

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

Guillaume ten Dam¹, Fujita Hiroyuki², Jeroen Markesteijn³, Chris van Wakeren¹ and Wim Traag¹

¹[DSP-Systems](#), Food Valley BTA12, Ede, The Netherlands, ²[Miura](#), 7 Horie, Matsuyama, Ehime, 799-2696, Japan, ³[QTI Services B.V.](#), Keenstraat 46, Rotterdam, The Netherlands

Goal

To establish the performance of a GO-eHT automated purification system for the analysis of Dioxins, Furans and all 209 PCBs

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 and EPA method 1668C 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 1668C² and additionally for Dioxins and Furans in agreement with EPA method 1613B¹.

The method applied on the [GO-eHT](#) is in agreement with official methods for analysis in the EU⁵ and is used for official analysis according the prescribed performance criteria^{6,7,8,9}. The precision and trueness (in EPA methods referred to as recovery) criteria in the EU for food⁶ and feed⁷ are 15% RSD (on-going precision and recovery) and 80% - 120% recovery.

The study focused on the purification system as such and standards were only added after extraction of samples. A total of 5 different environmental matrices and a fish oil were evaluated for initial precision and



Figure 1. System for automated purification of samples for dioxin, furan and PCB analysis, GO-6eHT ([Miura](#), Japan).

recovery (IPR) of all 209 native PCBs as well as labelled toxics/LOC/window-defining PCBs, labelled clean-up PCBs and labelled Dioxins and Furans. For Dioxins and Furans precision was calculated on the results of the duplicate analysis of the blank and with PCBs spiked sample according NEN:EN 7777⁴.

Experimental

Chemicals and reference materials

Solvents were from Biosolve and were of Dioxin, Furans & PCB analysis grade. Pre-made sets of two purification columns and two concentration columns were produced by [Miura](#) (Japan) and contained silica dispersed silver nitrate, sulfuric acid impregnated silica, carbon and aluminium oxide.

Reference standards for Dioxins, Furans, non-ortho PCBs, ¹³C₁₂ Dioxins, ¹³C₁₂ PCBs (EPA 1668 LOC, Clean-up and Injection) were from [Cambridge Isotope laboratories](#) (CIL) while all 209 native PCBs were from Accu Standard (US).

Samples

Samples were taken from the local environment and shops around Ede (The Netherlands) or provided by [QTI Services B.V.](#). A sample sediment was taken from the river Rhine (The Netherlands) from the shore at Wageningen. Soil was taken from a bag of top soil. Dust was collected from the vacuum cleaner of our own homes and sewage sludge was obtained as a slurry from the local sewage treatment plant. Ash and fish oil were obtained from QTI Services B.V..

The sample sediment and sewage sludge were dried in a stove at 100°C prior to analysis while all other samples were analysed as such.

Extraction

Except for fish oil, all samples were extracted using a [SER-158](#) (Velp Scientifca, Italy). No standards were added prior to extraction, after extraction samples were split into two to four parts; #1 for blank analysis and #2-4 for spiked with native PCBs. This was done to obtain homogeneous subsamples and to evaluate the purification apart from extraction.

Different weights of samples were measured into a cup; dust 2.5 gram, ash 5 gram, soil 10 gram, dried sewage sludge 10 gram and dried sediment 20 gram. Weights differed as to fit the sample into the volume

of the thimble (33x80 mm, Velp Scientifca, Italy) and availability of the sample. The thimble was placed in a Thimble holder and placed in an extraction beaker filled with 100ml of extraction solvent, toluene. The thimble was positioned above the bottom of the beaker, but in the extraction solvent. The beaker with the sample was placed in the SER-158 for extraction.

The extraction was according the Randall and Twisselman principle. Initially the sample was extracted in boiling toluene (immersion) for 1:15 hours. In 10 minutes toluene was removed through evaporation (removing) and the sample was extracted by vapor and re-condensate for 1:30 hours (washing). After the extraction process toluene was removed by evaporation in 15 minutes (removing). During the whole process the system was heated at level 12 to reach the boiling temperature of toluene.

The remaining extract was left to dry by air for a night to remove all residues of toluene. The remainders were reconstituted in 20 ml or 40 ml hexane. The sample ash was split into 4 subsamples and all other samples were split in 2.

For fish oil 1 gram was directly dissolved in 10 ml hexane. This was performed in duplicate. The two blanks consisted of 10 ml pure hexane.



Figure 2 Extraction of the samples according the Randall and Twisselman principle on a SER-158

Spiking

All blanks, samples and subsamples were spiked with ¹³C₁₂ internal standards [EC-4977](#) (0.1 ml of 5 ng/ml nonane), [EC-4978](#) (0.1 ml of 5 ng/ml nonane) and [EDF-5581](#) (0.02 ml of 10 ng/ml nonane). Of each sample 1 subsample was additionally spiked with all 209 native PCBs (0.05 ml 100 ng/ml nonane).

Due to the unknown and potentially high levels of PCBs in the samples, spike experiments were performed at relatively high levels to circumvent substantial uncertainty in the determination of the recovery.

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). 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); activated 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 column set was eluted with 85 ml hexane at a flowrate of 2.5 ml/min. During this step the temperature of the two purification columns was maintained at 60°C/140°F. 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 fraction (C) and alumina fraction (A), each contained about 1.5 ml extract.

Concentration and reconstitution

To each of the fractions, carbon, alumina and waste, 0.005 ml of dodecane was added to avoid evaporation of the most volatile PCBs. Consequently, vials containing the carbon fractions and alumina fractions were evaporated till dodecane remained in a [CentriVap Vacuum Concentrator](#) of Labconco (United States). The residues of the alumina fractions were reconstituted in 0.1 ml [EC-4979](#) (5 ng/ml nonane) and the carbon fractions in 0.05ml [EC-4979](#) (5 ng/ml nonane) and 0.05 ml [ED-911](#) (1 ng/ml nonane).

The waste was evaporated on a rotary evaporator from Heidolph (Germany) till approximately 1 ml. The residue was transferred to a 1.5 ml glass vial and 0.1 ml [EC-4979](#) (5 ng/ml nonane) was added.

Measurement

All extracts were measured on a APGC-MSMS v2 from Waters (United Kingdom) equipped with an Agilent (United States) A7890 GC with a split/spitless injector. The GC column installed was a HT8-PCB (60 m x 0.25 mm x 0.1 μ m) from Trajan.

Of each sample, 2 μ l was injected in spitless mode at an injector temperature of 280 °C and an oven temperature of 100 °C. The initial oven temperature was held for 1 min after which a ramp of 20 °C/min was applied till 180 °C. The ramp was decreased till 4 °C/min till 260 °C followed by a ramp of 5°C/min till 300 °C and 10 °C/min till 320°C. The temperature was held for 5 minutes at 320 °C. With 30 °C/min the temperature was raised till 330 °C and held at this temperature for a final 6 minutes. The transferline to the APGC-MSMS source was kept 310 °C. The Helium (5.0) gas flow was set to 2 ml/min.

The APGC was tuned for optimal sensitivity and reproducibility for pesticide analysis at 1.0 Da resolution. These settings were also used for the analysis of Dioxins, Furans and PCBs. In short, the make-up gas was set at 350 ml/min, the cone gas flow 210 L/Hr, the auxiliary gas flow 200 L/Hr, the corona current at 0.5 μ A and the source temperature at 150 °C. The MS settings are given in annex 8.

The data was processed using Masslynx Targetlynx from Waters (United Kingdom). For calibration, standards were analysed over a range of 0.05 ng/ml till 50 ng/ml.

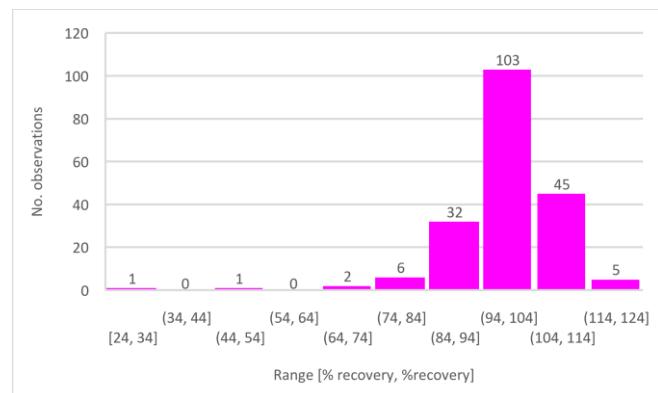


Figure 3 Histogram of the initial recovery for PCBs including those for internal standards.

Results and discussion

Recoveries of all analytes were calculated and used for calculation of IPR. In total, recoveries for native PCBs were obtained in 9 samples, including a spiked blank, while recoveries in 16 samples were obtained for internal standards (fig. 3 and fig. 4). For the precision of Dioxins and Furans, the number of datapoints vary. The level and number of datapoints depended on the occurrence of the analytes in the samples as they were

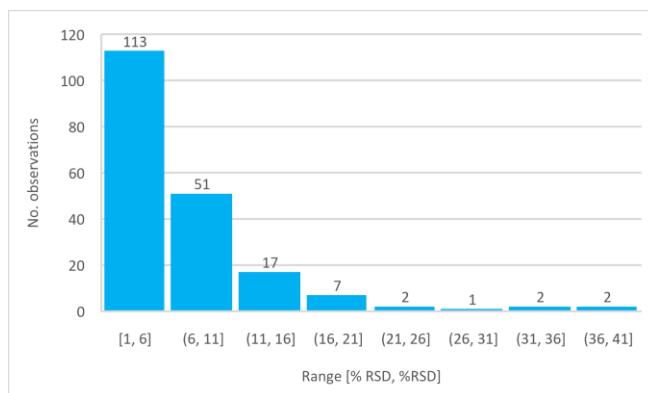


Figure 4 Histogram of the initial precision for PCBs including those for internal standards.

not spiked with native Dioxins and Furans. Results for levels of Dioxins, Furans and PCBs determined in samples are given in annex 5.

In annex 1 IPR are given for all native 209 PCBs, labelled toxic/LOC/window-defining PCBs, labelled clean-up PCBs and injection PCBs. In annex 2 IPR results are given for native Dioxins and Furans, labelled Dioxins and Furans and the injection standard.

All determined IPR for 209 PCBs, labelled PCBs, Dioxins, Furans and labelled Dioxins and Furans complied with the criteria set in EPA method 1613B¹ and EPA method 1668C². Native congeners were recovery corrected against the specified internal standard listed in annex 1. Dioxins and Furans were recovery corrected for their analogue internal standard.

The result for PCB12/13 was not corrected for an internal standard. This was due to the fractionation of these compounds over both the carbon and alumina column. The combined result of both fractions complied with IPR criteria.

EPA method 1668C PCBs

The recovery of ¹³C₁₂ PCB3, ¹³C₁₂ PCB209 and some non-ortho congeners varied more or were somewhat lower. These congeners fractionate over both columns or experience a stronger or weaker absorption to the silver nitrate and sulfuric acid column or the alumina column. At higher matrix loads, e.g. 5 gram soil or sewage sludge, 10 gram sediment or 1 gram fish oil, ¹³C₁₂ PCB209 was not retained sufficiently on the alumina column and eluted to the waste. The fractionation profiles for PCBs are given in annex 3 for all matrices.

For the sewage sludge and the Dust sample the alumina fraction was slightly turbid, yet this didn't have an effect on the chromatograms (annex 6). All Carbon fractions were clear. The volume of the carbon fraction is generally reduced to 10 µl. In this case the fraction was reduced to 100 µl to avoid saturation of the detection system.

In both EPA 1613B¹ and 1668C² a sample intake of 10 gram is suggested. Most sample intakes from the matrices tested were lower than 10 gram. In terms of homogeneity sufficient intake should be considered based on the material and grinding process. But, in terms of sufficient sensitivity, current detection systems have become much more sensitive reaching minimum levels (MLs) below 1 fg on-column according specifications⁸. In routine use 10 fg on-column is more likely to be continuously obtained. These detection limits allow for a smaller sample intake or injection of a smaller portion of the extract. Calculated MDLs for the analysis in this study varied between 0.1 pg/g and 1 pg/g based on a sample intake of 1 gram, a final volume of 100 µl and a signal to noise ratio (root mean square) (S/N_{RMS}) of 10.

Finally, also the method blanks should be considered, e.g. the lower the sample intake the higher the relative method blank contribution to the overall result. Based on 1 gram sample the majority of the PCBs complied to the EPA1668C paragraph 9.5.2 quality criteria for blanks (annex 4). A total of 12 PCBs (co-elutions) were above 2 times the MLs listed in table 2 of EPA1668C.

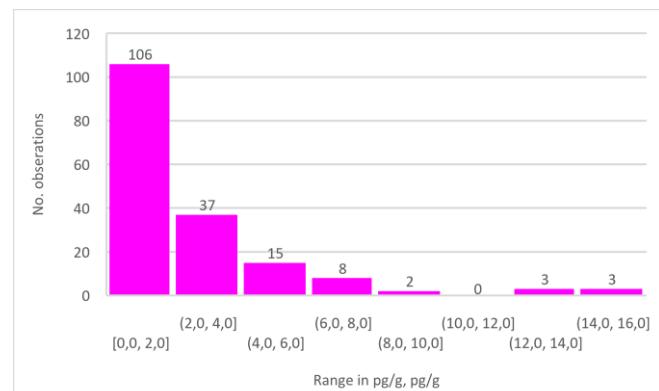


Figure 5. Histogram of the amounts found in the blank expressed on 1 gram sample.

The highest value found was 15.1 pg/g for PCB52/69/73. On extract base all PCBs comply to the blank quality criteria, e.g. calculated on a 20 µl extract

all results would be below 1 pg/µl. The levels found are within normal laboratory background and are expected to be of marginal influence at regulatory levels.

EPA method 1613B Dioxins and Furans

Recoveries for labelled Dioxins and Furans were in agreement with IPR criteria in EPA1613B¹ as well as the precision for the congeners naturally present in the samples. Dioxins and Furans were found in blank samples between 0.1 and 400 pg/g depending the sample and congener.

The MDL was set as for PCBs and Dioxins at S/N_{RMS} 10. Except for 1,2,3,4,7,8,9-HpCDF all MDLs were below the MLs. For this congener, the sensitivity of the detection system was considerably lower than for its constitutional isomer 1,2,3,4,6,7,8-HpCDF probably due to the temperature ramp of the GC. The lower sensitivity was also observed for OCDD and OCDF.

Dioxins as well as Furans were not detected in the blank and comply with the MLs. Considering the limit of detection of 1,2,3,4,7,8,9-HpCDF also this congener complies with its ML.

Conclusion

Purification for Dioxins, Furans and PCBs by [GO-eHT](#) can fulfil to IPR criteria set in EPA method 1613B¹ and 1668C².

The method allows for the determination of levels of Dioxins and Furans in combination with all 209 PCBs in environmental samples and fish oil in agreement with the required MDLs.

Furthermore, the separation of more toxic non-ortho PCBs and dioxins to the carbon fraction allows for low MDLs together with detection of high levels of other PCBs in the alumina fraction.

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 organic pollutants (POPs) in foods, Chemosphere 256 (2020) 127023
- 4) NEN 7777+C1:2012 en; Environment and food – Performance characteristics of measurement methods
- 5) NEN-EN 16215:2020, Animal feeding stuffs: Methods of sampling and analysis - Determination of dioxins and dioxin-like PCBs and of indicator PCBs by GC/HRMS
- 6) Commission Regulation (EU) 2017/644 of 5 April 2017 laying down methods of sampling and analysis for the control of levels of dioxins, dioxin-like PCBs and non-dioxin-like PCBs in certain foodstuffs.
- 7) Commission Regulation (EC) No 152/2009 of 27 January 2009 laying down the methods of sampling and analysis for the official control of feed.
- 8) NEN-EN 16167:2018+C1:2019 en, Soil, treated biowaste and sludge – Determination of dioxins and furans and dioxin-like polychlorinated biphenyls by gas chromatography with high resolution mass selective detection (HR GC-MS)
- 9) NEN-EN 16190:2019 en, Soil, treated biowaste and sludge – Determination of polychlorinated biphenyls by gas chromatography with mass selective detection (GC-MS) and gas chromatography with electron-capture detection (GC-ECD)
- 10) Guillaume ten Dam, MS Systems for the Analysis of Dioxins and PCBs, 2019, DSP Systems

Annexes

1. Initial precision and recovery (IPR) for PCBs – EPA 1668C
2. Initial precision and recovery (IPR) for Dioxins and Furans – EPA 1613B
3. Fractionation profiles* for all 209 PCBs in all samples
4. Results for PCBs for the system blank
5. Results and recovery in blank* samples for Dioxins and Furans (EPA 1613B) and PCBs (EPA 1668C)
6. Chromatograms of the analysis of PCBs in blank* samples
7. Chromatograms of blank* samples for Dioxins and Furans (EPA 1613B)
8. MS method

Annex 1. Initial precision and recovery (IPR) for PCBs – EPA 1668C

Name	Internal standard	Level ng/ml	n	Fraction	IPR			Xgem (%)		
					RSD			Xgem (%)		
					Experimental	IPR criterium	Assesment	Experimental	IPR criterium	Assesment
Native PCB										
Cl1*	13C PCB 1	150	9	Alumina	7	25	Compliant	75	70-130	Compliant
Cl2*	13C PCB 4	600	9	Alumina	7	25	Compliant	91	70-130	Compliant
Cl3*	13C PCB 19	1200	9	Alumina	5	25	Compliant	99	70-130	Compliant
Cl4*	13C PCB 54	2100	9	Alumina	3	25	Compliant	100	70-130	Compliant
Cl5*	13C PCB 104	2300	9	Alumina	7	25	Compliant	101	70-130	Compliant
Cl6*	13C PCB 155	2100	9	Alumina	5	25	Compliant	90	70-130	Compliant
Cl7*	13C PCB 188	1200	9	Alumina	4	25	Compliant	98	70-130	Compliant
Cl8*	13C PCB 202	600	9	Alumina	4	25	Compliant	98	70-130	Compliant
Cl9*	13C PCB 208	150	9	Alumina	5	25	Compliant	103	70-130	Compliant
Cl10*	13C PCB 209	50	9	Alumina	5	25	Compliant	100	70-130	Compliant
1										
PCB1	13C PCB 1	50	9	Alumina	3	25	Compliant	103	70-130	Compliant
PCB2	13C PCB 1	50	9	Alumina	9	25	Compliant	79	70-130	Compliant
PCB3	13C PCB 3	50	9	Carbon	2	25	Compliant	101	70-130	Compliant
2										
PCB10	13C PCB 4	50	9	Alumina	4	25	Compliant	95	70-130	Compliant
PCB4	13C PCB 4	50	9	Alumina	3	25	Compliant	103	70-130	Compliant
PCB9	13C PCB 4	50	9	Alumina	5	25	Compliant	101	70-130	Compliant
PCB7	13C PCB 4	50	9	Alumina	5	25	Compliant	99	70-130	Compliant
PCB6	13C PCB 4	50	9	Alumina	3	25	Compliant	114	70-130	Compliant
PCB8/5	13C PCB 4	100	9	Alumina	2	25	Compliant	101	70-130	Compliant
PCB14	13C PCB 4	50	9	Alumina	7	25	Compliant	97	70-130	Compliant
PCB11	13C PCB 4	50	9	Alumina	13	25	Compliant	96	70-130	Compliant
PCB12/13	External	100	9	Combined	9	25	Compliant	77	70-130	Compliant
PCB15	13C PCB 15	50	9	Carbon	3	25	Compliant	106	70-130	Compliant
3										
PCB19	13C PCB 19	50	9	Alumina	2	25	Compliant	102	70-130	Compliant
PCB30	13C PCB 19	50	9	Alumina	6	25	Compliant	102	70-130	Compliant
PCB18	13C PCB 19	50	9	Alumina	4	25	Compliant	101	70-130	Compliant
PCB17	13C PCB 19	50	9	Alumina	8	25	Compliant	111	70-130	Compliant
PCB24	13C PCB 19	50	9	Alumina	5	25	Compliant	100	70-130	Compliant
PCB27	13C PCB 19	50	9	Alumina	4	25	Compliant	109	70-130	Compliant
PCB32	13C PCB 19	50	9	Alumina	4	25	Compliant	114	70-130	Compliant
PCB16	13C PCB 19	50	9	Alumina	5	25	Compliant	98	70-130	Compliant
PCB23	13C PCB 19	50	9	Alumina	7	25	Compliant	94	70-130	Compliant
PCB34	13C PCB 19	50	9	Alumina	5	25	Compliant	109	70-130	Compliant
PCB29	13C PCB 19	50	9	Alumina	13	25	Compliant	113	70-130	Compliant

Name	Internal standard	Level ng/ml	n	Fraction	IPR			Xgem (%)		
					Experimental	IPR criterium	Assesment	Experimental	IPR criterium	Assesment
PCB26	13C PCB 19	50	9	Alumina	5	25	Compliant	109	70-130	Compliant
PCB25	13C PCB 19	50	9	Alumina	7	25	Compliant	113	70-130	Compliant
PCB31	13C PCB 19	50	9	Alumina	6	25	Compliant	111	70-130	Compliant
PCB28	13C PCB 19	50	9	Alumina	6	25	Compliant	110	70-130	Compliant
PCB21	13C PCB 19	50	9	Alumina	6	25	Compliant	101	70-130	Compliant
PCB20/33	13C PCB 19	100	9	Alumina	5	25	Compliant	105	70-130	Compliant
PCB22	13C PCB 19	50	9	Alumina	3	25	Compliant	102	70-130	Compliant
PCB36	13C PCB 19	50	9	Alumina	8	25	Compliant	95	70-130	Compliant
PCB39	13C PCB 19	50	9	Alumina	15	25	Compliant	88	70-130	Compliant
PCB38	13C PCB 19	50	9	Alumina	10	25	Compliant	95	70-130	Compliant
PCB35	13C PCB 19	50	9	Alumina	18	25	Compliant	74	70-130	Compliant
PCB37	13C PCB 37	50	9	Carbon	2	25	Compliant	103	70-130	Compliant
4										
PCB54	13C PCB 54	50	9	Alumina	4	25	Compliant	102	70-130	Compliant
PCB50	13C PCB 54	50	9	Alumina	3	25	Compliant	101	70-130	Compliant
PCB53	13C PCB 54	50	9	Alumina	5	25	Compliant	104	70-130	Compliant
PCB51	13C PCB 54	50	9	Alumina	3	25	Compliant	102	70-130	Compliant
PCB45	13C PCB 54	50	9	Alumina	4	25	Compliant	101	70-130	Compliant
PCB46	13C PCB 54	50	9	Alumina	7	25	Compliant	99	70-130	Compliant
PCB52/69/73	13C PCB 54	150	9	Alumina	4	25	Compliant	102	70-130	Compliant
PCB43/49	13C PCB 54	100	9	Alumina	11	25	Compliant	99	70-130	Compliant
PCB47/48/65/75	13C PCB 54	200	9	Alumina	3	25	Compliant	104	70-130	Compliant
PCB62	13C PCB 54	50	9	Alumina	7	25	Compliant	92	70-130	Compliant
PCB44/59	13C PCB 54	100	9	Alumina	5	25	Compliant	109	70-130	Compliant
PCB42	13C PCB 54	50	9	Alumina	11	25	Compliant	105	70-130	Compliant
PCB64/71/72	13C PCB 54	150	9	Alumina	5	25	Compliant	107	70-130	Compliant
PCB41	13C PCB 54	50	9	Alumina	15	25	Compliant	96	70-130	Compliant
PCB68	13C PCB 54	50	9	Alumina	6	25	Compliant	100	70-130	Compliant
PCB40	13C PCB 54	50	9	Alumina	8	25	Compliant	99	70-130	Compliant
PCB57	13C PCB 54	50	9	Alumina	7	25	Compliant	93	70-130	Compliant
PCB67	13C PCB 54	50	9	Alumina	7	25	Compliant	104	70-130	Compliant
PCB58/63	13C PCB 54	100	9	Alumina	8	25	Compliant	89	70-130	Compliant
PCB61/74	13C PCB 54	100	9	Alumina	6	25	Compliant	101	70-130	Compliant
PCB70	13C PCB 54	50	9	Alumina	6	25	Compliant	99	70-130	Compliant
PCB76	13C PCB 54	50	9	Alumina	8	25	Compliant	91	70-130	Compliant
PCB66/80	13C PCB 54	100	9	Alumina	8	25	Compliant	108	70-130	Compliant
PCB55	13C PCB 54	50	9	Alumina	3	25	Compliant	102	70-130	Compliant
PCB56/60	13C PCB 54	100	9	Alumina	4	25	Compliant	106	70-130	Compliant
PCB79	13C PCB 54	50	9	Alumina	18	25	Compliant	85	70-130	Compliant
PCB78	13C PCB 54	50	9	Alumina	8	25	Compliant	89	70-130	Compliant

