Certificate

of column set for GO system

MIURA CO., LTD.
Miura Institute of Environmental Science

This material is intended to be used for the determination of selected polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) in food/feed, environmental matrices, and similar matrices.

Material	18Φ Column set
Product Code	X300-002-1330
Lot No.	253205
Expiration Date	Jul/2027

Tests	Result	Criteria
Blank Values of PCDDs/PCDFs pg-TEQ/column set	< 0.34	<1.0
Blank values of Non-ortho-PCBs pg-TEQ/column set	< 0.026	< 0.05
Recovery PCDDs/PCDFs	87 to 101 %	70 to 120 %
Recovery Non-ortho-PCBs	87 to 93 %	70 to 120 %

Miura certifies that this product complies with all quality specifications. It was produced and inspected in accordance with the most current edition of the Miura Corporation Quality System Manual.
Contact: For any questions regarding your purchased product or the contents of this certificate, please contact your distributor.

DESCRIPTIONS

Lot Number: The number mentioned on the labels on the column bag is the lot production number.

Blank Level Values: Blank level values, expressed as mass fractions (pg-TEQ per a column set), for selected PCB congeners, PCDD, and PCDF congeners are provided in Table 2. Blank level values are a reference value for which MIURA has the highest confidence in its accuracy, in that all known or suspected sources of bias have been investigated or taken into account (JIS K0311 or JIS K0312).

Recovery Values (BLANK): Recovery values, expressed as percentages, are provided in Table 3 for selected mass labeled PCB congeners, and selected mass labeled PCDD and PCDF congeners, based on selected mass labeled recovery standards added before GC-MS measurement. Recovery values meet EU regulations; however, the values meet the MIURA criteria for this certification, which are stricter than what is required by EU regulations.

NOTICE AND WARNING TO USERS

THE GO SYSTEM COLUMN SET IS INTENDED FOR DIOXIN ANALYTICAL USE ONLY, INCLUDING HAZARDOUS MATERIALS. BEFORE USE, READ THE SDS CAREFULLY; HANDLE PRODUCT AS A HAZARDOUS MATERIAL CAPABLE OF SKIN CORROSION AND/OR EYE DAMAGE.

INSTRUCTIONS FOR STABILITY, STORAGE, AND USE

Stability and Storage: The column set should be stored at room temperatures below 25 °C until use. It should not be frozen or exposed to sunlight or ultraviolet radiation. After removing from the bags, the contents should be used immediately. Storing of the removed column set is not recommended.

Use: If storing in a cold room or refrigerator, bring them to room temperature (let stand for approximately 30 min), remove water condensed on the surface of the bags. Carefully remove the bags to avoid damage of the column. Use the same lot number with one column set. For more information of column set, refer to the operation manual.

ANALYTICAL METHODS USED AT MIURA

For preparation of blank test, several column sets chosen at random per lot production were allowed to reach ambient temperature; two types of the purification columns (upper: silver nitrate silica gel, and lower: sulfuric acid silica gel) were assembled, a known amount of internal standard solution (containing selected labeled PCB, PCDDs, and PCDFs congeners; as shown in Table 1) dissolved in 2 mL of n-hexane was added to the top of the column. Then, attach the concentration column (upper) to purification column and immediately set it to the system unit. Once eluate was obtained in the fraction, a known amount of the recovery standard solution was added. Finally, the fraction was concentrated to 0.01 mL using an evaporation system under nitrogen.

Table 1. Standard solutions used for recovery tests.

