Results from the first Round of the Korea-Interlab Calibration Study on PCDDs, PCDFs : Incineration Extract

Chang Han Joo[1], Seok Won Eom[2], Sam Cwan Kim[3], Seong Ki Jang[3], Jae Cheon You[1]

[1]Analytical Research Center, Environmental Management Co., Mt 26-4, Hasanwon-Dong, Boondang Gu, Sungnam Si, Kyonggi Do, Korea
[2]Metropolitan Government Institute of Health and Environment
[3]National Institute of Environmental Research (NIER)

Introduction

The accuracy of data in the PCDDs/PCDFs analysis is required to make the inventory and restrict the emission and establish the environmental policy. Dioxin analysis includes many steps such as pretreatment, cleanup, instrumental analysis. Due to various causes error would be produced and the interpretation of result will have error. Four Laboratories which having ability of Dioxin Analysis in Korea, analyzed the same samples and compared with the results from four laboratories. Through this study we knew about the repeatability of three injections in each lab and compared the results of two extracts A,B among the labs. RSDs of TEQ of the two samples were below 3%.

Material and Methods

Two flue gas samples in two kinds of MSWIs were prepared by soxhlet extration with toluene for over 16hrs for this calibration. The same amount of the extract was distributed to the four laboratories. They used the korean official method and also used the SP-2331 column for the comparision of result in same conditions except instrument [1]. Each lab analyzed two samples three times and got average TEQ values which calculated with i-Factor.

Results and Discussion

The results of three times injections to each sample in each lab was 4 8% of RSD in sample A, 5 10% in sample B except OCDF. The larger variation of OCDF than other isomers due to low sensitivity of OCDF in SP2331 did not influence total TEQ values because of its low i-TEQ factor. And another isomers which have more variation relatively (over RSD of 20%) were 2,3,7,8-TCDD, 1,2,3,7,8,9-HxCDF etc. As shown in Table 3 the RSD of total TEQ values were

3% and 1.6% in sample A and B, respectively. The recoveries of internal standard were obtained 5□117.2% which satisfied to the QC criteria of the method in Table 4.

Table 1. Analytical Results of Sample A unit : pg-TEQ

	L-1			L-2			L-3			L-4		
2,3,7,8-TCDD	3.37	4.38	4.47	7.2	7.8	8.4	4.19	5.16	4.78	5.29	4.71	4.06
1,2,3,7,8-PeCDD	6.24	6.64	6.9	8.8	7.4	7.6	8.51	7.79	8.08	7.18	4.71	4.79
1,2,3,4,7,8-HxCDD	2.12	2.23	2.12	2.6	2.6	2.6	2.27	2.19	2.02	2.63	2.18	2.25
1,2,3,6,7,8-HxCDD	4.70	4.90	4.30	5.2	5.2	4.8	4.73	4.56	4.37	4.94	4.32	4.27
1,2,3,7,8,9-HxCDD	2.41	2.50	2.64	3.0	3.2	3.0	3.10	3.29	3.19	3.56	2.76	2.58
1,2,3,4,6,7,8-HpCDD	3.29	3.27	3.23	3.6	3.8	3.6	2.91	2.97	2.91	3.22	3.13	3.14
OCDD	0.52	0.52	0.51	0.4	0.4	0.4	0.50	1.49	0.48	0.51	0.50	0.51
2,3.7,8-TCDF	1.58	1.80	1.79	1.8	2.2	1.6	1.82	1.63	1.71	1.57	1.41	1.62
1,2,3,7,8-PeCDF	4.17	4.31	4.15	3.2	3.0	3.0	3.64	3.63	3.6	2.64	2.22	2.26
2,3,4,7,8-PeCDF	41.4	40.92	40.39	30.4	33.6	32.8	39.32	39.99	38.26	42.60	39.28	39.90
1,2,3,4,7,8-HxCDF	10.62	10.48	9.99	10.6	11.2	11.4	9.59	9.38	9.26	10.43	9.46	10.02
1,2,3,6,7,8-HxCDF	11.66	10.70	11.4	12.8	11.2	12.0	10.08	10.64	10.09	11.19	10.82	10.94
2,3,4,6,7,8-HxCDF	16.09	16.75	15.89	17.6	18.6	17.8	16.84	14.92	15.42	16.47	16.19	16.06
1,2,3,7,8,9-HxCDF	1.70	1.43	1.64	2.0	1.8	1.8	1.55	2.01	1.95	2.28	1.57	1.55
1,2,3,4,6,7,8-HpCDF	4.96	4.98	4.86	4.0	4.4	4.2	4.32	4.29	4.29	4.58	4.55	4.53
1,2,3,4,7,8,9-HpCDF	0.61	0.66	0.62	0.6	0.8	0.6	0.68	0.61	0.63	0.59	0.55	0.58
OCDF	0.54	0.59	0.60	0	0	0	0	0.20	0.20	0.06	0.05	0
∑PCDD	22.65	24.44	24.17	30.80	30.40	30.40	26.21	26.45	25.83	27.33	22.31	21.60
∑PCDF	93.33	92.62	91.33	83.00	86.80	85.20	87.84	86.30	85.41	92.41	86.10	87.46
∑PCDD+∑PCDF	115.98	117.06	115.50	113.80	117.20	115.60	114.05	112.75	111.24	119.74	108.41	109.06

