SLOVENSKI STANDARD
SIST-TS ISO/TS 28581:2013

01-september-2013

Kakovost vode - Določevanje izbranih nepolarnih spojin - Metoda s plinsko kromatografijo z masno selektivnim detektorjem (GC/MS)

Water quality - Determination of selected non-polar substances - Method using gas chromatography with mass spectrometric detection (GC-MS)

Qualité de l'eau - Détermination de substances non polaires sélectionnées - Méthode par chromatographie en phase gazeuse avec détection par spectrométrie de masse (CG-SM)

Ta slovenski standard je istoveten z: **ISO/TS 28581:2012**

ICS:

13.060.50	Preiskava vode na kemične snovi	Examination of water for chemical substances

SIST-TS ISO/TS 28581:2013 **en,fr**

TECHNICAL SPECIFICATION

ISO/TS 28581

First edition
2012-02-15

Water quality — Determination of selected non-polar substances — Method using gas chromatography with mass spectrometric detection (GC-MS)

Qualité de l'eau — Détermination de substances non polaires sélectionnées — Méthode par chromatographie en phase gazeuse avec détection par spectrométrie de masse (CG-SM)

Reference number
ISO/TS 28581:2012(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of document:

— an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;

— an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TS 28581 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

Introduction

Non-polar substances 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 non-polar substances are polycyclic aromatic hydrocarbons (PAH). Some PAH are known or suspected to cause cancer. Maximum acceptable levels have been set in a number of countries. For instance, the European Council Directive 98/83/EC[10] on the quality of water intended for human consumption set the maximum acceptable level for benzo[*a*]pyrene at 0,010 µg/l, and for the sum of four specified PAH (benzo[*b*]fluoranthene, benzo[*k*]fluoranthene, benzo[*ghi*]perylene, indeno[1,2,3-*cd*]pyrene) at 0,100 µg/l.

There are further International Standards for the analytical determination of PAH in water and waste water.

ISO 6468 specifies methods for the determination of certain organochlorine insecticides, polychlorinated biphenyls and chlorobenzenes in drinking water, ground water, surface water and waste water.

ISO 17993[6] specifies methods for the determination of 15 PAH by high performance liquid chromatography in drinking water, ground water and surface water.

ISO 7981[2] specifies 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 17858[5] specifies methods for the determination of dioxin-like polychlorinated biphenyls in waters and waste waters.

ISO 28540[9] specifies the determination of PAH using gas chromatography with mass spectrometric detection (GC-MS).

Water quality — Determination of selected non-polar substances — Method using gas chromatography with mass spectrometric detection (GC-MS)

WARNING — The use of this Technical Specification may involve hazardous materials, operations and equipment.

Persons using this Technical Specification should be familiar with normal laboratory practice. This document 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 Technical Specification be carried out by suitably trained staff.

1 Scope

This Technical Specification specifies a method for the determination by gas chromatography with mass spectrometric detection (GC-MS) of polycyclic hydrocarbons and pesticide residues in drinking water and ground water at mass concentrations above 0,005 µg/l and surface water and waste water at mass concentrations above 0,01 µg/l (for each single compound).

This method can apply to non-polar substances other than polycyclic aromatic hydrocarbons (PAH) and pesticide residues. However, it is necessary to verify the applicability of this method for these compounds.

NOTE 1 A potentially suitable method for this verification is specified in ISO/TS 13530.[3]

This Technical Specification can be used for samples containing up to 150 mg/l of suspended matter.

NOTE 2 Determination of PAH using GC-MS lies within the scope of ISO 28540.[9]

2 Normative references

The following document, 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.

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

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

ISO 6468, *Water quality — Determination of certain organochlorine insecticides, polychlorinated biphenyls and chlorobenzenes — Gas-chromatographic method after liquid-liquid extraction*

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function*

3 Terms and definitions

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

3.1
analyte
substance to be determined

[SOURCE: ISO 15089:2000,[4] definition 3.2]

Note 1 to entry Substances covered by this specification are listed in Table 1.

