

Insulating liquids — Contamination by polychlorinated biphenyls (PCBs) — Method of determination by capillary column gas chromatography

The European Standard EN 61619:1997 has the status of a
British Standard

ICS 29.035.40

NO COPYING WITHOUT BSI PERMISSION EXCEPT AS PERMITTED BY COPYRIGHT LAW



Document Sharing Hub

National foreword

This British Standard is the English language version of EN 61619:1997. It is identical with IEC 1619:1997.

The UK participation in its preparation was entrusted to Technical Committee GEL/10, Fluids for electrotechnical applications, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

Cross-references

Attention is drawn to the fact that Annex ZA lists normative references to international publications with their corresponding European publications. The British Standards which implement these international or European publications may be found in the BSI Standards Catalogue under the section entitled “International Standards Correspondence Index”, or using the “Find” facility of the BSI Standards Electronic Catalogue.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the EN title page, pages 2 to 26, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

This British Standard, having been prepared under the direction of the Electrotechnical Sector Board, was published under the authority of the Standards Board and comes into effect on 15 July 1997

© BSI 09-1999

ISBN 0 580 27781 X

Amendments issued since publication

Amd. No.	Date	Comments

Contents

	Page
National foreword	Inside front cover
Foreword	2
Text of EN 61619	3

ICS 29.035.40

Descriptors: Electrical insulating materials, liquid electrical insulating materials, contamination, polychlorinated biphenyls, determination, gas chromatography, capillary columns

English version

Insulating liquids — Contamination by polychlorinated biphenyls (PCBs)
Method of determination by capillary column gas chromatography

(IEC 61619:1997)

Isolants liquides — Contamination par les polychlorobiphényles (PCB) — Méthode de détermination par chromatographie en phase gazeuse sur colonne capillaire (CEI 61619:1997)

Isolierflüssigkeiten — Verunreinigung durch polychlorierte Biphenyle (PCBs) Verfahren zur Bestimmung mittels Kapillar-Gaschromatographie (IEC 61619:1997)

This European Standard was approved by CENELEC on 1997-03-11. CENELEC members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CENELEC member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CENELEC member into its own language and notified to the Central Secretariat has the same status as the official versions.

CENELEC members are the national electrotechnical committees of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CENELEC

European Committee for Electrotechnical Standardization
Comité Européen de Normalisation Electrotechnique
Europäisches Komitee für Elektrotechnische Normung

Central Secretariat: rue de Stassart 35, B-1050 Brussels

© 1997 CENELEC — All rights of exploitation in any form and by any means reserved worldwide for CENELEC members.

Foreword

The text of document 10/379/FDIS, future edition 1 of IEC 61619, prepared by IEC TC 10, Fluids for electrotechnical applications, was submitted to the IEC-CENELEC parallel vote and was approved by CENELEC as EN 61619 on 1997-03-11.

The following dates were fixed:

- latest date by which the EN has to be implemented at national level by publication of an identical national standard or by endorsement (dop) 1998-01-01
- latest date by which the national standards conflicting with the EN have to be withdrawn (dow) 1998-01-01

Annexes designated “normative” are part of the body of the standard.

Annexes designated “informative” are given for information only.

In this standard, Annex A and Annex ZA are normative and Annex B and Annex C are informative.

Annex ZA has been added by CENELEC.

Endorsement notice

The text of the International Standard IEC 61619:1997 was approved by CENELEC as a European Standard without any modification.

In the official version, for Annex C, Bibliography, the following note has to be added for the standard indicated:

IEC 60567 NOTE Harmonized as EN 60567:1992 (not modified).

