ISO 17858:2007-02 (E)

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

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**