

STN	Kvalita vody. Stanovenie organochlórových pesticídov (OCP) v celých vzorkách vody. Metóda využívajúca extrakciu na tuhej fáze (SPE) s extrakčnými diskami SPE a plynovú chromatografiu s hmotnostnou spektrometriou (GC-MS).	STN EN 16693 75 7504
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Water quality - Determination of organochlorine pesticides (OCP) in whole water samples - Method using solid phase extraction (SPE) with SPE-disks combined with gas chromatography mass spectrometry (GC-MS)

Táto norma obsahuje anglickú verziu európskej normy.
This standard includes the English version of the European Standard.

Táto norma bola oznámená vo Vestníku ÚNMS SR č. 02/16

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EUROPEAN STANDARD

EN 16693

NORME EUROPÉENNE

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English Version

Water quality - Determination of organochlorine pesticides (OCP) in whole water samples - Method using solid phase extraction (SPE) with SPE-disks combined with gas chromatography mass spectrometry (GC-MS)

Qualité de l'eau - Dosage des pesticides organochlorés (POC) dans la totalité de l'échantillon d'eau - Méthode par extraction en phase solide (SPE) avec disques SPE, avec couplage chromatographie en phase gazeuse - spectrométrie de masse (CG-SM)

Wasserbeschaffenheit - Bestimmung von Organochlorpestiziden (OCP) in Gesamtwasserproben - Verfahren mittels Festphasenextraktion (SPE) mit SPE-Disks in Verbindung mit Gaschromatographie - Massenspektrometrie (GC-MS)

This European Standard was approved by CEN on 27 June 2015.

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COMITÉ EUROPÉEN DE NORMALISATION
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European foreword

This document (EN 16693:2015) has been prepared by Technical Committee CEN/TC 230 "Water analysis", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 2016, and conflicting national standards shall be withdrawn at the latest by March 2016.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

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Introduction

WARNING — Persons using this European Standard should be familiar with usual laboratory practice. This European Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted according to this European Standard be carried out by suitably trained staff.

Organochlorine pesticides (OCP) are priority substances listed in Annex X of the EU Water Framework Directive (WFD, Directive 2000/60/EC) for which environmental quality standards (EQS) have been set at EU level for inland waters as well as other surface waters to protect the aquatic environment against chemical pollution (Directive 2008/105/EC). With the exception of metals, the EQSs are expressed as total concentrations in the whole water sample. Furthermore, analytical methods used in WFD monitoring need to meet certain requirements as regards the minimum limit of quantification and the maximum tolerable measurement uncertainty (Directive 2009/90/EC). So far, there is no European-wide standardized method available for the determination of OCP in whole water samples fulfilling those requirements. Hence, the European Commission mandated CEN to develop or improve standards in support of the implementation of the monitoring requirements of WFD.

Directive 2008/105/EC has been amended by Directive 2013/39/EU, however this standard has been developed for the analysis of OCP as listed in Annex A of Directive 2008/105/EC.

The priority substances list in Annex X of the WFD includes various OCPs such as alachlor, endosulfan, hexachlorobenzene, hexachlorocyclohexane isomers, pentachlorobenzene, aldrin, dieldrin, endrin, isodrin, DDT and its metabolites. Annual average environmental quality standards (AA-EQS) values for individual OCP range from 0,000 5 µg/l to 0,3 µg/l and are defined for the concentration in the whole water sample, including suspended particulate matter (SPM) present in the sample. As long as compounds such as OCP, in particular the larger molecular weight ones, sorb strongly to environmental solids, the fraction bound to particles may be substantial. Therefore it is important to be able to handle whole water samples within the analytical process. Identification and quantification of OCP at trace level concentrations often require both high sensitive chromatographic equipment and effective enrichment steps.

