Results from the Third Round of the International Intercalibration Study on PCDDs, PCDFs and planar PCBs: Part 2 Soil/Sewage Sludge

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Introduction

As part of the third round of the international intercalibration, a laboratory inter-comparison study on soil and sewage sludge samples was organised. Both soil and sludge samples can contain dioxin levels in varying concentration. This introduces extra difficulties in the determination of PCDDs, PCDFs and planar PCBs for this kind of samples. To assure the quality of the analytical data it is important that inter-laboratory comparison studies are organised. The here presented study is an example of such a study.

Material and Methods

For the first part of the study three soil samples were distributed from two sample sites. Before distribution the samples were homogenised, sieved (0.5cm) and air-dried for 48 hours. The soils were than ground and homogenised again and put in small vials for shipment. Soil A and B originated from the same industrial site whereas soil C was taken from a former gasworks facility. Two sewage sludge samples prepared the same way, apart from a longer drying period (5 days), and a standard solution were send for the second part of the study. All participants were asked to consider the samples as a routine sample and use their own extraction and clean up protocols plus quantification standards.

Results and Discussion

In total 32 labs participated of which 29 were able to submit the results before the expiration of the dead line. The results for the best performing labs for first three soil are given in Table 1. The inter-laboratory variation between the samples from the same batch (A and B) was small compared to the variation between the different laboratories. The results for the highly contaminated showed good agreement between the participants. Although the samples were also highly contaminated with polychlorinated naphthalene's (PCNs) were most labs able to report the high levels. PCDFs mainly dominated the TEQs and larger variation in lower levels of PCDDs did not influence the TEQ results. The low-level soil C showed much larger variation at levels just above the detection limit for most labs. Also the extraction efficiency of this soil with an extreme high carbon content might contribute to the large variation in results.

Table 1 The results of intercalibration 2 Soil (Part 1)

Soil A
(Statistics of the results of the 28 best performing labs out of a total of 29)

	Average	Median	Min	Max	RSD	%RSD
2,3,7,8-TeCDD	0.014	0.015	0.003	0.030	0.008	56%
1,2,3,7,8-PeCDD	0.02	0.01	0.01	0.17	0.04	174%
1,2,3,4,7,8-HxCDD	0.01	0.01	0.00	0.03	0.01	62%
1,2,3,6,7,8-HxCDD	0.01	0.01	0.01	0.04	0.01	65%
1,2,3,7,8,9-HxCDD	0.02	0.01	0.00	0.16	0.04	186%
1,2,3,4,6,7,8-HpCDD	0.09	0.08	0.06	0.17	0.03	30%
OCDD	0.6	0.5	0.4	1.0	0.2	29%
2,3,7,8-TeCDF	22.20	22.50	15.00	29.59	4.82	22%
1,2,3,7,8-PeCDF	10.05	9.08	6.22	20.84	3.46	34%
2,3,4,7,8-PeCDF	8.29	7.91	5.67	14.30	1.71	21%
1,2,3,4,7,8-HxCDF	8.29	8.28	6.01	11.02	0.96	12%
1,2,3,6,7,8-HxCDF	1.79	1.72	1.34	2.56	0.29	16%
1,2,3,7,8,9-HxCDF	0.39	0.18	0.09	1.59	0.42	107%
2,3,4,6,7,8-HxCDF	0.66	0.72	0.16	1.00	0.25	39%
1,2,3,4,6,7,8-HpCDF	2.0	1.9	1.3	3.0	0.4	21%
1,2,3,4,7,8,9-HpCDF	0.6	0.6	0.480	0.9	0.1	16%
OCDF	4.3	4.1	2.864	10.1	1.5	34%
PCB #77	0.47	0.41	0.16	1.77	0.40	86%
PCB #126	0.06	0.05	0.02	0.20	0.04	68%
PCB #169	0.03	0.02	0.01	0.12	0.03	104%
TEQ	**7.99**	**7.89**	**6.11**	**11.20**	**1.25**	**16%**

Soil B
(Statistics of the results of all 29 reporting labs)