Compounds	Standard	Maker Code	Maker	Concentration
PCDDs and PCDFs, DL-PCBs	Internal Standard	DF-SS-A	Wellington Laboratories Inc.	10 ng/mL in decane
	Internal Standard	DFP-LCS-B		
	Recovery (Surrogate) Standard	DF-IS-J		

The concentrated fraction was analyzed using gas chromatography / high resolution mass spectrometry (GC/HRMS) operated in electron impact (EI) mode. A 0.25 mm ID × 60 m fused silica capillary (BPX-DXN, TRAJAN) was used. The injection volume was 2 μL using a splitless inlet. The results, blank level values, are provided in Table 2. The chromatograms of each compounds are shown after Table 3. Furthermore, the fraction was analyzed using gas chromatography / low resolution mass spectrometry operated in total ion scan (m/z 50 to 500) mode, to confirm if interferences may affect determination of target compounds by GC/HRMS are included in the fractions, the chromatograms are not shown here.

Table 2. Blank levels of dioxins (PCDDs/PCDFs and DL-PCBs) and NDL-PCBs per column set.

Congener	Concentration pg/column	LOQ pg/column	LOD pg/column	S/N=3 pg/column	TEQ* pg/column
2,3,7,8-TeCDD	ND	0.26	0.08	0.03	0.08
1,2,3,7,8-PeCDD	ND	0.25	0.07	0.05	0.07
1,2,3,4,7,8-HxCDD	ND	0.4	0.1	0.02	0.01
1,2,3,6,7,8-HxCDD	ND	0.8	0.2	0.02	0.02
1,2,3,7,8,9-HxCDD	ND	0.4	0.1	0.02	0.01
1,2,3,4,6,7,8-HpCDD	ND	0.7	0.2	0.1	0.002
OCDD	ND	1.3	0.4	0.2	0.00012
2,3,7,8-TeCDF	ND	0.7	0.2	0.1	0.02
1,2,3,7,8-PeCDF	ND	0.9	0.3	0.1	0.009
2,3,4,7,8-PeCDF	ND	0.3	0.1	0.1	0.03
1,2,3,4,7,8-HxCDF	ND	0.6	0.2	0.08	0.02
1,2,3,6,7,8-HxCDF	ND	0.7	0.2	0.08	0.02
1,2,3,7,8,9-HxCDF	ND	0.6	0.2	0.1	0.02
2,3,4,6,7,8-HxCDF	ND	0.6	0.2	0.07	0.02
1,2,3,4,6,7,8-HpCDF	ND	1.0	0.3	0.06	0.003
1,2,3,4,7,8,9-HpCDF	ND	0.7	0.2	0.08	0.002
OCDF	ND	1.7	0.5	0.2	0.00015
#81 (3,4,4',5-TeCB)	ND	0.4	0.1	0.06	0.00003
#77 (3,3',4,4'-TeCB)	ND	0.9	0.3	0.06	0.00003
#126 (3,3',4,4',5-PeCB)	ND	0.7	0.2	0.1	0.02
#169 (3,3',4,4',5,5'-HxCB)	ND	0.8	0.2	0.09	0.006

* TEQ： Toxicity Equivalents (are applied WHO-TEF(2006))

1. The figures in the parentheses in the concentration of actual measurement denote the concentration of the LOD or more and less than the LOQ.
2. ND in the concentration of actual measurement denotes less than the LOD.
3. TEQ was calculated with an actual measurement which is the concentration of the LOQ or more, and an actual measurement which is the concentration of the LOD or more and less than the LOQ, respectively. For values less than the LOD, TEQ was calculated with the LOD.

Table 3.Recoveries of labeled internal standards.

Congener	Blank
2,3,7,8-TeCDD	99 %
1,2,3,7,8-PeCDD	90 %
1,2,3,4,7,8-HxCDD	92 %
1,2,3,6,7,8-HxCDD	91 %
1,2,3,7,8,9-HxCDD	95 %
1,2,3,4,6,7,8-HpCDD	95 %
OCDD	101 %
2,3,7,8-TeCDF	94 %
1,2,3,7,8-PeCDF	94 %
2,3,4,7,8-PeCDF	91 %
1,2,3,4,7,8-HxCDF	88 %
1,2,3,6,7,8-HxCDF	87 %
1,2,3,7,8,9-HxCDF	94 %
2,3,4,6,7,8-HxCDF	97 %
1,2,3,4,6,7,8-HpCDF	95 %
1,2,3,4,7,8,9-HpCDF	100 %
OCDF	94 %
#81 (3,4,4',5-TeCB)	90 %
#77 (3,3',4,4'-TeCB)	93 %
#126 (3,3',4,4',5-PeCB)	92 %
#169 (3,3',4,4',5,5'-HxCB)	87 %

* These congeners are basically broken through the concentration column (upper), which a carbon is fulfilled.