Contents

	Page
Foreword	2
1 Scope and object	3
2 Normative reference	3
3 Definitions	3
4 Principle	3
5 Reagents and auxiliary materials	3
6 Apparatus	5
7 Sample	6
8 Chromatograph operating conditions	6
9 Data-processing system	7
10 Checks of instrumental performance	8
11 Procedure	10
12 Test report	14
13 Detection limit	14
14 Precision	14
Annex A (normative) Test mixtures	15
Annex B (informative) General informations	22
Annex C (informative) Bibliography	26
Annex ZA (normative) Normative references to international publications with their corresponding European publications	Inside back cover
Figure 1 — Linearity check	9
Figure A.1 — Chromatogram for test mixture 1242/1254/1260 (5.5)	15
Figure B.1 — Examples of chromatograms obtained for the three main Aroclors®	25
Table 1 — Typical amounts of the major congeners in Aroclor® mixtures	9
Table 2 — Reference peaks/congeners	12
Table 3 — Congeners for ERRF calibration	13
Table A.1 — List of congeners, retention times and relative response factors	17
Table A.2 — Elution order of PCB	20
Table B.1 — Systematic numbering of PCB-compounds	22

1 Scope and object

This International Standard specifies a method for the determination of polychlorinated biphenyl (PCB) concentration in non-halogenated insulating liquids by high-resolution capillary column gas chromatography using an electron capture detector (ECD).

The method gives the total PCB content and is especially useful when a detailed analysis of PCB congeners is necessary. Other methods, such as IEC 60997, may be used when a less detailed analysis is acceptable.

The method is applicable to unused, reclaimed (including dechlorinated and chemically and/or physically treated), or used insulating liquids contaminated by PCBs.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this normative document. At the time of publication, the edition indicated was valid. All normative documents are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative document indicated below. Members of IEC and ISO maintain registers of current valid International Standards.

IEC 60475:1974, *Method of sampling liquid dielectrics*.

3 Definitions

For the purposes of the International Standard, the following definitions apply:

3.1

polychlorinated biphenyl (PCB)

a biphenyl substituted by one to ten chlorine atoms

NOTE For legal purposes, congeners with one, two or ten chlorine atoms may be excluded from this definition.

3.2

congener

all the chlorine derivatives of biphenyl, irrespective of the number of chlorine atoms, are termed congeners

NOTE There are 209 possible PCB congeners. These are listed in Table B.1. The congener numbers (IUPAC)¹⁾ are for easy identification; they do not represent the order of chromatographic elution.

4 Principle

The PCB congeners are determined by temperature programmed gas chromatography. The chromatograph uses a high-efficiency capillary column to separate the PCBs into single or small groups of overlapping congeners.

The sensitivity of the electron capture detector (ECD) may be reduced by the presence of mineral oil. In this method the sample is diluted 100 times to reduce this effect to a minimum.

A sample preparation (clean-up) procedure is used to remove most of the impurities likely to interfere with the determination.

Reference compounds are included to enable calculation of experimental relative retention times (ERRTs) which are compared to a data file of peak ERRTs to identify individual or groups of unresolved congeners. An internal standard is added for quantification.

Relative response factors (RRF), taken from the data files (9.1), corrected by experimental relative response factors (ERRFs) obtained from reference compounds, are applied to identified peaks to quantify the individual (or groups of) congeners, and the values summed to give total PCB content.

5 Reagents and auxiliary materials

5.1 Reagents and standards

All reagents and materials, including those for clean-up, shall be free from PCB contamination and compounds responding to the ECD.

¹⁾ International Union of Pure and Applied Chemistry.

5.1.1 Solvent

Hexane, heptane, cyclohexane or isooctane (2,2,4-trimethylpentane), high purity, free from PCB contamination and low in compounds that respond to the ECD.

5.1.2 Hexachlorobenzene

Purity 99 % at least, used for checking detector sensitivity.

5.1.3 Insulating liquid

An insulating liquid, checked to be free from PCBs or other interfering substances, of the same type as is present in the sample.

5.1.4 Congener 30 solution (C 30)

10 mg/l in solvent (5.1.1) purchased in solution or prepared from pure material (purity 99 % at least).

5.1.5 Congener 209 (DCB), decachlorobiphenyl solution

10 mg/l in solvent (5.1.1) purchased in solution or prepared from pure material (purity 99 % at least).

5.1.6 Calibration solution of selected PCB congeners

Certified calibration mixture in solvent (5.1.1) containing at least the following PCB congeners at a concentration of 10 mg/l each: 18, 28, 31, 44, 52, 101, 118, 138, 149, 153, 170, 180, 194 and 209 (see B.3).

5.2 Commercial PCB standards (see B.4)

5.2.1 Solutions of Aroclors[®] 1242, 1254 and 1260 in solvent (5.1.1)

Concentration required 50 mg/l or more, typically 1 000 mg/l.