	Average	Median	Min	Max	RSD	%RSD
2,3,7,8-TeCDD	0.012	0.013	0.002	0.020	0.006	51%
1,2,3,7,8-PeCDD	0.03	0.01	0.00	0.16	0.05	159%
1,2,3,4,7,8-HxCDD	0.02	0.01	0.00	0.12	0.03	161%
1,2,3,6,7,8-HxCDD	0.02	0.01	0.01	0.16	0.03	183%
1,2,3,7,8,9-HxCDD	0.02	0.01	0.00	0.14	0.04	163%
1,2,3,4,6,7,8-HpCDD	0.11	0.08	0.06	0.25	0.06	57%
OCDD	1	1	0	2	0	41%
2,3,7,8-TeCDF	20.87	19.78	13.26	29.90	4.63	22%
1,2,3,7,8-PeCDF	9.41	8.63	5.27	18.22	3.05	32%
2,3,4,7,8-PeCDF	7.96	7.99	4.58	11.83	1.31	16%
1,2,3,4,7,8-HxCDF	7.79	7.95	4.76	10.21	1.15	15%
1,2,3,6,7,8-HxCDF	1.67	1.68	1.20	2.41	0.27	16%
1,2,3,7,8,9-HxCDF	0.50	0.17	0.11	3.42	0.73	146%
2,3,4,6,7,8-HxCDF	0.65	0.66	0.15	1.66	0.30	47%
1,2,3,4,6,7,8-HpCDF	1.8	1.8	1.2	2.9	0.4	21%
1,2,3,4,7,8,9-HpCDF	0.6	0.6	0.4	0.9	0.1	18%
OCDF	4.0	3.8	2.7	6.6	0.8	19%
PCB #77	0.43	0.34	0.13	1.46	0.34	78%
PCB #126	0.05	0.05	0.02	0.10	0.02	45%
PCB #169	0.03	0.02	0.01	0.13	0.04	124%
TEQ	**7.62**	**7.80**	**5.00**	**9.75**	**1.07**	**14%**

Table 1 Continued.

Soil C
(Statistics of the results of the all 29 reporting labs)*

	Average	Median	Min	Max	RSD	%RSD
2,3,7,8-TeCDD	0.002	0.001	0.000	0.010	0.002	132%
1,2,3,7,8-PeCDD	0.003	0.002	0.001	0.008	0.002	68%
1,2,3,4,7,8-HxCDD	0.002	0.001	0.001	0.007	0.002	91%
1,2,3,6,7,8-HxCDD	0.004	0.003	0.001	0.010	0.002	66%
1,2,3,7,8,9-HxCDD	0.003	0.002	0.001	0.015	0.003	96%
1,2,3,4,6,7,8-HpCDD	0.034	0.010	0.005	0.490	0.100	290%
OCDD	0.14	0.03	0.01	2.45	0.50	349%
2,3,7,8-TeCDF	0.016	0.011	0.006	0.063	0.013	77%
1,2,3,7,8-PeCDF	0.007	0.006	0.003	0.017	0.004	54%
2,3,4,7,8-PeCDF	0.006	0.005	0.003	0.014	0.003	50%
1,2,3,4,7,8-HxCDF	0.008	0.006	0.004	0.018	0.005	57%
1,2,3,6,7,8-HxCDF	0.005	0.003	0.002	0.014	0.004	84%
1,2,3,7,8,9-HxCDF	0.003	0.001	0.000	0.011	0.004	138%
2,3,4,6,7,8-HxCDF	0.003	0.002	0.000	0.018	0.004	120%
1,2,3,4,6,7,8-HpCDF	0.02	0.01	0.00	0.15	0.03	131%
1,2,3,4,7,8,9-HpCDF	0.002	0.00	0.00	0.01	0.00	137%
OCDF	0.02	0.01	0.00	0.13	0.03	119%
PCB #77	0.044	0.016	0.000	0.230	0.066	150%
PCB #126	0.009	0.002	0.000	0.050	0.016	182%
PCB #169	0.010	0.000	0.000	0.050	0.018	179%
TEQ	**0.010**	**0.007**	**0.001**	**0.02**	**0.01**	**63%**

* Three participants only reported detection limits for this low contaminated soil.

Conclusions

The analysis of soil samples with high concentrations of PCDFs showed good agreement among all participants (RSD 14-16%). Interference's of high levels of PCNs were present but did not seem to influence the results. The results from a low-level soil with a high carbon content showed much larger variation among the laboratories.

Intercalibration exercises are an essential tool in the assurance of the quality of dioxin analysis. These kinds of studies enable laboratories to improve their analytical capacity or confirm their capability. This way data acquired by different laboratories will be directly compatible, both form a scientific and a legislative point of view.

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Table 2. Participants in the third round of the International Intercalibration

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