 0,01 cm^3 divisions.

A.1.3 Procedure

Dissolve 35,5 g ± 0,1 g of the liquid under test in 25 cm^3 toluene in a 150 cm^3 beaker by stirring with a small glass rod. Transfer the solution to a separatory funnel. Rinse the beaker several times with a total of 25 cm^3 toluene and add the rinses to the funnel.

Add an excess (approximately 30 cm^3 is usually sufficient) of sodium diphenyl solution to the contents of the separatory funnel. The excess is indicated by a colour change of blue or green. Stopper the vessel and gently shake to thoroughly mix the solution, venting occasionally to release the excess pressure build-up.

Allow the blue-green mixture to stand 5 min to ensure complete reaction. Remove stopper, add 2 cm^3 of isopropyl alcohol, and swirl with stopper removed until excess reagent is destroyed.

Add slowly 50 cm^3 of the nitric acid solution. Ensure that the organic and aqueous phases are in intimate and uniform contact by gentle swirling and rocking for 5 min. Loosen the stopper occasionally to release slight pressure. Drain the aqueous phase into a beaker. Extract the organic phase twice more with 50 cm^3 portions of nitric acid solution. Drain the aqueous phases into the beaker containing the first extract.

Place the beaker containing the aqueous phase on the titration stand and insert the electrode system. Start the stirrer and record the initial value of potential or pH. Titrate slowly with $AgNO_3$ solution (0,025 mol/dm^3), recording readings after the addition of each drop of $AgNO_3$ solution.

Continue titrating until the point of maximum change in potential or pH scale reading is reached. Plot the volume of silver nitrate as abscissa and voltage or pH reading as ordinates. The end-point is selected at the point of inflection of the curve.

Titrate the same volume of solvent without the sample, as a blank.

A.1.4 Calculation

Calculate the amount of total chlorine as follows:

Total chlorine (mg/kg) = $[(A - B)\ N/m]$ 35,5 × 10^3

where

A is the number of cubic centimetres of $AgNO_3$ solution required for titration of the sample;

B is the number of cubic centimetres of $AgNO_3$ solution required for titration of the blank;

N is the molarity of the $AgNO_3$ solution;

m is the mass in grams of sample used;

35,5 is the atomic mass of chlorine.

A.2 Test method for determination of organic chloride

This test method is based on UOP 779/08.

Annex B
(informative)

IR spectra of typical synthetic aromatic hydrocarbons

NOTE All IR spectra conditions are: KBr cells between wavelength 400 cm^{-1} to 4 000 cm^{-1}.

B.1 IR spectra typical of the family of alkylbenzenes

See Figure B.1 and Figure B.2.