Table 2. Analytical Results of Sample B unit:pg-TEQ

	L-1			L-2			L-3			L-4		
2,3,7,8-TCDD	4.00	4.12	4.02	5.6	5.6	5.2	6.17	4.75	8.31	3.45	3.52	3.12
1,2,3,7,8-PeCDD	13.9	13.51	13.29	13.8	10.8	14.4	16.72	13.52	15.17	13.77	13.79	13.22
1,2,3,4,7,8-HxCDD	3.55	3.37	3.66	4.2	4.6	4.4	3.85	4.62	4.44	3.59	3.60	3.47
1,2,3,6,7,8-HxCDD	6.38	5.81	6.77	7.6	7.2	7.4	6.39	7.16	7.61	6.53	6.64	6.40
1,2,3,7,8,9-HxCDD	3.16	3.12	3.18	5.4	4.6	4.6	3.83	4.29	5.32	3.59	4.00	3.31
1,2,3,4,6,7,8-HpCDD	1.88	1.83	1.87	2.2	2.2	2.4	1.72	2.13	2.02	1.85	1.82	1.82
OCDD	0.12	0.12	0.12	0.2	0.2	0.2	0.14	0.17	0.20	0.13	0.13	0.12
2,3.7,8-TCDF	2.35	2.57	2.37	2	3.6	3.2	2.82	3.06	2.92	2.35	2.54	2.61
1,2,3,7,8-PeCDF	7.33	7.19	7.33	7.8	7.8	7	6.49	6.51	6.87	6.68	6.82	6.85
2,3,4,7,8-PeCDF	103.84	103.19	106.26	86.8	77.2	82.8	101.19	102.23	95.48	106.72	102.94	101.04
1,2,3,4,7,8-HxCDF	19.30	18.96	19.12	20	21.4	23.4	17.33	17.97	18.65	18.17	18.84	18.68
1,2,3,6,7,8-HxCDF	24.00	24.48	23.64	28.4	27.2	25.6	21.70	22.23	22.48	22.62	23.19	22.81
2,3,4,6,7,8-HxCDF	45.51	43.67	45.55	52.6	54.4	52.2	45.50	50.18	47.53	46.31	45.82	45.48
1,2,3,7,8,9-HxCDF	2.32	1.43	2.2	2.4	4.4	3.4	2.17	2.34	3.70	2.06	2.38	2.22
1,2,3,4,6,7,8-HpCDF	3.65	4.98	3.79	4.6	4.4	4.4	3.36	3.60	3.99	3.72	3.57	3.63
1,2,3,4,7,8,9-HpCDF	0.55	0.66	0.55	0.6	0.6	0.4	0.70	0.75	0.71	0.52	0.53	0.55
OCDF	0.24	0.59	0.22	0.2	0	0	0	0.20	0	0	0	0
∑PCDD	32.99	31.88	32.91	39.00	34.20	37.60	38.82	36.64	43.07	32.91	33.5	31.46
∑PCDF	209.08	207.72	211.03	205.4	199.4	202.4	201.26	209.07	202.33	209.15	206.63	203.87
∑PCDD+∑PCDF	242.08	239.6	243.94	244.4	233.6	240.0	240.08	245.71	245.4	242.06	240.13	235.33