Compound View

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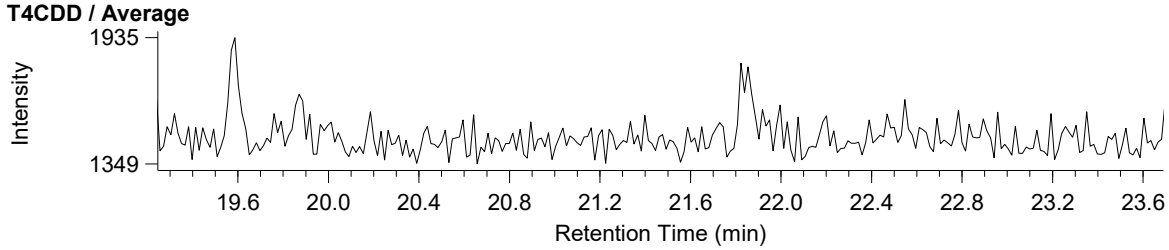
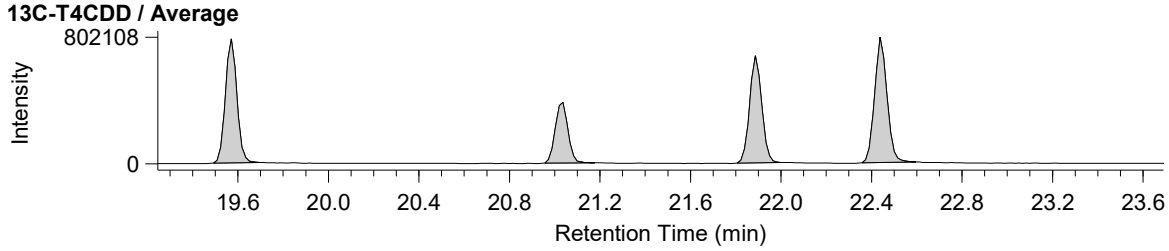
Injection : C5EF05001X

T4CDD / Average



13C-T4CDD / Average



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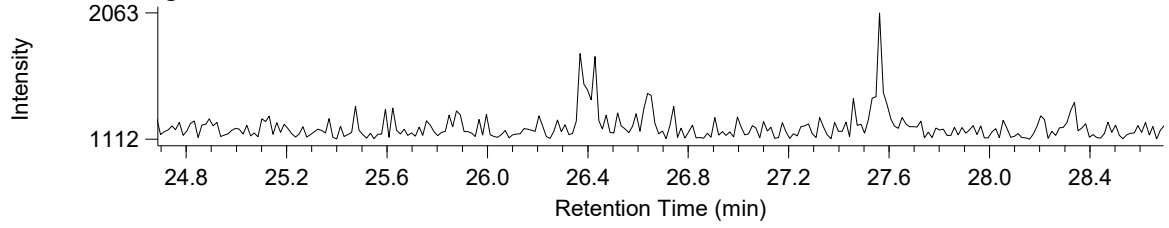

