
Water quality — Determination of dioxin-like polychlorinated biphenyls — Method using gas chromatography/mass spectrometry

Qualité de l'eau — Dosage des biphényles polychlorés de type dioxine — Méthode par chromatographie en phase gazeuse/spectrométrie de masse



PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

This document is a preview generated by EVS

© ISO 2007

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

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.....	vi
Introduction	vii
1 Scope	1
2 Normative references	1
3 Terms, definitions and abbreviated terms	2
3.1 Terms and definitions.....	2
3.2 Abbreviated terms	3
4 Principle	4
4.1 Spiking and extraction	4
4.2 Clean-up.....	4
4.3 Concentration.....	4
4.4 Identification.....	5
4.5 Quantification	5
4.6 Analytical quality	5
5 Contamination and interferences.....	5
6 Reagents and standards	6
7 Apparatus and materials.....	10
7.1 Sampling equipment for discrete sampling	10
7.2 Equipment for sample preparation	11
7.3 Extraction apparatus	11
7.4 Filtration apparatus	12
7.5 Clean-up apparatus	12
7.6 Concentration apparatus	13
7.7 Other equipment	13
8 Sample collection, preservation, storage and holding times	14
9 Quality assurance (QA)/quality control (QC)	14
9.1 General.....	14
9.2 Initial precision and recovery (IPR).....	15
9.3 Spiking	15
9.4 Recovery of labelled compounds assessment.....	16
9.5 Method blanks.....	16
9.6 QC check sample	16
9.7 Method precision	16
10 Calibration	17
10.1 Operating conditions.....	17
10.2 Mass spectrometer (MS) resolution.....	17
10.3 Ion abundance ratios, minimum levels, signal-to-noise ratios, and absolute retention times.....	17
10.4 Retention time	18
10.5 Isomer specificity.....	18
10.6 Calibration by isotope dilution	18
10.7 Calibration by internal standard.....	19
10.8 Combined calibration	19
11 Sample preparation	20
11.1 General.....	20

11.2	Determination of percent suspended solids	20
11.3	Preparation of aqueous samples containing 1 % of suspended solids or less	21
12	Extraction and concentration	22
12.1	Separatory funnel extraction of filtrates and of aqueous samples that are visibly absent of particles.....	22
12.2	Solid-phase extraction (SPE) of samples containing less than 1 % suspended solids	22
12.3	Soxhlet extraction of filters and/or disks	23
12.4	Back-extraction with acid and base	24
12.5	Macro-concentration.....	24
12.6	Micro-concentration and solvent exchange.....	26
13	Extract clean-up	26
13.1	General.....	26
13.2	Gel permeation chromatography (GPC)	27
13.3	Silica clean-up	28
13.4	Alumina clean-up	28
13.5	Carbon column.....	29
13.6	High performance liquid chromatography (HPLC).....	29
13.7	Florisil clean-up.....	30
13.8	Silver nitrate/silica column.....	31
14	HRGC/HRMS analysis	31
15	System and laboratory performance.....	31
15.1	General.....	31
15.2	MS resolution.....	31
15.3	Calibration verification	31
15.4	GC resolution.....	32
15.5	Blank.....	32
16	Qualitative determination	32
17	Quantitative determination.....	32
17.1	Isotope dilution quantification.....	32
17.2	Internal standard quantification and labelled-compound recovery.....	33
17.3	Concentration in sample	34
17.4	Results and reporting	35
17.5	Toxic equivalents (TEQ)	35
18	Analysis of complex samples	36
18.1	General.....	36
18.2	Recovery of labelled compounds.....	36
19	Pollution prevention	36
20	Waste management	37
21	Precision	37
Annex A (informative)	Example chromatograms	45
Annex B (informative)	Use of HRGC/LRMS.....	47
Annex C (informative)	Precision data	50
Bibliography		54
Table 1 —	Dioxin-like PCBs determined by this method	38
Table 2 —	Suggested quantification relationships.....	39
Table 3 —	Suggested calibration standard concentrations	40
Table 4 —	Suggested concentration of dioxin-like PCBs in stock and spiking solutions	41
Table 5 —	Typical GC columns and temperature programs	42

Table 6 — Examples of toxic equivalent factors	43
Table 7 — Congener function groups and ions	44
Table B.1 — TetraCBs	49
Table B.2 — PentaCBs	49
Table B.3 — HexaCBs	49
Table B.4 — HeptaCBs	49
Table C.1 — Spiking amounts transferred to sample bottles	50
Table C.2 — Samples 1 and 2 (fortified industrial effluent) — Statistical summary	51
Table C.3 — Sample 3 (unfortified industrial effluent) — Statistical summary	52
Table C.4 — Sample 4 (HPLC water) — Statistical summary	53

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.

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 17858 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

Introduction

When using this International Standard, it may be necessary in some cases to determine whether and to what extent particular problems will require the specification of minor additional conditions.

This document is a preview generated by EVS

This document is a preview generated by EVS

Water quality — Determination of dioxin-like polychlorinated biphenyls — Method using gas chromatography/mass spectrometry

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International 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.

Attention is drawn to any relevant national safety regulations. The non-*ortho* and mono-*ortho* PCBs are co-planar and are among the most toxic of chemicals. All work with dioxin-like PCBs requires therefore the utmost care; the national safety measures which correspond to those for toxic substances shall be strictly adhered to.

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

1 Scope

This International Standard specifies a method for the determination of dioxin-like tetra- to hepta-chlorinated biphenyls (PCBs) in waters and wastewaters (containing less than 1 % suspended solids) using high-resolution gas chromatography/high-resolution mass spectrometry (HRGC/HRMS). The method is optimized for dioxin-like PCBs, but can include other co-planar compounds such as polychlorinated dioxins and furans (PCDDs/PCDFs) and polychlorinated naphthalenes (PCNs). This method can be used to determine dioxin-like PCBs in other matrices (e.g. biota, sediments, air); however, additional clean-up steps and techniques can be required for samples with high organic loadings.

This method is applicable to the twelve non- and mono-*ortho* PCBs designated by the World Health Organization, as well as to other PCBs and co-planar compounds.

The detection limits and quantification levels in this method are dependent on the level of interferences as well as instrumental limitations. The minimum levels (ML) in Table 2 are the levels at which the dioxin-like PCBs can typically be determined with no interferences present.

This method is “performance based”. The analyst is permitted to modify the method to overcome interferences or lower the cost of measurements, provided that all performance criteria in this method are met. The requirements for establishing method equivalency are given in 9.2.

2 Normative references

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

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

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

ISO 5667-2, *Water quality — Sampling — Part 2: Guidance on sampling techniques*