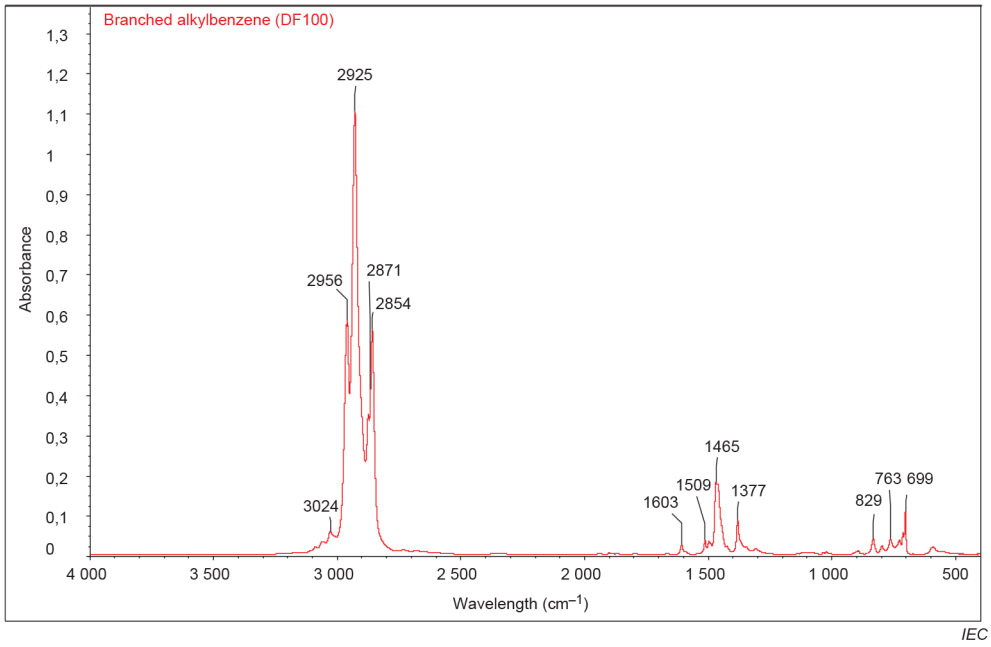

Figure B.1 – IR spectra of branched alkylbenzenes

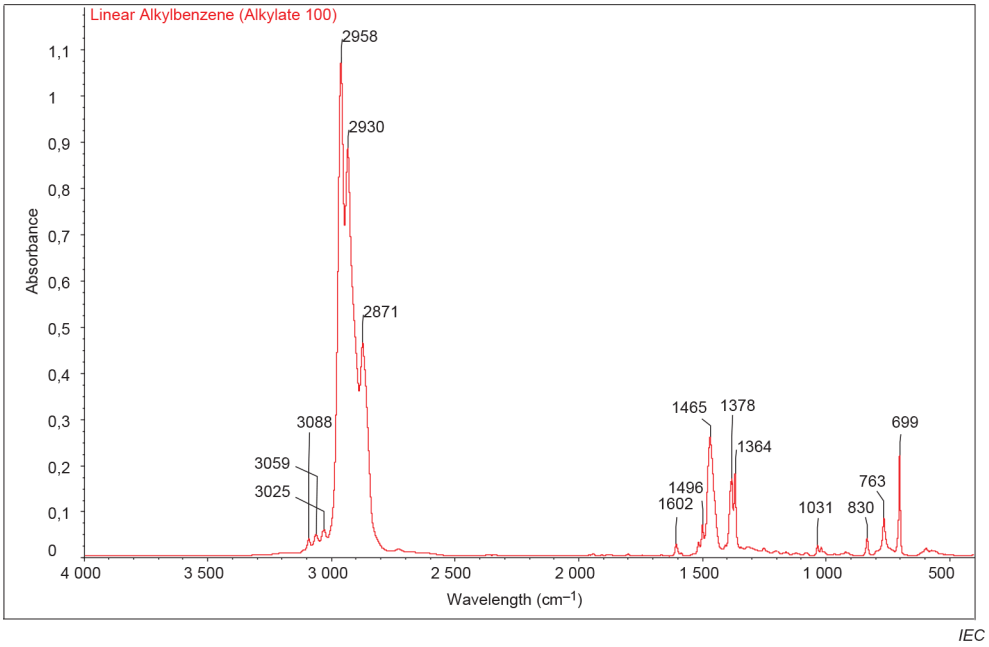

Figure B.2 – IR spectra of linear alkylbenzenes

NOTE The two types of alkylbenzene shown in Figure B.1 and Figure B.2 can be distinguished by infrared spectrophotometric analysis. The straight-chain type shows a single absorption peak in the region 1 360 cm^{-1} to 1 380 cm^{-1} and the branched-chain type shows a double peak in that region.

B.2 Alkyldiphenylethanes

See Figure B.3.