Table 3. The Result of Statistics of Sample A,B unit:pg-TEQ

	Sample A					Sample B				
	Max	**Min**	**Avg**	**SD**	**%RSD**	**Max**	**Min**	**Avg**	**SD**	**%RSD**
2,3,7,8-TCDD	8.4	3.37	5.32	1.6	30.1	8.31	3.12	4.66	1.45	31.1
1,2,3,7,8-PeCDD	8.8	4.71	7.05	1.3	18.5	16.72	10.8	13.82	1.37	9.9
1,2,3,4,7,8-HxCDD	2.63	2.02	2.32	0.22	9.7	4.62	3.37	3.95	0.47	12.0
1,2,3,6,7,8-HxCDD	5.2	4.27	4.69	0.33	7.1	7.61	5.81	6.82	0.57	8.3
1,2,3,7,8,9-HxCDD	3.56	2.41	2.94	0.36	12.1	5.4	3.12	4.03	0.82	20.2
1,2,3,4,6,7,8-HpCDD	3.8	2.91	3.26	0.28	8.7	2.4	1.72	1.98	0.21	10.5
OCDD	0.52	0.4	0.48	0.05	10.2	0.2	0.12	0.15	0.04	23.7
2,3.7,8-TCDF	2.2	1.41	1.71	0.2	11.5	3.6	2	2.7	0.44	16.4
1,2,3,7,8-PeCDF	4.31	2.22	3.32	0.72	21.7	7.8	6.49	7.06	0.44	6.3
2,3,4,7,8-PeCDF	42.6	30.4	38.16	3.8	10	106.72	77.2	97.47	9.81	10.1
1,2,3,4,7,8-HxCDF	11.4	9.26	10.2	0.7	6.9	23.4	17.33	19.32	1.65	8.5
1,2,3,6,7,8-HxCDF	12.8	10.08	11.13	0.78	7	28.4	21.7	24.03	2.07	8.6
2,3,4,6,7,8-HxCDF	18.6	14.92	16.55	1.04	6.3	54.4	43.67	47.9	3.51	7.3
1,2,3,7,8,9-HxCDF	2.28	1.43	1.77	0.25	14	3.7	1.43	2.45	0.6	24.6
1,2,3,4,6,7,8-HpCDF	4.98	4	4.5	0.31	6.9	4.98	3.36	3.97	0.5	12.6
1,2,3,4,7,8,9-HpCDF	0.8	0.55	0.63	0.06	10.2	0.75	0.4	0.59	0.1	16.6
OCDF	0.6	0	0.19	0.25	132	0.59	0	0.12	0.18	149.5
Σ PCDD	30.8	21.6	26.05	3.22	12.3	43.07	31.46	35.42	3.58	10.1
Σ PCDF	93.33	83	88.15	3.41	3.9	211.03	199.4	205.6	3.73	1.8
Σ PCDD+ Σ PCDF	119.74	108.41	114.20	3.38	3	245.7	233.6	241.03	3.76	1.6

Table 4. Internal Standard Recovery % (QC Criteria : 50□120%)

	Sample A				Sample B			
	L-1	**L-2**	**L-3**	**L-4**	**L-1**	**L-2**	**L-3**	**L-4**
13C-2,3,7,8-TCDD	73.2	67.3	100	97.6	79.1	69.7	100	97.1
13C-1,2,3,7,8-PeCD	93.7	117.2	92.4	111.1	99.3	97.1	92.9	107.8
13C-1,2,3,4,7,8-HxC	77.5	51.1	78.5	93.5	84.4	61.2	95.0	94.1
13C-1,2,3,6,7,8-HxC	76.3	76.8	75.5	94.1	83.7	61.8	91.0	94.5
13C-1,2,3,4,6,7,8-Hp	78.4	54.1	79.5	96.6	86.1	53.8	73.0	95.0
13C-OCDD	81.2	65.5	80.9	94.0	94.6	65.1	63.0	92.6
13C-2,3.7,8-TCDF	71.8	67.6	99.8	96.9	79.0	59.7	105.5	98.5
13C-1,2,3,7,8-PeCDF	80.6	81.2	92.1	96.2	85.5	67.5	94.5	96.6
13C-2,3,4,7,8-PeCDF	73.8	68.4	99.4	97.1	80.4	51.0	92.8	97.5
13C-1,2,3,4,7,8-HxC	77.1	64.2	85.6	85.0	84.2	51.3	107.2	92.2
13C-1,2,3,6,7,8-HxC	76.6	66.6	84.0	94.2	85.9	53.8	103.1	96.5
13C-2,3,4,6,7,8-HxC	67.9	60.0	105.2	96.8	78.7	59.1	108.5	100.4
13C-1,2,3,7,8,9-HxC	71.7	56.8	103.3	88.9	82.4	58.2	107.9	90.3
13C-1,2,3,4,6,7,8-Hp	74.5	97.3	87.4	81.8	87.1	73.9	80.0	81.4
13C-1,2,3,4,7,8,9-Hp	93.9	51.9	75.9	54.9	107.1	52.6	59.0	54.4
Max	93.9	117.2	105.2	111.1	107.1	97.1	108.5	107.8
Min	67.9	51.1	75.5	54.9	78.7	51.0	59.0	54.4

Conclusion

The results of this study showed a good consistency in the each lab and RSDs of total TEQ of the two samples were below 3%. But only OCDF was variated too much relatively in the mentioned condition of analysis. It is important to certify the reliability of results analyzed by the different laboratories through this kind of study.

Acknowledgement

Thanks for all participants, Seung Jin Kim(Analytical Research Center in Environmental Management Co.), Kyung Seop Kim(Korea Testing-Lab. for industrial Technolory), Byung Uk Kim(Research Institude of Industrial Science & Technology), In Chul Ryu(Metropolitan Government Institude of Health and Environment) and Jae Mun Yang who working for the Ministry of Environmental in Korea who made this chance.

Reference

1. S. K. Shin, Y. H. Chung, S. C. Kim, W.S. Lee, L. J. Lee, J. B. Lee, S. G. Jang, 18th Symposium on Halogenated Environmental Organic Pollutant, Vol 36(1998), 307-311