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Dioxin, Furan and PCB Testing of Environmental and Food Samples using a GO-EHT Automated Clean-Up System

Compliance study for EPA method 1613B and EPA method 1668C – part 1: in-house tests

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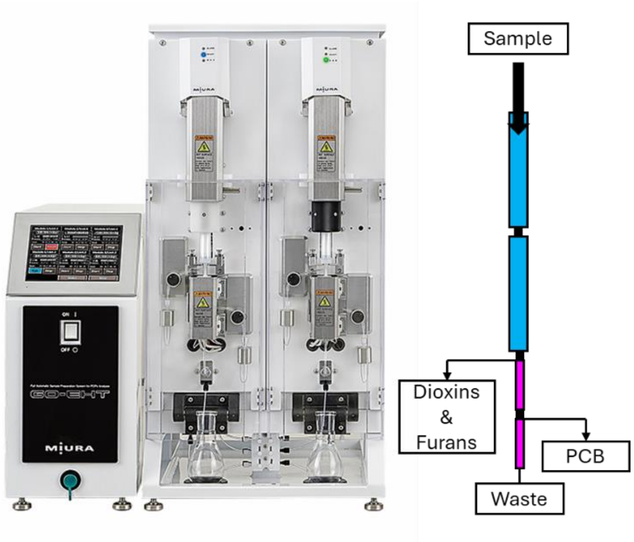

Figure 1. GO-2EHT automated clean-up system for Dioxin, furan and PCB testing, and the sample/analyte schematic flow path (Miura, Japan).

Goal

The goal of this study is to establish the performance of the GO-EHT automated clean-up systems for the testing of Dioxins, Furans and all 209 PCBs in environmental and food samples.

Introduction

In earlier times, mostly manual methods were developed or semi-automated systems were constructed in-house. Currently, well established automated systems are available for purification of sample extracts for Dioxin, Furan and PCB analysis. Such systems allow for systematic performance, fast turnaround times, increased capacity and lower solvent consumption.

Methods of analysis for regulatory purposes are a.o. described in EPA method 1613B[1] and EPA method 1668C[2] respectively, for Dioxins and Furans, and PCBs. These methods are performance based and modification or automatization is allowed.

In this study the GO-EHT (fig. 1) of Miura (Japan) is evaluated for its use for analysis of PCBs in accordance with EPA method 1668C[2] and additionally for Dioxins and Furans in agreement with EPA method 1613B[1].

The GO-EHT is an advanced, modular purification system designed to revolutionize sample clean-up. The GO-EHT features a sophisticated controller and up to three processing modules, each equipped with dual units for parallel and independent sample purification. Utilizing a consumable column set of 4 columns, Dioxins and Furans, and PCB can be split over two fractions of each less than 1.5mL of toluene. Samples are loaded off-line to prevent cross-contamination and all analyte-contacting tubing and vials are disposable.

The fractionation of Dioxins, Furans and PCBs is determined by the column set and the traditional set was developed to recover non-ortho-PCBs together with Dioxins and Furans due to the level of relevance in the EU. For the North-American market column sets were developed to separate PCBs completely from Dioxins and Furans.

The traditional method applied on the GO-EHT is in accordance with official methods for analysis in the EU and is widely used for official testing in compliance with regulatory performance criteria. The precision and trueness criteria in the EU for food and feed are strict at 15% RSD on-going precision and 80% - 120% on-going recovery.

In prior studies[3,4] the performance of the traditional method was considered suitable for EPA 1613B and EPA 1668C, but as PCBs were split over two fractions research was continued for a more straightforward application.

In this study, the performance of the newly developed column set for complete separation of PCBs from Dioxins and Furans is established.

Experimental

A total of 11 tests, a blank and 5 different environmental matrices and a fish oil, were evaluated for initial precision and recovery (IPR) of labelled PCBs, and labelled Dioxins and Furans.

The method and materials in this study were comparable or the same as in ten Dam et al[4]. PCBs, Dioxins and Furans analytical standards were purchased from Cambridge Isotope laboratories (United States), samples were extracted using a SER-158 (Velp Scientifca, Italy), evaporation was performed with a CentriVap Vacuum Concentrator of Labconco (United States), PCBs were measured on a APGC-MSMS v2 from Waters (United Kingdom) equipped with an Agilent (United States) A7890 GC and Dioxins and Furans were measured on a Thermo DFS sector HRMS. The laboratory work was facilitated by QTI Services B.V (The Netherlands).

Purification

All extracts were purified on a GO-EHT (fig. 1) from Miura (Japan) using for each sample a column set for environmental samples (18 mm ϕ EPA 209PCB) (fig. 2). Each column set consisted of four columns and were connected in the following order: silica gel impregnated with silver nitrate (1st); silica gel impregnated with sulfuric acid (2nd); “carbon” (3rd) and “alumina” (4th).

Sample extracts were transferred on top of the first column and after complete absorption, the set of columns was placed in the automated sample purification system. Next, the silver nitrate column was heated at 60°C/140°F for 10 minutes. After cooling

Figure 2 Column sets after clean-up procedure, from left to right sewage sludge, soil, ash and house dust.

down to 40°C/104°F the columns were eluted with 85 ml hexane at a flowrate of 2.5 ml/min. The hexane was collected at the outlet of the four columns in a flask (waste (W)). After purging the columns with air to remove all hexane, both the alumina and the carbon column were eluted in backflush using a small amount of toluene and collected in a 1.5 ml glass vial. During the whole process, both the carbon and alumina column were heated till 90°C/194°F. The resulting two fractions, e.g. a “carbon”/Dioxin fraction (C) and

