

Tab 3: PCPh- and PCN-recovery rates for the two samples A and B spiked with native compounds

	Added amount of the native compound [ng/sample]	Analytically detected amount of the native compound [ng/sample]		Recovery rate of the native compound [%]	
Sample	A/B	A	B	A	B
Chlorophenols					
2,4,6-Trichlorophenol	1000	1013	1041	101	104
2,3,6-Trichlorophenol	1000	945	962	95	96
2,3,5-Trichlorophenol	1000	957	937	96	94
2,4,5-Trichlorophenol	1000	994	965	99	97
2,3,4-Trichlorophenol	1000	1057	994	106	99
3,4,5-Trichlorophenol	1000	988	968	99	97
2,3,5,6-Tetrachlorophenol	1000	916	1030	92	103
2,3,4,6-Tetrachlorophenol	1000	1018	1068	102	107
2,3,4,5-Tetrachlorophenol	1000	1087	1125	109	112
Pentachlorophenol (PCP)	448	462	476	103	106
PCN compounds					
2-MonoCN	1000	967	1036	97	104
1-MonoCN	1000	1051	1026	105	103
1,4-DiCN	1000	945	924	95	92
1,5-DiCN	1000	990	959	99	96
2,7-DiCN	1000	969	917	97	92
1,2-DiCN	1000	1024	876	102	88
2,3-DiCN	1000	1020	898	102	90
1,8-DiCN	1000	1032	909	103	91
1,2,7-TriCN	1000	1023	1028	102	103
1,2,3,4-TetraCN	1000	906	781	91	78
1,2,3,6,7-PentaCN	1000	1265	1022	126	102
1,2,3,4,6,7/1,2,3,5,6,7-HexaCN	2000	2518	2333	126	117
1,2,3,4,5,6,7-HeptaCN	1000	1228	1151	123	115
OctaCN	1000	1235	1239	124	124

The recoveries for PCDD/Fs are in the range of the required percentage with 76 to 120 % for Tetra- through HexaCDD/F and 68 to 92 % for Hepta- through OctaCDD/F. All other organic components show high recovery rates mostly between 90 and 110 %. This confirms that the extraction procedure acc. to the EN 1948 standard provides high extraction efficiency for other organic trace compounds as well.

References

1. DIN EN 1948, *Stationary Source Emissions*, Part 2: Extraction and clean-up, Ref.-No. EN 1948-2:2006 D

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EXTRACTION EFFICIENCY ON THE ANALYSIS OF “NON-DIOXIN-COMPOUNDS” BY THE EN 1948 METHOD

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Abstract

The European EN 1948 standard procedure provides good extraction efficiencies performing PCDD/F analyses of exhaust gas samples. Good recoveries are also stated for other organic trace contaminants but not yet verified. This paper reports about some data on the extraction efficiencies by the EN 1948 procedure for PCBs, PAHs, PCBzs, PCPhs, PCNs and Lindane.

Introduction

The basics of sampling and analysis of polychlorinated dibenzo(p)dioxins and dibenzofurans (PCDD/F) in exhaust gas from stationary sources are laid down in the European standard EN 1948.¹ Part 1 describes the sampling while parts 2 and 3 are dealing with the extraction, the clean-up procedure and the analysis by HRGC/HRMS.

The EN 1948 points out that the described methods are also valid to collect and analyse for more volatile compounds e.g. dioxin-like PCBs (12 non-ortho and mono-ortho polychlorinated biphenyls as specified by the WHO), although there are no performance characteristics available yet. Above this, compounds like polycyclic aromatic hydrocarbons (PAHs), polychlorinated benzenes (PCBzs), polychlorinated phenols (PCPhs), polychlorinated naphthalenes (PCNs) and Lindane (γ-hexachloro-cyclohexane) in practice are as well collected and analysed within the same sample. Of course, the respective analytical procedures are substance specific.

Since the relevant ranges of concentrations of these “Non-Dioxin-Compounds” are, as a rule, significantly higher than the PCDD/F concentrations, always small amounts of the sample extracts (10 %) are used for clean-up and analysis. For the same reason internal standards of these compounds are not added until the raw extract has been divided. This procedure obviously excludes the direct control of the extraction efficiency. On the other hand the recovery rates of the routinely determined PCDD/F extraction standards provide a principal evidence for a directive-concurring procedure.

The herewith described investigations shall serve as a verification for the extraction efficiency of compounds “beyond” dioxins but according to the extraction procedure given in EN 1948, part 2.