Name	Internal standard	Level ng/ml	n	Fraction	IPR			Xgem (%)		
					Experimental	IPR criterium	Assesment	Experimental	IPR criterium	Assesment
PCB81	13C PCB 81	50	9	Carbon	4	25	Compliant	101	70-130	Compliant
PCB77	13C PCB 77	50	9	Carbon	4	25	Compliant	99	70-130	Compliant
5										
PCB104	13C PCB 104	50	9	Alumina	2	25	Compliant	100	70-130	Compliant
PCB96	13C PCB 104	50	9	Alumina	5	25	Compliant	106	70-130	Compliant
PCB103	13C PCB 104	50	9	Alumina	3	25	Compliant	101	70-130	Compliant
PCB100	13C PCB 104	50	9	Alumina	3	25	Compliant	102	70-130	Compliant
PCB94	13C PCB 104	50	9	Alumina	3	25	Compliant	105	70-130	Compliant
PCB93/95/98/102	13C PCB 104	200	9	Alumina	5	25	Compliant	102	70-130	Compliant
PCB88	13C PCB 104	50	9	Alumina	10	25	Compliant	101	70-130	Compliant
PCB91/121	13C PCB 104	100	9	Alumina	7	25	Compliant	100	70-130	Compliant
PCB92	13C PCB 104	50	9	Alumina	15	25	Compliant	119	70-130	Compliant
PCB84	13C PCB 104	50	9	Alumina	19	25	Compliant	103	70-130	Compliant
PCB89	13C PCB 104	50	9	Alumina	19	25	Compliant	116	70-130	Compliant
PCB90/101	13C PCB 104	100	9	Alumina	14	25	Compliant	92	70-130	Compliant
PCB113	13C PCB 104	50	9	Alumina	12	25	Compliant	99	70-130	Compliant
PCB99	13C PCB 104	50	9	Alumina	16	25	Compliant	99	70-130	Compliant
PCB112/119	13C PCB 104	100	9	Alumina	18	25	Compliant	99	70-130	Compliant
PCB83	13C PCB 104	50	9	Alumina	16	25	Compliant	81	70-130	Compliant
PCB108	13C PCB 104	50	9	Alumina	18	25	Compliant	113	70-130	Compliant
PCB86/97/116/117	13C PCB 104	200	9	Alumina	8	25	Compliant	105	70-130	Compliant
PCB87/115/125	13C PCB 104	150	9	Alumina	11	25	Compliant	99	70-130	Compliant
PCB111	13C PCB 104	50	9	Alumina	15	25	Compliant	91	70-130	Compliant
PCB85	13C PCB 104	50	9	Alumina	13	25	Compliant	103	70-130	Compliant
PCB110/120	13C PCB 104	100	9	Alumina	9	25	Compliant	100	70-130	Compliant
PCB82	13C PCB 104	50	9	Alumina	6	25	Compliant	104	70-130	Compliant
PCB124	13C PCB 104	50	9	Alumina	11	25	Compliant	107	70-130	Compliant
PCB107/109	13C PCB 104	100	9	Alumina	13	25	Compliant	105	70-130	Compliant
PCB123	13C PCB 123	50	9	Alumina	9	25	Compliant	99	70-130	Compliant
PCB106/118	13C PCB 118	100	9	Alumina	5	25	Compliant	101	70-130	Compliant
PCB114	13C PCB 114	50	9	Alumina	3	25	Compliant	102	70-130	Compliant
PCB122	13C PCB 104	50	9	Alumina	9	25	Compliant	103	70-130	Compliant
PCB105/127	13C PCB 105	100	9	Alumina	4	25	Compliant	94	70-130	Compliant
PCB126	13C PCB 126	50	9	Carbon	6	25	Compliant	99	70-130	Compliant
6										
PCB155	13C PCB 155	50	9	Alumina	1	25	Compliant	90	70-130	Compliant
PCB150	13C PCB 155	50	9	Alumina	2	25	Compliant	91	70-130	Compliant
PCB152	13C PCB 155	50	9	Alumina	2	25	Compliant	92	70-130	Compliant
PCB145	13C PCB 155	50	9	Alumina	3	25	Compliant	93	70-130	Compliant
PCB136/148	13C PCB 155	100	9	Alumina	2	25	Compliant	90	70-130	Compliant

Name	Internal standard	Level ng/ml	n	Fraction	IPR			Xgem (%)		
					Experimental	IPR criterium	Assesment	Experimental	IPR criterium	Assesment
PCB154	13C PCB 155	50	9	Alumina	4	25	Compliant	96	70-130	Compliant
PCB151	13C PCB 155	50	9	Alumina	6	25	Compliant	91	70-130	Compliant
PCB135	13C PCB 155	50	9	Alumina	8	25	Compliant	81	70-130	Compliant
PCB144/147	13C PCB 155	100	9	Alumina	4	25	Compliant	97	70-130	Compliant
PCB139/149	13C PCB 155	100	9	Alumina	6	25	Compliant	94	70-130	Compliant
PCB140	13C PCB 155	50	9	Alumina	11	25	Compliant	93	70-130	Compliant
PCB143	13C PCB 155	50	9	Alumina	11	25	Compliant	94	70-130	Compliant
PCB134	13C PCB 155	50	9	Alumina	10	25	Compliant	98	70-130	Compliant
PCB131/133/142	13C PCB 155	150	9	Alumina	7	25	Compliant	94	70-130	Compliant
PCB165	13C PCB 155	50	9	Alumina	12	25	Compliant	91	70-130	Compliant
PCB146	13C PCB 155	50	9	Alumina	12	25	Compliant	92	70-130	Compliant
PCB132/161	13C PCB 155	100	9	Alumina	10	25	Compliant	93	70-130	Compliant
PCB153	13C PCB 155	50	9	Alumina	9	25	Compliant	86	70-130	Compliant
PCB168	13C PCB 155	50	9	Alumina	10	25	Compliant	94	70-130	Compliant
PCB141	13C PCB 155	50	9	Alumina	5	25	Compliant	91	70-130	Compliant
PCB137	13C PCB 155	50	9	Alumina	8	25	Compliant	93	70-130	Compliant
PCB130	13C PCB 155	50	9	Alumina	10	25	Compliant	94	70-130	Compliant
PCB164/163	13C PCB 155	100	9	Alumina	11	25	Compliant	97	70-130	Compliant
PCB138	13C PCB 155	50	9	Alumina	25	25	Compliant	100	70-130	Compliant
PCB158/160	13C PCB 155	100	9	Alumina	21	25	Compliant	89	70-130	Compliant
PCB129	13C PCB 155	50	9	Alumina	6	25	Compliant	91	70-130	Compliant
PCB166	13C PCB 155	50	9	Alumina	7	25	Compliant	96	70-130	Compliant
PCB159	13C PCB 155	50	9	Alumina	11	25	Compliant	95	70-130	Compliant
PCB128/162	13C PCB 155	100	9	Alumina	9	25	Compliant	100	70-130	Compliant
PCB167	13C PCB 167	50	9	Alumina	6	25	Compliant	104	70-130	Compliant
PCB156	13C PCB 156	50	9	Alumina	5	25	Compliant	100	70-130	Compliant
PCB157	13C PCB 157	50	9	Alumina	5	25	Compliant	102	70-130	Compliant
PCB169	13C PCB 169	50	9	Carbon	3	25	Compliant	97	70-130	Compliant
7										
PCB188	13C PCB 188	50	9	Alumina	2	25	Compliant	100	70-130	Compliant
PCB184	13C PCB 188	50	9	Alumina	2	25	Compliant	99	70-130	Compliant
PCB179	13C PCB 188	50	9	Alumina	3	25	Compliant	98	70-130	Compliant
PCB176	13C PCB 188	50	9	Alumina	4	25	Compliant	97	70-130	Compliant
PCB186	13C PCB 188	50	9	Alumina	4	25	Compliant	96	70-130	Compliant
PCB178	13C PCB 188	50	9	Alumina	2	25	Compliant	100	70-130	Compliant
PCB175	13C PCB 188	50	9	Alumina	7	25	Compliant	96	70-130	Compliant
PCB182/187	13C PCB 188	100	9	Alumina	4	25	Compliant	96	70-130	Compliant
PCB183	13C PCB 188	50	9	Alumina	3	25	Compliant	98	70-130	Compliant
PCB185	13C PCB 188	50	9	Alumina	10	25	Compliant	96	70-130	Compliant
PCB174	13C PCB 188	50	9	Alumina	9	25	Compliant	99	70-130	Compliant

IPR										
Name	Internal standard	Level ng/ml	n	Fraction	RSD			Xgem (%)		
					Experimental	IPR criterium	Assesment	Experimental	IPR criterium	Assesment
PCB181	13C PCB 188	50	9	Alumina	11	25	Compliant	95	70-130	Compliant
PCB177	13C PCB 188	50	9	Alumina	11	25	Compliant	103	70-130	Compliant
PCB171	13C PCB 188	50	9	Alumina	4	25	Compliant	102	70-130	Compliant
PCB173	13C PCB 188	50	9	Alumina	4	25	Compliant	100	70-130	Compliant
PCB172	13C PCB 188	50	9	Alumina	3	25	Compliant	98	70-130	Compliant
PCB192	13C PCB 188	50	9	Alumina	6	25	Compliant	98	70-130	Compliant
PCB180/193	13C PCB 188	100	9	Alumina	4	25	Compliant	99	70-130	Compliant
PCB191	13C PCB 188	50	9	Alumina	4	25	Compliant	97	70-130	Compliant
PCB170	13C PCB 188	50	9	Alumina	4	25	Compliant	106	70-130	Compliant
PCB190	13C PCB 188	50	9	Alumina	4	25	Compliant	103	70-130	Compliant
PCB189	13C PCB 189	50	9	Alumina	3	25	Compliant	104	70-130	Compliant
8										
PCB202	13C PCB 202	50	9	Alumina	2	25	Compliant	102	70-130	Compliant
PCB201	13C PCB 202	50	9	Alumina	3	25	Compliant	95	70-130	Compliant
PCB204	13C PCB 202	50	9	Alumina	4	25	Compliant	104	70-130	Compliant
PCB197	13C PCB 202	50	9	Alumina	3	25	Compliant	99	70-130	Compliant
PCB200	13C PCB 202	50	9	Alumina	5	25	Compliant	90	70-130	Compliant
PCB198/199	13C PCB 202	100	9	Alumina	3	25	Compliant	99	70-130	Compliant
PCB196/203	13C PCB 202	100	9	Alumina	4	25	Compliant	100	70-130	Compliant
PCB195	13C PCB 202	50	9	Alumina	3	25	Compliant	100	70-130	Compliant
PCB194	13C PCB 202	50	9	Alumina	6	25	Compliant	101	70-130	Compliant
PCB205	13C PCB 205	50	9	Alumina	2	25	Compliant	101	70-130	Compliant
9										
PCB208	13C PCB 208	50	9	Alumina	12	25	Compliant	100	70-130	Compliant
PCB207	13C PCB 208	50	9	Alumina	2	25	Compliant	100	70-130	Compliant
PCB206	13C PCB 206	50	9	Alumina	2	25	Compliant	102	70-130	Compliant
10										
PCB209	13C PCB 209	50	9	Alumina	5	25	Compliant	100	70-130	Compliant
TOXIC/LOC/WINDOW DEFINING										
13C PCB 1	13C PCB 9	5	16	Alumina	3	70	Compliant	107	20-135	Compliant
13C PCB 3	13C PCB 9	5	16	Carbon	39	70	Compliant	24	20-135	Compliant
13C PCB 4	13C PCB 9	5	16	Alumina	4	70	Compliant	111	20-135	Compliant
13C PCB 15	13C PCB 9	5	16	Carbon	35	70	Compliant	70	20-135	Compliant
13C PCB 19	13C PCB 9	5	16	Alumina	4	70	Compliant	113	20-135	Compliant
13C PCB 37	13C PCB 9	5	16	Carbon	30	70	Compliant	78	20-135	Compliant
13C PCB 54	13C PCB 52	5	16	Alumina	5	70	Compliant	110	20-135	Compliant
13C PCB 81	13C PCB 52	7	16	Carbon	34	50	Compliant	50	45-135	Compliant
13C PCB 77	13C PCB 52	7	16	Carbon	22	50	Compliant	97	45-135	Compliant
13C PCB 104	13C PCB 101	5	16	Alumina	5	50	Compliant	110	45-135	Compliant
13C PCB 123	13C PCB 101	5	16	Alumina	6	50	Compliant	115	45-135	Compliant

IPR										
Name	Internal standard	Level ng/ml	n	Fraction	RSD			Xgem (%)		
					Experimental	IPR criterium	Assesment	Experimental	IPR criterium	Assesment
13C PCB 118	13C PCB 101	5	16	Alumina	6	50	Compliant	114	45-135	Compliant
13C PCB 114	13C PCB 101	5	16	Alumina	5	50	Compliant	112	45-135	Compliant
13C PCB 105	13C PCB 101	5	16	Alumina	4	50	Compliant	113	45-135	Compliant
13C PCB 126	13C PCB 101	7	16	Carbon	15	50	Compliant	105	45-135	Compliant
13C PCB 155	13C PCB 138	5	16	Alumina	7	50	Compliant	121	45-135	Compliant
13C PCB 167	13C PCB 138	5	16	Alumina	6	50	Compliant	108	45-135	Compliant
13C PCB 156	13C PCB 138	5	16	Alumina	4	50	Compliant	109	45-135	Compliant
13C PCB 157	13C PCB 138	5	16	Alumina	3	50	Compliant	109	45-135	Compliant
13C PCB 169	13C PCB 138	7	16	Carbon	9	50	Compliant	108	45-135	Compliant
13C PCB 188	13C PCB 138	5	16	Alumina	5	50	Compliant	112	45-135	Compliant
13C PCB 189	13C PCB 138	5	16	Alumina	5	50	Compliant	111	45-135	Compliant
13C PCB 202	13C PCB 194	5	16	Alumina	5	50	Compliant	113	45-135	Compliant
13C PCB 205	13C PCB 194	5	16	Alumina	3	50	Compliant	113	45-135	Compliant
13C PCB 208	13C PCB 194	5	16	Alumina	6	50	Compliant	112	45-135	Compliant
13C PCB 206	13C PCB 194	5	16	Alumina	5	50	Compliant	110	45-135	Compliant
13C PCB 209	13C PCB 194	5	16	Alumina	40	50	Compliant	77	45-135	Compliant
CLEAN-UP										
13C PCB 28	13C PCB 9	5	16	Alumina	7	70	Compliant	115	20-135	Compliant
13C PCB 111	13C PCB 101	5	16	Alumina	4	50	Compliant	109	45-135	Compliant
13C PCB 178	13C PCB 138	5	16	Alumina	2	50	Compliant	110	45-135	Compliant
INJECTION										
13C PCB 9	External	5*	16	Carbon & Alumina	10	-	-	69	-	-
13C PCB 52	External	5*	16	Carbon & Alumina	7	-	-	74	-	-
13C PCB 101	External	5*	16	Carbon & Alumina	8	-	-	75	-	-
13C PCB 138	External	5*	16	Carbon & Alumina	12	-	-	80	-	-
13C PCB 194	External	5*	16	Carbon & Alumina	11	-	-	80	-	-

* Total constitutional isomer results (Cl1, Cl2, etc) are based on integration of the full chromatographic window and are not the sum of individual congeners.

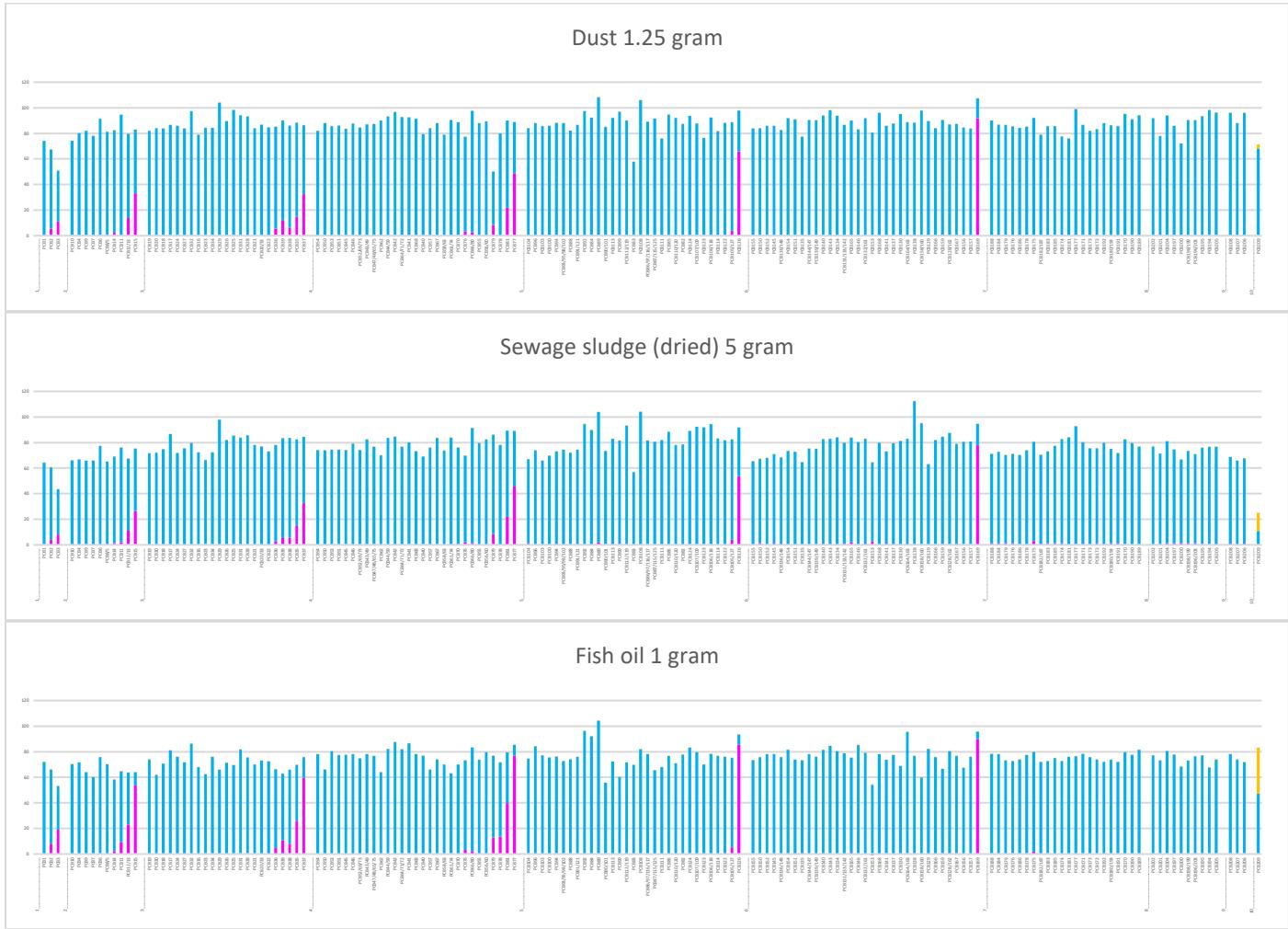
Annex 2. Initial precision and recovery (IPR) for Dioxins and Furans – EPA 1613B

CDD/CDF	Level ng/kg (12C) ng/ml (13C)	n	Fraction	IPR					
				RSD			Xgem (%)		
				Experimental	IPR criterium	Assesment	Experimental	IPR criterium	
2378-TCDF	0.1 - 2.6	6	Carbon	9.0	20.0	Compliant	-	-	
12378-PeCDF	-	0	Carbon	-	-	-	-	-	
23478-PeCDF	0.9 - 3.2	3	Carbon	3.9	17.2	Compliant	-	-	
123478-HxCDF	0.3 - 1.1	6	Carbon	11.9	17.4	Compliant	-	-	
123678-HxCDF	0.2 - 2.6	6	Carbon	13.1	13.4	Compliant	-	-	
234678-HxCDF	0.1 - 1.1	7	Carbon	14.0	14.8	Compliant	-	-	
123789-HxCDF	-	0	Carbon	-	-	-	-	-	
1234678-HpCDF	1.0 - 13	4	Carbon	8.3	12.6	Compliant	-	-	
1234789-HpCDF	-	0	Carbon	-	-	-	-	-	
OCDF	6.3 - 17	4	Carbon	6.0	27.0	Compliant	-	-	
2378-TCDD	0.6 - 0.6	1	Carbon	2.4	28.0	Compliant	-	-	
12378-PeCDD	2.7 - 2.8	1	Carbon	2.0	15.0	Compliant	-	-	
123478-HxCDD	2.0 - 2.3	2	Carbon	7.9	18.8	Compliant	-	-	
123678-HxCDD	0.9 - 6.6	5	Carbon	11.1	15.4	Compliant	-	-	
123789-HxCDD	0.5 - 4.2	5	Carbon	11.9	22.2	Compliant	-	-	
1234678-HpCDD	0.6 - 48	6	Carbon	8.9	15.4	Compliant	-	-	
OCDD	5.4 - 380	6	Carbon	5.3	19.0	Compliant	-	-	
Internal standards									
13C-2378-TCDF	2	16	Carbon	4	35	Compliant	90	31-113	Compliant
13C-12378-PeCDF	2	16	Carbon	5	34	Compliant	90	27-156	Compliant
13C-23478-PeCDF	2	16	Carbon	5	38	Compliant	92	16-279	Compliant
13C-123478-HxCDF	2	16	Carbon	7	43	Compliant	86	27-152	Compliant
13C-123678-HxCDF	2	16	Carbon	8	35	Compliant	85	30-122	Compliant
13C-234678-HxCDF	2	16	Carbon	6	37	Compliant	86	29-136	Compliant
13C-123789-HxCDF	2	16	Carbon	13	40	Compliant	90	24-157	Compliant
13C-1234678-HpCDF	2	16	Carbon	6	41	Compliant	85	32-110	Compliant
13C-1234789-HpCDF	2	16	Carbon	14	40	Compliant	106	28-141	Compliant
13C-OCDF	2	16	Carbon	21	-	-	110	-	-
13C-2378-TCDD	2	16	Carbon	4	37	Compliant	88	28-134	Compliant
13C-12378-PeCDD	2	16	Carbon	7	39	Compliant	88	27-184	Compliant
13C-123478-HxCDD	2	16	Carbon	9	41	Compliant	86	29-147	Compliant
13C-123678-HxCDD	2	16	Carbon	10	38	Compliant	89	34-122	Compliant
13C-123789-HxCDD	2	16	Carbon	5	-	-	86	-	-
13C-1234678-HpCDD	2	16	Carbon	6	35	Compliant	86	34-129	Compliant
13C-OCDD	2	16	Carbon	9	95	Compliant	88	41-276	Compliant
Injection standard									
13C-1234-TCDD	0.5	16	Carbon	5	-	-	90	-	-

Annex 3. Fractionation profiles* for all 209 PCBs in all samples

*Recoveries are not corrected for Injection recovery, toxic/loc/window defining or clean-up standard. **Pink** is the recovery in the carbon fraction, **blue** the recovery in the alumina fraction and **yellow** the recovery in the waste. The Y-axes are the recovery in % and each line represents an increments of 20%.





Annex 4. Results for PCBs for the system blank

Name	IS correction	Fraction final result	Final result pg/g	ML	Assessment**
<u>209PCB</u>					
Cl1*	13C PCB 1	Alumina	13,5	9	Compliant
Cl2*	13C PCB 4	Alumina	58,5	43	Compliant
Cl3*	13C PCB 19	Alumina	104,9	70	Compliant
Cl4*	13C PCB 54	Alumina	96,5	217	Compliant
Cl5*	13C PCB 104	Alumina	97,4	247	Compliant
Cl6*	13C PCB 155	Alumina	72,7	270	Compliant
Cl7*	13C PCB 188	Alumina	39,3	135	Compliant
Cl8*	13C PCB 202	Alumina	20,9	70	Compliant
Cl9*	13C PCB 208	Alumina	5,8	15	Compliant
Cl10*	13C PCB 209	Alumina	2,2	5	Compliant
<u>1</u>					
PCB1	13C PCB 1	Alumina	2,8	2	Compliant
PCB2	13C PCB 1	Alumina	1,1	2	Compliant
PCB3	13C PCB 3	Carbon	4,2	5	Compliant
<u>2</u>					
PCB10	13C PCB 4	Alumina	0,0	5	Compliant
PCB4	13C PCB 4	Alumina	3,5	5	Compliant
PCB9	13C PCB 4	Alumina	1,5	2	Compliant
PCB7	13C PCB 4	Alumina	2,0	2	Compliant
PCB6	13C PCB 4	Alumina	3,0	2	Compliant
PCB8/5	13C PCB 4	Alumina	14,6	5	Non-compliant
PCB14	13C PCB 4	Alumina	2,9	2	Compliant
PCB11	13C PCB 4	Alumina	6,2	10	Compliant
PCB12/13	External	Combined	8,1	5	Compliant
PCB15	13C PCB 15	Carbon	4,7	5	Compliant
<u>3</u>					
PCB19	13C PCB 19	Alumina	1,6	2	Compliant
PCB30	13C PCB 19	Alumina	0,7	5	Compliant
PCB18	13C PCB 19	Alumina	13,4	5	Non-compliant
PCB17	13C PCB 19	Alumina	4,6	2	Non-compliant
PCB24	13C PCB 19	Alumina	1,0	2	Compliant
PCB27	13C PCB 19	Alumina	1,5	2	Compliant
PCB32	13C PCB 19	Alumina	4,3	2	Non-compliant
PCB16	13C PCB 19	Alumina	5,2	2	Non-compliant
PCB23	13C PCB 19	Alumina	1,1	2	Compliant
PCB34	13C PCB 19	Alumina	1,1	2	Compliant
PCB29	13C PCB 19	Alumina	1,1	5	Compliant
PCB26	13C PCB 19	Alumina	3,8	5	Compliant
PCB25	13C PCB 19	Alumina	1,8	2	Compliant
PCB31	13C PCB 19	Alumina	14,6	5	Non-compliant