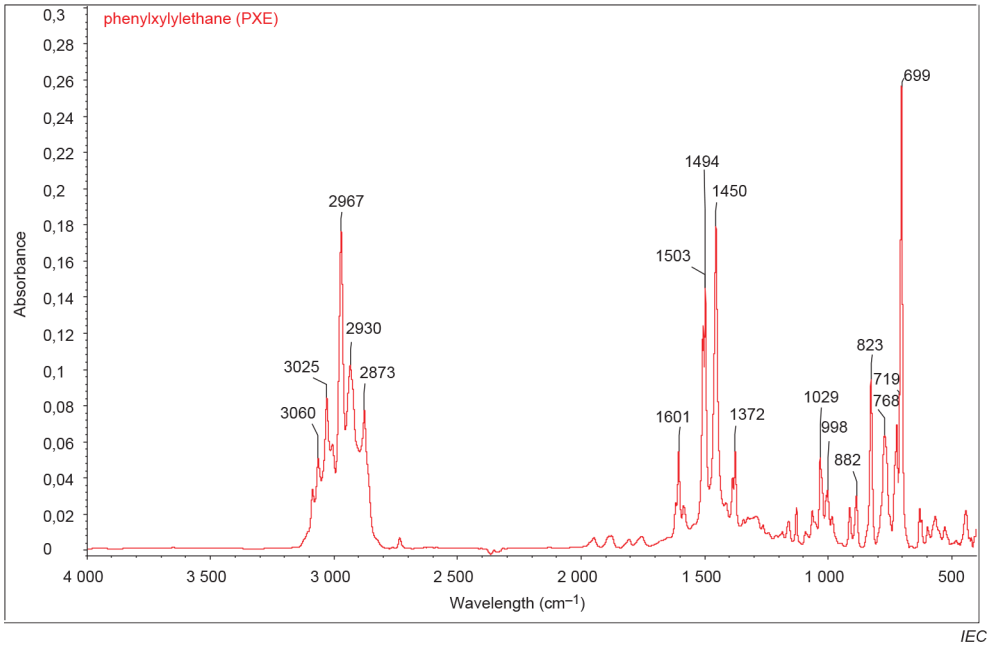

Figure B.3 – IR spectrum of phenylxylylethane (PXE)

NOTE Phenylxylylethane (PXE) is characterized by infrared absorption bands at 3 070 cm^{-1}, 1 606 cm^{-1} and 705 cm^{-1}.

B.3 Alkylnaphtalenes – typified by 1-methylnaphthalene

See Figure B.4.

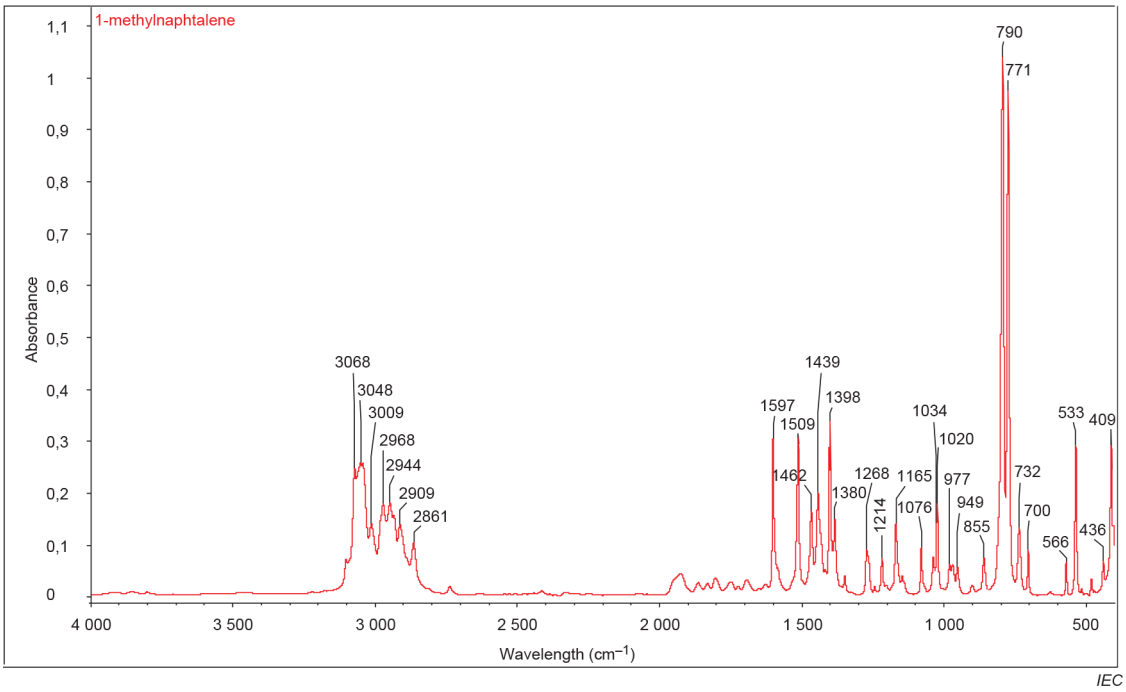

Figure B.4 – IR spectrum of 1-methylnaphthalene

B.4 Methylpolyarylmethanes

See Figure B.5 and Figure B.6.

