ICS 13.060.50 SLOVENSKÁ TECHNICKÁ NORMA Marec 2016

STN	**Kvalita vody. Stanovenie vybraných polycyklických aromatických uhľovodíkov (PAU) 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 16691** 75 7515

Water quality - Determination of selected polycyclic aromatic hydrocarbons (PAH) 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

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EUROPÄISCHE NORM

EN 16691

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English Version

Water quality - Determination of selected polycyclic aromatic hydrocarbons (PAH) 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 hydrocarbures aromatiques polycycliques (HAP) sélectionnés dans des échantillons d'eau totale - Méthode par extraction en phase solide (SPE) avec disques SPE, avec couplage chromatographie en phase gazeuse-spectrométrie de masse (CG-SM)

Wasserbeschaffenheit - Bestimmung von ausgewählten polycyclischen aromatischen Kohlenwasserstoffen (PAK) 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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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (EN 16691:2015) 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of an identical text or by endorsement, at the latest by March 2016, and conflicting national standards shall be withdrawn at the latest by March 2016.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.

Polycyclic aromatic hydrocarbons (PAH) 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 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 standardized method available for the determination of PAH 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 PAH as listed in Annex A of Directive 2008/105/EC.

Organic compounds as specified in the WFD occur in nearly all types of water. These substances are adsorbed on solids (sediments, suspended matter) as well as dissolved in the liquid phase. A large group of these compounds are polycyclic aromatic hydrocarbons (PAH). There are further standards for the analytical determination of PAH in water and waste water:

— EN ISO 17993 describes a method for the determination of 15 PAH by high performance liquid chromatography/UV detection in drinking water, ground water and surface water;

— ISO 7981-1 and ISO 7981-2 describe methods for the determination of 6 PAH by high performance thin layer chromatography or by high performance liquid chromatography in drinking water and ground water;

— ISO 28540 describes a method for at least 16 PAH using gas chromatography with mass spectrometric detection (GC-MS) in drinking water, ground water and surface water;

— ISO/TS 28581 describes a method for the determination of polycyclic hydrocarbons and pesticide residues in drinking water, ground water surface water and waste water.

1 Scope

This European Standard specifies a method for the determination of 7 polycyclic aromatic hydrocarbons (PAH) in whole water samples listed in Table 1. The method uses solid-phase disk extraction with SPE-disks followed by gas chromatography-mass spectrometry (GC-MS). It is applicable to the analysis of PAHs in surface water containing suspended particulate matter (SPM) up to 500 mg/l (whole water samples), drinking water and groundwater.

The lower and upper 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. The limit of quantification (LOQ) determined in the validation is given in Table 1. The upper limit of the working range is approximately 2 000 ng/l.

This method is, with some modifications suitable for the analysis of waste water. This method is applicable to other PAH[1)], provided the method is validated for each PAH.

Table 1 — Polycyclic aromatic hydrocarbons (PAH) determined by this method

Substance	Molecular formula	Molar mass g/mol	EC number[a]	CAS-RN[b]	LOQ[c] ng/l
anthracene	$C_{14}H_{10}$	178,23	204–371–1	120–12–7	0,24
fluoranthene	$C_{16}H_{10}$	202,26	205–912–4	206–44–0	2,1
benzo[*b*]fluoranthene	$C_{20}H_{12}$	252,32	205–911–9	205–99–2	0,56
benzo[*k*]fluoranthene	$C_{20}H_{12}$	252,32	205–916–6	207–08–9	0,44
benzo[*a*]pyrene	$C_{20}H_{12}$	252,32	200–028–5	50–32–8	0,33
benzo[*ghi*]perylene	$C_{22}H_{12}$	276,34	205–883–8	191–24–2	0,44
indeno[*1,2,3-cd*]pyrene	$C_{22}H_{12}$	276,34	205–893–2	193–39–5	0,42

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 For the determination of the LOQ the procedure given in NEN 7777+C1:2012 [12] was used.

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 1042, *Laboratory glassware - One-mark volumetric flasks (ISO 1042)*

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

EN ISO 5667-1, *Water quality - Sampling - Part 1: Guidance on the design of sampling programmes and sampling techniques (ISO 5667-1)*

1) During the inter-laboratory validation trial the method was tested for all 16 EPA PAH (see Annex B).

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