Name	IS correction	Fraction final result	Final result pg/g	ML	Assessment**
PCB28	13C PCB 19	Alumina	6,7	5	Compliant
PCB21	13C PCB 19	Alumina	1,6	5	Compliant
PCB20/33	13C PCB 19	Alumina	12,2	5	Non-compliant
PCB22	13C PCB 19	Alumina	5,6	2	Non-compliant
PCB36	13C PCB 19	Alumina	3,0	2	Compliant
PCB39	13C PCB 19	Alumina	3,3	2	Compliant
PCB38	13C PCB 19	Alumina	3,7	2	Compliant
PCB35	13C PCB 19	Alumina	4,1	2	Non-compliant
PCB37	13C PCB 37	Carbon	5,8	2	Non-compliant
4					
PCB54	13C PCB 54	Alumina	0,9	5	Compliant
PCB50	13C PCB 54	Alumina	0,9	10	Compliant
PCB53	13C PCB 54	Alumina	1,9	10	Compliant
PCB51	13C PCB 54	Alumina	2,3	5	Compliant
PCB45	13C PCB 54	Alumina	1,4	5	Compliant
PCB46	13C PCB 54	Alumina	0,7	2	Compliant
PCB52/69/73	13C PCB 54	Alumina	15,1	5	Non-compliant
PCB43/49	13C PCB 54	Alumina	6,4	5	Compliant
PCB47/48/65/75	13C PCB 54	Alumina	12,8	5	Non-compliant
PCB62	13C PCB 54	Alumina	0,4	10	Compliant
PCB44/59	13C PCB 54	Alumina	5,8	10	Compliant
PCB42	13C PCB 54	Alumina	2,3	5	Compliant
PCB64/71/72	13C PCB 54	Alumina	6,6	5	Compliant
PCB41	13C PCB 54	Alumina	0,4	10	Compliant
PCB68	13C PCB 54	Alumina	1,0	5	Compliant
PCB40	13C PCB 54	Alumina	1,7	10	Compliant
PCB57	13C PCB 54	Alumina	0,8	5	Compliant
PCB67	13C PCB 54	Alumina	1,9	5	Compliant
PCB58/63	13C PCB 54	Alumina	3,1	5	Compliant
PCB61/74	13C PCB 54	Alumina	5,2	20	Compliant
PCB70	13C PCB 54	Alumina	4,1	20	Compliant
PCB76	13C PCB 54	Alumina	0,8	20	Compliant
PCB66/80	13C PCB 54	Alumina	7,3	5	Compliant
PCB55	13C PCB 54	Alumina	2,2	5	Compliant
PCB56/60	13C PCB 54	Alumina	3,4	5	Compliant
PCB79	13C PCB 54	Alumina	2,8	5	Compliant
PCB78	13C PCB 54	Alumina	2,2	5	Compliant
PCB81	13C PCB 81	Carbon	0,8	5	Compliant
PCB77	13C PCB 77	Carbon	1,8	5	Compliant
5					
PCB104	13C PCB 104	Alumina	1,5	5	Compliant
PCB96	13C PCB 104	Alumina	1,7	5	Compliant

Name	IS correction	Fraction final result	Final result pg/g	ML	Assessment**
PCB103	13C PCB 104	Alumina	1,3	5	Compliant
PCB100	13C PCB 104	Alumina	1,4	20	Compliant
PCB94	13C PCB 104	Alumina	1,5	5	Compliant
PCB93/95/98/102	13C PCB 104	Alumina	9,5	20	Compliant
PCB88	13C PCB 104	Alumina	1,2	5	Compliant
PCB91/121	13C PCB 104	Alumina	3,2	5	Compliant
PCB92	13C PCB 104	Alumina	1,5	5	Compliant
PCB84	13C PCB 104	Alumina	0,8	2	Compliant
PCB89	13C PCB 104	Alumina	1,9	5	Compliant
PCB90/101	13C PCB 104	Alumina	7,4	20	Compliant
PCB113	13C PCB 104	Alumina	1,1	20	Compliant
PCB99	13C PCB 104	Alumina	2,2	10	Compliant
PCB112/119	13C PCB 104	Alumina	2,7	5	Compliant
PCB83	13C PCB 104	Alumina	0,3	10	Compliant
PCB108	13C PCB 104	Alumina	1,7	10	Compliant
PCB86/97/116/117	13C PCB 104	Alumina	7,8	10	Compliant
PCB87/115/125	13C PCB 104	Alumina	4,2	10	Compliant
PCB111	13C PCB 104	Alumina	0,7	5	Compliant
PCB85	13C PCB 104	Alumina	0,8	10	Compliant
PCB110/120	13C PCB 104	Alumina	2,8	5	Compliant
PCB82	13C PCB 104	Alumina	1,9	5	Compliant
PCB124	13C PCB 104	Alumina	1,0	10	Compliant
PCB107/109	13C PCB 104	Alumina	2,4	5	Compliant
PCB123	13C PCB 123	Alumina	1,8	5	Compliant
PCB106/118	13C PCB 118	Alumina	6,3	5	Compliant
PCB114	13C PCB 114	Alumina	0,0	5	Compliant
PCB122	13C PCB 104	Alumina	1,9	5	Compliant
PCB105/127	13C PCB 105	Alumina	4,9	5	Compliant
PCB126	13C PCB 126	Carbon	0,9	5	Compliant
6					
PCB155	13C PCB 155	Alumina	1,8	5	Compliant
PCB150	13C PCB 155	Alumina	1,8	5	Compliant
PCB152	13C PCB 155	Alumina	1,8	5	Compliant
PCB145	13C PCB 155	Alumina	1,6	5	Compliant
PCB136/148	13C PCB 155	Alumina	3,6	5	Compliant
PCB154	13C PCB 155	Alumina	1,4	10	Compliant
PCB151	13C PCB 155	Alumina	0,8	10	Compliant
PCB135	13C PCB 155	Alumina	1,6	10	Compliant
PCB144/147	13C PCB 155	Alumina	1,3	5	Compliant
PCB139/149	13C PCB 155	Alumina	5,0	10	Compliant
PCB140	13C PCB 155	Alumina	1,1	10	Compliant
PCB143	13C PCB 155	Alumina	1,6	10	Compliant

Name	IS correction	Fraction final result	Final result pg/g	ML	Assessment**
PCB134	13C PCB 155	Alumina	1,5	10	Compliant
PCB131/133/142	13C PCB 155	Alumina	4,5	5	Compliant
PCB165	13C PCB 155	Alumina	0,5	5	Compliant
PCB146	13C PCB 155	Alumina	0,1	5	Compliant
PCB132/161	13C PCB 155	Alumina	3,3	5	Compliant
PCB153	13C PCB 155	Alumina	2,8	10	Compliant
PCB168	13C PCB 155	Alumina	1,0	10	Compliant
PCB141	13C PCB 155	Alumina	1,6	5	Compliant
PCB137	13C PCB 155	Alumina	0,9	5	Compliant
PCB130	13C PCB 155	Alumina	1,4	5	Compliant
PCB164/163	13C PCB 155	Alumina	3,6	20	Compliant
PCB138	13C PCB 155	Alumina	2,8	20	Compliant
PCB158/160	13C PCB 155	Alumina	2,5	5	Compliant
PCB129	13C PCB 155	Alumina	1,4	20	Compliant
PCB166	13C PCB 155	Alumina	1,0	10	Compliant
PCB159	13C PCB 155	Alumina	1,5	5	Compliant
PCB128/162	13C PCB 155	Alumina	3,4	5	Compliant
PCB167	13C PCB 167	Alumina	0,9	5	Compliant
PCB156	13C PCB 156	Alumina	2,1	10	Compliant
PCB157	13C PCB 157	Alumina	1,8	10	Compliant
PCB169	13C PCB 169	Carbon	0,4	5	Compliant

7

PCB188	13C PCB 188	Alumina	2,0	5	Compliant
PCB184	13C PCB 188	Alumina	1,1	5	Compliant
PCB179	13C PCB 188	Alumina	2,3	5	Compliant
PCB176	13C PCB 188	Alumina	2,1	5	Compliant
PCB186	13C PCB 188	Alumina	0,8	5	Compliant
PCB178	13C PCB 188	Alumina	1,8	5	Compliant
PCB175	13C PCB 188	Alumina	1,7	5	Compliant
PCB182/187	13C PCB 188	Alumina	1,9	5	Compliant
PCB183	13C PCB 188	Alumina	1,9	10	Compliant
PCB185	13C PCB 188	Alumina	1,4	10	Compliant
PCB174	13C PCB 188	Alumina	2,1	5	Compliant
PCB181	13C PCB 188	Alumina	0,6	5	Compliant
PCB177	13C PCB 188	Alumina	1,9	5	Compliant
PCB171	13C PCB 188	Alumina	1,2	10	Compliant
PCB173	13C PCB 188	Alumina	0,9	10	Compliant
PCB172	13C PCB 188	Alumina	0,6	5	Compliant
PCB192	13C PCB 188	Alumina	0,3	5	Compliant
PCB180/193	13C PCB 188	Alumina	3,1	10	Compliant
PCB191	13C PCB 188	Alumina	1,1	5	Compliant
PCB170	13C PCB 188	Alumina	1,7	5	Compliant

Name	IS correction	Fraction final result	Final result pg/g	ML	Assessment**
PCB190	13C PCB 188	Alumina	1,1	5	Compliant
PCB189	13C PCB 189	Alumina	1,2	5	Compliant
<u>8</u>					
PCB202	13C PCB 202	Alumina	1,3	10	Compliant
PCB201	13C PCB 202	Alumina	0,5	5	Compliant
PCB204	13C PCB 202	Alumina	2,1	5	Compliant
PCB197	13C PCB 202	Alumina	1,1	10	Compliant
PCB200	13C PCB 202	Alumina	1,9	10	Compliant
PCB198/199	13C PCB 202	Alumina	2,9	10	Compliant
PCB196/203	13C PCB 202	Alumina	1,5	5	Compliant
PCB195	13C PCB 202	Alumina	1,2	5	Compliant
PCB194	13C PCB 202	Alumina	1,5	5	Compliant
PCB205	13C PCB 205	Alumina	0,5	5	Compliant
<u>9</u>					
PCB208	13C PCB 208	Alumina	2,4	5	Compliant
PCB207	13C PCB 208	Alumina	0,8	5	Compliant
PCB206	13C PCB 206	Alumina	1,1	5	Compliant
<u>10</u>					
PCB209	13C PCB 209	Alumina	2,4	5	Compliant

* Total constitutional isomer results (Cl1, Cl2, etc) are based on integration of the full chromatographic window and are not the sum of individual congeners.

** Assessment according EP 1668C; Blank < 2x ML

*** Assessment according EP 1668C; Blank < 1/3 x Regulatory level

Annex 5a. Results and recovery in blank* samples for Dioxins and Furans (EPA 1613B)

* Blank is referred to as not spiked with native PCBs

	Blank	Ash	Sediment (dried)	Soil	Fish oil	Dust	Sewage sludge (dried)
Sample intake (gram)	1	1,25	10	5	1	1,25	5
Extract volume (ml)	0,1	0,1	0,1	0,1	0,1	0,1	0,1
Dioxins	pg/g	pg/g	pg/g	pg/g	pg/g	pg/g	pg/g
2378-TCDD	<0,30	<0,24	0,05	<0,40	<0,30	<0,80	<0,14
12378-PeCDD	<0,70	<2,40	<0,09	0,5	0,68	<0,64	<0,40
123478-HxCDD	<0,30	2	<0,05	0,36	<0,40	0,18	<0,20
123678-HxCDD	<0,30	6,56	<0,04	1,5	0,45	0,88	1,28
123789-HxCDD	<0,40	4,24	<0,05	0,78	<0,50	0,46	0,58
1234678-HpCDD	<5,00	28,8	0,69	46	<2,00	12	36
OCDD	<1,00	21,6	6,4	300	<1,00	70,4	360
Furans	pg/g	pg/g	pg/g	pg/g	pg/g	pg/g	pg/g
2378-TCDF	<0,40	0,68	0,12	1,82	4,2	2,24	2
12378-PeCDF	<0,60	<0,64	<0,10	<0,60	0,8	<2,40	<0,60
23478-PeCDF	<0,60	0,74	0,11	0,98	3,2	<2,40	0,98
123478-HxCDF	<0,30	0,42	0,35	0,98	<0,30	1,12	0,88
123678-HxCDF	<0,30	1,12	0,24	2,6	0,34	1,44	1,22
234678-HxCDF	<0,30	0,74	0,07	1,06	0,4	1,04	0,68
123789-HxCDF	<0,40	<0,40	<0,07	<0,40	<0,50	<0,40	<0,20
1234678-HpCDF	<4,00	<3,20	1,1	12,2	<4,00	5,92	8,8
1234789-HpCDF	<9,00	<5,60	<0,50	<1,00	<7,00	<4,00	<1,00
OCDF	<10,00	<7,20	7,2	14	<9,00	8,8	15,6
Internal standards 13C	%	%	%	%	%	%	%
13C-2378-TCDD	79	86	91	91	86	86	92
13C-12378-PeCDD	75	91	94	92	84	76	85
13C-123478-HxCDD	84	94	96	89	81	92	81
13C-123678-HxCDD	81	85	103	99	72	82	89
13C-123789-HxCDD	77	82	91	91	79	87	88
13C-1234678-HpCDD	76	83	91	93	78	89	87
13C-OCDD	69	83	94	97	80	101	92
13C-2378-TCDF	80	89	97	94	86	89	94
13C-12378-PeCDF	85	93	97	92	81	90	89
13C-23478-PeCDF	86	99	99	90	84	91	89
13C-123478-HxCDF	81	87	92	94	77	82	76
13C-123678-HxCDF	68	89	95	83	83	84	83
13C-234678-HxCDF	78	87	91	91	79	85	83
13C-123789-HxCDF	79	90	103	99	79	100	97
13C-1234678-HpCDF	74	83	86	91	76	86	82
13C-1234789-HpCDF	78	94	112	122	82	124	110
13C-OCDF	67	85	116	131	90	148	135
linjection standard							
13C-1234-TCDD	95	94	88	86	90	79	86

Annex 5b. Results and recovery in blank* samples for PCBs (EPA 1668C)

A figure of the congener profiles is given below the table.

* Blank is referred to as not spiked with native PCBs

Sample		Blank	Ash	Sediment (dried)	Soil	Fish oil	Dust	Sewage sludge (dried)
Sample intake (g)		1	1,25	10	5	1	1,25	5
Volume extract (ml)		0,1	0,1	0,1	0,1	0,1	0,1	0,1
Name	IS correction	Fraction						
209PCB								
Cl1*	13C PCB 1	Alumina	13	7	1	4	1	3
Cl2*	13C PCB 4	Alumina	59	42	13	238	24	313
Cl3*	13C PCB 19	Alumina	105	89	119	402	329	152
Cl4*	13C PCB 54	Alumina	97	84	273	1011	1091	357
Cl5*	13C PCB 104	Alumina	97	72	341	1500	2973	515
Cl6*	13C PCB 155	Alumina	73	57	396	1957	3667	694
Cl7*	13C PCB 188	Alumina	39	32	178	917	1024	306
Cl8*	13C PCB 202	Alumina	21	15	30	131	108	51
Cl9*	13C PCB 208	Alumina	6	6	3	13	11	4
Cl10*	13C PCB 209	Alumina	2	6	14	12	9	3
LOC WINDOW DEFINING 13C								
13C PCB 1	13C PCB 9	Alumina	103	106	103	106	100	104
13C PCB 3	13C PCB 9	Carbon	50	67	53	32	58	37
13C PCB 4	13C PCB 9	Alumina	114	112	116	117	111	119
13C PCB 15	13C PCB 9	Carbon	92	103	100	85	94	93
13C PCB 19	13C PCB 9	Alumina	114	112	114	114	111	119
13C PCB 37	13C PCB 9	Carbon	103	114	119	112	111	103
13C PCB 54	13C PCB 52	Alumina	119	117	116	124	119	121
13C PCB 81	13C PCB 52	Carbon	109	117	126	116	120	105
13C PCB 77	13C PCB 52	Carbon	104	124	128	137	120	116
13C PCB 104	13C PCB 101	Alumina	108	109	116	105	97	118
13C PCB 123	13C PCB 101	Alumina	111	111	113	108	116	129
13C PCB 118	13C PCB 101	Alumina	94	97	103	100	105	111
13C PCB 114	13C PCB 101	Alumina	106	114	111	105	116	114
13C PCB 105	13C PCB 101	Alumina	103	109	105	105	111	107
13C PCB 126	13C PCB 101	Carbon	114	129	132	131	127	124
13C PCB 155	13C PCB 138	Alumina	116	116	123	128	113	129
13C PCB 167	13C PCB 138	Alumina	103	105	103	108	105	111
13C PCB 156	13C PCB 138	Alumina	100	105	108	111	110	107
13C PCB 157	13C PCB 138	Alumina	100	105	105	108	108	114
13C PCB 169	13C PCB 138	Carbon	102	122	115	119	114	117
								100

Sample			Blank	Ash	Sediment (dried)	Soil	Fish oil	Dust	Sewage sludge (dried)
Sample intake (g)			1	1,25	10	5	1	1,25	5
Volume extract (ml)			0,1	0,1	0,1	0,1	0,1	0,1	0,1
Name	IS correction	Fraction							
13C PCB 188	13C PCB 138	Alumina	111	111	115	117	105	129	109
13C PCB 189	13C PCB 138	Alumina	103	103	110	114	115	121	109
13C PCB 202	13C PCB 194	Alumina	116	111	116	117	113	106	111
13C PCB 205	13C PCB 194	Alumina	111	105	118	111	110	112	109
13C PCB 208	13C PCB 194	Alumina	114	108	113	111	108	103	97
13C PCB 206	13C PCB 194	Alumina	111	95	108	108	103	115	103
13C PCB 209	13C PCB 194	Alumina	97	95	100	78	92	100	24
CLEAN-UP 13C									
13C PCB 28	13C PCB 9	Alumina	111	112	114	117	105	122	128
13C PCB 111	13C PCB 101	Alumina	106	106	105	103	105	114	112
13C PCB 178	13C PCB 138	Alumina	108	108	113	114	108	118	109
INJECTION 13C									
13C PCB 9		Carbon and Alumina	72	72	72	68	72	60	68
13C PCB 52		Carbon and Alumina	80	76	76	68	76	68	76
13C PCB 101		Carbon and Alumina	80	80	76	72	80	68	76
13C PCB 138		Carbon and Alumina	92	84	88	84	88	76	84
13C PCB 194		Carbon and Alumina	88	92	92	84	88	76	80

* Total constitutional isomer results (Cl1, Cl2, etc) are based on integration of the full chromatographic window and are not the sum of individual congeners. The result represents the sum of amounts found in the carbon fraction and alumina fraction.

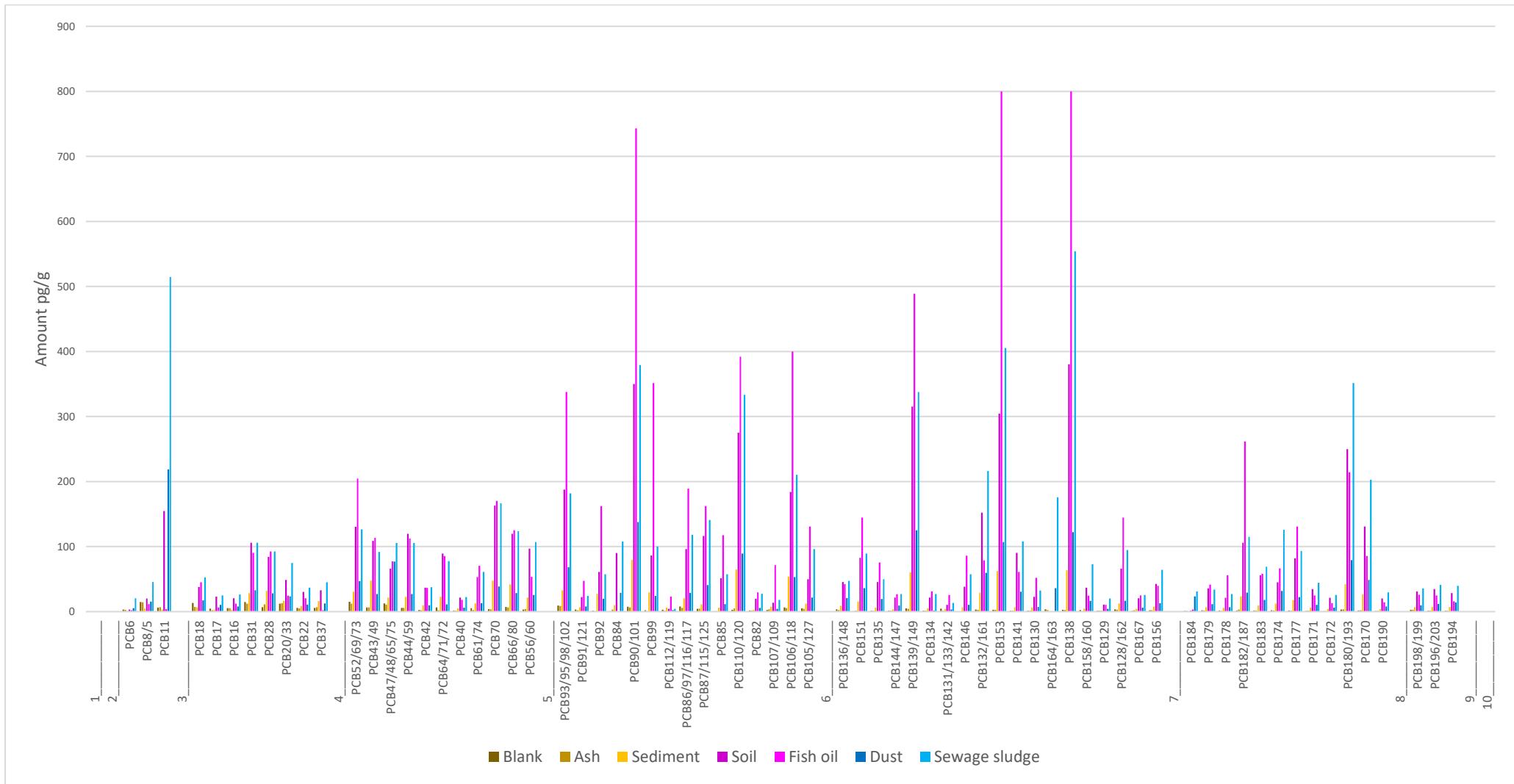
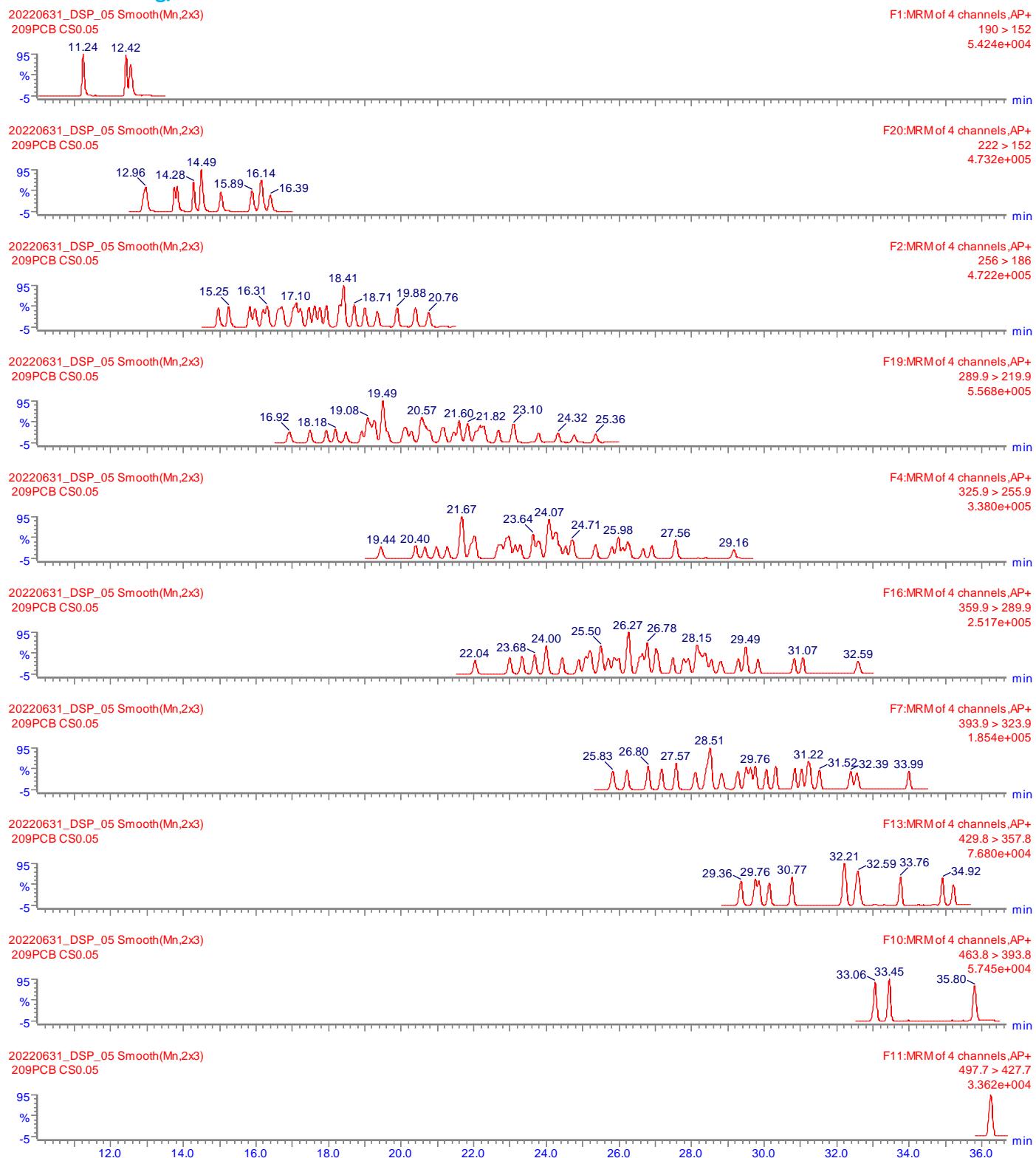


Figure annex 5b. Amounts of PCBs determined in blank samples with a cut off value for the congener of 20 pg/g on the maximum amount found in a sample

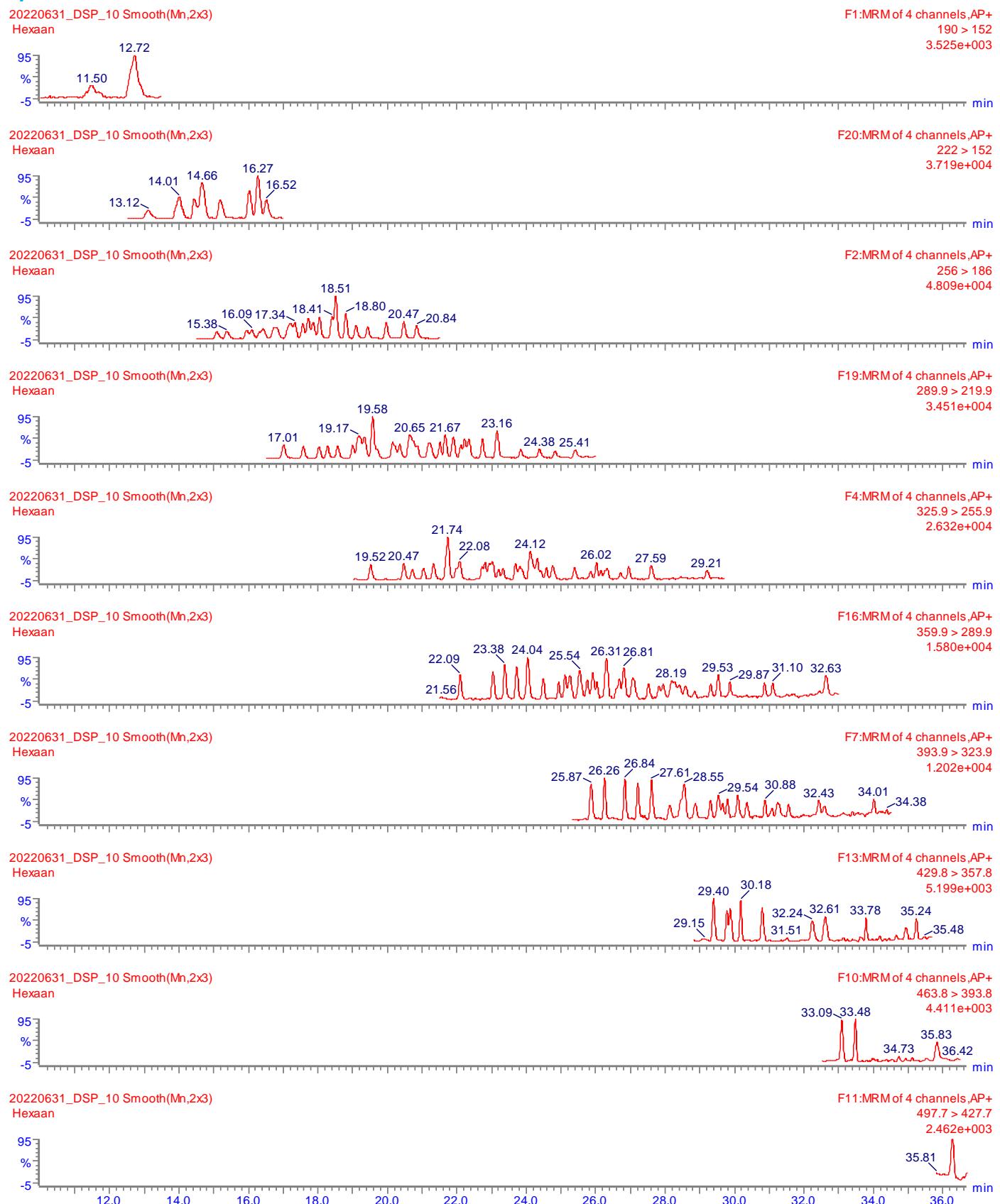
Annex 6. Chromatograms of the analysis of PCBs in blank* samples

* Blank is referred to as not spiked with native PCBs. The detection system was contaminated due to the high levels in the standards and spiked samples. The blank analysis didn't show a representative view of the blank analysis, but was sufficiently low to determine IPR and levels in samples.

