THE USE OF INTERCALIBRATIONS AS A TOOL TO BE ABLE TO CONTROL AND CONFIRM THAT THE QUALITY IS UNDER CONTROL AFTER RENEWING PERSONNEL AND LOCATION

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Introduction

The mean of this study is to give an explanation how we confirmed and assured that the laboratory quality was not disturbed by the changes in location and new personnel. The methods used were not changed and are based on EPA 1613[1] and SFS-EN 1948:Part 1-3. The CND1, 2013 exercise was performed in Raisio and the CIND2, 2014 was performed in Kaustinen[6]. We had to teach new personnel the extraction procedures and clean-up steps used[2,4,5]. None of the existing personnel moved to the new location. The High Resolution Instrument, a Jeol SX 102, was not moved. In this presentation it is proved that the quality of the analysis for PCDD/F and PCBs has been maintained.

Materials and methods

Both the sediment samples and the fly ash samples were extracted using toluene Soxhlet extraction for 24 hours. The fly ash was acid threated with HCL before the extraction[9]. All corresponding C^{13}-labelled internal standards were added before the extraction (S_6, S_7 and WHO12PCB). The extracts were primary cleaned by silica/acid silica column chromatography. After this stage the dioxins and furans (PCDD/F) were separated from the PCBs using Florisil column chromatography[5]. The dioxin fraction was further cleaned using basic alumina (Al_2O_3, Super I active) and injection standards were added to the vial. The volume was reduced to 25 µl and then analyzed on a High Resolution GC-MS. The column used was a 60 m DB-5ms capillary column (60m, 0.25mm, 0.25µm): Injector temperature 290 °C, split-less-injection 1.0 minute, transfer line temperature 295 °C, detector temperature 250 °C. Carrier gas He 4.6 and pressure 30 psi (at 180 °C). The oven temperature program used was:180 °C (2 min)-4 °C/min-220 °C (12 min)-5 °C/min-235 °C (7 min)- 5 °C/min-330 °C (2,8 min). The PCBs were analyzed directly after the Florisil clean-up, final volume 50µl dodecane, on the High Resolution GC-MS. Same column and temperatures were used as for the PCDD/F analyzes[3]. Optionally PAH compounds were analyzed from a separate extract obtained by ASE 300 extraction using n-hexane or n-hexane:acetone (1:1 V/V) analyzed using SIM on a GC-MSD[7].

Results and discussion

The data given for CIND1 have already been threated and z-scores are available. The data for the fly ash have not yet been confirmed but we are confident that they are of good quality. The data is presented in the Table 1. Also our laboratory identification is presented in the Table 1. The standard deviations for the three separate extractions of the same material are good. It is shown that we have a good reproducibility. The conclusion of the studies shows that we have been able to maintain the quality of the analyses, even after the changes in location and personnel, for the presented compounds. This was of very great importance for us and our customers. In this way is it possible to test, control and verify quality, when making changes to locations and personnel. In the hard economic climate this is of great importance in the future. We want to show an efficient and economical way to tackle the problems, with verifying that the problems created by moving the Laboratory from one part of the country to another part, are under control.

Table 1. The results for the different compounds reported for the two studies are presented in this table. The compounds analyzed are given in the table.

PARTICIPANT CODE:	**LAB 063**			**LAB 36**		
	CIND01			**CIND02**		
Sample:	**SEDIMENT**	**2013**		**FLY ASH**	**2014**	
PCDD/F	**A**	**B**	**C**	**A**	**B**	**C**
Weight Analysed (g):	**8,96**	**6,99**	**6,94**	**5,06**	**5,06**	**5,03**
	Sample	**Sample**	**Sample**	**Sample**	**Sample**	**Sample**
	(pg/g)	**(pg/g)**	**(pg/g)**	**(pg/g)**	**(pg/g)**	**(pg/g)**
2,3,7,8-TeCDD	0,91	1,01	0,34	2162,46	2212,38	2048,44
1,2,3,7,8-PeCDD	0,00	0,00	0,00	9771,48	10588,96	11391,67
1,2,3,4,7,8-HxCDD	< 0,0010	< 0,0010	< 0,0010	11800,05	9850,68	11074,04
1,2,3,6,7,8-HxCDD	0,98	0,42	0,36	22412,64	20141,09	20665,91
1,2,3,7,8,9-HxCDD	0,68	0,48	0,29	24368,16	24915,63	23645,83
1,2,3,4,6,7,8-HpCDD	11,61	5,73	4,99	127389,65	110341,61	121911,16
OCDD	62,96	27,35	32,73	120077,72	107849,28	120597,16
2,3,7,8-TeCDF	1,07	2,24	1,25	10100,97	9798,76	9574,87
1,2,3,7,8-PeCDF	1,86	2,49	2,32	14423,53	14616,19	14827,77
2,3,4,7,8-PeCDF	2,39	2,90	2,33	31585,12	28385,06	29535,31
1,2,3,4,7,8-HxCDF	10,15	9,58	8,86	22979,06	23047,40	24193,79
1,2,3,6,7,8-HxCDF	6,43	4,48	4,53	23501,05	21537,39	24484,23
1,2,3,7,8,9-HxCDF	3,64	4,15	3,67	7651,41	7072,74	8685,86
2,3,4,6,7,8-HxCDF	0,58	0,95	0,90	39290,20	34280,42	39136,34
1,2,3,4,6,7,8-HpCDF	43,94	37,07	42,59	77939,67	67280,81	72449,54
1,2,3,4,7,8,9-HpCDF	6,41	6,75	5,72	16409,52	13342,86	14920,27
OCDF	112,16	110,04	102,13	31722,60	32261,38	31825,51
TEQ (PCDD/F)	4,71	4,72	3,67	40315,46	38771,43	41030,17
TEQ (PCDD/F) Upperbound (UpB)	4,71	4,72	3,67	40315,46	38771,43	41030,17
Z-score	-0.47	- 0.46	- 1,34	ny*	ny	ny
PCB-DL	**A**	**B**	**C**	**A**	**B**	**C**
Weight Analysed (g):	**8,96**	**6,99**	**6,94**	**5,06**	**5,06**	**5,03**
	Sample	**Sample**	**Sample**	**Sample**	**Sample**	**Sample**
	(pg/g)	(pg/g)	(pg/g)	(pg/g)	(pg/g)	(pg/g)
PCB #77	14,87	12,99	11,78	5359,67	5936,21	5979,86
PCB #81	< 0,05	< 0,05	< 0,05	1433,54	1591,15	1579,85
PCB #105	69,42	46,07	47,61	1841,09	1901,24	1851,06
PCB #114	2,06	2,10	< 0,05	478,32	327,83	479,18
PCB #118	237,82	190,99	180,35	2230,45	2406,94	2349,24
PCB #123	13,00	6,00	6,32	484,70	480,11	502,31
PCB #126	4,41	3,56	3,76	3881,34	4041,19	4183,43