3.2
calibration standard
solution prepared from a secondary standard and/or stock solutions and used to calibrate the response of the instrument with respect to analyte concentration

[SOURCE: ISO 18073:2004,[7] definition 3.1.2]

3.3
diagnostic ion
selected fragment ion, molecular ion or other characteristic ion from the mass spectrum of the target compound with the highest possible specificity

[SOURCE: ISO 22892:2006,[8] definition 3.6]

3.4
injection standard
standard mixture added to a sample before injection into the GC-MS apparatus, to monitor variability of instrument response and to calculate internal standard recovery

3.5
internal standard
isotopically labelled standard or a non-polar substance added to samples prior to extraction, unlikely to be present in the sample, against which the concentrations of native substances are calculated

Note 1 to entry The substance is added to the sample before extraction and is used for quantification of the components to be measured. Recoveries of these standards are also calculated and used to check the performance of the procedure.

3.6
native compound
non-labelled compound

3.7
selected ion mode
SIM
selected ion recording
SIR
measuring the intensity of selected diagnostic ions only

[SOURCE: ISO 22892:2006,[8] definition 3.8, modified — the last two synonyms have been added.]

4 Principle

The non-polar substances determinable by the method specified in this Technical Specification are listed in Table 1.

The non-polar substances present in the aqueous sample are extracted from the water sample by liquid-liquid extraction with hexane. An internal standard mixture is added to the sample prior to extraction. The extract is concentrated by evaporation and the residue taken up in a solvent appropriate for clean-up or gas chromatography (GC).

Other volatile solvents can also be used if it is proven that there is equal or better recovery (recovery between 70 % and 110 %).

NOTE Other possible suitable solvents are: isohexane C_6H_{15} (CAS: 107-83-5); cyclohexane: C_6H_{12} (CAS: 110-82-7); pentane: C_5H_{12} (CAS: 109-66-0); petroleum ether: boiling range 40 °C to 60 °C.

The liquid-liquid extraction method shall not be used with samples containing more than 150 mg/l of suspended matter.

If necessary, extracts of surface water or waste water samples can be cleaned by column chromatography prior to analysis. Prior to injection, injection standards are added to each extract, and an aliquot of the extract is injected into the gas chromatograph.

The non-polar substances are separated on a suitable fused silica capillary column, coated with a film of cross-linked non-polar polysiloxane or slightly polar modified polysiloxane with an efficient separation. The column shall be suitable for separating critical and isomeric pairs of substances. Identification and quantification is performed by means of mass spectrometry (MS) using electron impact ionization (EI).

Table 1 — Non-polar substances determinable that can be determined by using this Technical Specification

Name	Molecular formula	Molar mass g/mol	CAS number
PAH			
Naphthalene	$C_{10}H_8$	128,17	91-20-3
Acenaphthylene	$C_{12}H_8$	152,20	208-96-8
Acenaphthene	$C_{12}H_{10}$	154,21	83-32-9
Fluorene	$C_{13}H_{10}$	166,22	86-73-7
Phenanthrene	$C_{14}H_{10}$	178,23	85-01-8
Anthracene	$C_{14}H_{10}$	178,23	120-12-7
Pyrene	$C_{16}H_{10}$	202,26	129-00-0
Fluoranthene	$C_{16}H_{10}$	202,26	206-44-0
Chrysene	$C_{18}H_{12}$	228,29	218-01-9
Benzo[*a*]anthracene	$C_{18}H_{12}$	228,29	56-55-3
Benzo[*b*]fluoranthene	$C_{20}H_{12}$	252,32	205-99-2
Benzo[*k*]fluoranthene	$C_{20}H_{12}$	252,32	207-08-9
Benzo[*a*]pyrene	$C_{20}H_{12}$	252,32	50-32-8
Dibenzo[*a,h*]anthracene	$C_{22}H_{14}$	278,35	053-70-3
Benzo[*ghi*]perylene	$C_{22}H_{12}$	276,34	191-24-2
Indeno[1,2,3-*cd*]pyrene	$C_{22}H_{12}$	276,34	193-39-5
PCB			
PCB-28: 2,4,4′-trichlorobiphenyl	$C_{12}H_7Cl_3$	257,54	7012-37-5
PCB-52: 2,2′,5,5′-tetrachlorobiphenyl	$C_{12}H_6Cl_4$	291,99	35693-99-3
PCB-101: 2,2′,4,5,5′-pentachlorobiphenyl	$C_{12}H_5Cl_5$	326,43	37680-73-2
PCB-118: 2,3′,4,4′,5-pentachlorobiphenyl	$C_{12}H_5Cl_5$	326,43	31508-00-6
PCB-138: 2,2′,3,4,4′,5′-hexachlorobiphenyl	$C_{12}H_4Cl_6$	360,88	35065-28-2
PCB-153: 2,2′,4,4′,5,5′-hexachlorobiphenyl	$C_{12}H_4Cl_6$	360,88	35065-27-1
PCB-180: 2,2′,3,4,4′,5,5′-heptachlorobiphenyl	$C_{12}H_3Cl_7$	395,33	35065-29-3
OCP			
Hexachlorobenzene (HCB)	C_6Cl_6	284,78	118-74-1
α-Hexachlorocyclohexane (α-HCH)	$C_6H_6Cl_6$	290,83	319-84-6
β-Hexachlorocyclohexane (β-HCH)	$C_6H_6Cl_6$	290,83	319-85-7