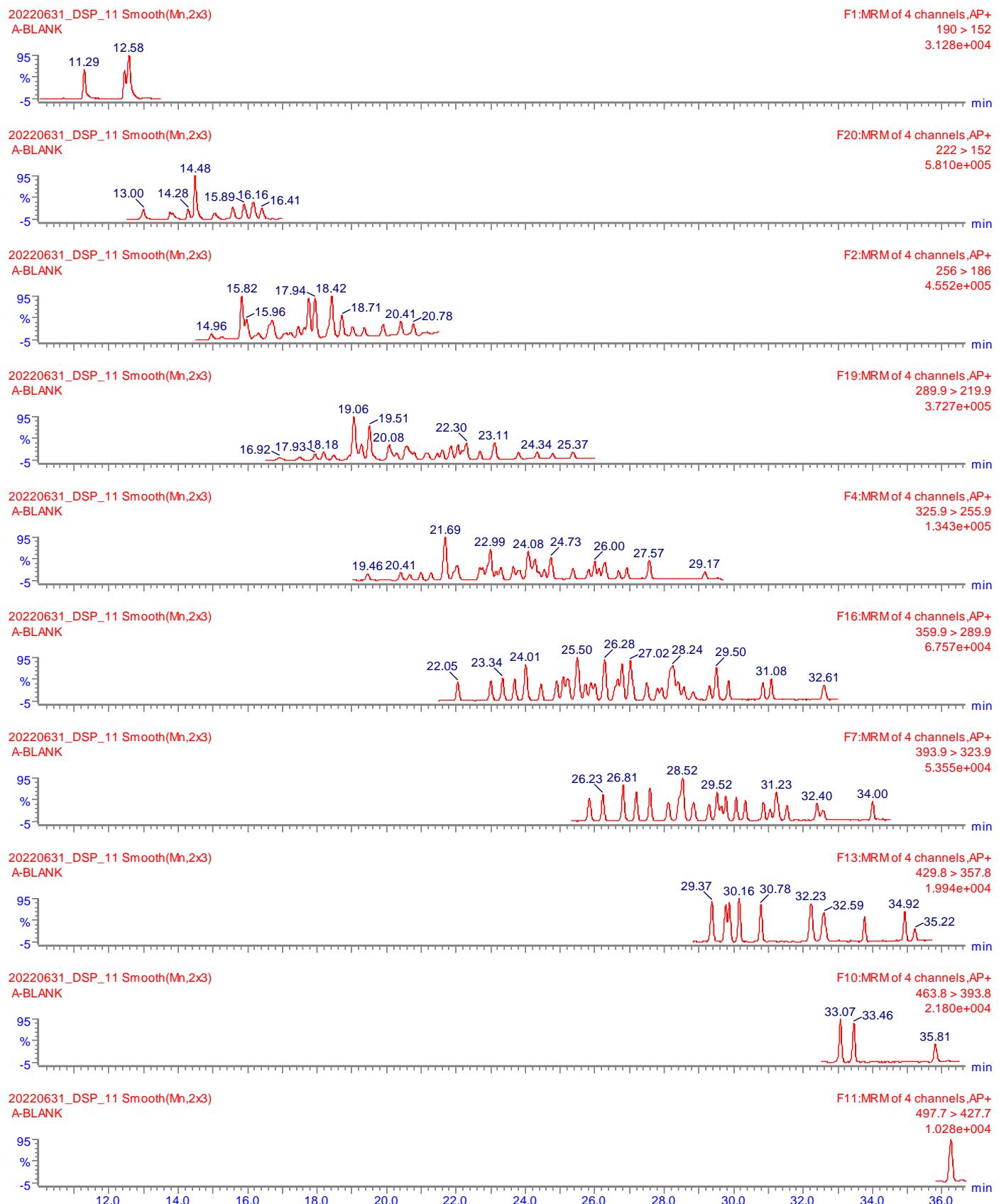
Standard 0.05ng/ml



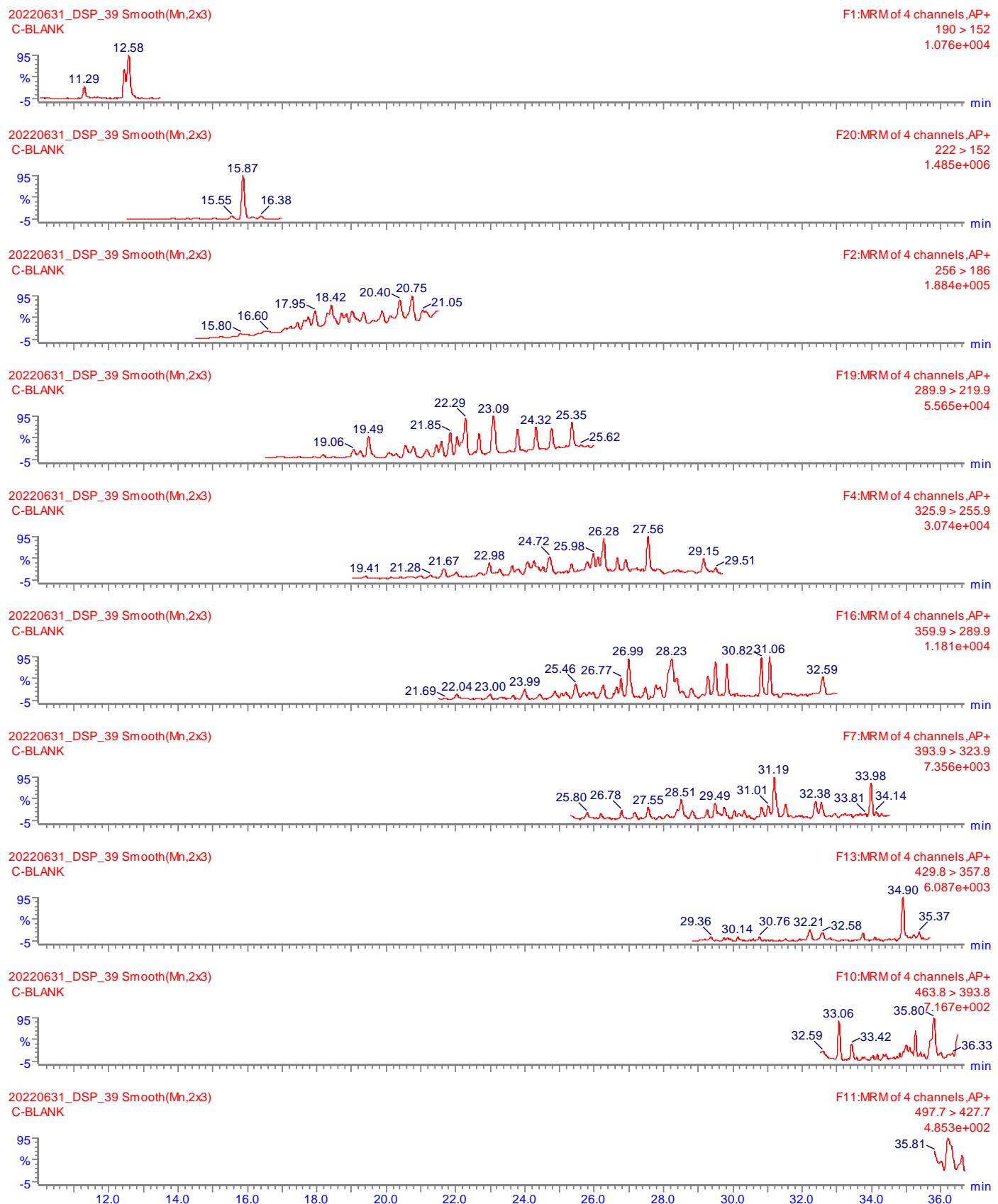
System blank – APGC-MS - Hexane



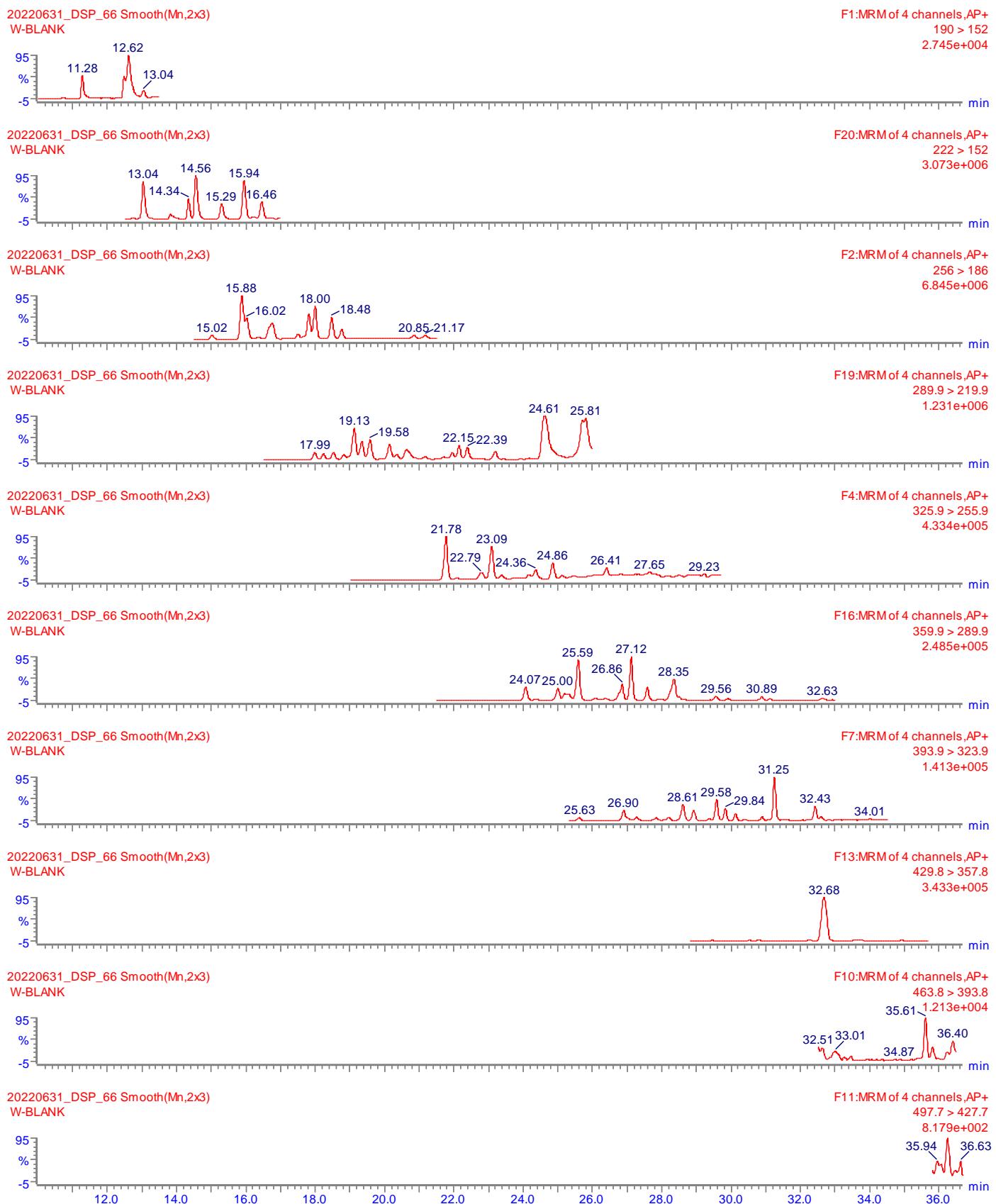
Blank - Alumina



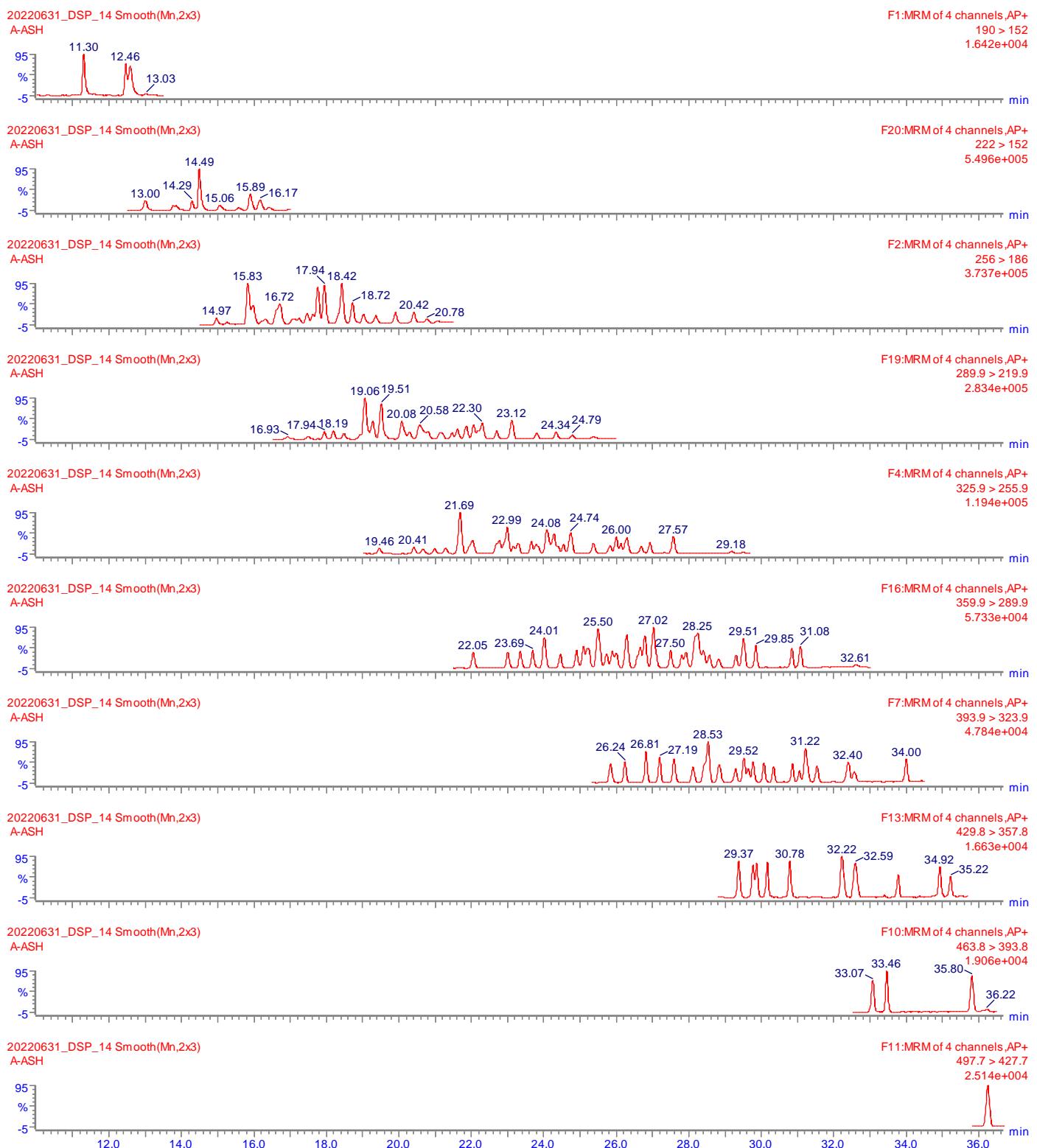
Blank - Carbon



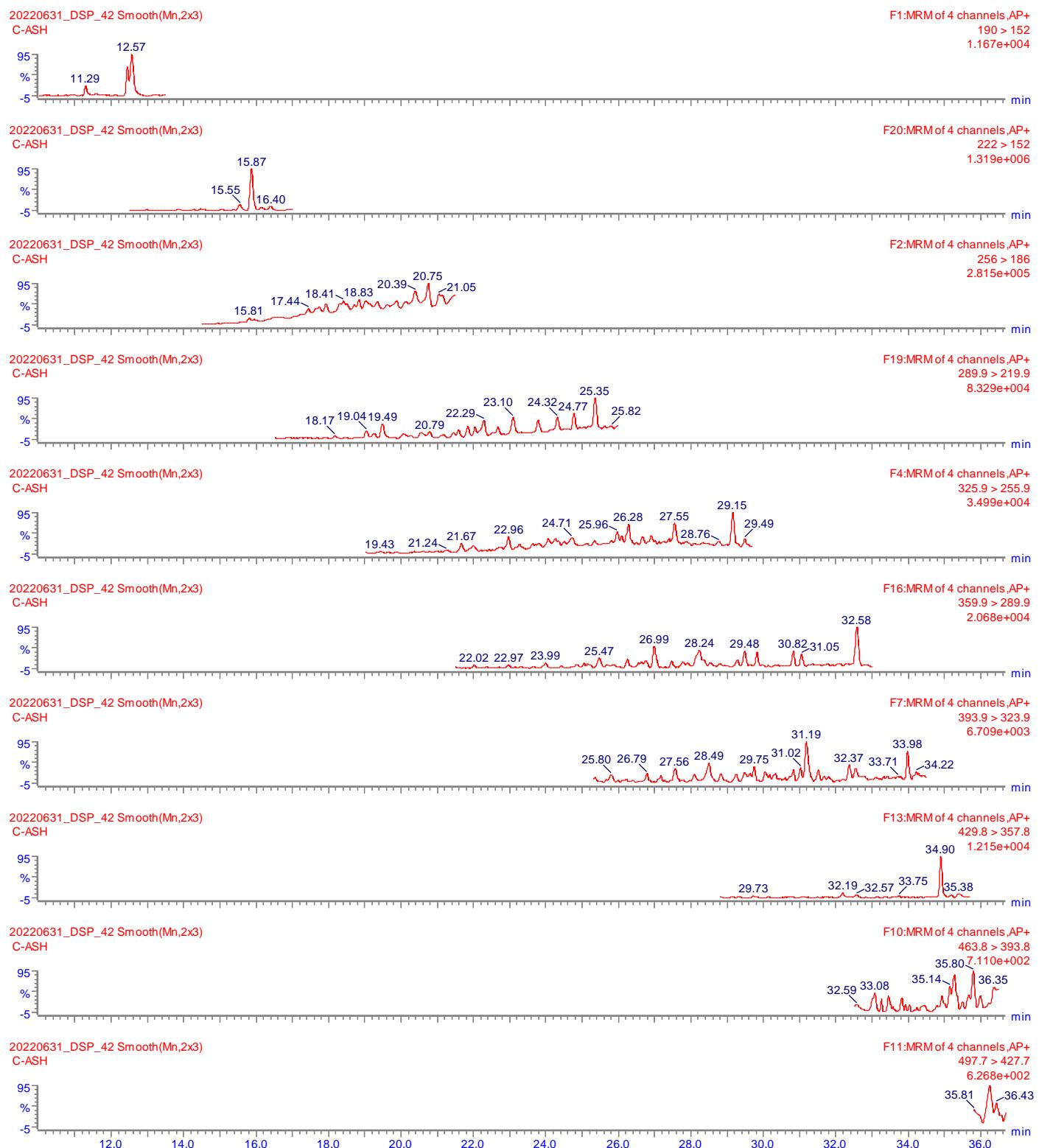
Blank – Waste



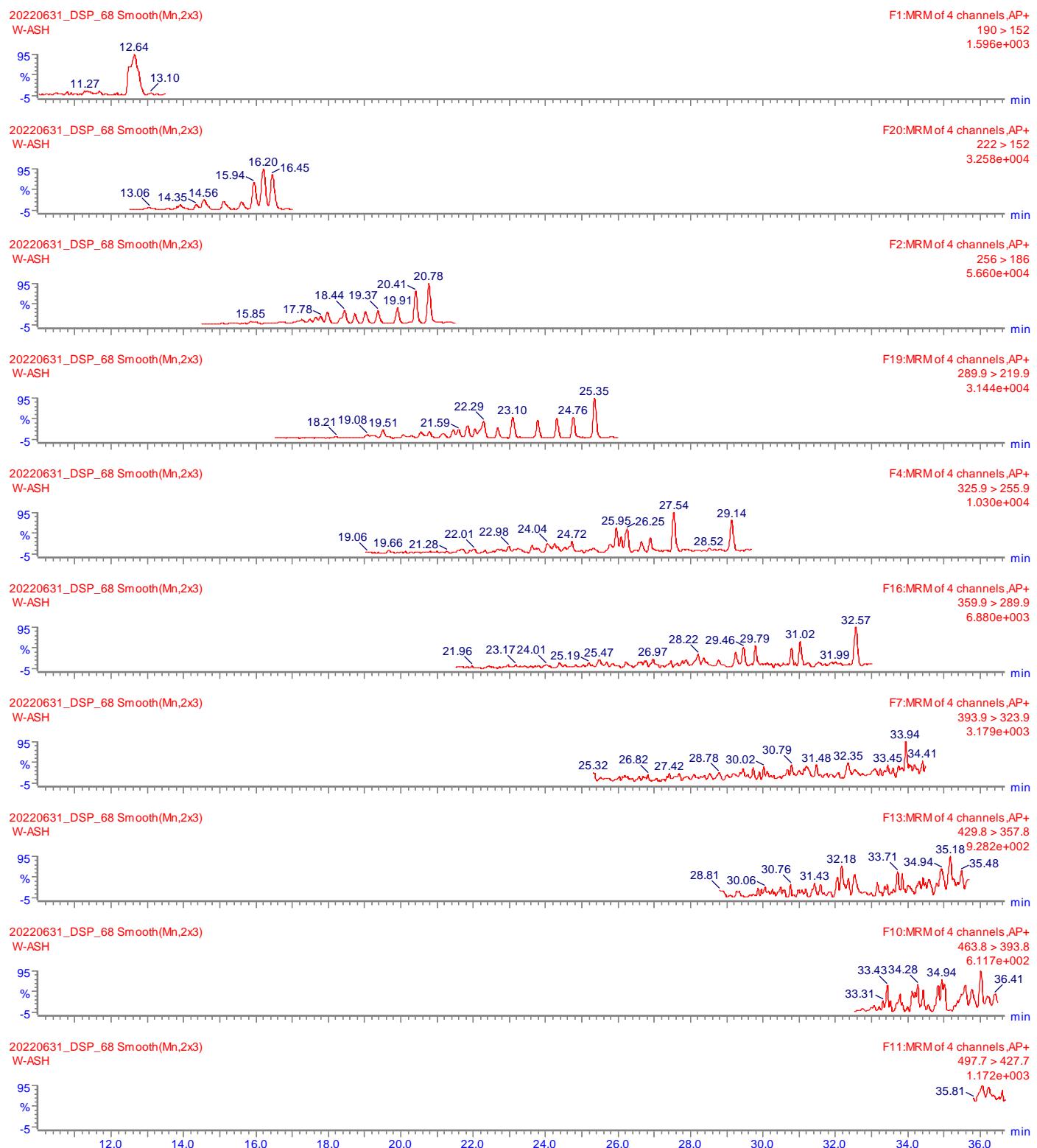
Ash - Alumina



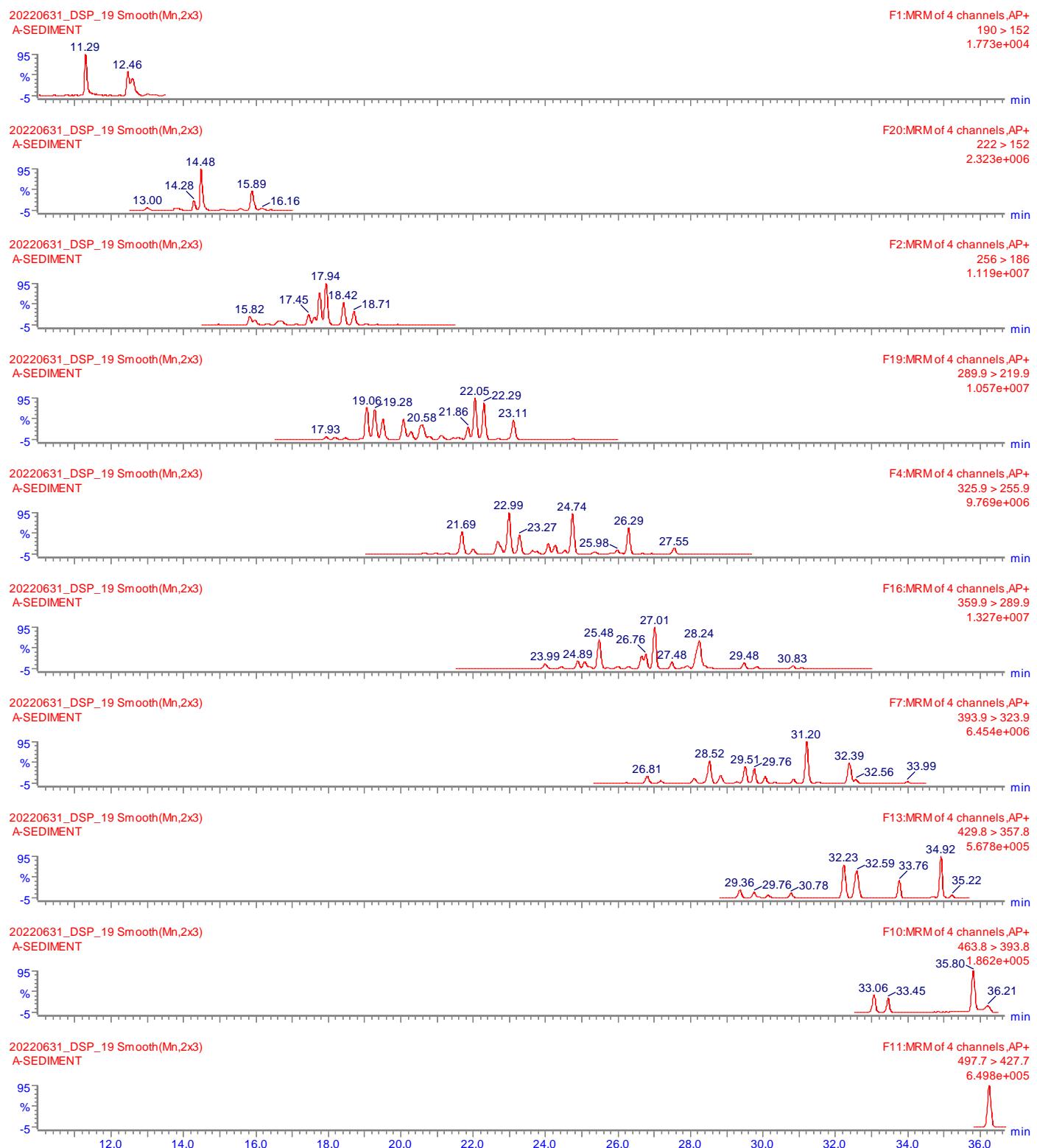
Ash - Carbon



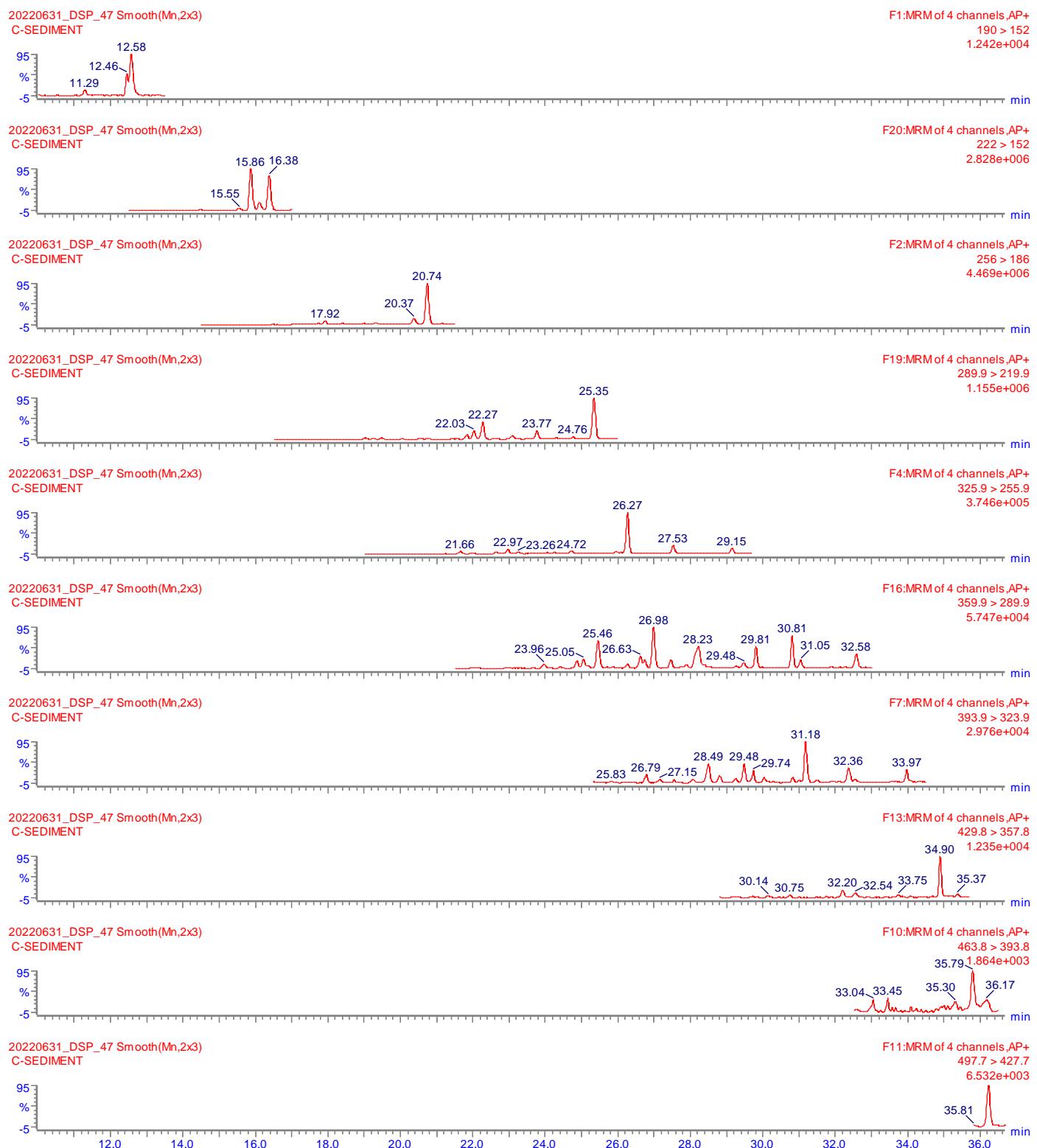
Ash – Waste