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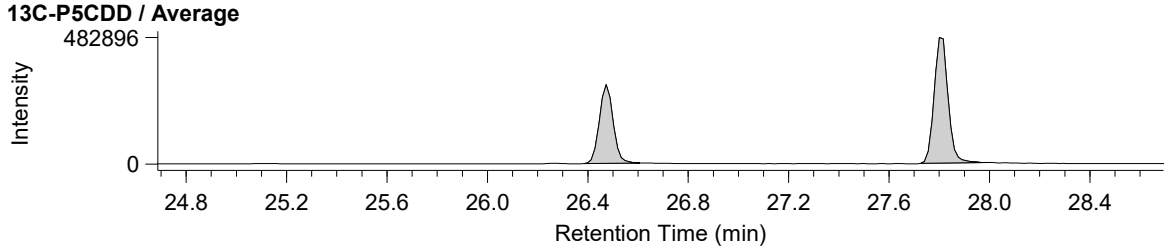

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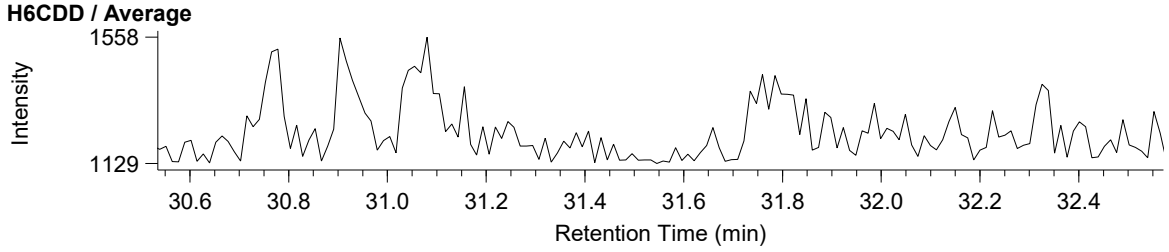

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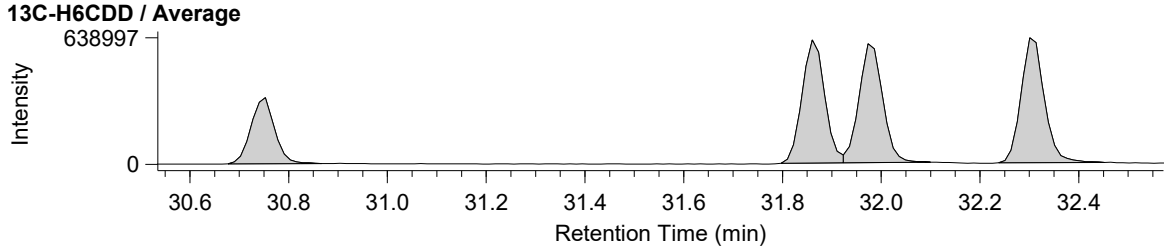

Compound View

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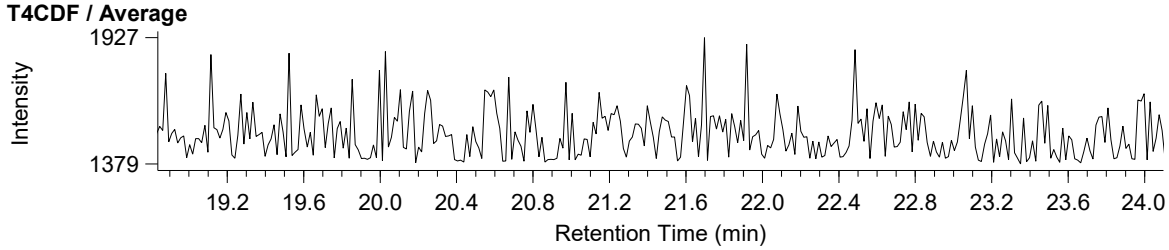
Injection : C5EF05001X