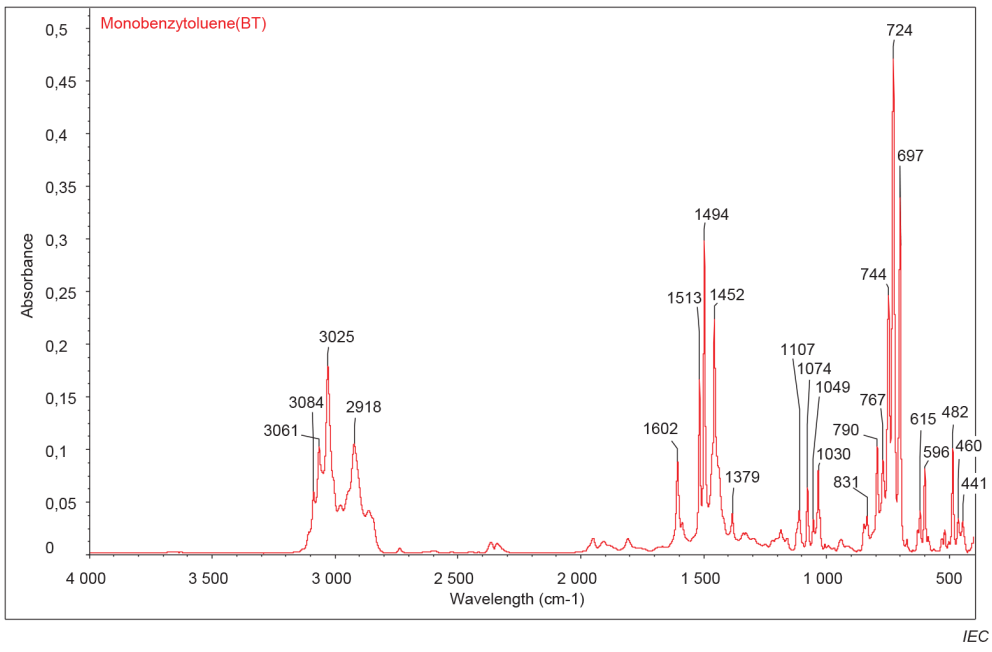

Figure B.5 – IR spectrum of monobenzyltoluene (MBT)

NOTE Methylpolyarylmethanes are characterized by infrared absorption bands at 3 025 cm^{-1}, 1 606 cm^{-1} and 705 cm^{-1}.

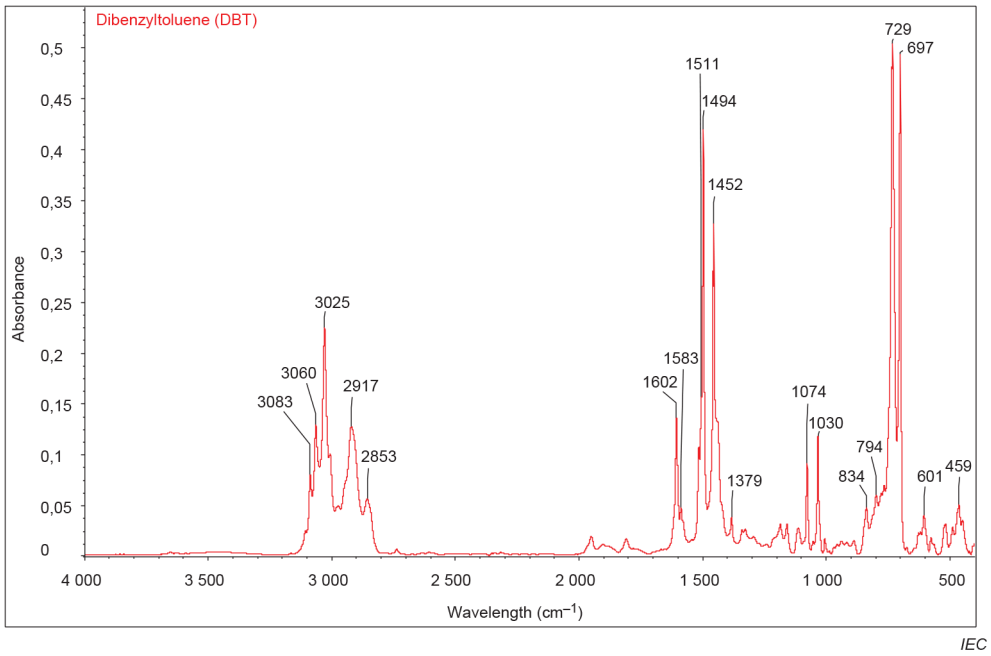

Figure B.6 – IR spectrum of dibenzyltoluene (DBT)

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