

# DIN EN 1948-4:2010-12 (E)

## Stationary source emissions - Determination of the mass concentration of PCDDs/PCDFs and dioxin-like PCBs - Part 4: Sampling and analysis of dioxin-like PCBs

---

<b>Contents</b>		<b>Page</b>
Foreword .....		4
Introduction .....		5
1	Scope .....	6
2	Normative references .....	7
3	Terms and definitions .....	7
4	Symbols and abbreviations .....	10
4.1	General .....	10
4.2	Polychlorinated biphenyls .....	10
5	Principle of the measurement procedure .....	11
6	Device, materials and 13 C12-labelled standards .....	12
6.1	Device and materials .....	12
6.2	13 C12-labelled standards .....	12
7	Safety measures .....	13
8	Measurement procedure .....	13
8.1	Sampling .....	13
8.2	Extraction .....	13
8.3	Clean-up .....	14
8.4	Final concentration of the sample extracts .....	14
8.5	Addition of recovery standards .....	15
8.6	Principle of identification and quantification .....	15
8.7	Calibration of the HRGC/HRMS .....	15
8.8	Quantification of HRGC/HRMS results .....	17
8.8.1	Quantification of the sample .....	17
8.8.2	Calculation of the recovery rates of the extraction standards .....	18
8.8.3	Calculation of the recovery rates of the sampling standards .....	19
8.9	Calculation of the measurement results .....	19
8.10	Analytical report .....	20
9	Method validation .....	21
9.1	General .....	21
9.2	Validation of sampling .....	21
9.3	Validation of Analytical Extraction and Clean-up .....	22
9.3.1	Extraction .....	22
9.3.2	Clean-up .....	22
10	Quality control requirements for the measurement .....	22
10.1	Use of a validated method .....	22
10.2	Use of 13 C12-labelled standards .....	22
10.3	Minimum requirements for sampling .....	23
10.4	Minimum requirements for extraction and clean-up .....	23

<b>10.5</b>	<b>Minimum requirements for identification of PCB congeners .....</b>	<b>24</b>
<b>10.6</b>	<b>Minimum requirements for quantification .....</b>	<b>25</b>
<b>11</b>	<b>Quality assurance criteria for extraction/clean-up/quantification procedure blanks .....</b>	<b>25</b>
<b>11.1</b>	<b>Analytical blank .....</b>	<b>25</b>
<b>11.2</b>	<b>If the analytical blank values exceed the values mentioned above, the laboratory specific quantification limit has to be adopted (increased) correspondingly. HRGC/HRMS blank ..</b>	<b>26</b>
<b>12</b>	<b>Performance characteristics .....</b>	<b>26</b>
<b>12.1</b>	<b>General .....</b>	<b>26</b>
<b>12.2</b>	<b>Results of the validation campaign .....</b>	<b>27</b>
<b>13</b>	<b>Interferences (informative) .....</b>	<b>28</b>
<b>Annex A (informative) Toxicity and toxic equivalency .....</b>		<b>29</b>
<b>Annex B (informative) Examples of extraction and clean-up procedures .....</b>		<b>30</b>
<b>Annex C (informative) Evaluation of the performance characteristics .....</b>		<b>41</b>
<b>Annex D (informative) Recommendations for measuring high concentrations of dioxin-like PCBs ..</b>		<b>48</b>
<b>Annex E (informative) Possible interferences in dioxin-like PCB analysis .....</b>		<b>49</b>
<b>Annex F (informative) Measurement of the marker PCBs 28, 52, 101, 138, 153, and 180 in addition to the 12 dioxin-like PCBs .....</b>		<b>52</b>
<b>Annex G (informative) Measurement of hexachlorobenzene (HCB) .....</b>		<b>55</b>
<b>Annex H (informative) Significant technical changes .....</b>		<b>56</b>
<b>Annex ZA (informative) Relationship between this European Standard and the essential requirements of EU Directives .....</b>		<b>57</b>
<b>Bibliography .....</b>		<b>58</b>