T4CDF / Average



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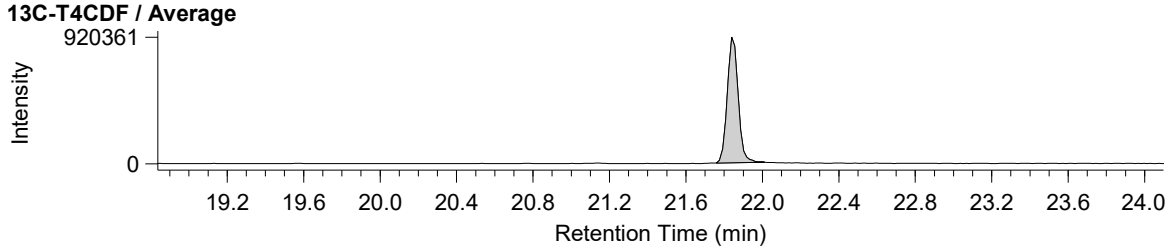

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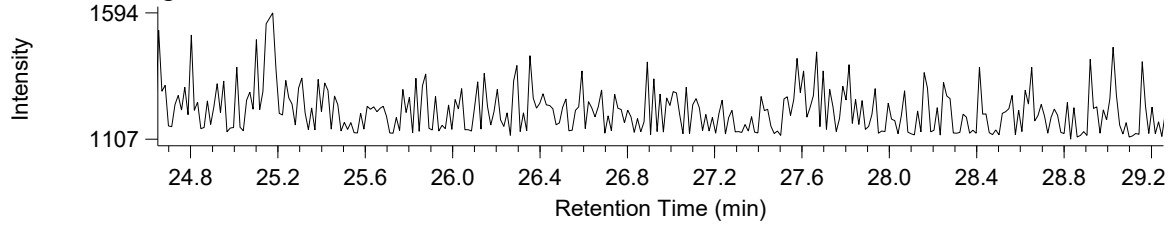

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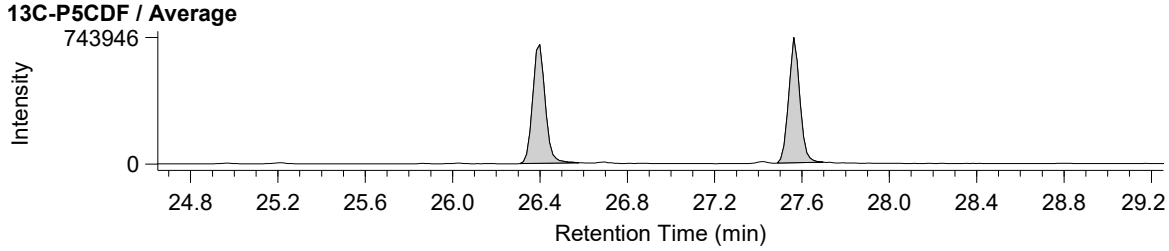

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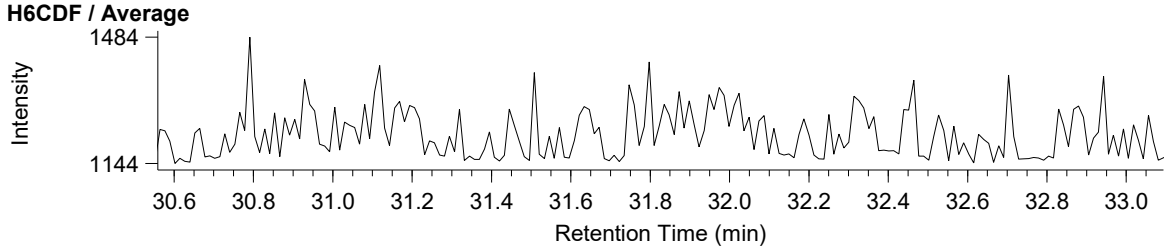

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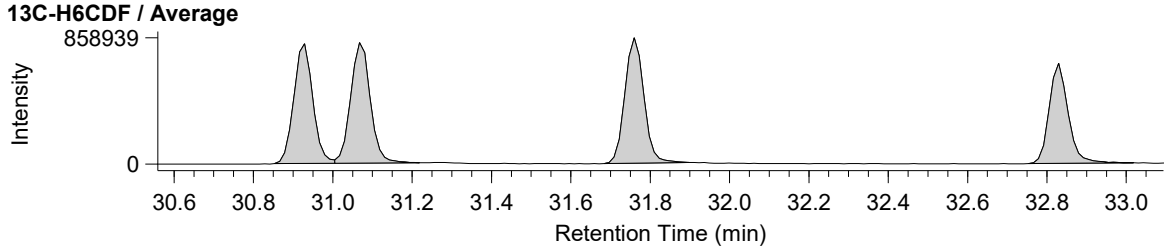