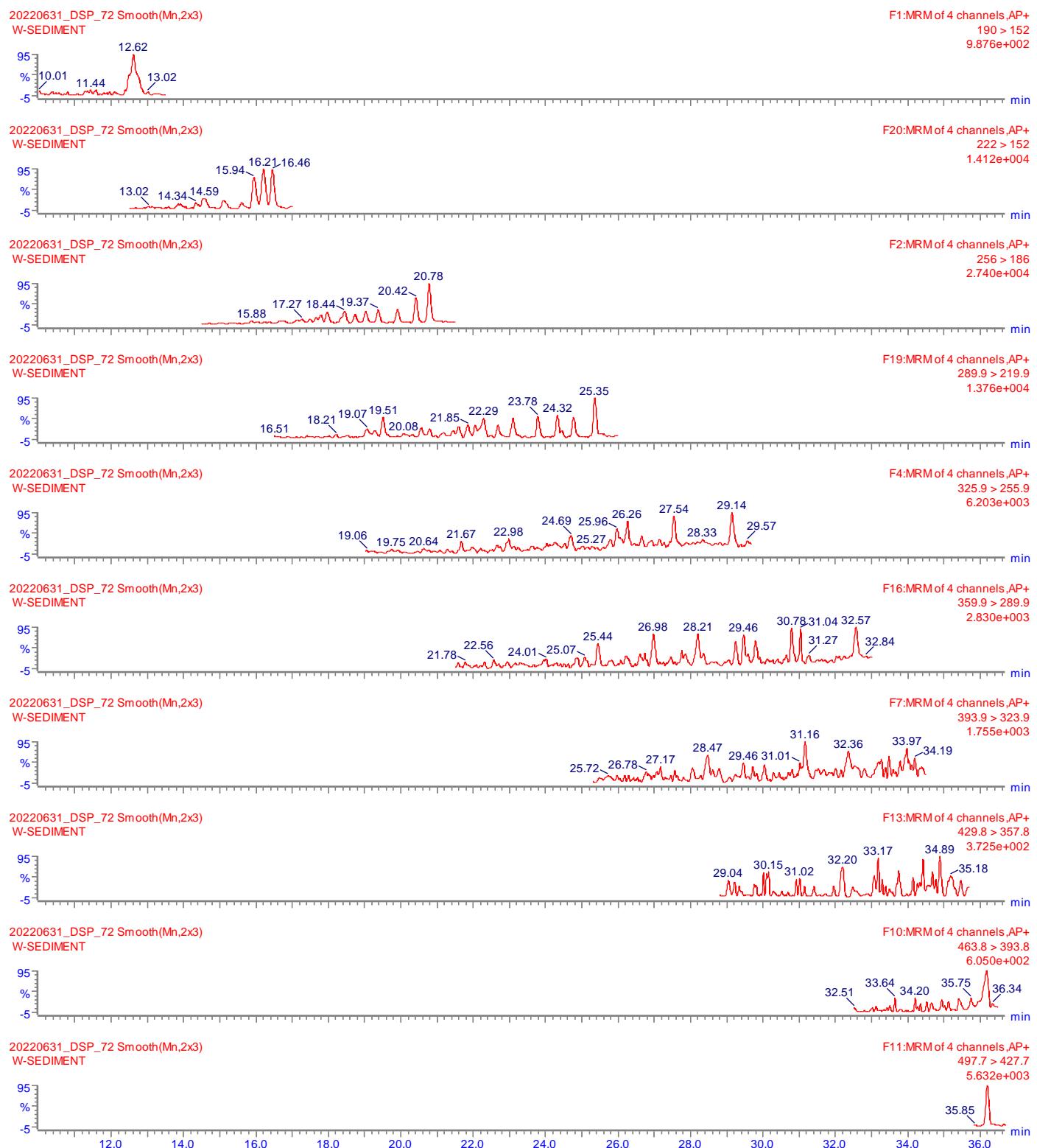
Sediment - Alumina



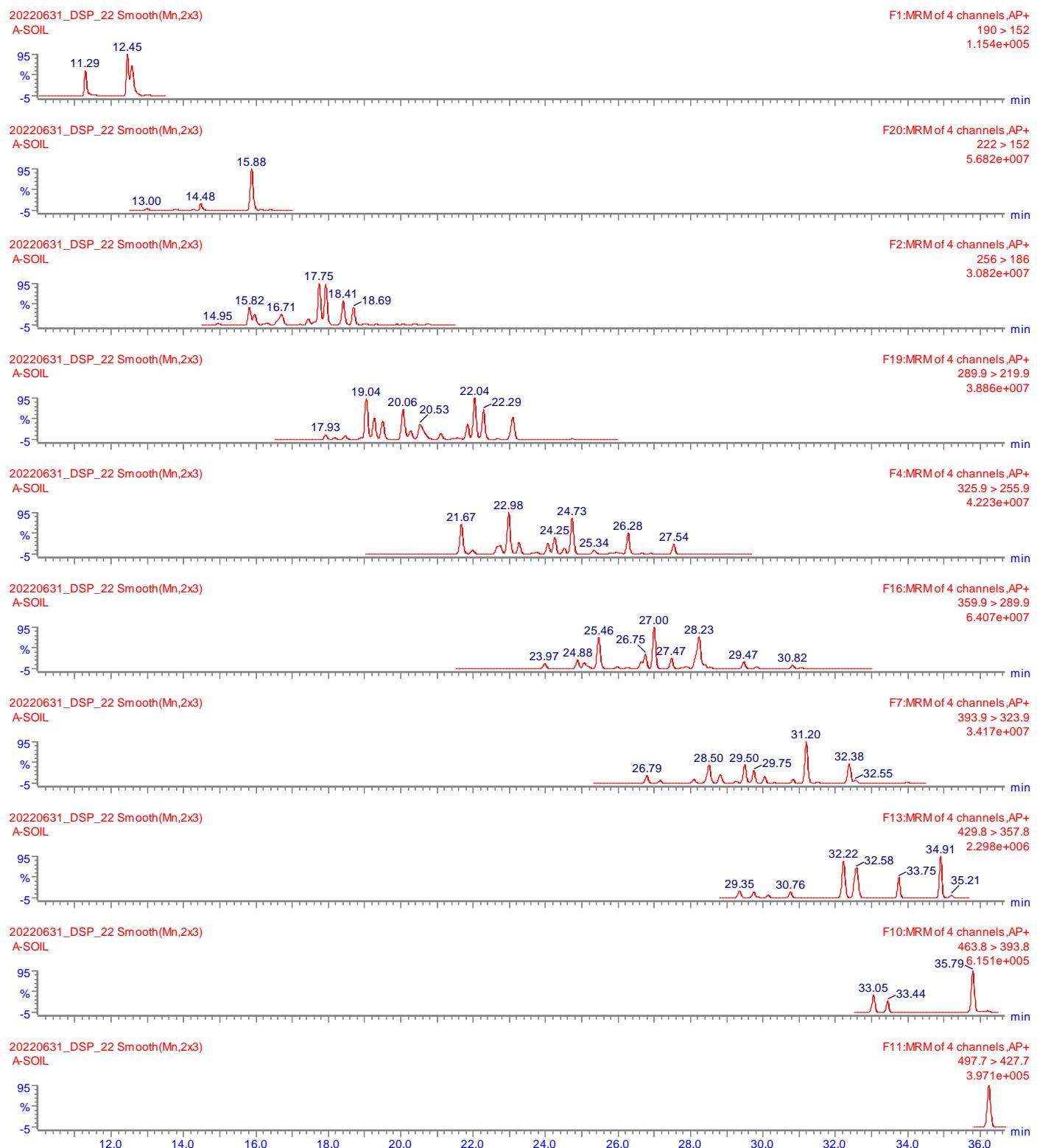
Sediment - Carbon



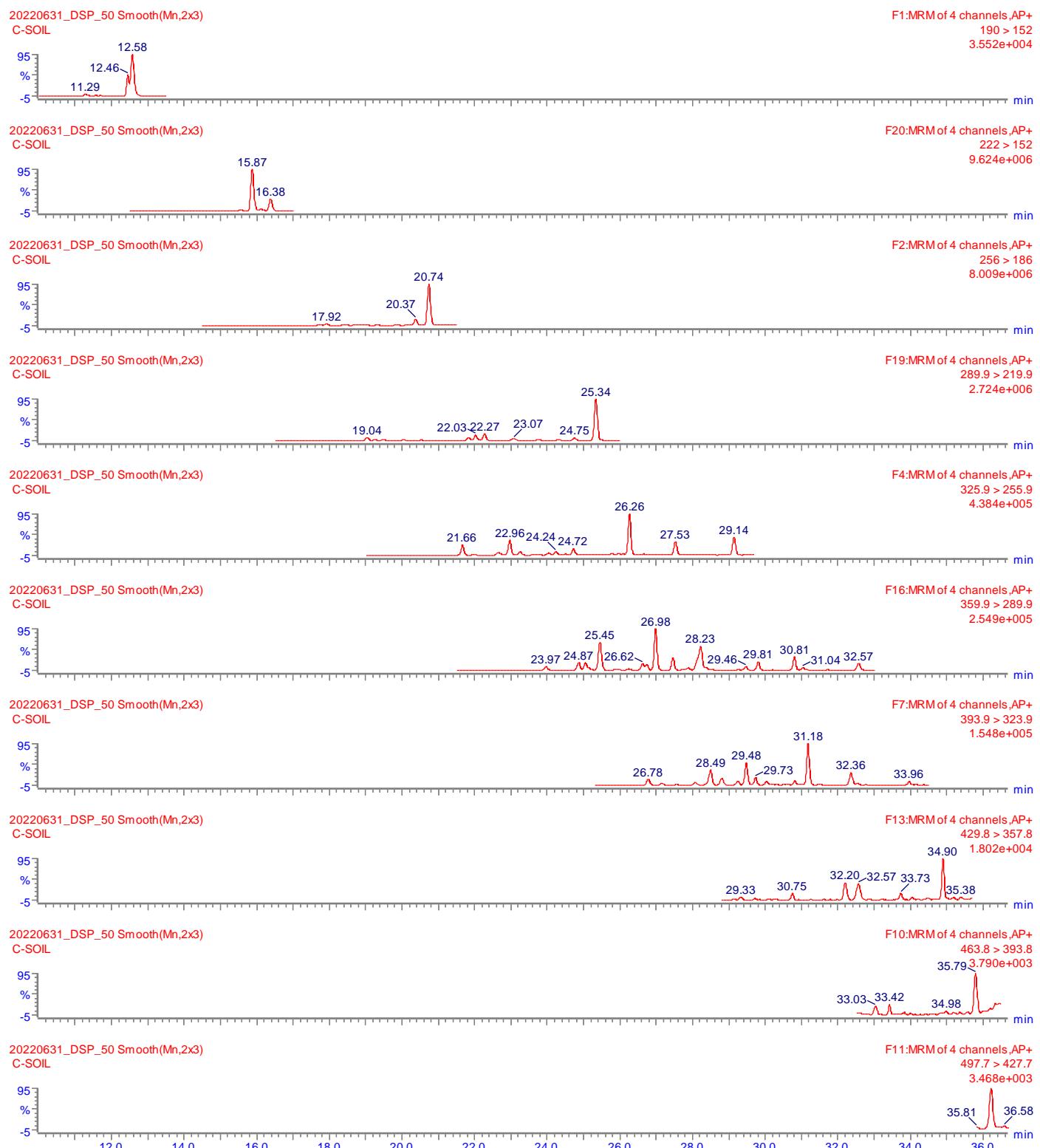
Sediment - Waste



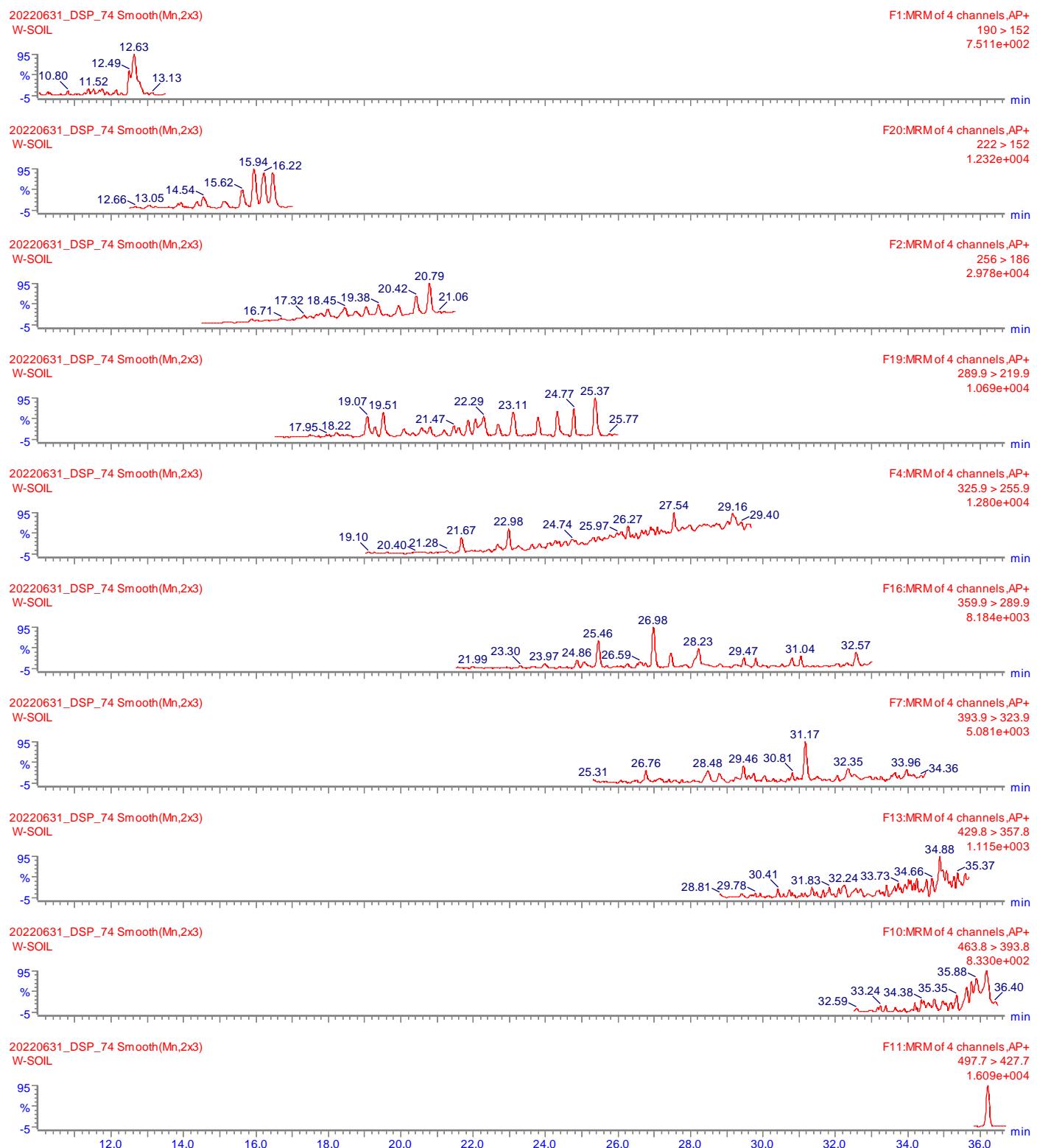
Soil - Alumina



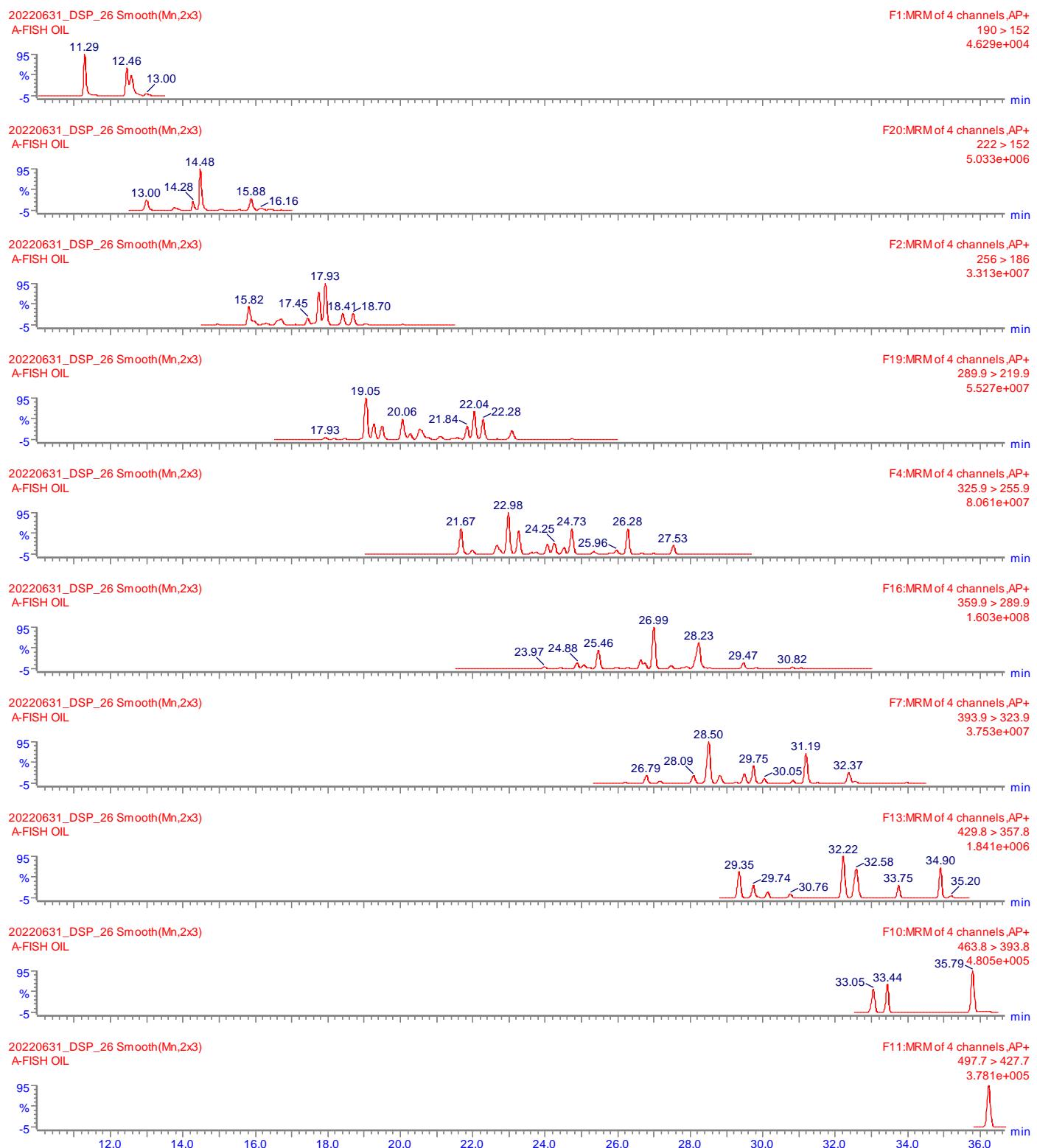
Soil - Carbon



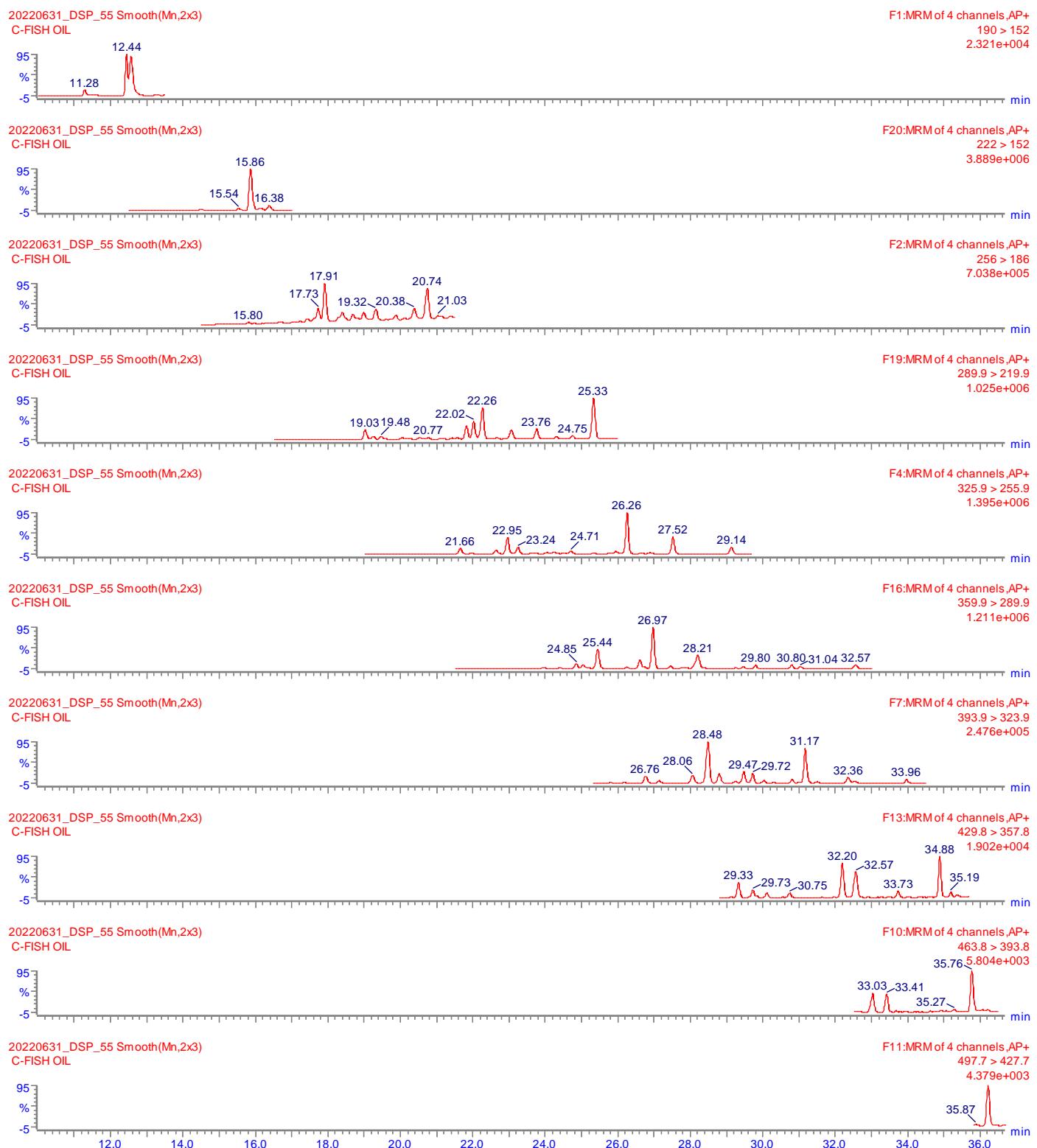
Soil - Waste



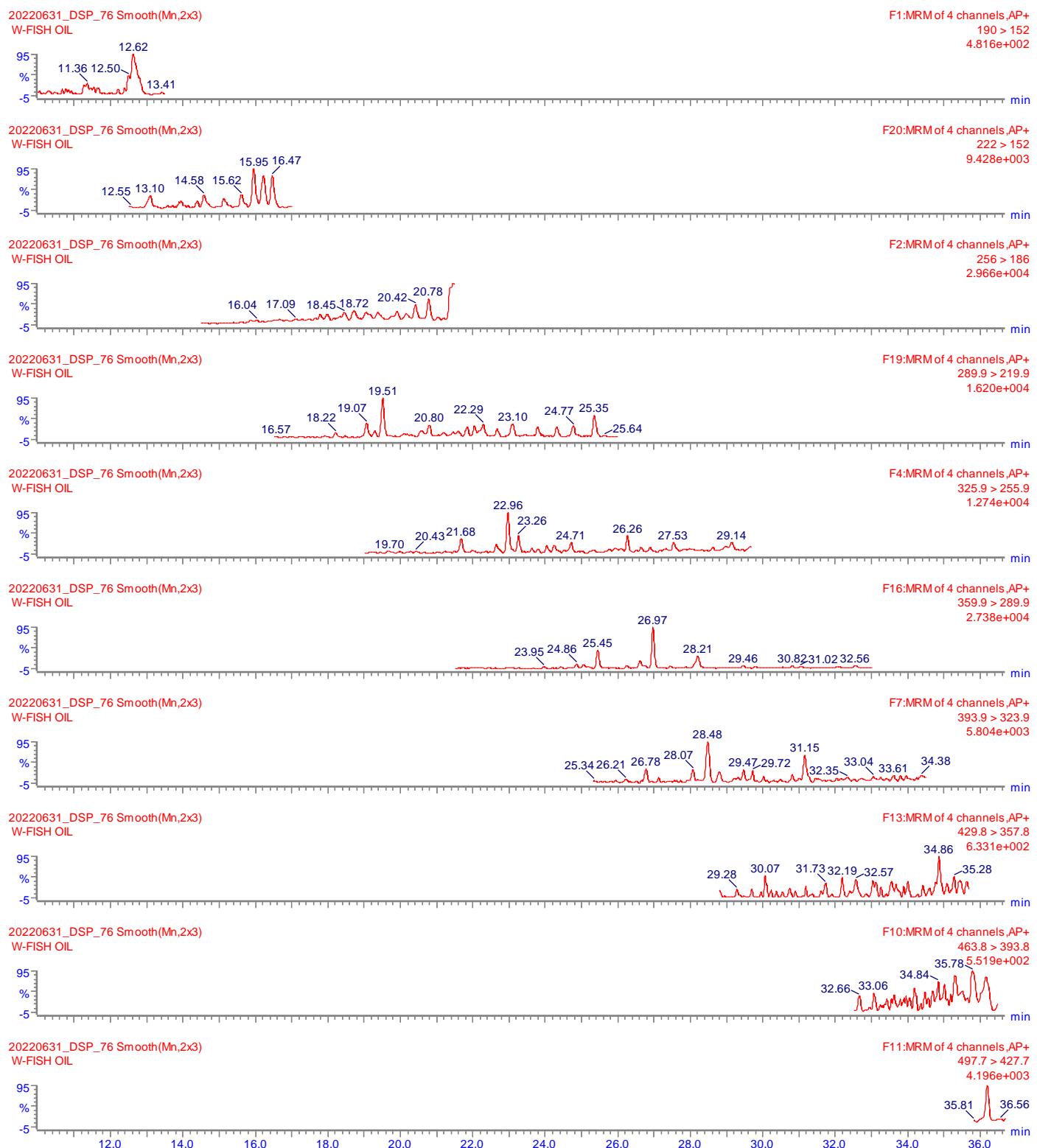
Fish oil - Alumina



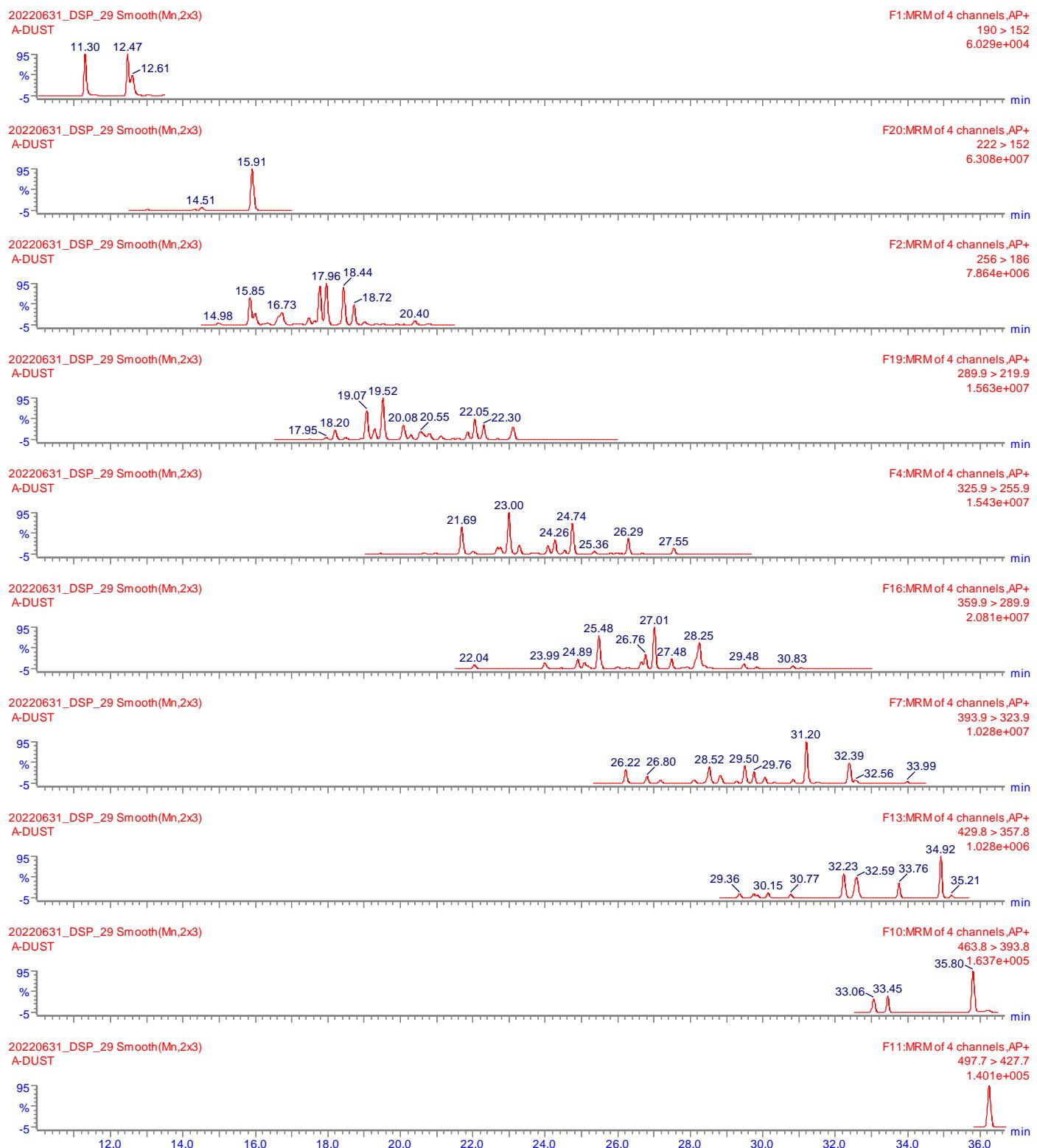
Fish oil – Carbon-