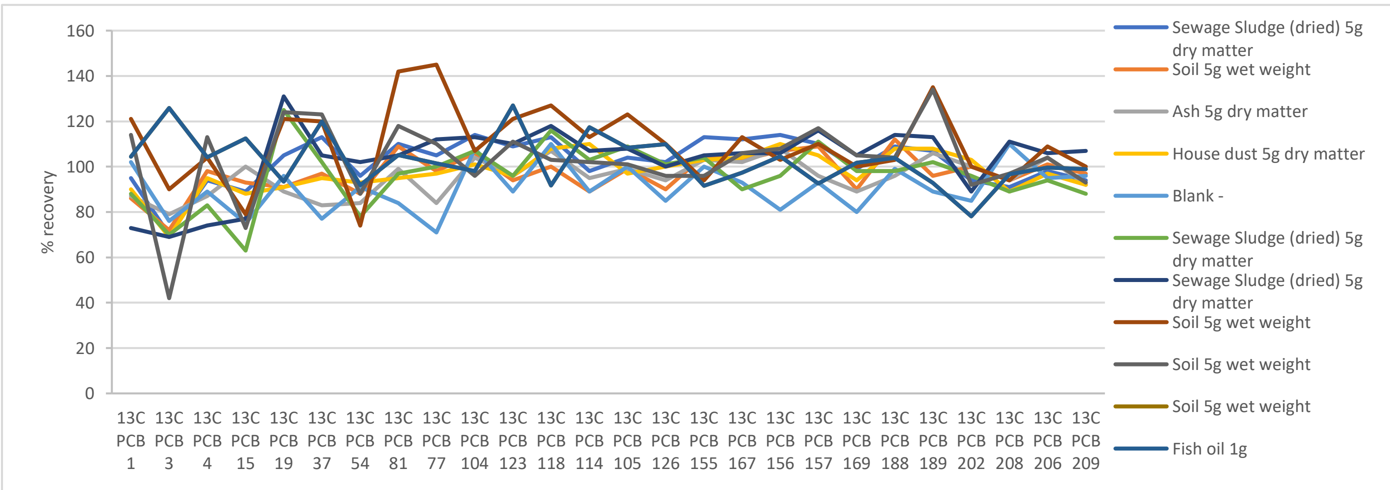

Figure 3 Recoveries for labelled PCBs in 11 in-house tested samples.

"alumina"/PCB fraction (A), each contained about 1.5 ml extract.

environmental samples and fish oil in agreement with the required MDLs.

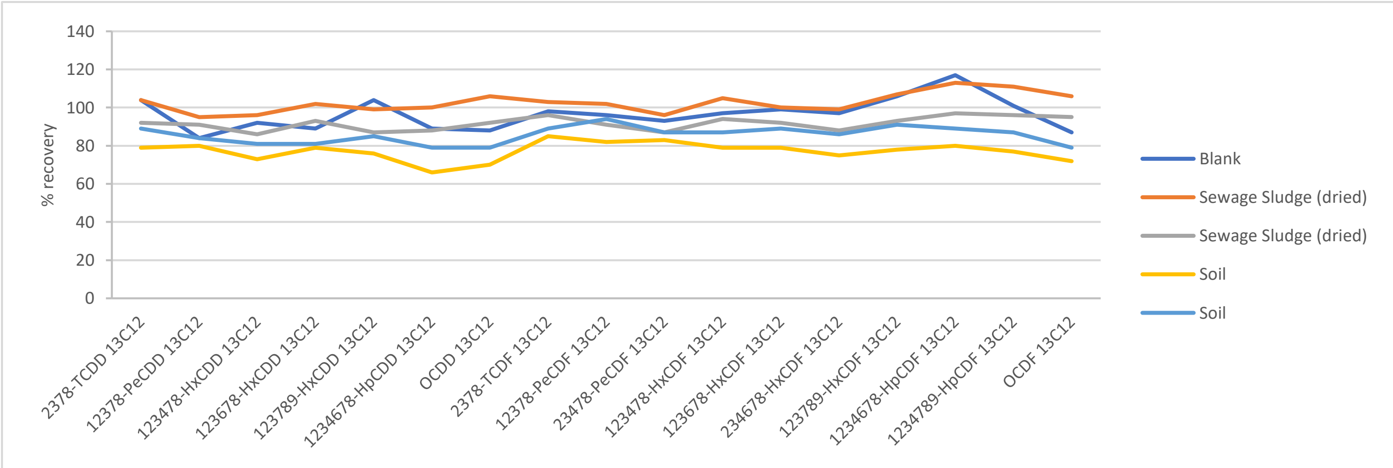

Figure 4 Recoveries for labelled Dioxins and Furans in 4 in-house tested samples.

Results and discussion

Recoveries of all internal standards were calculated and used for calculation of IPR. In total, recoveries of internal standards were obtained from 11 samples tested in-house (fig. 3 and 4). Recoveries were within 60 to 140% for PCBs except for 13C PCB3 in Fish oil (fig. 3 and table 2). For Dioxins Recoveries were between 60 and 120% (Fig. 4 and table 2).

EPA 1668C IPR criteria for internal standards are at stringent 45% - 135% for average recovery and 50% for RSD. With 81% recovery and 25% SD PCB3 has the largest error, yet still complies amply to these criteria (table 1).

EPA 1613 revision B IPR criteria are in the range of 30% till 150% average recovery and over 30% RSD. With average recoveries around 90% and SDs around 10% the method complies amply to the criteria set in this method (table 1).

Conclusion

The performance of GO-EHT purification for Dioxins, Furans and PCBs comply with IPR criteria set in EPA method 1613B[1] and 1668C[2].

The method allows for the determination of levels of Dioxins and Furans in combination with all 209 PCBs.

References

1) EPA; U.S. Environmental Protection Agency (1994) Method 1613: Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS. Revision B

2) US Environmental Protection Agency, Method 1668C, chlorinated biphenyl congeners in water, soil, sediment, biosolids and tissue by HRGC/HRMS, revision C, (2010)

3) Douglas G. Hayward and Willem Traag, New approach for removing co-extracted lipids before mass spectrometry measurement of persistent of organic pollutants (POPs) in foods, Chemosphere 256 (2020) 127023

4) Guillaume ten Dam, Compliance study for automated purification of environmental samples and fish oil for the analysis of Dioxins, Furans and all 209 PCBs according EPA method 1613B and EPA method 1668C, 2022, DSP Systems

Table 1 Average recovery internal standards and standard deviation of 11 in-house tested samples.