Compound View

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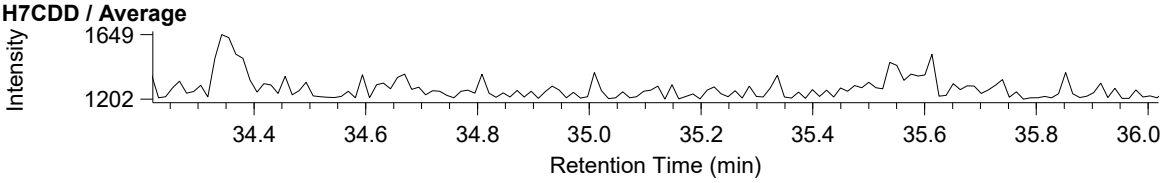
Injection : C5EF05001X

H7CDD / Average



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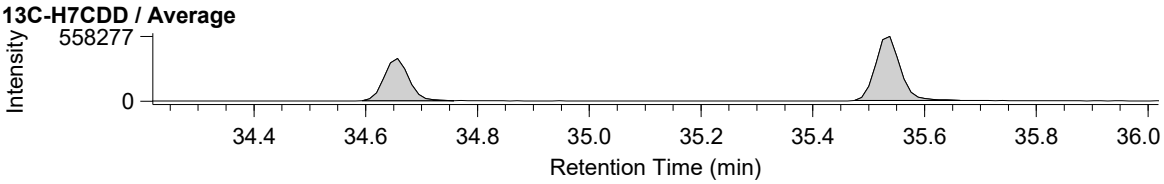
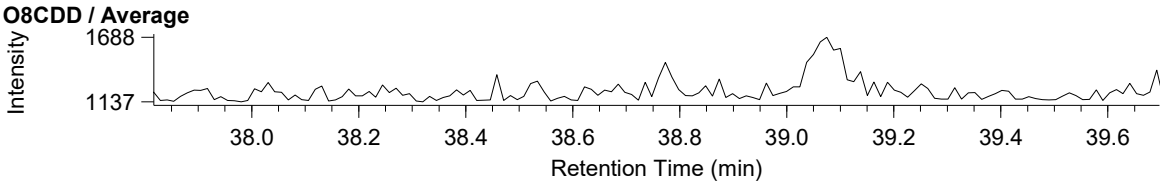
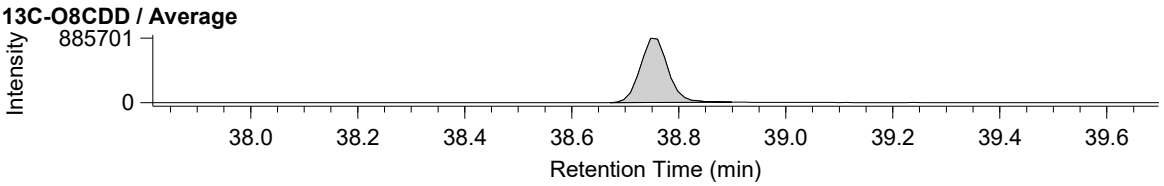


