

# ISO 17858:2007-02 (E)

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

---

<b>Contents</b>		<b>Page</b>
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

<b>Bibliography .....</b>	<b>54</b>
<b>Table 1 -- Dioxin-like PCBs determined by this method .....</b>	<b>38</b>
<b>Table 2 -- Suggested quantification relationships .....</b>	<b>39</b>
<b>Table 3 -- Suggested calibration standard concentrations .....</b>	<b>40</b>
<b>Table 4 -- Suggested concentration of dioxin-like PCBs in stock and spiking solutions .....</b>	<b>41</b>
<b>Table 5 -- Typical GC columns and temperature programs .....</b>	<b>42</b>
<b>Table 6 -- Examples of toxic equivalent factors .....</b>	<b>43</b>
<b>Table 7 -- Congener function groups and ions .....</b>	<b>44</b>
<b>Table B.1 -- TetraCBs .....</b>	<b>49</b>
<b>Table B.2 -- PentaCBs .....</b>	<b>49</b>
<b>Table B.3 -- HexaCBs .....</b>	<b>49</b>
<b>Table B.4 -- HeptaCBs .....</b>	<b>49</b>
<b>Table C.1 -- Spiking amounts transferred to sample bottles .....</b>	<b>50</b>
<b>Table C.2 -- Samples 1 and 2 (fortified industrial effluent) -- Statistical summary .....</b>	<b>51</b>
<b>Table C.3 -- Sample 3 (unfortified industrial effluent) -- Statistical summary .....</b>	<b>52</b>
<b>Table C.4 -- Sample 4 (HPLC water) -- Statistical summary .....</b>	<b>53</b>