Analyte	Recovery	SD	n
2378-TCDD 13C12	94	11	5
12378-PeCDD 13C12	87	6	5
123478-HxCDD 13C12	86	9	5
123678-HxCDD 13C12	89	9	5
123789-HxCDD 13C12	90	11	5
1234678-HpCDD 13C12	84	13	5
OCDD 13C12	87	14	5
2378-TCDF 13C12	94	7	5
12378-PeCDF 13C12	93	7	5
23478-PeCDF 13C12	89	5	5
123478-HxCDF 13C12	92	10	5
123678-HxCDF 13C12	92	9	5
234678-HxCDF 13C12	89	10	5
123789-HxCDF 13C12	95	12	5
1234678-HpCDF 13C12	99	16	5
1234789-HpCDF 13C12	94	13	5
OCDF 13C12	88	13	5
13C PCB 1	97	14	11
13C PCB 3	81	25	11
13C PCB 4	95	11	11
13C PCB 15	87	16	11
13C PCB 19	105	16	11
13C PCB 37	105	16	11
13C PCB 54	89	8	11
13C PCB 81	106	15	11
13C PCB 77	102	18	11
13C PCB 104	105	6	11
13C PCB 123	107	14	11
13C PCB 118	108	11	11
13C PCB 114	104	10	11
13C PCB 105	105	7	11
13C PCB 126	100	8	11
13C PCB 155	101	7	11
13C PCB 167	102	7	11
13C PCB 156	104	9	11
13C PCB 157	105	9	11
13C PCB 169	97	8	11
13C PCB 188	105	6	11
13C PCB 189	107	16	11
13C PCB 202	92	9	11
13C PCB 205	-	-	-
13C PCB 208	97	7	11
13C PCB 206	101	5	11
13C PCB 209	96	5	11

Analyte	Recovery	SD	n
CB_Group	-	-	-
Cl1	85	6	6
Cl2	100	4	6
Cl3	107	13	6
Cl4	101	11	6
Cl5	107	8	6
Cl6	112	8	6
Cl7	113	9	6
Cl8	100	3	6
Cl9	104	7	6
Cl10	101	10	6
INJECTION 13C____	-	-	-
13C PCB 9	107	28	11
13C PCB 52	89	14	11
13C PCB 101	85	15	11
13C PCB 138	88	14	11
13C PCB 194	100	24	11

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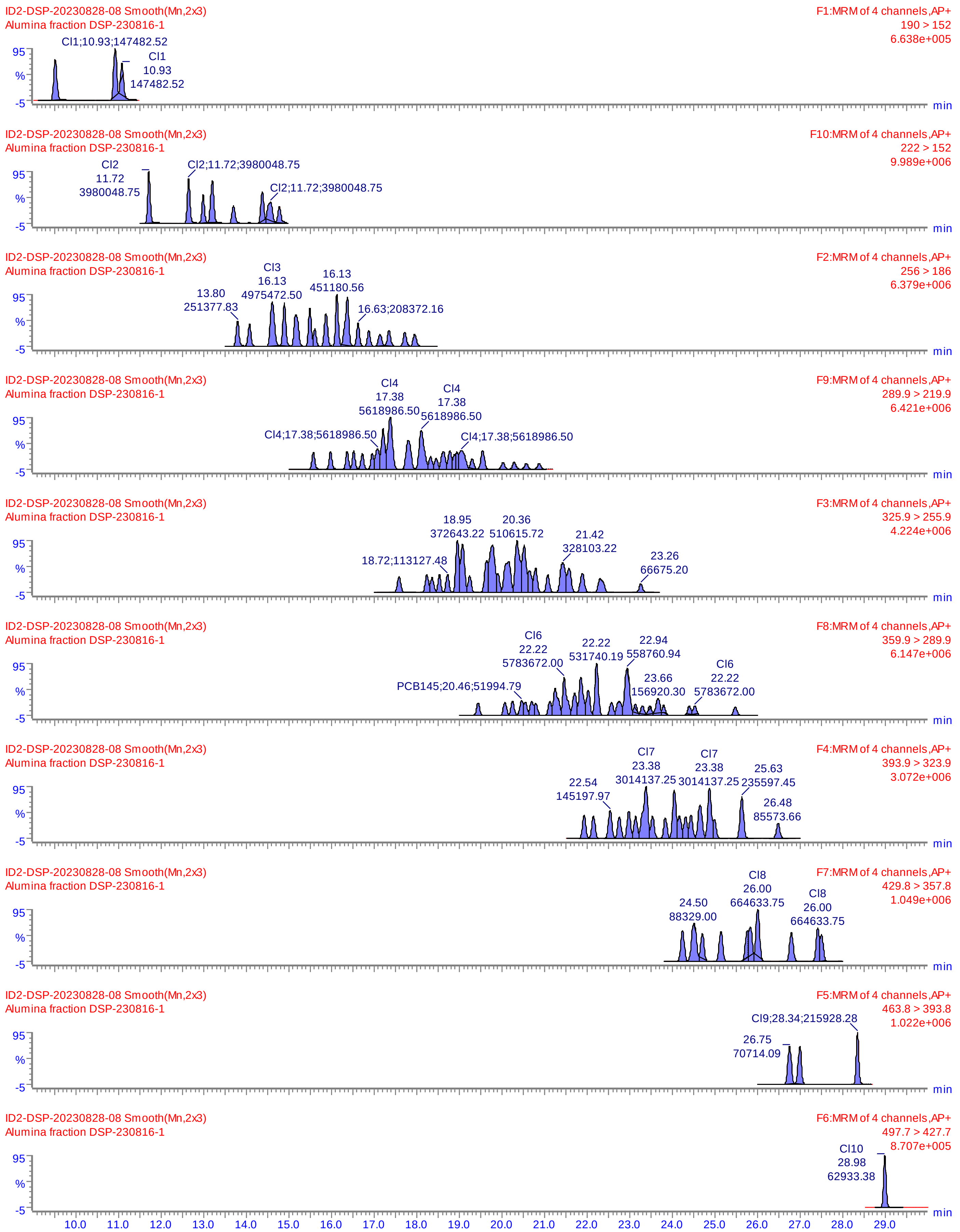
Table 2.1 Individual recoveries of the 11 in-house tested samples.