1 Scope

This European Standard specifies a method for the determination of selected organochlorine pesticides (OCP) (see Table 1), in water samples. The method uses solid-phase extraction with SPE-disks followed by gas chromatography-mass spectrometry (GC-MS). It is applicable to the analysis of OCPs in surface water containing suspended particulate matter (SPM) up to 500 mg/l (whole water samples), drinking water and groundwater. The lower limit of the working range depends on the matrix, on the specific compound to be analyzed and on the sensitivity of the mass spectrometric detection unit. For compounds listed in Table 1 the limit of determination (LOQ) is at least 30 % of the corresponding AA-EQS value (0,000 15 µg/l to 0,1 µg/l) according to the requirements of the European Quality Standards Directive (Directive 2008/105/EC) for both inland surface waters and other surface waters.

This method may be used for the analysis of other OCPs not listed in Table 1 or other types of water. However, it is important to verify its applicability before use.

Table 1 — Organochlorine pesticides (OCP) determined by this method

Substance	Molecular formula	Molar mass g/mol	EC Number ^a	CAS RN ^b
Alachlor	C ₁₄ H ₂₀ ClNO ₂	269,77	240-110-8	15972-60-8
<u>Cyclodiene pesticides:</u>				
Aldrin	C ₁₂ H ₈ Cl ₆	364,91	206-215-8	309-00-2
Dieldrin	C ₁₂ H ₈ Cl ₆ O	380,91	200-484-5	60-57-1
Endrin	C ₁₂ H ₈ Cl ₆ O	380,91	200-775-7	72-20-8
Isodrin	C ₁₂ H ₈ Cl ₆	364,91	207-366-2	465-73-6
<u>DDT-total:</u>				
op'-DDT	C ₁₄ H ₉ Cl ₅	354,49	212-332-5	789-02-6
pp'-DDT	C ₁₄ H ₉ Cl ₅	354,49	200-024-3	50-29-3
pp'-DDD	C ₁₄ H ₉ Cl ₄	320,04	200-783-0	72-54-8
pp'-DDE	C ₁₄ H ₉ Cl ₄	318,03	200-784-6	72-55-9
Hexachlorobenzene (HCB)	C ₆ Cl ₆	284,80	204-273-9	118-74-1
Hexachlorobutadiene (HCBd)	C ₄ Cl ₆	260,76	201-765-5	87-68-3
<u>Hexachlorocyclohexane ^c:</u>				
alpha-HCH	C ₆ H ₆ Cl ₆	290,83	206-270-8	319-84-6
beta-HCH	C ₆ H ₆ Cl ₆	290,83	206-271-3	319-85-7
delta-HCH	C ₆ H ₆ Cl ₆	290,83	206-272-9	319-86-8
gamma-HCH	C ₆ H ₆ Cl ₆	290,83	200-401-2	58-89-9
Pentachlorobenzene	C ₆ HCl ₅	250,34	210-172-0	608-93-5
<u>Trichlorobenzene ^c:</u>				
1,2,3-TCB	C ₆ H ₃ Cl ₃	181,45	201-757-1	87-61-6
1,2,4-TCB	C ₆ H ₃ Cl ₃	181,45	204-428-0	120-82-1
1,3,5-TCB	C ₆ H ₃ Cl ₃	181,45	203-608-6	108-70-3
<u>Endosulfan ^c:</u>				
Endosulfan-I (alpha)	C ₉ H ₆ Cl ₆ O ₃ S	406,93	-	959-98-8
Endosulfan-II (beta)	C ₉ H ₆ Cl ₆ O ₃ S	406,93	-	33213-65-9
^a EC Number: European inventory of existing commercial substances (EINECS) or European list of notified chemical substances (ELINCS). ^b CAS RN: Chemical Abstracts Service Registry Number. ^c Mixture of isomers.				

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696)*

EN ISO 5667-3, *Water quality - Sampling - Part 3: Preservation and handling of water samples (ISO 5667-3)*

koniec náhľadu – text ďalej pokračuje v platenej verzii STN