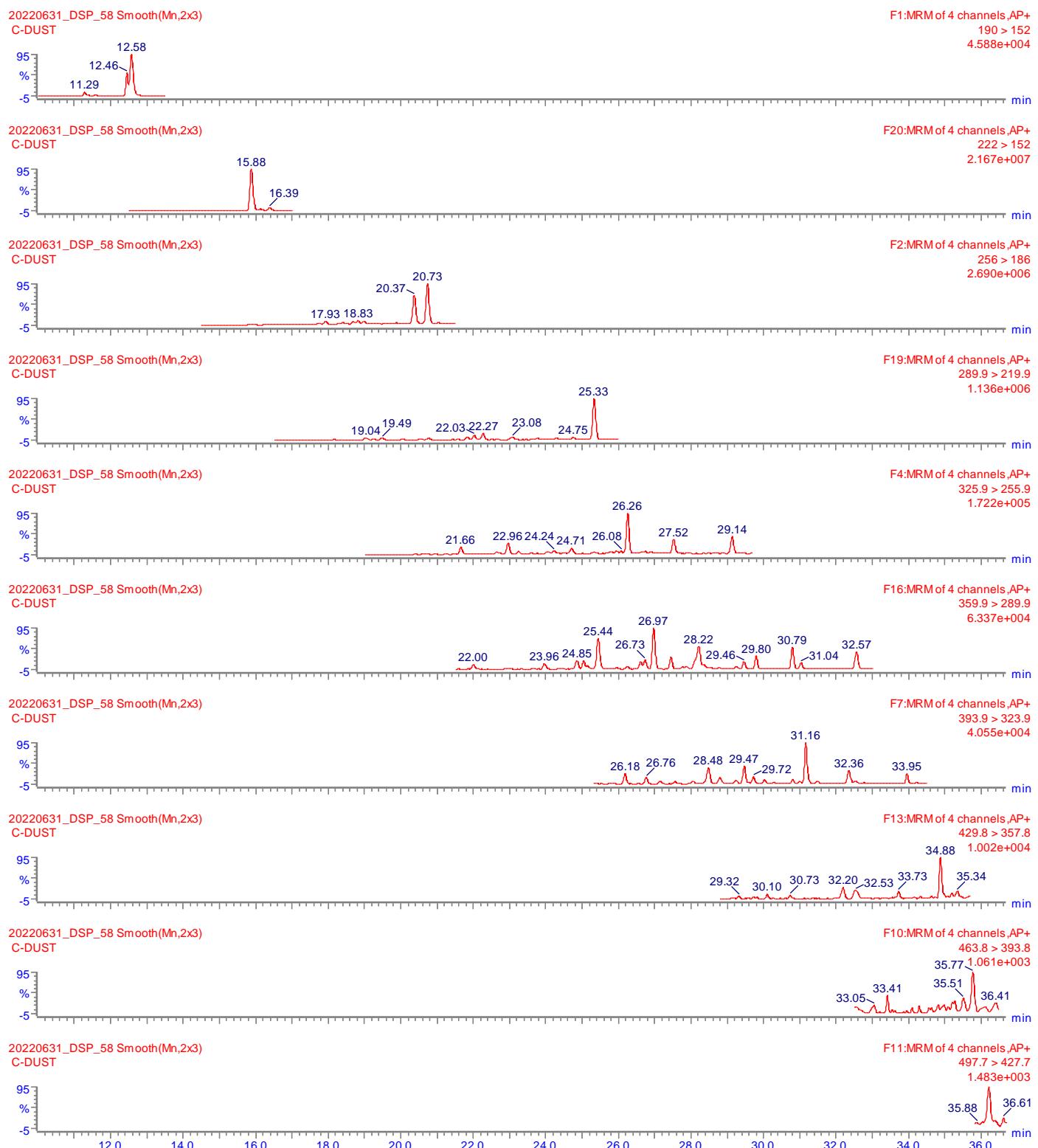
Fish oil - Waste



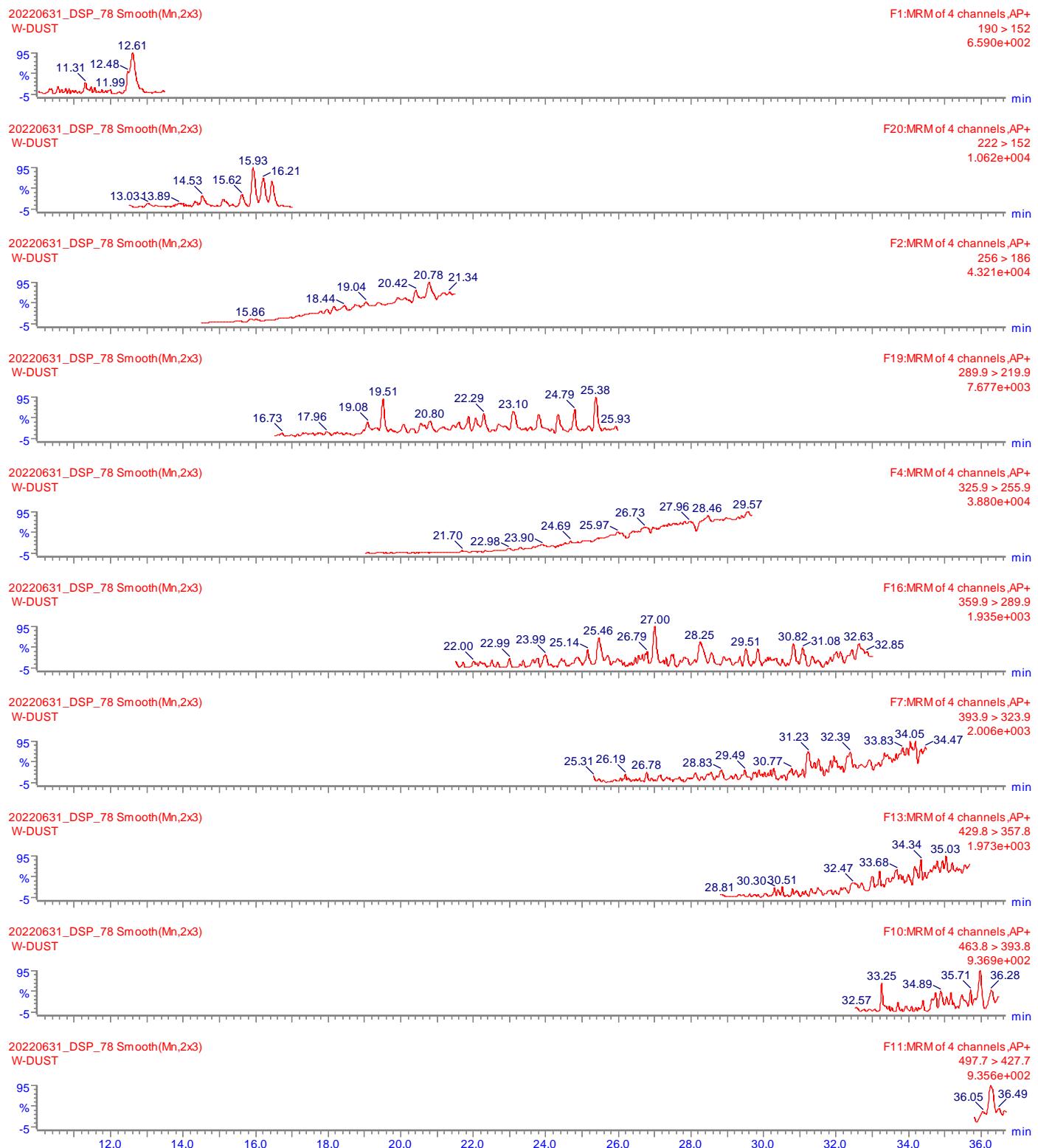
Dust - Alumina



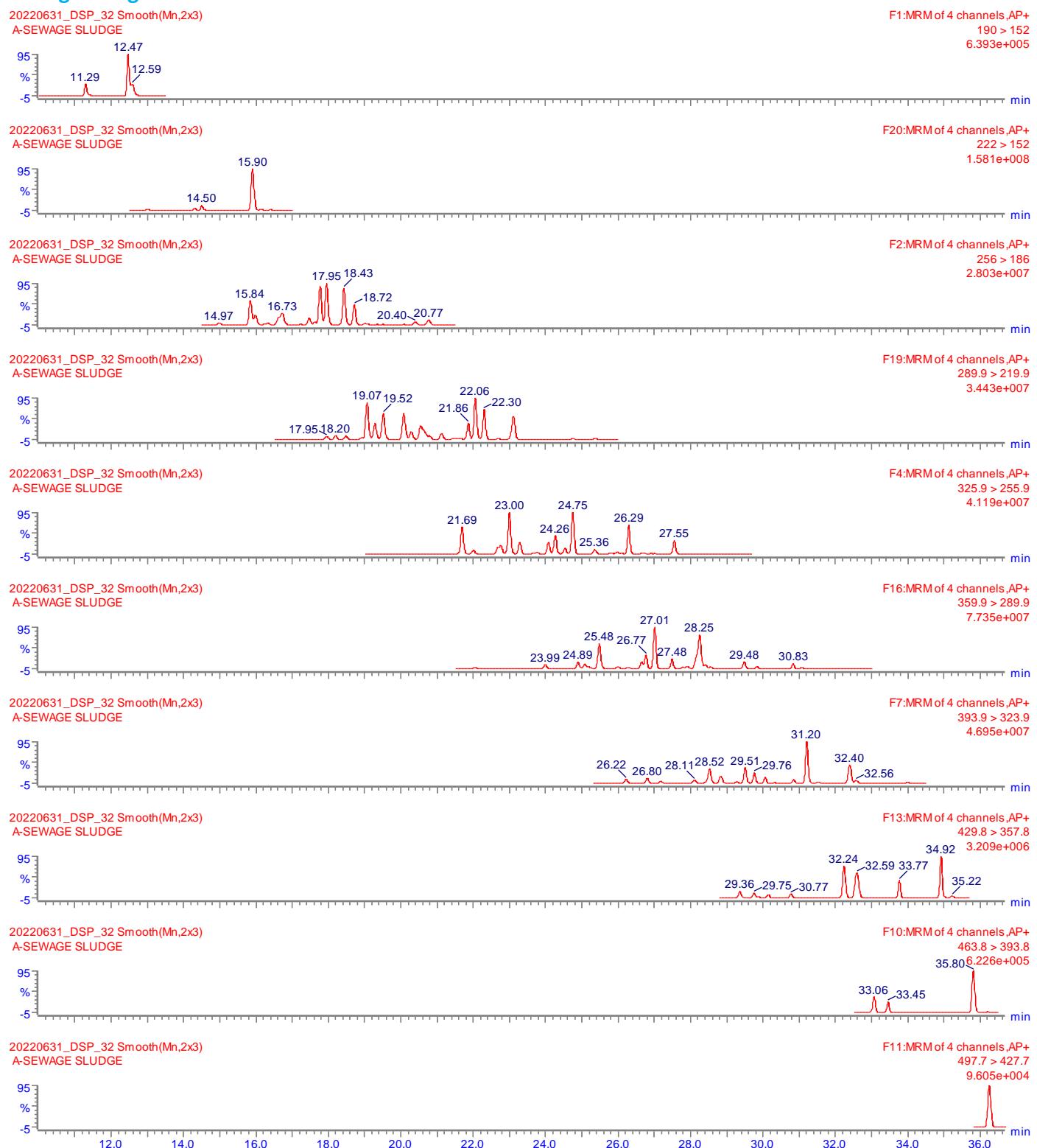
Dust - Carbon



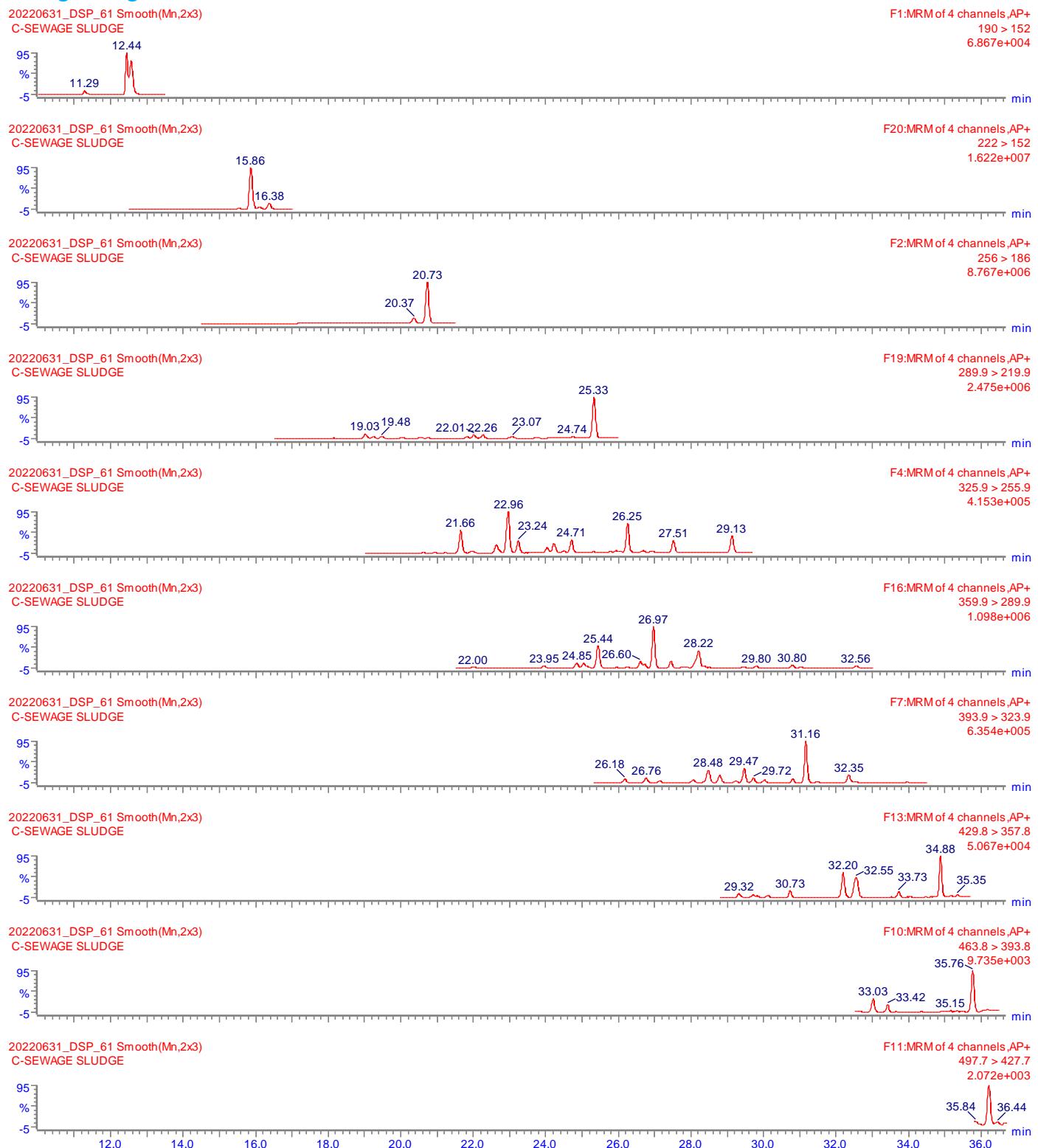
Dust – Waste



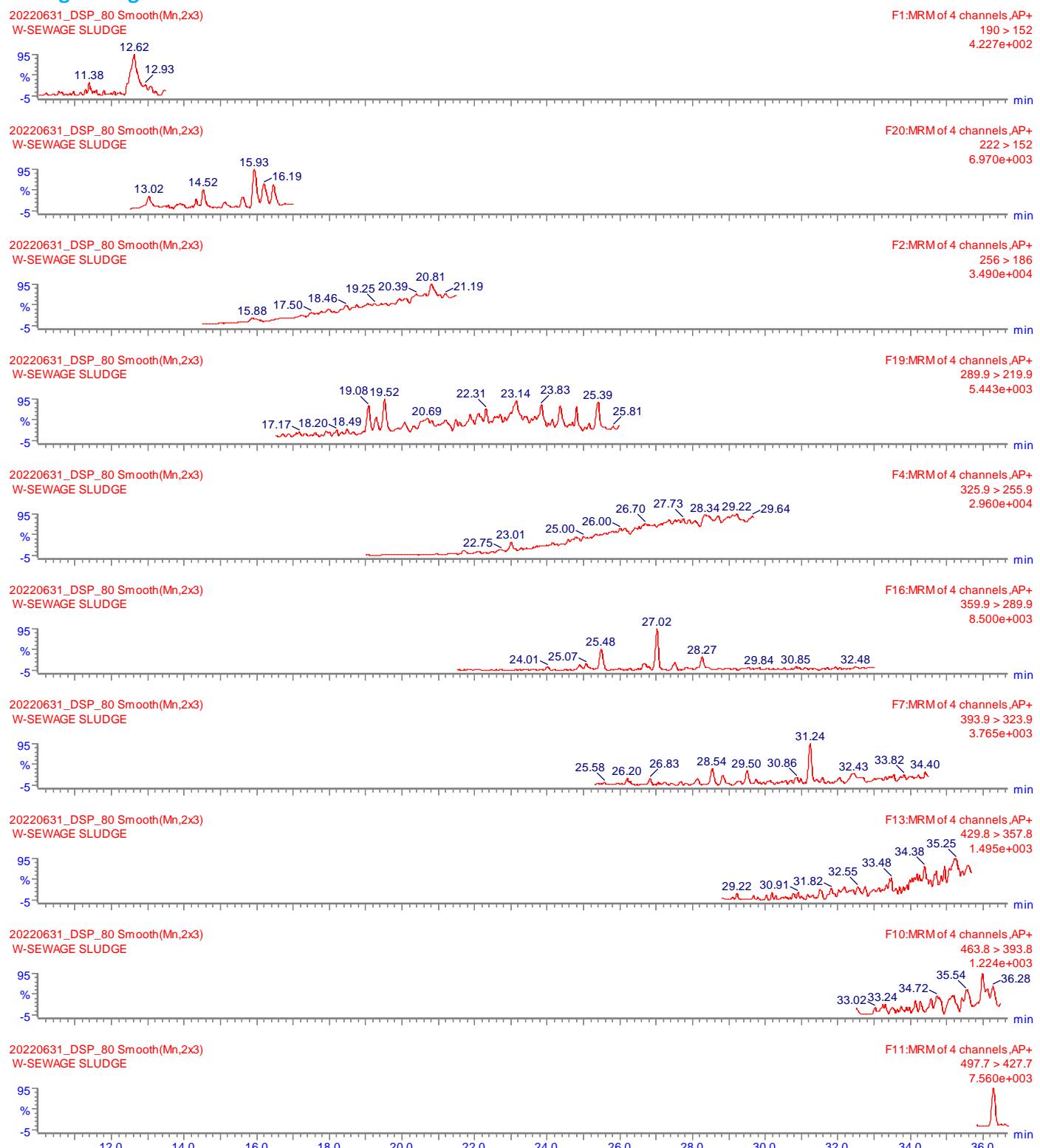
Sewage sludge - Alumina



Sewage sludge - Carbon



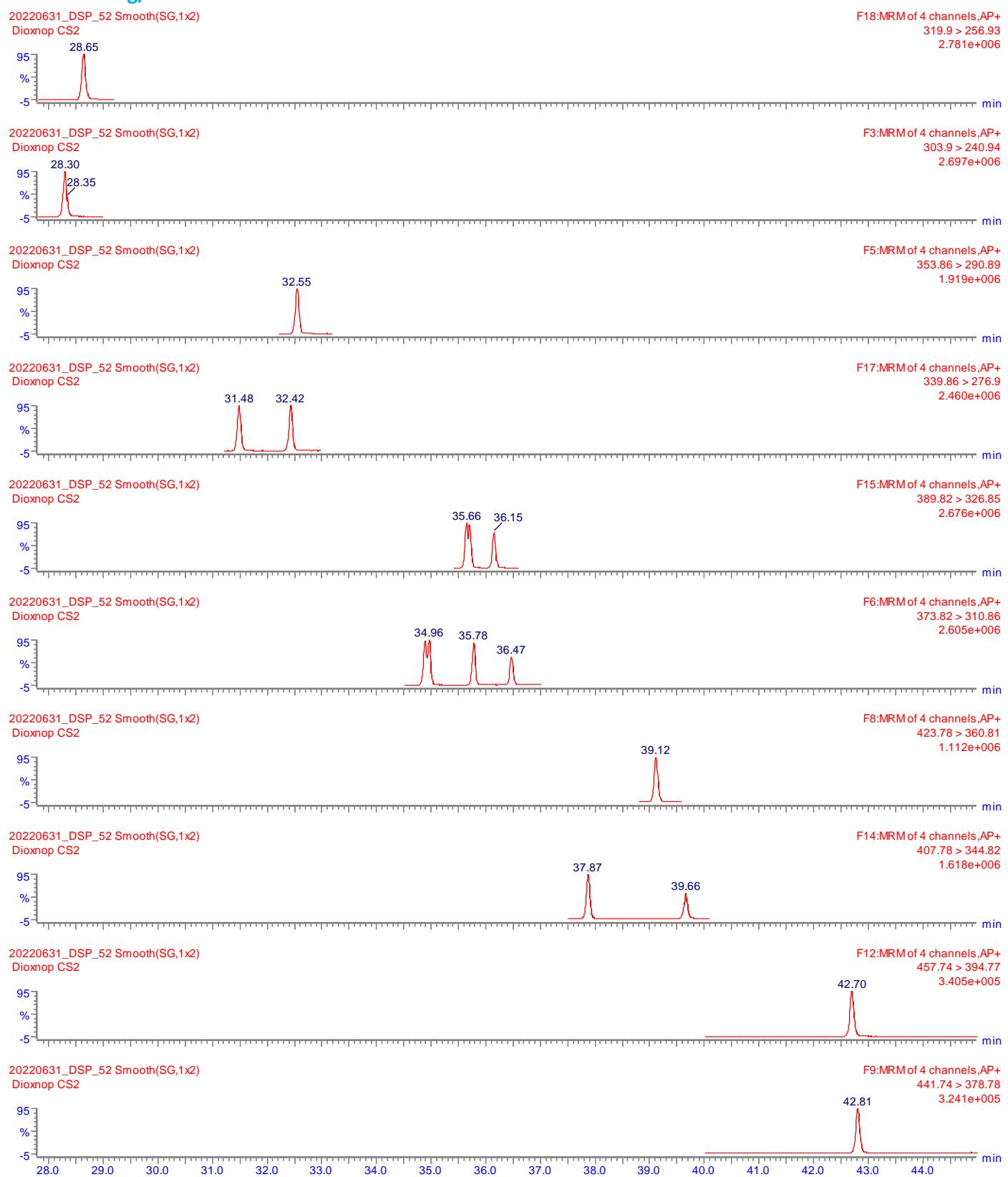
Sewage sludge - Waste



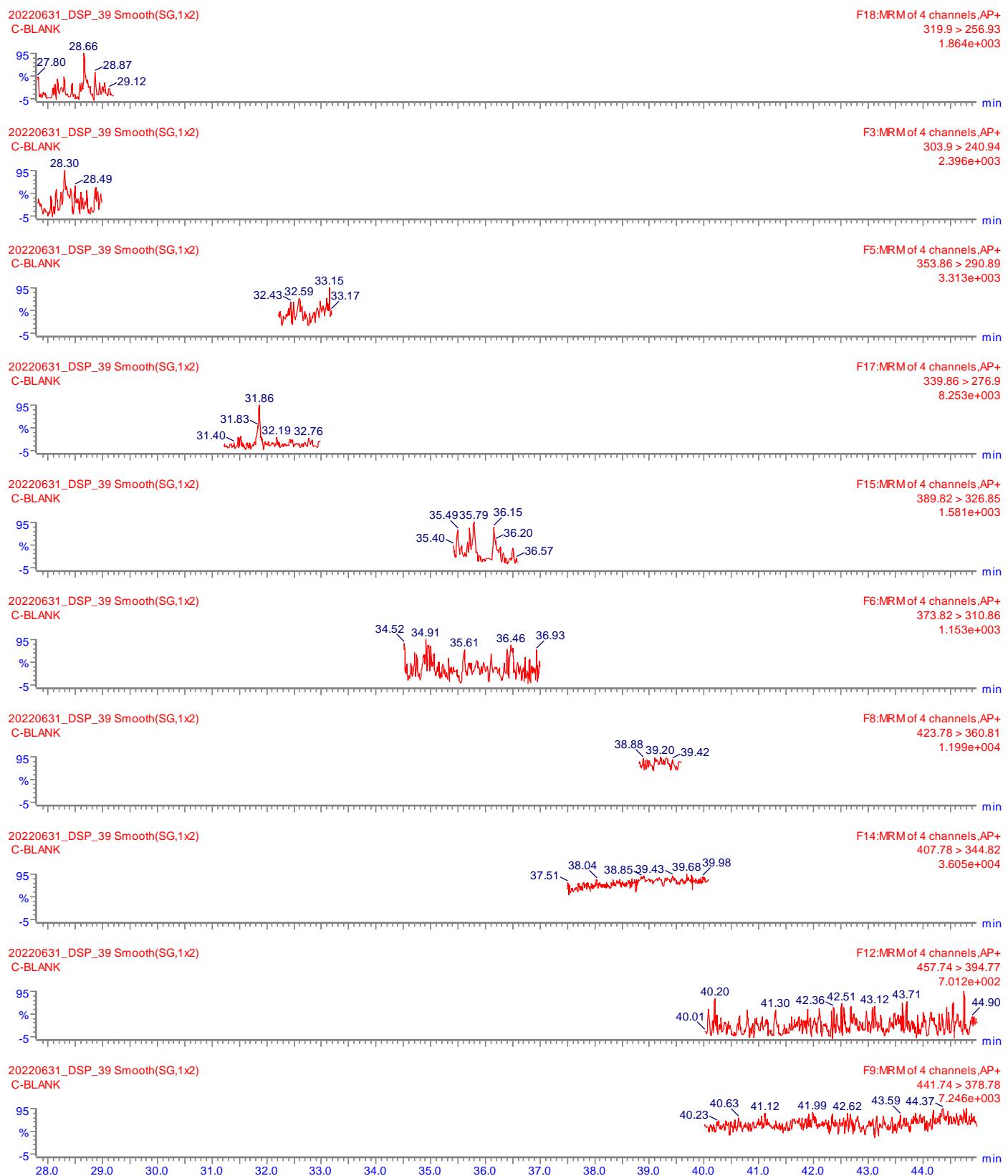
Annex 7. Chromatograms of blank* samples for Dioxins and Furans (EPA 1613B)