Sample nr.	1	2	3	4	5	6	7	8	9	10	11
Product	Sewage Sludge (dried)	Soil	Ash	House dust	Blank	Sewage Sludge (dried)	Sewage Sludge (dried)	Soil	Soil	Soil	Fish oil
System/Channel	JP-83260147-3	JP-83260147-3	JP-83260147-3	JP-83260147-3	JP-83260147-1	JP-83260147-3	JP-83260147-4	JP-83260147-1	JP-83260147-2	JP-83260147-2	QTI-1
Intake	5g dry matter	5g wet weight	5g dry matter	5g dry matter	-	5g dry matter	5g dry matter	5g wet weight	5g wet weight	5g wet weight	1g
V extract (Diox&PCB)	30/1000ul	30/1000ul	30/1000ul	30/1000ul	30/1000ul	30/1000ul	30/1000ul	30/1000ul	30/1000ul	30/1000ul	-/1000ul
Detection type	DFS-HRMS/APGC-MSMS	DFS-HRMS/APGC-MSMS	DFS-HRMS/APGC-MSMS	DFS-HRMS/APGC-MSMS	DFS-HRMS/APGC-MSMS	DFS-HRMS/APGC-MSMS	DFS-HRMS/APGC-MSMS	DFS-HRMS/APGC-MSMS	DFS-HRMS/APGC-MSMS	DFS-HRMS/APGC-MSMS	-/APGC-MSMS
Injection volume Diox/PCB	1ul	1ul	1ul	1ul	1ul	1ul	1ul	1ul	1ul	1ul	1ul
2378-TCDD 13C12	-	-	-	-	104	104	92	79	89	-	-
12378-PeCDD 13C12	-	-	-	-	84	95	91	80	84	-	-
123478-HxCDD 13C12	-	-	-	-	92	96	86	73	81	-	-
123678-HxCDD 13C12	-	-	-	-	89	102	93	79	81	-	-
123789-HxCDD 13C12	-	-	-	-	104	99	87	76	85	-	-
1234678-HpCDD 13C12	-	-	-	-	89	100	88	66	79	-	-
OCDD 13C12	-	-	-	-	88	106	92	70	79	-	-
2378-TCDF 13C12	-	-	-	-	98	103	96	85	89	-	-
12378-PeCDF 13C12	-	-	-	-	96	102	91	82	94	-	-
23478-PeCDF 13C12	-	-	-	-	93	96	87	83	87	-	-
123478-HxCDF 13C12	-	-	-	-	97	105	94	79	87	-	-
123678-HxCDF 13C12	-	-	-	-	99	100	92	79	89	-	-
234678-HxCDF 13C12	-	-	-	-	97	99	88	75	86	-	-
123789-HxCDF 13C12	-	-	-	-	106	107	93	78	91	-	-
1234678-HpCDF 13C12	-	-	-	-	117	113	97	80	89	-	-
1234789-HpCDF 13C12	-	-	-	-	101	111	96	77	87	-	-
OCDF 13C12	-	-	-	-	87	106	95	72	79	-	-
Cl1	85	80	86	80	-	-	82	-	96	-	-
Cl2	104	96	96	104	-	-	101	-	97	-	-
Cl3	113	94	92	99	-	-	122	-	120	-	-
Cl4	108	93	87	94	-	-	112	-	113	-	-
Cl5	116	100	101	103	-	-	117	-	105	-	-
Cl6	123	112	97	114	-	-	115	-	111	-	-
Cl7	119	111	99	109	-	-	120	-	121	-	-
Cl8	97	101	101	98	-	-	104	-	101	-	-
Cl9	100	102	99	96	-	-	113	-	111	-	-
Cl10	98	101	93	91	-	-	117	-	106	-	-
13C PCB 1	95	86	87	90	102	88	73	121	114	104	104
13C PCB 3	70	72	79	69	76	70	69	90	42	126	126
13C PCB 4	94	98	87	95	89	83	74	104	113	104	104
13C PCB 15	89	93	100	88	75	63	77	79	73	112	112
13C PCB 19	105	91	89	91	96	125	131	121	124	93	93
13C PCB 37	113	97	83	95	77	102	105	120	123	120	120
13C PCB 54	96	88	84	93	91	78	102	74	88	92	92
13C PCB 81	110	109	99	95	84	97	105	142	118	105	105
13C PCB 77	105	98	84	97	71	100	112	145	110	101	101
13C PCB 104	114	106	104	101	108	107	113	107	96	98	98
13C PCB 123	109	94	96	96	89	96	110	121	111	127	127
13C PCB 118	113	100	107	108	110	116	118	127	103	92	92
13C PCB 114	98	89	95	110	89	103	107	113	102	117	117
13C PCB 105	104	99	99	97	100	109	108	123	101	108	108
13C PCB 126	102	90	94	100	85	101	100	110	96	110	110
13C PCB 155	113	105	103	103	100	104	105	94	96	92	92

Table 2.2 Individual recoveries of the 11 in-house tested samples. (continued)