Materials and Methods

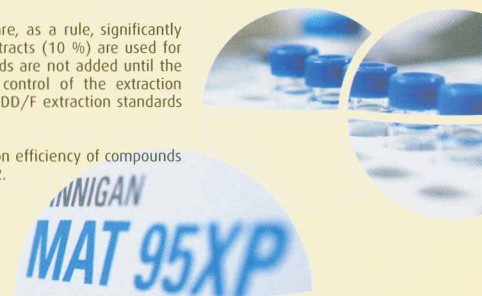
Two collection units (A and B) were used for the tests, each consisting of

- filter cartridge plugged with quartz wool
- cartridge filled with XAD resin (30 g) and a plane filter inlet
- cartridge filled with XAD resin (30 g)

Prior to extraction each collection unit was spiked with ¹³C₁₂-labelled PCDD/F-standards acc. to EN 1948 and D8-Naphthalene. In addition the units were spiked with defined amounts of native standards of all substance classes regarded here. The sample preparation and extraction was performed according to the EN 1948 method as well as the clean-up and analysis of the PCDD/F. Variations have been made for the clean-up procedure of the other substance classes according to validated and accredited in-house methods. All components were identified and quantified by means of GC/HRMS (Thermo Finnigan MAT 95 XP : PCDD/Fs and PCBs) resp. GC/LRMS analysis (Thermo Finnigan Trace GC Ultra : others).

Results and Discussion

The following tables and figures summarize the results of the recoveries of the native compounds by which the collection units have been spiked prior to sample preparation and extraction.



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Fig 1: PCDD/F-recovery rates of the sampling standards and the extraction internal standards in two samples A and B (spiked amount of $^{13}\text{C}_{12}$ -labelled PCDD/F: 800 ng/sample for Hepta-/OctaCDD/F, 400 ng/sample for others)



Tab 1: PCB-recovery rates for the two samples A and B spiked with native PCBs

	Added amount of the native PCB [ng/sample]	Analytically detected amount of the native PCB [ng/sample]		Recovery rate of the native PCB [%]	
Sample	A/B	A	B	A	B
Non-ortho PCB					
PCB 77	400	390	381	98	95
PCB 81	400	396	372	99	93
PCB 126	400	374	347	94	87
PCB 169	400	379	351	95	88
Mono-ortho PCB					
PCB 105	400	402	379	101	95
PCB 114	400	401	389	100	97
PCB 118	400	359	355	90	89
PCB 123	400	406	373	102	93
PCB 156	400	376	350	94	88
PCB 157	400	384	359	96	90
PCB 167	400	421	373	105	93
PCB 189	400	401	386	100	97

Tab 2: PAH-/PCBz- and Lindane-recovery rates for the two samples A and B spiked with native compounds

	Added amount of the native compound [ng/sample]	Analytically detected amount of the native compound [ng/sample]		Recovery rate of the native compound [%]	
Sample	A/B	A	B	A	B
PAH compounds					
D ₈ -Naphthalene	5000	4632	4576	93	92
Acenaphthylene	800	776	800	97	100
Acenaphthene	800	824	999	103	125
Fluorene	800	945	990	118	124
Phenanthrene	800	815	917	102	115
Anthracene	800	795	804	99	101
Fluoranthene	800	894	1063	112	133
Pyrene	800	792	810	99	101
Benzo(a)anthracene	800	780	789	98	99
Chrysene	800	788	800	99	100
Benzo(b/j)fluoranthene	800	784	800	98	100
Benzo(k)fluoranthene	800	785	796	98	100
Benzo(a)pyrene	800	774	767	97	96
Dibenzo(a,h/a,c)-anthracene	800	819	810	102	101
Benzo(g,h,i)perylene	800	827	804	103	101
Indeno(1,2,3-c,d)-pyrene	800	789	819	99	102
Chlorobenzenes					
1,3,5-Trichlorobenzene	800	668	694	84	87
1,2,4-Trichlorobenzene	800	984	920	123	115
1,2,3-Trichlorobenzene	800	908	854	114	107
1,2,3,5-Tetrachlorobenzene	800	806	799	101	100
1,2,4,5-Tetrachlorobenzene	800	832	840	104	105
1,2,3,4-Tetrachlorobenzene	800	908	873	114	109
1,2,3,4,5-Pentachlorobenzene	800	803	823	100	103
1,2,3,4,5,6-Hexachlorobenzene	800	823	831	103	104
Lindane	1000	1024	1015	102	101