5.2.2 Solutions of Aroclors[®] 1242, 1254 and 1260 in oil

50 mg/kg solutions of Aroclors[®] 1242, 1254 and 1260 in unused insulating liquid, either purchased as standardized solutions or prepared from pure material.

5.3 Gas chromatography gases

5.3.1 Carrier gas: helium or hydrogen, purity 99,99 % at least.

5.3.2 Make-up gas: argon/methane, 95 %/5 %. Alternatively, 99,99 % minimum purity nitrogen can be used.

5.4 Internal standard/reference solutions

NOTE Standards should be stored in a cool, dark place.

5.4.1 Internal standard solution 2 (IS 2)

2 mg/l C209 (DCB), 2 mg/l C30.

Pipette (5.8.3) 5 ml of DCB solution (5.1.5) and 5 ml C30 solution (5.1.4) into a 25 ml volumetric flask, make up to the mark with solvent (5.1.1).

5.4.2 Internal standard solution 0,5 (IS 0,5)

0,5 mg/l C209 (DCB), 0,5 mg/l C30.

Follow 5.4.1 using a 100 ml volumetric flask.

5.5 Test mixture solution (for system evaluation)

Into a 20 ml volumetric flask: weigh, to the nearest 0,001 g, 0,50 g of 50 mg/kg Aroclor[®] 1260, plus 0,50 g of 50 mg/kg Aroclor[®] 1254 plus 1,00 g of 50 mg/kg Aroclor[®] 1242 solutions in insulating liquid (5.2.2).

Add by pipette 1 ml of IS 2 solution (5.4.1) and make up to volume with solvent.

Prior to use this solution shall be treated as per 11.1.3.

5.6 Calibration — congener mix stock solution

Into a 20 ml volumetric flask: weigh, to the nearest 0,001 g, 2,0 g of insulating liquid (5.1.3) and add 1 ml of the calibration PCB congener mix (5.1.6). Make up to the mark with solvent (5.1.1).

5.7 Congener mix calibration solution (for response factors)

Submit 500 µl of solution (5.6) to the clean-up (11.1.3). The final solution is suitable for the determination of relative factors.

Prepare a fresh solution monthly.

5.8 Glassware

5.8.1 Volumetric flasks 100, 50, 25, 10 and 5 ml (tolerance better than $\pm 0,4\%$)

5.8.2 Syringes and pipettes:

500 μl $\pm$ 5 μl syringe or pipette,

1 μl and 5 μl gas chromatography precision syringes.

5.8.3 Bulb pipettes (volumetric) 1, 2 and 5 ml class A

5.9 Columns and accessories for sample preparation

5.9.1 Commercial or self-packed solid-phase extraction columns:

3 ml silica gel column, adsorbent weight 500 mg, particle size 40 μm ,

3 ml benzenesulphonic acid column, adsorbent weight 500 mg, particle size 40 μm .

5.9.2 Column adapter, for joining two columns

5.9.3 Vacuum manifold column processor — optional

6 Apparatus

6.1 Gas chromatograph (GC)

A high-resolution gas chromatograph with accurately reproducible oven temperature control, capable, when used with the appropriate column and conditions, of resolving the test mixture (5.5) at least as well as in Figure A.1 (90 peaks observed) and of reproducing relative retention times to within $\pm 0,0015$.

The gas lines (carrier gas and make-up gas) shall be fitted with water vapour and oxygen traps.

The carrier gas supply system shall be capable of running with a 50 m column at maximum efficiency using He or H₂ carrier gas, e.g. adequate column head pressure.

The oven temperature programmer shall have a range that can be set to attain the required resolution.

6.1.1 Injector

Either an “on-column” injector or a “split/splitless” injector may be used.

6.1.2 Columns

Either a cross-linked 5 % phenyl-methyl silicone, stationary phase coated onto fused silica capillary column or a similar chemically bonded phase column. Their dimensions shall be as follows:

length = 50 m to 60 m;

internal diameter = 0,2 mm to 0,35 mm;

film thickness = 0,1 μm to 0,25 μm .