Sample nr.	1	2	3	4	5	6	7	8	9	10	11
Product	Sewage Sludge (dried)	Soil	Ash	House dust	Blank	Sewage Sludge (dried)	Sewage Sludge (dried)	Soil	Soil	Soil	Fish oil
System/Channel	JP-83260147-3	JP-83260147-3	JP-83260147-3	JP-83260147-3	JP-83260147-1	JP-83260147-3	JP-83260147-4	JP-83260147-1	JP-83260147-2	JP-83260147-2	QTI-1
Intake	5g dry matter	5g wet weight	5g dry matter	5g dry matter	-	5g dry matter	5g dry matter	5g wet weight	5g wet weight	5g wet weight	1g
V extract (Diox&PCB)	30/1000ul	30/1000ul	30/1000ul	30/1000ul	30/1000ul	30/1000ul	30/1000ul	30/1000ul	30/1000ul	30/1000ul	-/1000ul
Detection type	DFS-HRMS/APGC-MSMS	DFS-HRMS/APGC-MSMS	DFS-HRMS/APGC-MSMS	DFS-HRMS/APGC-MSMS	DFS-HRMS/APGC-MSMS	DFS-HRMS/APGC-MSMS	DFS-HRMS/APGC-MSMS	DFS-HRMS/APGC-MSMS	DFS-HRMS/APGC-MSMS	DFS-HRMS/APGC-MSMS	-/APGC-MSMS
Injection volume Diox/PCB	1ul	1ul	1ul	1ul	1ul	1ul	1ul	1ul	1ul	1ul	1ul
13C PCB 167	112	103	102	104	93	90	106	113	106	97	97
13C PCB 156	114	107	108	110	81	96	106	103	108	105	105
13C PCB 157	110	109	96	105	93	111	116	110	117	93	93
13C PCB 169	99	90	89	94	80	98	105	100	105	102	102
13C PCB 188	109	112	96	108	99	98	114	103	104	104	104
13C PCB 189	107	96	106	108	89	102	113	135	134	93	93
13C PCB 202	94	100	100	103	85	96	89	100	92	78	78
13C PCB 205	-	-	-	-	-	-	-	-	-	-	-
13C PCB 208	91	94	94	89	110	89	111	94	97	97	97
13C PCB 206	98	101	104	97	95	94	106	109	104	100	100
13C PCB 209	94	97	92	92	96	88	107	100	93	99	99
INJECTION 13C____	-	-	-	-	-	-	-	-	-	-	-
13C PCB 9	118	101	71	102	102	117	113	122	172	78	78
13C PCB 52	111	104	75	101	96	68	72	83	102	86	86
13C PCB 101	100	103	66	96	88	65	69	85	108	80	80
13C PCB 138	97	96	65	93	87	73	74	101	114	87	87
13C PCB 194	115	102	65	102	102	90	85	132	145	81	81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F1:MRM of 4 channels,AP+
190 > 152
6.638e+005
Cl1;10.93;147482.52
Cl1
10.93
147482.52
ID2-DSP-20230828-08 Smooth(Mn,2x3)
Alumina fraction DSP-230816-1
F10:MRM of 4 channels,AP+
222 > 152
9.989e+006
Cl2
11.72
3980048.75
Cl2;11.72;3980048.75
Cl2;11.72;3980048.75
ID2-DSP-20230828-08 Smooth(Mn,2x3)
Alumina fraction DSP-230816-1
F2:MRM of 4 channels,AP+
256 > 186
6.379e+006
13.80
251377.83
Cl3
16.13
4975472.50
16.13
451180.56
16.63;208372.16
ID2-DSP-20230828-08 Smooth(Mn,2x3)
Alumina fraction DSP-230816-1
F9:MRM of 4 channels,AP+
289.9 > 219.9
6.421e+006
Cl4
17.38
5618986.50
Cl4
17.38
5618986.50
Cl4;17.38;5618986.50
Cl4;17.38;5618986.50
ID2-DSP-20230828-08 Smooth(Mn,2x3)
Alumina fraction DSP-230816-1
F3:MRM of 4 channels,AP+
325.9 > 255.9
4.224e+006
18.95
372643.22
20.36
510615.72