* Blank is referred to as not spiked with native PCBs

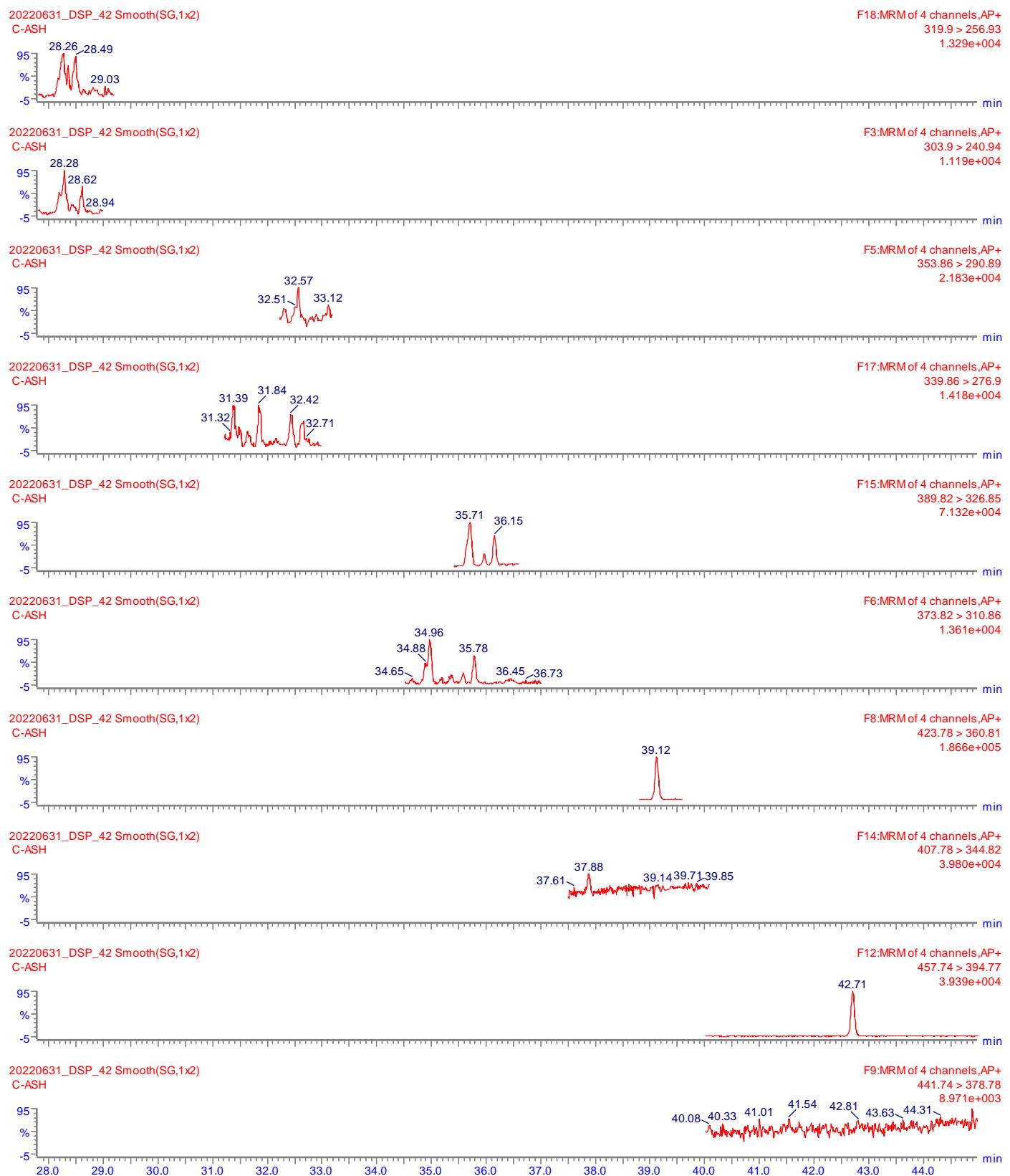
Standard 2 ng/ml



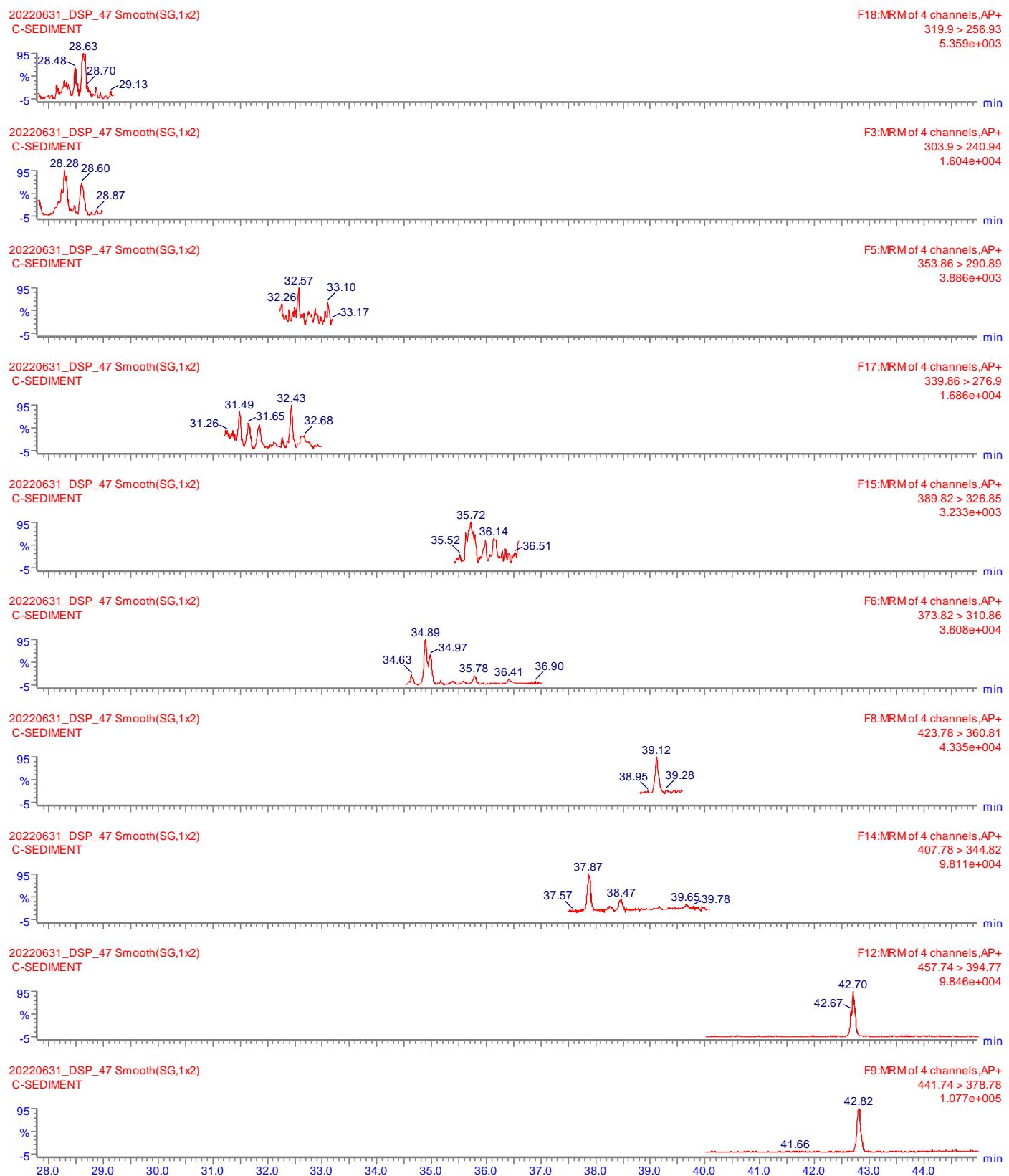
Blank - Carbon



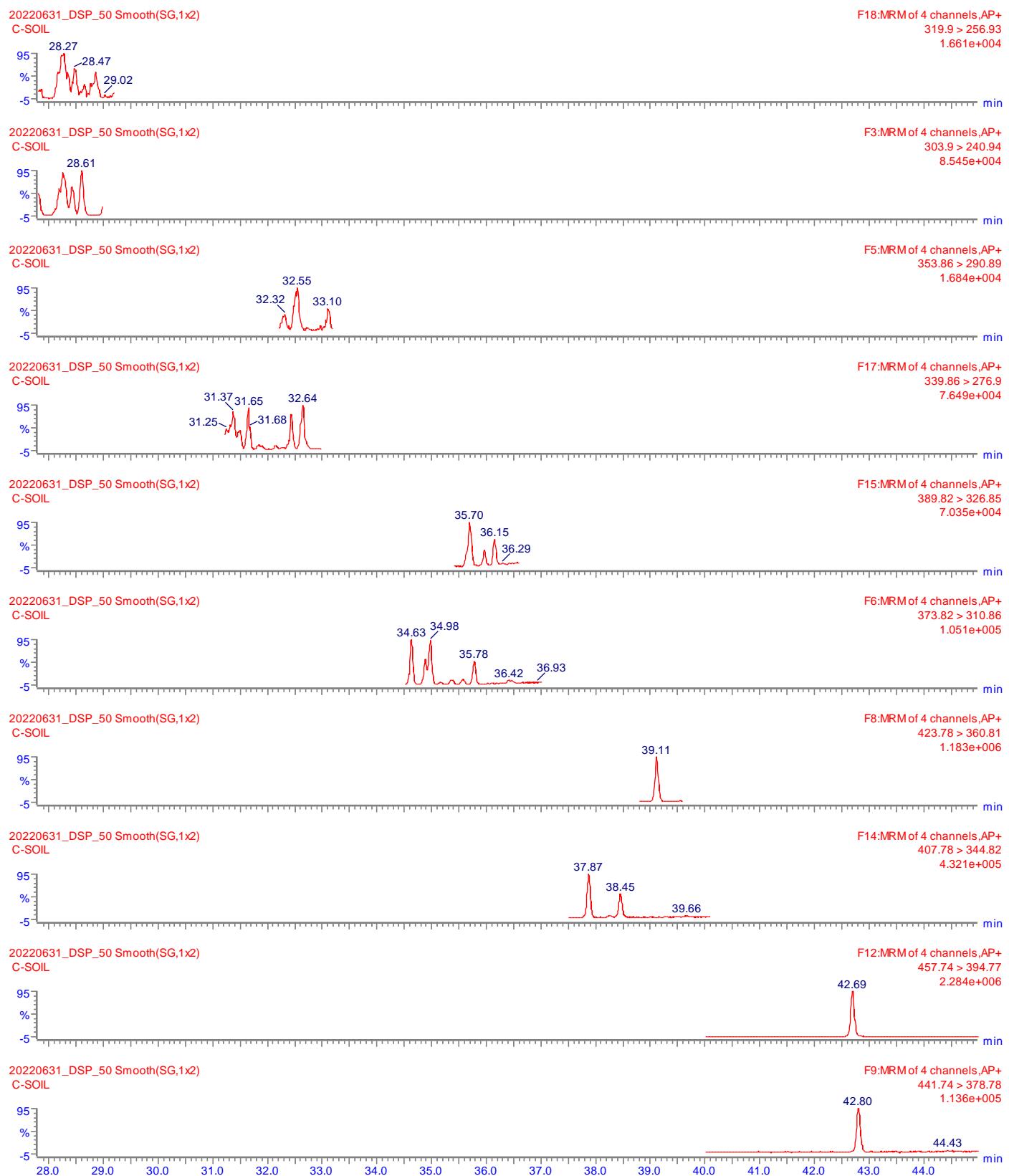
Ash - Carbon



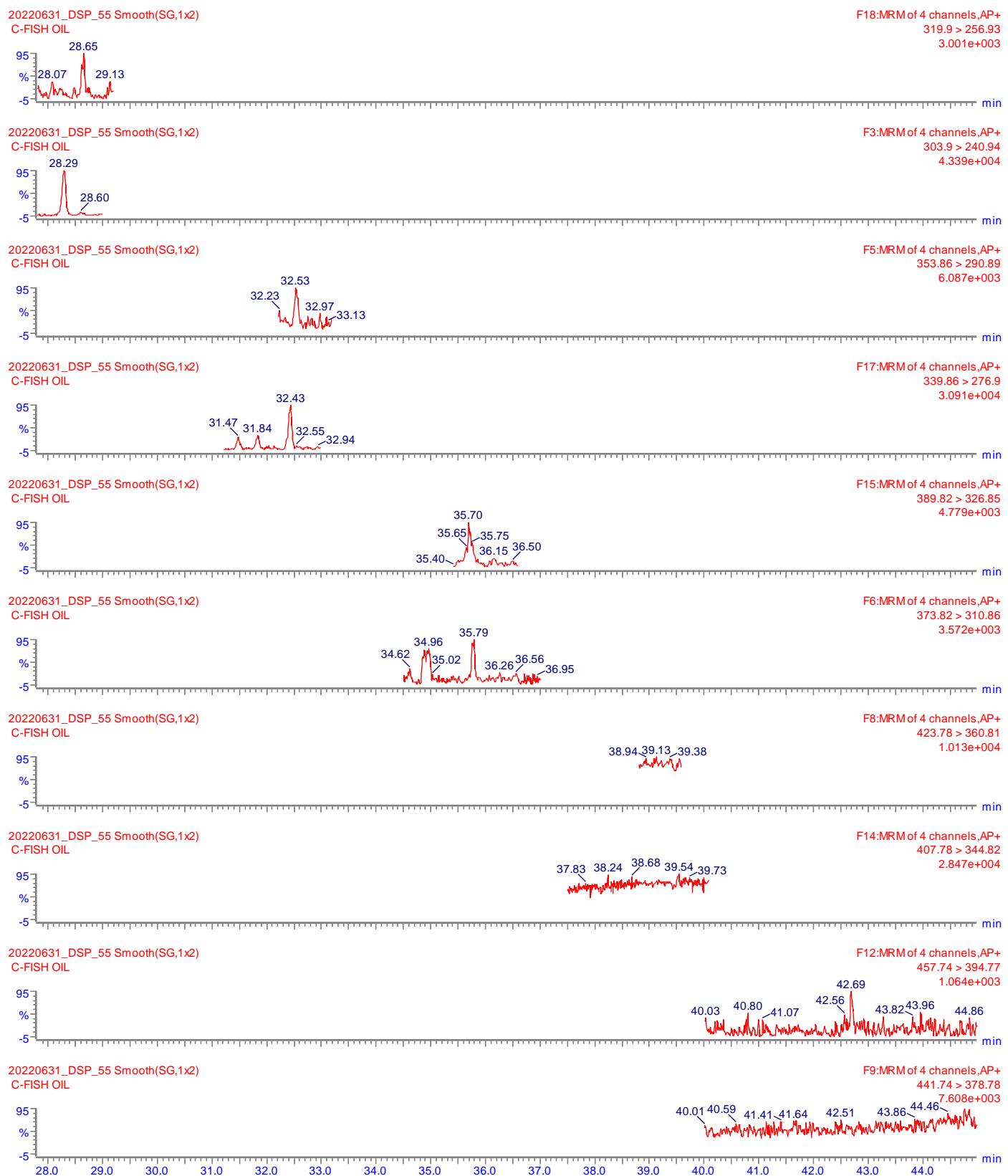
Sediment - Carbon



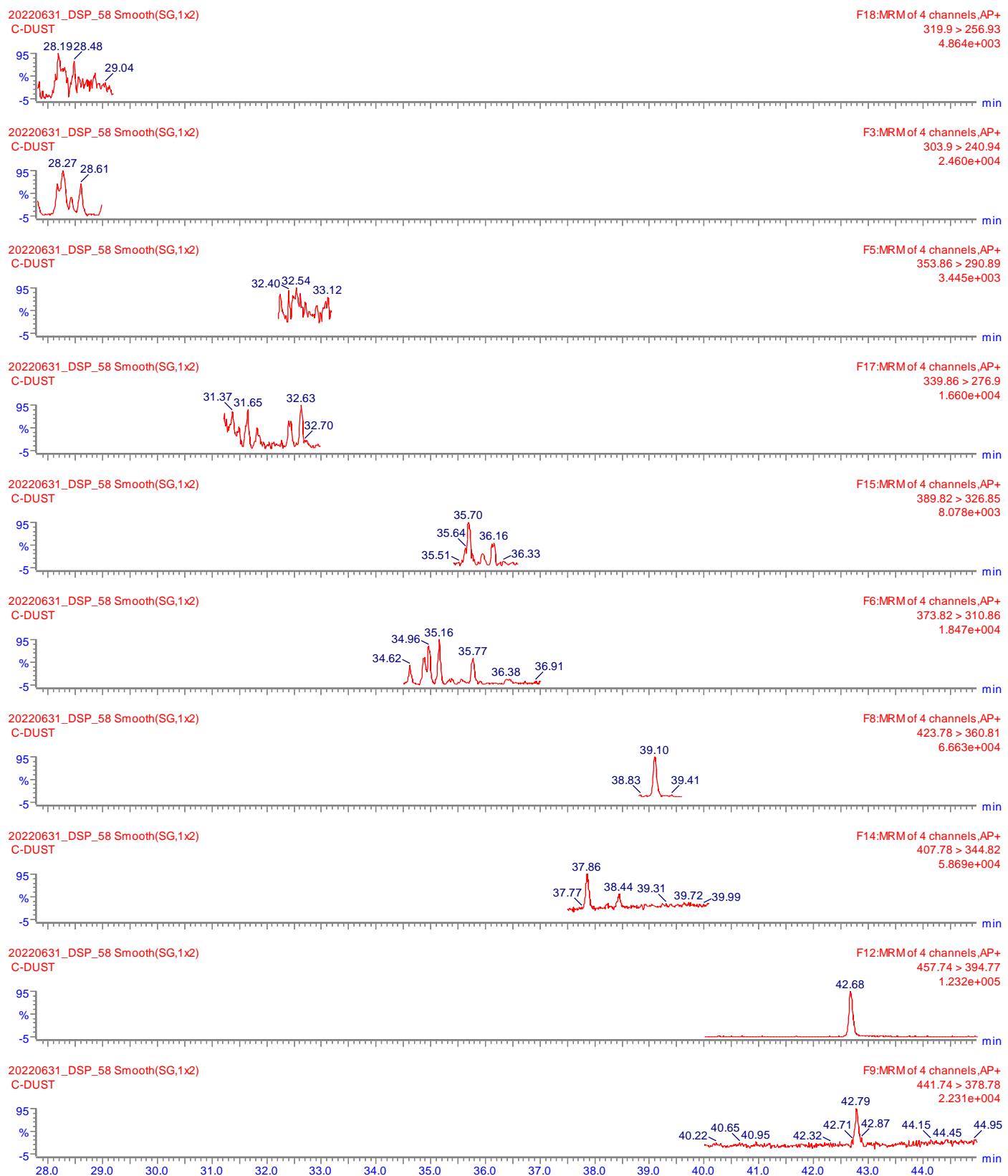
Soil - Carbon



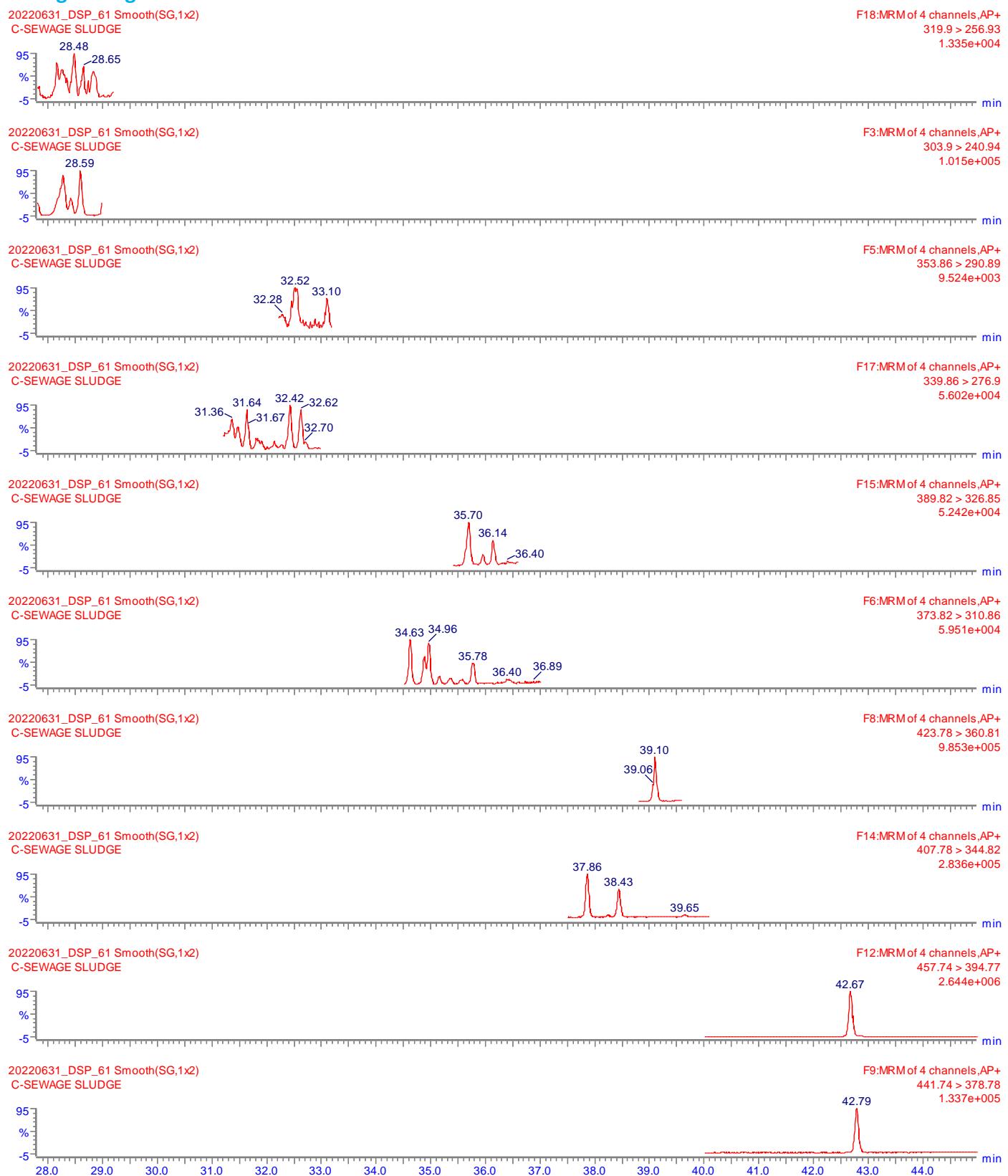
Fish oil - Carbon



Dust - Carbon



Sewage Sludge - Carbon



Annex 8. MS method

Dataset: C:\MassLynx\209PCB 202206.PRO\20220631 209PCB.qld

Last Altered: Thursday, July 07, 2022 13:20:06 W. Europe Summer Time

Printed: Thursday, July 07, 2022 19:27:13 W. Europe Summer Time

Method: C:\MassLynx\209PCB 202206.PRO\MethDB\Targetlynx 209PCB HT8 60mx025x01 v3.mdb 05 Jul 2022 12:41:26

Calibration: 07 Jul 2022 13:17:17

Function 1

Scans in function: 560
Cycle time (secs): Automatic
Inter Scan Delay (secs): Automatic
Inter Channel Delay (secs): Automatic
Span (Da): 0.000
Start and End Time(mins): 10.000 to 13.500
Ionization mode: AP+
Data type: Enhanced SIR or MRM
Function type: MRM of 4 channels
Chan Reaction Dwell(secs) Cone Volt. Col.Energy Delay(secs) Compound
1 : 188.00 > 152.00 0.080 40.0 28.0 Auto 1-CB
2 : 200.00 > 164.00 0.080 40.0 28.0 Auto 1-CB
3 : 202.00 > 164.00 0.080 40.0 28.0 Auto 1-CB
4 : 190.00 > 152.00 0.080 40.0 28.0 Auto 1-CB

Function 2

Scans in function: 940
Cycle time (secs): Automatic
Inter Scan Delay (secs): Automatic
Inter Channel Delay (secs): Automatic
Span (Da): 0.000
Start and End Time(mins): 14.500 to 21.500
Ionization mode: AP+
Data type: Enhanced SIR or MRM
Function type: MRM of 4 channels
Chan Reaction Dwell(secs) Cone Volt. Col.Energy Delay(secs) Compound
1 : 256.00 > 186.00 0.052 40.0 28.0 Auto 3-CB
2 : 268.00 > 198.00 0.052 40.0 28.0 Auto 3-CB
3 : 270.00 > 198.00 0.052 40.0 28.0 Auto 3-CB
4 : 258.00 > 186.00 0.052 40.0 28.0 Auto 3-CB

Function 3

Scans in function: 129
Cycle time (secs): Automatic
Inter Scan Delay (secs): Automatic
Inter Channel Delay (secs): Automatic
Span (Da): 0.000
Start and End Time(mins): 27.800 to 29.000
Ionization mode: AP+
Data type: Enhanced SIR or MRM
Function type: MRM of 4 channels
Chan Reaction Dwell(secs) Cone Volt. Col.Energy Delay(secs) Compound
1 : 303.90 > 240.94 0.024 40.0 40.0 Auto TCDF
2 : 315.94 > 251.97 0.024 40.0 40.0 Auto TCDF
3 : 317.94 > 253.97 0.024 40.0 40.0 Auto TCDF
4 : 305.90 > 242.93 0.024 40.0 40.0 Auto TCDF

Function 4

Scans in function: 1546
Cycle time (secs): Automatic
Inter Scan Delay (secs): Automatic
Inter Channel Delay (secs): Automatic
Span (Da): 0.000
Start and End Time(mins): 19.000 to 29.700
Ionization mode: AP+
Data type: Enhanced SIR or MRM
Function type: MRM of 4 channels
Chan Reaction Dwell(secs) Cone Volt. Col.Energy Delay(secs) Compound
1 : 323.90 > 253.90 0.024 40.0 28.0 Auto 5-CB
2 : 335.90 > 265.90 0.024 40.0 28.0 Auto 5-CB
3 : 337.90 > 267.90 0.024 40.0 28.0 Auto 5-CB
4 : 325.90 > 255.90 0.024 40.0 28.0 Auto 5-CB

Function 5

Scans in function: 106
Cycle time (secs): Automatic
Inter Scan Delay (secs): Automatic

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Inter Channel Delay (secs): Automatic
Span (Da): 0.000
Start and End Time(mins): 32.200 to 33.200
Ionization mode: AP+
Data type: Enhanced SIR or MRM
Function type: MRM of 4 channels
Chan Reaction Dwell(secs) Cone Volt. Col.Energy Delay(secs) Compound
1 : 353.86 > 290.89 0.024 40.0 28.0 Auto PeCDD
2 : 365.90 > 301.93 0.024 40.0 28.0 Auto PeCDD
3 : 367.89 > 303.93 0.024 40.0 28.0 Auto PeCDD
4 : 355.85 > 292.89 0.024 40.0 28.0 Auto PeCDD
Function 6
Scans in function: 366
Cycle time (secs): Automatic
Inter Scan Delay (secs): Automatic
Inter Channel Delay (secs): Automatic
Span (Da): 0.000
Start and End Time(mins): 34.500 to 37.000
Ionization mode: AP+
Data type: Enhanced SIR or MRM
Function type: MRM of 4 channels
Chan Reaction Dwell(secs) Cone Volt. Col.Energy Delay(secs) Compound
1 : 373.82 > 310.86 0.043 40.0 40.0 Auto HxCDF
2 : 385.86 > 321.89 0.043 40.0 40.0 Auto HxCDF
3 : 387.86 > 323.89 0.043 40.0 40.0 Auto HxCDF
4 : 375.82 > 312.85 0.043 40.0 40.0 Auto HxCDF
Function 7
Scans in function: 1406
Cycle time (secs): Automatic
Inter Scan Delay (secs): Automatic
Inter Channel Delay (secs): Automatic
Span (Da): 0.000
Start and End Time(mins): 25.300 to 34.500
Ionization mode: AP+
Data type: Enhanced SIR or MRM
Function type: MRM of 4 channels
Chan Reaction Dwell(secs) Cone Volt. Col.Energy Delay(secs) Compound
1 : 391.90 > 321.90 0.024 30.0 35.0 Auto 7-CB
2 : 405.90 > 333.90 0.024 30.0 35.0 Auto 7-CB
3 : 407.90 > 335.90 0.024 30.0 35.0 Auto 7-CB
4 : 393.90 > 323.90 0.024 30.0 35.0 Auto 7-CB
Function 8
Scans in function: 86
Cycle time (secs): Automatic
Inter Scan Delay (secs): Automatic
Inter Channel Delay (secs): Automatic
Span (Da): 0.000
Start and End Time(mins): 38.800 to 39.600
Ionization mode: AP+
Data type: Enhanced SIR or MRM
Function type: MRM of 4 channels
Chan Reaction Dwell(secs) Cone Volt. Col.Energy Delay(secs) Compound
1 : 423.78 > 360.81 0.080 40.0 28.0 Auto HpCDD
2 : 435.82 > 371.85 0.080 40.0 28.0 Auto HpCDD
3 : 437.81 > 373.85 0.080 40.0 28.0 Auto HpCDD
4 : 425.77 > 362.81 0.080 40.0 28.0 Auto HpCDD
Function 9
Scans in function: 677
Cycle time (secs): Automatic
Inter Scan Delay (secs): Automatic
Inter Channel Delay (secs): Automatic
Span (Da): 0.000
Start and End Time(mins): 40.000 to 45.000
Ionization mode: AP+
Data type: Enhanced SIR or MRM
Function type: MRM of 4 channels
Chan Reaction Dwell(secs) Cone Volt. Col.Energy Delay(secs) Compound

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1 : 441.74 > 378.78	0.052	40.0	40.0	Auto	OCDF
2 : 453.78 > 389.82	0.052	40.0	40.0	Auto	OCDF
3 : 455.78 > 391.81	0.052	40.0	40.0	Auto	OCDF
4 : 443.74 > 380.78	0.052	40.0	40.0	Auto	OCDF

Function 10

Scans in function:	556
Cycle time (secs):	Automatic
Inter Scan Delay (secs):	Automatic
Inter Channel Delay (secs):	Automatic
Span (Da):	0.000