PCB #156	41,35	28,50	28,83	2391,41	2607,35	2733,30
PCB #157	11,64	6,13	5,11	1400,66	1537,17	1610,08
PCB #167	23,36	13,07	16,21	1006,98	1109,67	1183,66
PCB #169	0,82	3,96	3,90	1570,05	1807,27	1811,62
PCB #189	6,22	5,49	4,31	1435,82	1564,96	1787,16
TEQ (PCB)	0,48	0,48	0,50	436,54	459,77	474,14
TEQ (PCB) Upperbound	0,48	0,48	0,50	436,54	459,77	474,14
TEQ Total (PCDD/F+PCB)	5,19	5,20	4,17	40752,00	39231,20	41504,31
TEQ Total (PCDD/F+PCB) UpB	5,19	5,20	4,17	40752,00	39231,20	41504,31
PCB ICES-6	**A**	**B**	**C**	**A**	**B**	**C**
Weight Analysed (g):	**8,96**	**6,99**	**6,94**	**1,09**	**1,09**	**1,01**
	Sample	**Sample**	**Sample**	**Sample**	**Sample**	**Sample**
	(ng/g)	**(ng/g)**	**(ng/g)**	**(ng/g)**	**(ng/g)**	**(ng/g)**
PCB #28	0,25	0,21	0,24	0,79	0,53	0,81
PCB #52	0,24	0,27	0,26	0,55	0,55	0,36
PCB #101	0,41	0,32	0,43	0,95	0,86	0,95
PCB #138	0,64	0,53	0,48	1,27	1,18	1,75
PCB #153	0,66	0,55	0,55	0,96	0,85	0,89
PCB #180	0,35	0,27	0,20	0,80	0,78	0,69
Total PCB ICES-6	**2,53**	**2,17**	**2,16**	**5,32**	**4,74**	**5,45**
PAH	**A**	**B**	**C**	**A**	**B**	**C**
Weight Analysed (g):	**8,96**	**6,99**	**6,94**	**1,01**	**0,98**	**1,01**
	Sample	**Sample**	**Sample**	**Sample**	**Sample**	**Sample**
	(ng/g)	**(ng/g)**	**(ng/g)**	**(ng/g)**	**(ng/g)**	**(ng/g)**
Benzo[a]anthracene	11,58	10,31	11,01	89,53	63,56	83,96
Chrysene	16,19	14,94	13,71	481,68	402,36	470,76
Benzo[b+j+k]fluoranthene	33,52	30,72	29,51	517,93	467,12	489,78
Benzo[a]pyrene	9,91	8,54	8,95	23,24	27,80	25,47
Indeno[1,2,3cd]pyrene	14,27	13,46	12,76	177,95	161,89	165,10
Dibenzo[a,h]anthracene	3,29	2,92	2,54	27,72	27,25	25,30
Benzo[ghi]perylene	18,54	17,60	16,15	153,69	166,41	205,32
TOTAL ΣPAH	107,30	98,50	94,63	1471,74	1316,39	1465,69
TEQ (PAH)	0,01	0,01	0,01	0,18	0,16	0,17
TEQ (PAH) Upperbound	0,01	0,01	0,01	0,18	0,16	0,17
TEQ Total (PCDD/F+PCB+PAH)	19,68	18,34	17,01	40929,05	39392,77	41672,42
TEQ Total (PCDD/F+PCB+PAH) UpB	19,68	18,34	17,01	40929,05	39392,77	41672,42
Z-scores	-0.87	-1.10	-1.34	ny*	ny	ny

*ny = Not yet known

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