18.72;113127.48
21.42
328103.22
23.26
66675.20
ID2-DSP-20230828-08 Smooth(Mn,2x3)
Alumina fraction DSP-230816-1
F8:MRM of 4 channels,AP+
359.9 > 289.9
6.147e+006
Cl6
22.22
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558760.94
Cl6
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23.66
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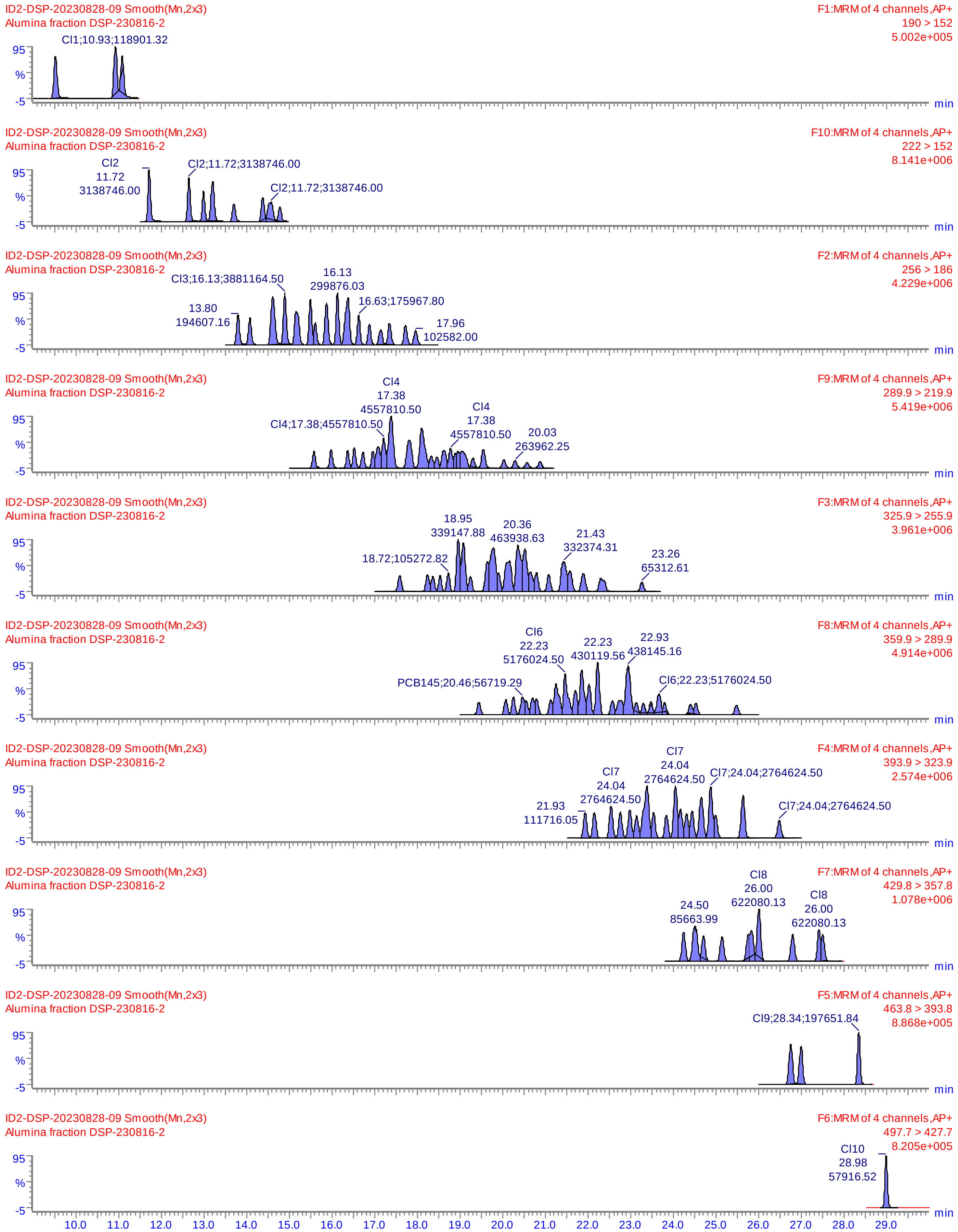
ID2-DSP-20230828-08 Smooth(Mn,2x3)
Alumina fraction DSP-230816-1
F4:MRM of 4 channels,AP+
393.9 > 323.9
3.072e+006
Cl7
23.38
3014137.25
Cl7
23.38
3014137.25
25.63
235597.45
22.54
145197.97
26.48
85573.66
ID2-DSP-20230828-08 Smooth(Mn,2x3)
Alumina fraction DSP-230816-1
F7:MRM of 4 channels,AP+
429.8 > 357.8
1.049e+006
Cl8
26.00
664633.75
24.50
88329.00
Cl8
26.00
664633.75
ID2-DSP-20230828-08 Smooth(Mn,2x3)
Alumina fraction DSP-230816-1
F5:MRM of 4 channels,AP+
463.8 > 393.8
1.022e+006
Cl9;28.34;215928.28
26.75
70714.09
ID2-DSP-20230828-08 Smooth(Mn,2x3)
Alumina fraction DSP-230816-1
F6:MRM of 4 channels,AP+
497.7 > 427.7
8.707e+005
Cl10
28.98
62933.38
95
%
-5
min
10.0 11.0 12.0 13.0 14.0 15.0 16.0 17.0 18.0 19.0 20.0 21.0 22.0 23.0 24.0 25.0 26.0 27.0 28.0 29.0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190 > 152
5.002e+005
Cl1;10.93;118901.32
F10:MRM of 4 channels,AP+
222 > 152
8.141e+006
Cl2
11.72
3138746.00
Cl2;11.72;3138746.00
Cl2;11.72;3138746.00
F2:MRM of 4 channels,AP+
256 > 186
4.229e+006
Cl3;16.13;3881164.50
16.13
299876.03
13.80
194607.16
16.63;175967.80
17.96
102582.00
F9:MRM of 4 channels,AP+
289.9 > 219.9
5.419e+006
Cl4
17.38
4557810.50
Cl4;17.38;4557810.50
Cl4
17.38
4557810.50
20.03
263962.25
F3:MRM of 4 channels,AP+
325.9 > 255.9
3.961e+006
18.95
339147.88
20.36
463938.63
21.43
332374.31
18.72;105272.82
23.26
65312.61
F8:MRM of 4 channels,AP+
359.9 > 289.9
4.914e+006
Cl6
22.23
5176024.50
22.23
430119.56
22.93
438145.16
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Cl6;22.23;5176024.50
F4:MRM of 4 channels,AP+
393.9 > 323.9
2.574e+006
Cl7
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Cl7
24.04
2764624.50
Cl7;24.04;2764624.50
21.93
111716.05
Cl7;24.04;2764624.50
F7:MRM of 4 channels,AP+
429.8 > 357.8
1.078e+006
Cl8
26.00
622080.13
24.50
85663.99
Cl8
26.00
622080.13
F5:MRM of 4 channels,AP+
463.8 > 393.8
8.868e+005
Cl9;28.34;197651.84
F6:MRM of 4 channels,AP+
497.7 > 427.7
8.205e+005
Cl10
28.98
57916.52
95
%
-5
min
10.0
11.0
12.0
13.0
14.0
15.0
16.0
17.0
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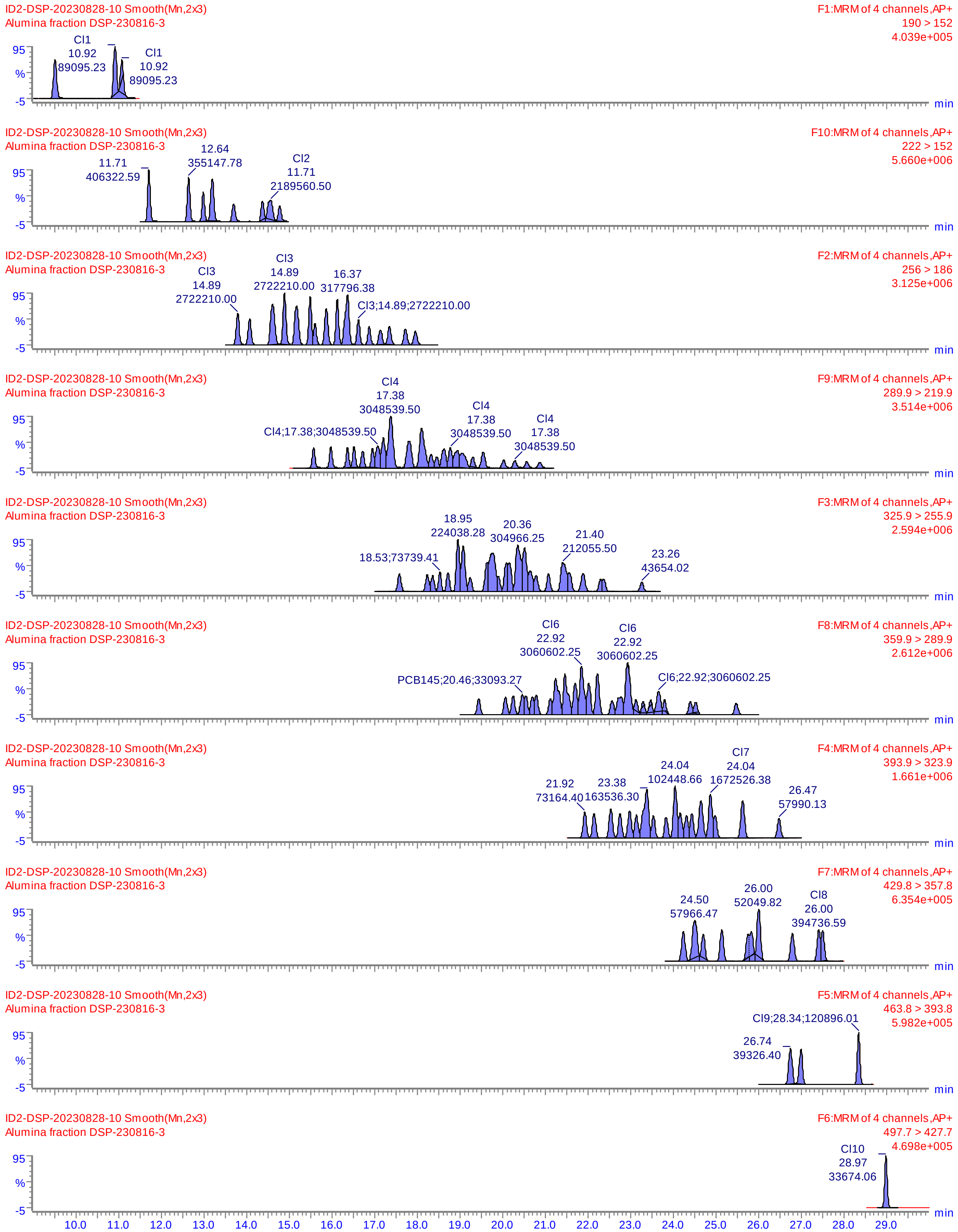
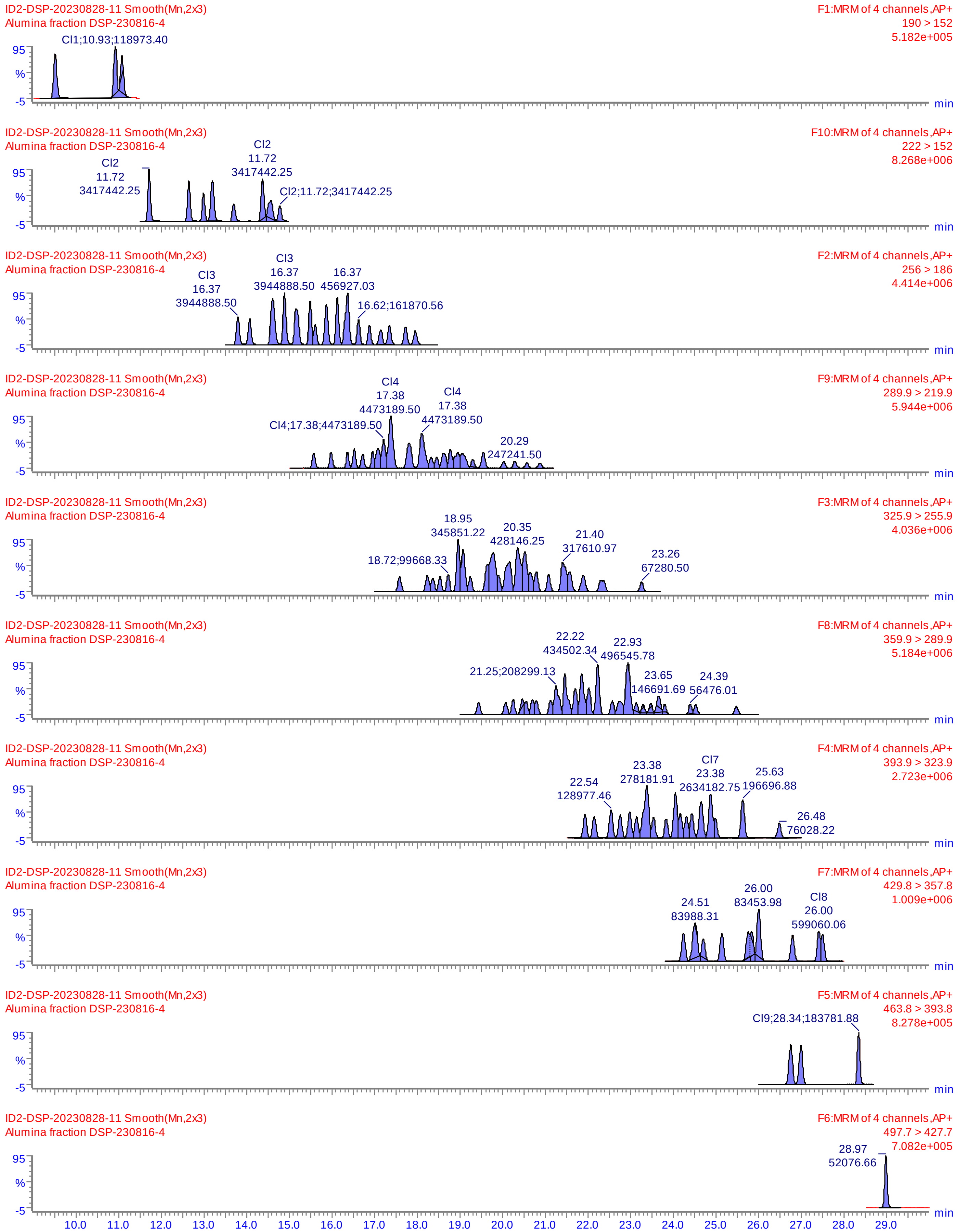
29.0


