Data quality of the serum analysis of PCDDs, PCDFs and DL-PCBs in the French Dioxin and Incinerators Study

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Introduction

The French Dioxin and Incinerators Study was launched to determine whether the emissions of the waste incinerators contribute to the body-burden of PCDDs, PCDFs, and PCBs in the surrounding population.

The study involved 8 areas surrounding 8 incinerators and included 1030 adults (30-65 years old).

Thousands of individual data were collected and data quality was a leading concern. This poster presents the work done to ensure the accuracy of the analysis of PCDDs, PCDFs and DL-PCBs levels in serum.

Methods

Sample collection and Analytical methods

1030 serums samples:
- 200 mL of blood
- 7 PCDDs, 10 PCDFs, 12 DL-PCBs
- GC – HRMS method by CART laboratory
- blood lipids analysed by enzymatic method
- results expressed in WHO TEFs 1998.

Internal QC

The concentrations of the QC (spiked foeatal beef serum) were of :
- 157.3 pgTEQ/l for PCDD/F
- 122.0 pgTEQ/l for DL-PCB

Blind QC

Twenty blind QC samples were prepared from a pool of human blood.

Estimation of the target value in iterative steps:
1) Initial target value: mean of the 8 first results
2) Acceptable range = mean ± 2 standard deviations of the 8 first samples
3) When a result fell in this acceptable range, it was included in the computation of a new target value and acceptable range.

Limit of quantification (LOQ)

Two definition were compared:

EU dioxin directive 2004/44/EC for the official control of PCDDs/Fs and DL-PCBs in foodstuffs: LOQ = concentration with a signal/noise ratio of 3:1 and fulfilment of the requirements described in EPA method 1613 revision B.

Definition base on the blanks:

LOD=mean blanks level+1.64 standard deviation of the blanks
LOQ=mean blanks level+3.28 standard deviation of the blanks

Results

Internal QC

All internal QCs and blanks respected the quality criteria.

The internal QC allowed the assessment of the laboratory performances for the upper side of the concentration range encountered in the study.

Blind QC

The target values of the blind QC were of:
- 6.9 pgTEQ/g lipids for PCDD/Fs
- 6.3 pgTEQ/g lipids for DL-PCBs

For each congener the target values were close or below the mean concentrations obtained in our study. A coefficient of variation (CV) was computed on at least 17 results from the blind QC. CV were below 20% for most of the congeners (Table 1).

TABLE 1 Coefficients of variation from the blind QC

	Target value of the blind QC (pgTEQ/g lipids)	CV (%) at the target value
1, 2, 3, 7, 8 - PentaCDD	1.7	12.1
1, 2, 3, 4, 7, 8 - HexaCDD	1.5	15.8
1, 2, 3, 6, 7, 8 - HexaCDD	10.2	12.6
1, 2, 3, 7, 8, 9 - HexaCDD	1.7	12.4
1, 2, 3, 4, 6, 7, 8 - HeptaCDD	23.9	15.5
OctaCDD (OCDD)	158.5	14.0
2, 3, 4, 7, 8 - PentaCDF	5.3	12.4
1, 2, 3, 4, 7, 8 - HexaCDF	1.8	10.9
1, 2, 3, 6, 7, 8 - HexaCDF	2.65	9.1
2, 3, 4, 6, 7, 8 - HexaCDF	1.2	14.4
1, 2, 3, 4, 6, 7, 8 - HeptaCDF	5.2	15.5
PCB 126 (non-ortho)	14.5	41.7
PCB 169 (non-ortho)	23.7	11.8
PCB 114 (ortho)	384.7	21.3
PCB 118 (ortho)	6201.3	23.3
PCB 123 (ortho)	52.6	43.6
PCB 156 (ortho)	5616.1	11.5
PCB 157 (ortho)	1743.0	10.6
PCB 167 (ortho)	2919.0	9.7
PCB 189 (ortho)	1277.7	10.3
PCB 138	35105.3	13.9
PCB 153	79364.1	10.5
PCB 180	114668.25	12.1

Limit of quantification (LOQ)

The differences between LOQ values computed with the two definitions can be as large as one or two orders of magnitude (Table 2).

However, even with the highest LOQ the percentage of non-quantified values remains acceptable (<20%) for most congeners.

In the final statistic analysis, the more conservative definition based on the blanks levels was preferred.

TABLE 2 Comparison of LOQ definition and its impact on the number of non quantified values

	LOQ defined by the EU dioxin directive 2004/44/EC		LOQ defined by the mean blank level +3.28 SD blanks	
	LOQ pg/L	% <LOQ	LOQ pg/L	% <LOQ
2, 3, 7, 8 - TetraCDD	2	6.5	2	6.5
1, 2, 3, 7, 8 - PentaCDD	2	0.1	2	0.1
1, 2, 3, 4, 7, 8 - HexaCDD	5	1.8	5	1.7
1, 2, 3, 6, 7, 8 - HexaCDD	5	0.0	5	0.0
1, 2, 3, 7, 8, 9 - HexaCDD	5	1.0	5	0.9
1, 2, 3, 4, 6, 7, 8 - HeptaCDD	15	0.9	32	2.8
OctaCDD (OCDD)	80	0.2	89	0.2
2, 3, 7, 8 – TetraCDF	2	64.7	2	64.7
1, 2, 3, 7, 8 - PentaCDF	2	72.5	2	72.5
2, 3, 4, 7, 8 - PentaCDF	2	0.0	2	0.0
1, 2, 3, 4, 7, 8 - HexaCDF	5	0.7	11	11.6
1, 2, 3, 6, 7, 8 - HexaCDF	5	0.0	5	0.0
1, 2, 3, 7, 8, 9 - HexaCDF	5	81.8	5	81.8
2, 3, 4, 6, 7, 8 - HexaCDF	5	13.6	5	13.6
1, 2, 3, 4, 6, 7, 8 - HeptaCDF	10	8.1	20	29.4
1, 2, 3, 4, 7, 8, 9 - HeptaCDF	10	99.8	10	99.8
OctaCDF (OCDF)	10	90.5	48	99.0
PCB 77 (non-ortho)	200	88.7	1600	99.2
PCB 81 (ortho)	200	93.0	400	96.3
PCB 126 (non-ortho)	20	2.6	20	2.6
PCB 169 (non-ortho)	20	0.1	20	0.1
PCB 105 (ortho)	200	7.9	8000	37.3
PCB 114 (ortho)	20	0.1	900	2.0
PCB 118 (ortho)	200	1.0	24000	10.6
PCB 123 (ortho)	20	12.9	20	13.0
PCB 156 (ortho)	20	0.0	900	0.0
PCB 157 (ortho)	20	0.0	250	0.0
PCB 167 (ortho)	20	0.0	600	0.0
PCB 189 (ortho)	20	0.1	20	0.1
PCB 138	200	0.1	5000	0.1
PCB 153	200	0.0	5000	0.0
PCB 180	200	0.0	5000	0.0

Conclusions

A close collaboration between the laboratory and health professionals is required to successfully manage the quality assurance of a large study.

Internal QC should be set at levels close to the concentrations expected in the studied population. Assessment of the data quality at low concentrations is an issue, for which the use of blind QC is relevant.

The method used to define the LOQ should always be stated. A conservative definition taking into account the blanks levels and their variability is preferred.

Finally, the good performances obtained by the laboratory in this study at very low levels, especially for PCDDs and PCDFs, should be underlined.

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