NOTE For suitable columns and manufacturers see B.2.

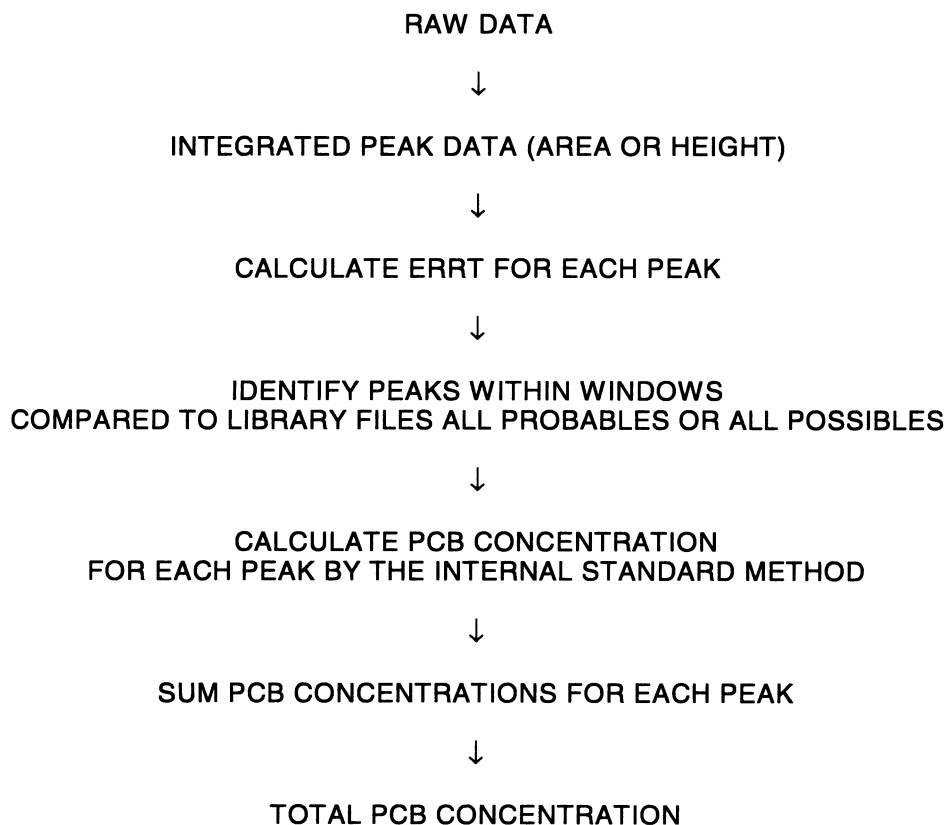
6.1.3 Detector

High-temperature Ni 63 electron capture detector (ECD) with adequate sensitivity to give a signal-to-noise ratio greater than 20 for one picogram of hexachlorobenzene (5.1.2) injected into the column.

The detector shall be operated within its linear range.

6.2 Data-processing system

Any data-processing system may be used with suitable software that can be programmed to process the operations shown below.



7 Sample

7.1 Sampling

Sampling shall be made in accordance with procedures described in IEC 60475.

To avoid cross-contamination of the samples, it is recommended that all the auxiliary material used (tubing, fittings, corks, connections, etc.) be disposable (single-use) and free from interfering compounds.

7.2 Sample preparation

Only glass or metal apparatus is suitable for sample preparation and the determination except for disposable pipette tips and columns made of plastic. All equipment shall be free from PCBs and interfering substances.

If the samples have a free-water phase, it shall be separated from the oil phase prior to further analysis, for example by centrifuging. Emulsified water perceived as opacity can be removed by adding sodium sulfate in portions and shaking until a clear sample is obtained.

The sample should be homogenized, e.g. by shaking the sample by hand for 3 min. An ultrasonic bath may also be used for this purpose.

8 Chromatograph operating conditions

8.1 General

The operating conditions given below have been found to be adequate but they should be optimized with each GC system so that gas chromatograms similar to the one shown in Annex A can be obtained from dilutions of the test mixture in 5.5. Using hydrogen as carrier gas, a satisfactory separation will be obtained in 30–40 min (Figure A.1). With helium carrier gas, the separation will take longer, 55–60 min.