ID2-DSP-20230828-10 Smooth(Mn,2x3)
Alumina fraction DSP-230816-3
F1:MRM of 4 channels,AP+
190 > 152
4.039e+005
Cl1
10.92
89095.23
Cl1
10.92
89095.23
ID2-DSP-20230828-10 Smooth(Mn,2x3)
Alumina fraction DSP-230816-3
F10:MRM of 4 channels,AP+
222 > 152
5.660e+006
11.71
406322.59
12.64
355147.78
Cl2
11.71
2189560.50
ID2-DSP-20230828-10 Smooth(Mn,2x3)
Alumina fraction DSP-230816-3
F2:MRM of 4 channels,AP+
256 > 186
3.125e+006
Cl3
14.89
2722210.00
Cl3
14.89
2722210.00
16.37
317796.38
Cl3;14.89;2722210.00
ID2-DSP-20230828-10 Smooth(Mn,2x3)
Alumina fraction DSP-230816-3
F9:MRM of 4 channels,AP+
289.9 > 219.9
3.514e+006
Cl4
17.38
3048539.50
Cl4;17.38;3048539.50
Cl4
17.38
3048539.50
Cl4
17.38
3048539.50
ID2-DSP-20230828-10 Smooth(Mn,2x3)
Alumina fraction DSP-230816-3
F3:MRM of 4 channels,AP+
325.9 > 255.9
2.594e+006
18.95
224038.28
20.36
304966.25
21.40
212055.50
18.53;73739.41
23.26
43654.02
ID2-DSP-20230828-10 Smooth(Mn,2x3)
Alumina fraction DSP-230816-3
F8:MRM of 4 channels,AP+
359.9 > 289.9
2.612e+006
Cl6
22.92
3060602.25
Cl6
22.92
3060602.25
PCB145;20.46;33093.27
Cl6;22.92;3060602.25
ID2-DSP-20230828-10 Smooth(Mn,2x3)
Alumina fraction DSP-230816-3
F4:MRM of 4 channels,AP+
393.9 > 323.9
1.661e+006
21.92
73164.40
23.38
163536.30
24.04
102448.66
Cl7
24.04
1672526.38
26.47
57990.13
ID2-DSP-20230828-10 Smooth(Mn,2x3)
Alumina fraction DSP-230816-3
F7:MRM of 4 channels,AP+
429.8 > 357.8
6.354e+005
24.50
57966.47
26.00
52049.82
Cl8
26.00
394736.59
ID2-DSP-20230828-10 Smooth(Mn,2x3)
Alumina fraction DSP-230816-3
F5:MRM of 4 channels,AP+
463.8 > 393.8
5.982e+005
Cl9;28.34;120896.01
26.74
39326.40
ID2-DSP-20230828-10 Smooth(Mn,2x3)
Alumina fraction DSP-230816-3
F6:MRM of 4 channels,AP+
497.7 > 427.7
4.698e+005
Cl10
28.97
33674.06
95
%
-5
min
10.0
11.0
12.0
13.0
14.0
15.0
16.0
17.0
18.0
19.0
20.0
21.0
22.0
23.0
24.0
25.0
26.0
27.0
28.0
29.0