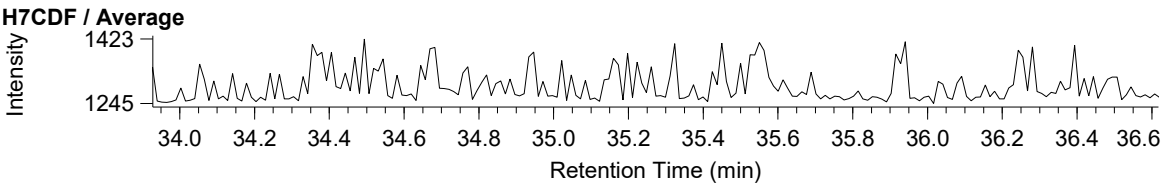
O8CDD / Average



13C-O8CDD / Average



H7CDF / Average



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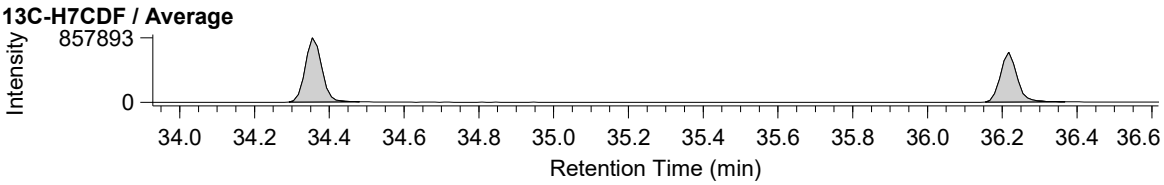

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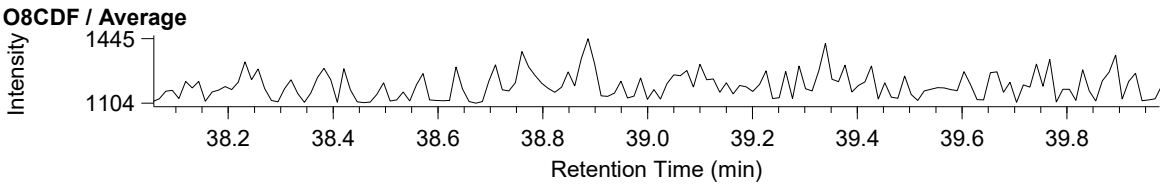

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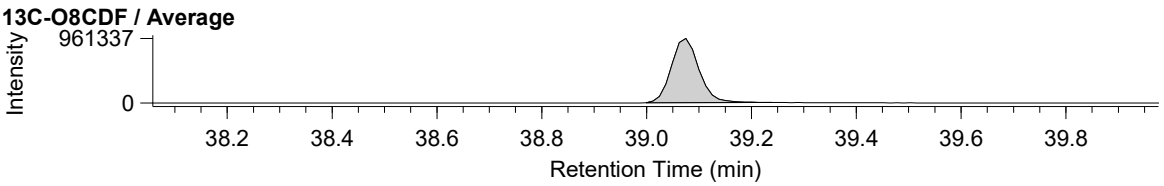

Compound View

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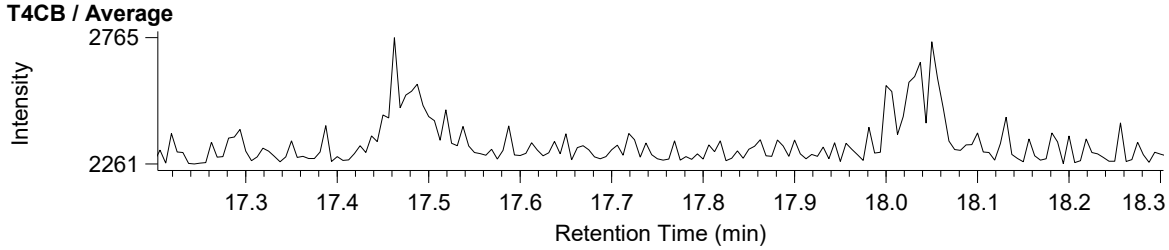
Injection : C5EF05001X