Table 1 *(continued)*

Name	Molecular formula	Molar mass g/mol	CAS number
γ-Hexachlorocyclohexane (γ-HCH)	$C_6H_6Cl_6$	290,83	58-89-9
δ-Hexachlorocyclohexane (δ-HCH)	$C_6H_6Cl_6$	290,83	319-86-8
ε-Hexachlorocyclohexane (ε-HCH)	$C_6H_6Cl_6$	290,83	6108-10-7
Aldrin	$C_{12}H_8Cl_6$	364,93	309-00-2
Dieldrin	$C_{12}H_8Cl_6O$	380,91	60-57-1
Endrin	$C_{12}H_8Cl_6O$	380,91	72-20-8
Heptachlor	$C_{10}H_5Cl_7$	373,32	76-44-8
Heptachlor epoxide (exo-, *cis*- or β-isomer)	$C_{10}H_5Cl_7O$	389,30	28044-83-9
Heptachlor epoxide (endo-, *trans*- or α-isomer)	$C_{10}H_5Cl_7O$	389,30	1024-57-3
α-Endosulfan	$C_9H_6Cl_6O_3S$	406,92	959-98-8
β-Endosulfan	$C_9H_6Cl_6O_3S$	406,92	33213-65-9
p,*p*′-DDE	$C_{14}H_8Cl_4$	318,02	72-55-9
o,*p*′-DDD	$C_{14}H_{10}Cl_4$	320,04	53-19-0
o,*p*′-DDT	$C_{14}H_9Cl_5$	354,49	784-02-6
p,*p*′-DDD	$C_{14}H_{10}Cl_4$	320,04	72-54-8
o,*p*′-DDE	$C_{14}H_8Cl_4$	318,02	3424-82-6
p,*p*′-DDT	$C_{14}H_9Cl_5$	354,49	50-29-3
Methoxychlor	$C_{16}H_{15}Cl_3O_2$	345,65	72-43-5
Chlorobenzenes			
1,2,4-Trichlorobenzene	$C_6H_3Cl_3$	181,45	120-82-1
1,2,3-Trichlorobenzene	$C_6H_3Cl_3$	181,45	87-61-6
1,3,5-Trichlorobenzene	$C_6H_3Cl_3$	181.45	108-70-3
1,2,3,4-Tetrachlorobenzene	$C_6H_2Cl_4$	215,89	634-66-2
1,2,3,5-Tetrachlorobenzene	$C_6H_2Cl_4$	215,89	634-90-2
1,2,4,5-Tetrachlorobenzene	$C_6H_2Cl_4$	215,89	95-94-3
Pentachlorobenzene	C_6HCl_5	250,34	608-93-5
Pentachloronitrobenzene	$C_6Cl_5NO_2$	295,34	82-68-8
Organophosphorus			
Azinphos-ethyl	$C_{12}H_{16}N_3O_3PS_2$	345,40	2642-71-9
Bromofenvinphos-ethyl	$C_{12}H_{14}BrCl_2O_4P$	404,02	33399-00-7
Chlorofenvinphos	$C_{12}H_{14}Cl_3O_4P$	359,57	470-90-6
Chloropyriphos-ethyl	$C_9H_{11}Cl_3NO_3PS$	350,59	2921-88-2
Chloropyriphos-methyl	$C_7H_7Cl_3NO_3PS$	322,53	5598-13-0
Heptenophos	$C_9H_{12}ClO_4P$	250,02	23560-59-0

5 Interferences

5.1 Interferences with sampling, extraction and concentration

Use sampling containers of materials that do not affect the analyte content during the contact time (preferably of stainless steel or glass). Avoid plastics and organic materials other than polytetrafluoroethene (PTFE) during sampling, sample storage or extraction. Care should be taken with the use of surfactants for cleaning sample containers because they may lead to the formation of emulsions during liquid-liquid extraction.