SLOVENSKI STANDARD
SIST EN 61619:1998

01-november-1998

Insulating liquids - Contamination by polychlorinated biphenyls (PCBs) - Method of determination by capillary column chromatography (IEC 61619:1997)

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

Isolierflüssigkeiten - Verunreinigung durch polychlorierte Biphenyle (PCBs) - Verfahren zur Bestimmung mittels Kapillar-Gaschromatographie

Isolants liquides - Contamination par les polychlorobiphényles (PCB) - Méthode de détermination par chromatographie en phase gazeuse sur colonne capillaire

Ta slovenski standard je istoveten z: **EN 61619:1997**

ICS:

29.040.10	Izolacijska olja	Insulating oils

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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)

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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.

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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

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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, annexes A and ZA are normative and annexes B and 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).

Annex ZA (normative)

Normative references to international publications with their corresponding European publications

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

NOTE: When an international publication has been modified by common modifications, indicated by (mod), the relevant EN/HD applies.

Publication	Year	Title	EN/HD	Year
IEC 60475	1974	Method of sampling liquid dielectrics	-	-

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Isolants liquides –

Contamination par les polychlorobiphényles (PCB) – Méthode de détermination par chromatographie en phase gazeuse sur colonne capillaire

Insulating liquids –

Contamination by polychlorinated biphenyls (PCBs) – Method of determination by capillary column gas chromatography

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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INSULATING LIQUIDS – CONTAMINATION BY POLYCHLORINATED BIPHENYLS (PCBs) – METHOD OF DETERMINATION BY CAPILLARY COLUMN GAS CHROMATOGRAPHY

FOREWORD

1) The IEC (International Electrotechnical Commission) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of the IEC is to promote international co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, the IEC publishes International Standards. Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International, governmental and non-governmental organizations liaising with the IEC also participate in this preparation. The IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.

2) The formal decisions or agreements of the IEC on technical matters express, as nearly as possible, an international consensus of opinion on the relevant subjects since each technical committee has representation from all interested National Committees.

3) The documents produced have the form of recommendations for international use and are published in the form of standards, technical reports or guides and they are accepted by the National Committees in that sense.

4) In order to promote international unification, IEC National Committees undertake to apply IEC International Standards transparently to the maximum extent possible in their national and regional standards. Any divergence between the IEC Standard and the corresponding national or regional standard shall be clearly indicated in the latter.

5) The IEC provides no marking procedure to indicate its approval and cannot be rendered responsible for any equipment declared to be in conformity with one of its standards.

6) Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. The IEC shall not be held responsible for identifying any or all such patent rights.

International Standard IEC 61619 has been prepared by technical committee 10: Fluids for electrotechnical applications.

The text of this standard is based on the following documents:

FDIS	Report on voting
10/379/FDIS	10/408/RVD

Full information on the voting for the approval of this standard can be found in the report on voting indicated in the above table.

Annex A forms an integral part of this standard.

Annexes B and C are for information only.

INSULATING LIQUIDS – CONTAMINATION BY POLYCHLORINATED BIPHENYLS (PCBs) – METHOD OF DETERMINATION BY CAPILLARY COLUMN GAS CHROMATOGRAPHY

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)* are for easy identification; they do not represent the order of chromatographic elution.

* International Union of Pure and Applied Chemistry.

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.

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 1000 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).