ID2-DSP-20230828-11 Smooth(Mn,2x3)
Alumina fraction DSP-230816-4
F1:MRM of 4 channels,AP+
190 > 152
5.182e+005
Cl1;10.93;118973.40
ID2-DSP-20230828-11 Smooth(Mn,2x3)
Alumina fraction DSP-230816-4
F10:MRM of 4 channels,AP+
222 > 152
8.268e+006
Cl2
11.72
3417442.25
Cl2
11.72
3417442.25
Cl2;11.72;3417442.25
ID2-DSP-20230828-11 Smooth(Mn,2x3)
Alumina fraction DSP-230816-4
F2:MRM of 4 channels,AP+
256 > 186
4.414e+006
Cl3
16.37
3944888.50
Cl3
16.37
3944888.50
16.37
456927.03
16.62;161870.56
ID2-DSP-20230828-11 Smooth(Mn,2x3)
Alumina fraction DSP-230816-4
F9:MRM of 4 channels,AP+
289.9 > 219.9
5.944e+006
Cl4
17.38
4473189.50
Cl4
17.38
4473189.50
Cl4;17.38;4473189.50
20.29
247241.50
ID2-DSP-20230828-11 Smooth(Mn,2x3)
Alumina fraction DSP-230816-4
F3:MRM of 4 channels,AP+
325.9 > 255.9
4.036e+006
18.95
345851.22
20.35
428146.25
21.40
317610.97
18.72;99668.33
23.26
67280.50
ID2-DSP-20230828-11 Smooth(Mn,2x3)
Alumina fraction DSP-230816-4
F8:MRM of 4 channels,AP+
359.9 > 289.9
5.184e+006
22.22
434502.34
22.93
496545.78
21.25;208299.13
23.65
146691.69
24.39
56476.01
ID2-DSP-20230828-11 Smooth(Mn,2x3)
Alumina fraction DSP-230816-4
F4:MRM of 4 channels,AP+
393.9 > 323.9
2.723e+006
23.38
278181.91
Cl7
23.38
2634182.75
25.63
196696.88
22.54
128977.46
26.48
76028.22
ID2-DSP-20230828-11 Smooth(Mn,2x3)
Alumina fraction DSP-230816-4
F7:MRM of 4 channels,AP+
429.8 > 357.8
1.009e+006
26.00
83453.98
24.51
83988.31
Cl8
26.00
599060.06
ID2-DSP-20230828-11 Smooth(Mn,2x3)
Alumina fraction DSP-230816-4
F5:MRM of 4 channels,AP+
463.8 > 393.8
8.278e+005
Cl9;28.34;183781.88
ID2-DSP-20230828-11 Smooth(Mn,2x3)
Alumina fraction DSP-230816-4
F6:MRM of 4 channels,AP+
497.7 > 427.7
7.082e+005
28.97
52076.66
95
%
-5
min
10.0
11.0
12.0
13.0
14.0
15.0
16.0
17.0
18.0
19.0
20.0
21.0
22.0
23.0
24.0
25.0
26.0
27.0
28.0
29.0