Start and End Time(mins):	32.500 to 36.500
Ionization mode:	AP+
Data type:	Enhanced SIR or MRM
Function type:	MRM of 4 channels
Chan Reaction	Dwell(secs) Cone Volt. Col.Energy Delay(secs) Compound
1 : 461.80 > 391.80	0.024 30.0 35.0 Auto 9-CB
2 : 463.80 > 393.80	0.024 30.0 35.0 Auto 9-CB
3 : 475.80 > 405.80	0.024 30.0 35.0 Auto 9-CB
4 : 473.80 > 403.80	0.024 30.0 35.0 Auto 9-CB

Function 11

Scans in function:	90
Cycle time (secs):	Automatic
Inter Scan Delay (secs):	Automatic
Inter Channel Delay (secs):	Automatic
Span (Da):	0.000
Start and End Time(mins):	35.800 to 36.700
Ionization mode:	AP+
Data type:	Enhanced SIR or MRM
Function type:	MRM of 4 channels
Chan Reaction	Dwell(secs) Cone Volt. Col.Energy Delay(secs) Compound
1 : 495.70 > 425.70	0.043 30.0 35.0 Auto 10-CB
2 : 509.70 > 439.70	0.043 30.0 35.0 Auto 10-CB
3 : 511.70 > 441.70	0.043 30.0 35.0 Auto 10-CB
4 : 497.70 > 427.70	0.043 30.0 35.0 Auto 10-CB

Function 12

Scans in function:	676
Cycle time (secs):	Automatic
Inter Scan Delay (secs):	Automatic
Inter Channel Delay (secs):	Automatic
Span (Da):	0.000
Start and End Time(mins):	40.000 to 45.000
Ionization mode:	AP+
Data type:	Enhanced SIR or MRM
Function type:	MRM of 4 channels
Chan Reaction	Dwell(secs) Cone Volt. Col.Energy Delay(secs) Compound
1 : 457.74 > 394.77	0.052 40.0 28.0 Auto OCDD
2 : 469.78 > 405.81	0.052 40.0 28.0 Auto OCDD
3 : 471.78 > 407.81	0.052 40.0 28.0 Auto OCDD
4 : 459.73 > 396.77	0.052 40.0 28.0 Auto OCDD

Function 13

Scans in function:	1040
Cycle time (secs):	Automatic
Inter Scan Delay (secs):	Automatic
Inter Channel Delay (secs):	Automatic
Span (Da):	0.000
Start and End Time(mins):	28.800 to 35.700
Ionization mode:	AP+
Data type:	Enhanced SIR or MRM
Function type:	MRM of 4 channels
Chan Reaction	Dwell(secs) Cone Volt. Col.Energy Delay(secs) Compound
1 : 427.80 > 355.80	0.024 30.0 35.0 Auto 8-CB
2 : 439.80 > 367.80	0.024 30.0 35.0 Auto 8-CB
3 : 441.80 > 369.80	0.024 30.0 35.0 Auto 8-CB
4 : 429.80 > 357.80	0.024 30.0 35.0 Auto 8-CB

Function 14

Scans in function:	559
Cycle time (secs):	Automatic

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Inter Scan Delay (secs): Automatic
Inter Channel Delay (secs): Automatic
Span (Da): 0.000
Start and End Time(mins): 37.500 to 40.100
Ionization mode: AP+
Data type: Enhanced SIR or MRM
Function type: MRM of 4 channels
Chan Reaction Dwell(secs) Cone Volt. Col.Energy Delay(secs) Compound
1 : 407.78 > 344.82 0.052 40.0 40.0 Auto HpCDF
2 : 419.82 > 355.85 0.052 40.0 40.0 Auto HpCDF
3 : 421.82 > 357.85 0.052 40.0 40.0 Auto HpCDF
4 : 409.78 > 346.82 0.052 40.0 40.0 Auto HpCDF

Function 15
Scans in function: 117
Cycle time (secs): Automatic
Inter Scan Delay (secs): Automatic
Inter Channel Delay (secs): Automatic
Span (Da): 0.000
Start and End Time(mins): 35.400 to 36.600
Ionization mode: AP+
Data type: Enhanced SIR or MRM
Function type: MRM of 4 channels
Chan Reaction Dwell(secs) Cone Volt. Col.Energy Delay(secs) Compound
1 : 389.82 > 326.85 0.043 40.0 28.0 Auto HxCDD
2 : 401.86 > 337.89 0.043 40.0 28.0 Auto HxCDD
3 : 403.85 > 339.89 0.043 40.0 28.0 Auto HxCDD
4 : 391.81 > 328.85 0.043 40.0 28.0 Auto HxCDD

Function 16
Scans in function: 1738
Cycle time (secs): Automatic
Inter Scan Delay (secs): Automatic
Inter Channel Delay (secs): Automatic
Span (Da): 0.000
Start and End Time(mins): 21.500 to 33.000
Ionization mode: AP+
Data type: Enhanced SIR or MRM
Function type: MRM of 4 channels
Chan Reaction Dwell(secs) Cone Volt. Col.Energy Delay(secs) Compound
1 : 357.90 > 287.90 0.024 30.0 35.0 Auto 6-CB
2 : 369.90 > 299.90 0.024 30.0 35.0 Auto 6-CB
3 : 371.90 > 301.90 0.024 30.0 35.0 Auto 6-CB
4 : 359.90 > 289.90 0.024 30.0 35.0 Auto 6-CB

Function 17
Scans in function: 217
Cycle time (secs): Automatic
Inter Scan Delay (secs): Automatic
Inter Channel Delay (secs): Automatic
Span (Da): 0.000
Start and End Time(mins): 31.200 to 33.000
Ionization mode: AP+
Data type: Enhanced SIR or MRM
Function type: MRM of 4 channels
Chan Reaction Dwell(secs) Cone Volt. Col.Energy Delay(secs) Compound
1 : 337.86 > 274.90 0.024 40.0 40.0 Auto PeCDF
2 : 349.86 > 285.90 0.024 40.0 40.0 Auto PeCDF
3 : 351.90 > 287.93 0.024 40.0 40.0 Auto PeCDF
4 : 339.86 > 276.90 0.024 40.0 40.0 Auto PeCDF

Function 18
Scans in function: 151
Cycle time (secs): Automatic
Inter Scan Delay (secs): Automatic
Inter Channel Delay (secs): Automatic
Span (Da): 0.000
Start and End Time(mins): 27.800 to 29.200
Ionization mode: AP+
Data type: Enhanced SIR or MRM
Function type: MRM of 4 channels

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Chan Reaction	Dwell(secs)	Cone	Volt.	Col.Energy	Delay(secs)	Compound
1 : 319.90 > 256.93	0.024	40.0	28.0	Auto		TCDD
2 : 331.94 > 267.97	0.024	40.0	28.0	Auto		TCDD
3 : 333.93 > 269.97	0.024	40.0	28.0	Auto		TCDD
4 : 321.89 > 258.93	0.024	40.0	28.0	Auto		TCDD

Function 19

Scans in function: 1353

Cycle time (secs): Automatic

Inter Scan Delay (secs): Automatic

Inter Channel Delay (secs): Automatic

Span (Da): 0.000

Start and End Time(mins): 16.500 to 26.000

Ionization mode: AP+

Data type: Enhanced SIR or MRM

Function type: MRM of 4 channels

Chan Reaction	Dwell(secs)	Cone	Volt.	Col.Energy	Delay(secs)	Compound
1 : 289.90 > 219.90	0.038	40.0	28.0	Auto		4-CB
2 : 301.90 > 231.90	0.038	40.0	28.0	Auto		4-CB
3 : 303.90 > 233.90	0.038	40.0	28.0	Auto		4-CB
4 : 291.90 > 221.90	0.038	40.0	28.0	Auto		4-CB

Function 20

Scans in function: 703

Cycle time (secs): Automatic

Inter Scan Delay (secs): Automatic

Inter Channel Delay (secs): Automatic

Span (Da): 0.000

Start and End Time(mins): 12.500 to 17.000

Ionization mode: AP+

Data type: Enhanced SIR or MRM

Function type: MRM of 4 channels

Chan Reaction	Dwell(secs)	Cone	Volt.	Col.Energy	Delay(secs)	Compound
1 : 222.00 > 152.00	0.052	40.0	28.0	Auto		2-CB
2 : 234.00 > 164.00	0.052	40.0	28.0	Auto		2-CB
3 : 236.00 > 164.00	0.052	40.0	28.0	Auto		2-CB
4 : 224.00 > 152.00	0.052	40.0	28.0	Auto		2-CB