T4CB / Average



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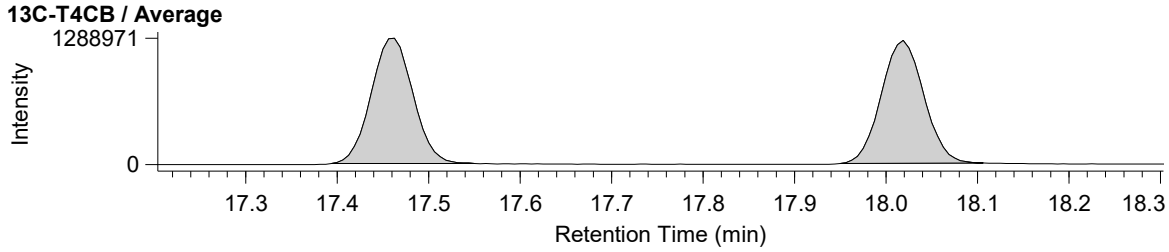
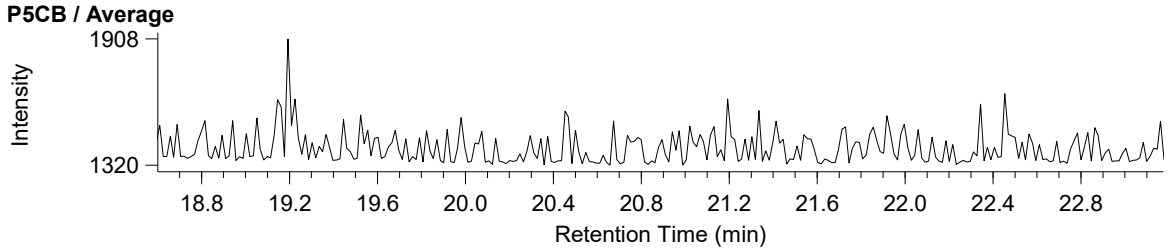


P5CB / Average



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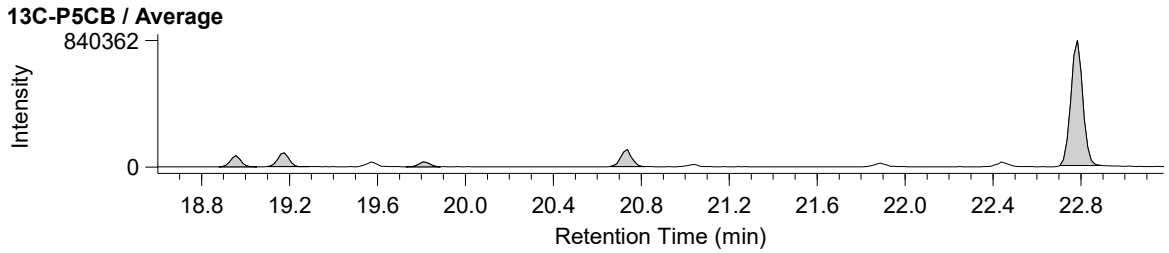
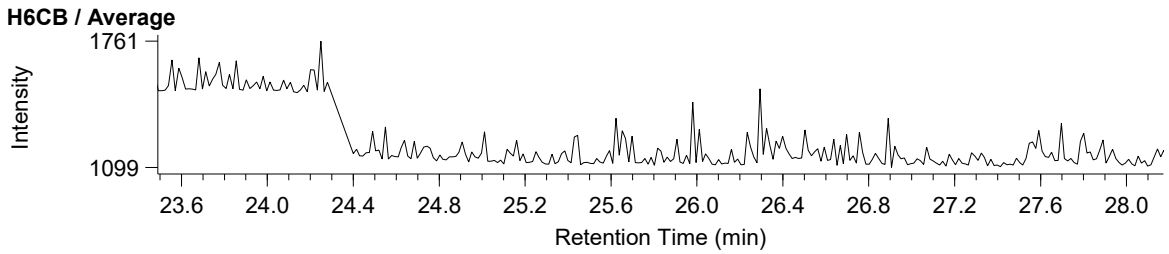


H6CB / Average



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