MIURA

Certificate

of column set for GO system

MIURA CO., LTD.
Environmental Business Headquarters

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

Material	20Φ Column set
Product Code	X300-002-2100
Lot No.	193045
Expiration Date	Mar./2021

Tests	Result	Criteria
Blank Values of PCDDs/PCDFs pg-TEQ/column set	< 0.40	< 0.5
Blank values of DL-PCBs pg-TEQ/column set	< 0.026	< 0.05
Blank values of NDL-PCBs pg-congener /column set	The highest isomer (#28) < 1.1	Each isomer < 200
Recovery PCDDs/PCDFs/ DL-PCBs	71 to 92 %	70 to 120 %
Recovery NDL-PCBs	89 to 98 %	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.

Expiration of Certification: The expiration date displayed in the first page means that the certification of the column set is valid, until the expiration date, provided the column set is stored in accordance with instructions given in this certificate (see "Instructions for Stability, Storage, and Use"). The certification is nullified if the column set is damaged, contaminated, or otherwise modified.

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 (Sample): 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, especially, because the concentration column (lower) "Alumina" can be deactivated under high-humidity. Storing of the removed column set is not recommended.

Use: If storing in a cold room or refrigerator, bring 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, and 10 mL of n-hexane was added to wet the top of the column with the designated column cap and disposable syringe. Then, a known amount of internal standard solution (containing selected labeled PCB, PCDDs, and PCDFs congeners; as shown in Table 1) dissolved in 6 mL of n-hexane was added to the top of the column with disposable syringe, and the syringe was washed with 2 x 2 mL of n-hexane; the n-hexane was injected into the column again. Then, the purification columns assembled with the concentration columns (upper) and (lower) were set to the each system unit immediately. After two fractions (dioxin and PCB fractions) were obtained from each system unit, a known amount of the recovery standard solution was added to each concentration vessel. Finally, both fractions were concentrated using an evaporation system under nitrogen to 0.01 mL.

Table 1. Standard solutions used for recovery tests.

Compounds	Standard	Maker Code	Maker	Diluted 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		
NDL-PCBs	Internal Standard	MBP-28		
		MBP-52		
		MBP-101		
		MBP-138		
		MBP-153		
	Recovery (Surrogate) Standard	MBP-19		
		MBP-70		
		MBP-111		
		MBP-162		
		MBP-178		

The concentrated dioxin 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 concentrated PCB fraction was analyzed using GC/HRMS operated in EI mode. A 0.25 mm ID × 60 m fused silica capillary (HT8-PCB, TRAJAN) was used. All injections were 2 µL using a splitless inlet. The results, blank level values, are provided in Table 2. The chromatograms of each compounds are shown at page 6 and after. Furthermore, the dioxin and PCB fractions were 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.

For the recovery test (sample), 0.5 or 1.0 g of fish oil from menhaden (F8020-1L, Sigma-Aldrich) was dissolved in 2 mL of n-hexane. A known amount of the internal standard solution was added to the flask, mixed, and allowed to equilibrate. First, several column sets chosen at random per lot production were allowed to reach ambient temperature; the purification columns (upper) and (lower) were assembled. 10 mL of n-hexane was added to wet the top of the column with the designated column cap and disposable syringe. Then, the sample with the internal standard was injected into the top of the column with the disposable syringe, and the syringe was washed with 2 x 2 mL of n-hexane; the n-hexane was injected into the column again. The purification column was assembled with the concentration column (upper) and (lower), and set to the each system unit immediately. After obtaining two fractions from the system unit, the dioxin and PCB fractions were concentrated using an evaporation system under nitrogen to approximately 0.01 mL. After the addition of a known amount of recovery standard solution, the both fractions were concentrated to 0.02 mL; then dioxin and PCB in each fractions were analyzed using GC/HRMS as mentioned above test. The inspection results is displayed in Table 3.

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	0.29	0.09	0.09	0.09
1,2,3,7,8-PeCDD	ND	0.27	0.08	0.06	0.08
1,2,3,4,7,8-HxCDD	ND	0.5	0.1	0.04	0.01
1,2,3,6,7,8-HxCDD	ND	0.8	0.2	0.05	0.02
1,2,3,7,8,9-HxCDD	ND	0.7	0.2	0.05	0.02
1,2,3,4,6,7,8-HpCDD	ND	0.9	0.3	0.07	0.003
OCDD	ND	1.3	0.4	0.07	0.00012
2,3,7,8-TeCDF	ND	0.6	0.2	0.06	0.02
1,2,3,7,8-PeCDF	ND	0.6	0.2	0.08	0.006
2,3,4,7,8-PeCDF	ND	0.6	0.2	0.07	0.06
1,2,3,4,7,8-HxCDF	ND	0.6	0.2	0.06	0.02
1,2,3,6,7,8-HxCDF	ND	0.7	0.2	0.06	0.02
1,2,3,7,8,9-HxCDF	ND	0.6	0.2	0.08	0.02
2,3,4,6,7,8-HxCDF	ND	0.7	0.2	0.06	0.02
1,2,3,4,6,7,8-HpCDF	ND	1.0	0.3	0.07	0.003
1,2,3,4,7,8,9-HpCDF	ND	1.2	0.4	0.09	0.004
OCDF	ND	1.3	0.4	0.2	0.00012
#81 (3,4,4',5-TeCB)	ND	0.6	0.2	0.06	0.00006
#77 (3,3',4,4'-TeCB)	ND	0.8	0.2	0.06	0.00002
#126 (3,3',4,4',5-PeCB)	ND	0.8	0.2	0.1	0.02
#169 (3,3',4,4',5,5'-HxCB)	ND	0.7	0.2	0.07	0.006
#123 (2',3,4,4',5-PeCB)	ND	0.7	0.2	0.01	0.000006
#118 (2,3',4,4',5-PeCB)	(0.4)	0.5	0.1	0.01	0.000012
#105 (2,3,3',4,4'-PeCB)	ND	0.4	0.1	0.01	0.000003
#114 (2,3,4,4',5-PeCB)	ND	0.6	0.2	0.01	0.000006
#167 (2,3',4,4',5,5'-HxCB)	ND	0.9	0.3	0.01	0.000009
#156 (2,3,3',4,4',5-HxCB)	ND	0.28	0.08	0.01	0.0000024
#157 (2,3,3',4,4',5'-HxCB)	ND	0.7	0.2	0.01	0.000006
#189 (2,3,3',4,4',5,5'-HpCB)	ND	1.0	0.3	0.01	0.000009
#28 (2,4,4'-TrCB)	1.1	0.23	0.07	0.02	-
#52 (2,2',5,5'-TeCB)	(0.5)	0.7	0.2	0.02	-
#101 (2,2',4,5,5'-PeCB)	(0.2)	0.5	0.1	0.02	-
#138 (2,2',3,4,4',5'-HxCB)	ND	0.6	0.2	0.01	-
#153 (2,2',4,4',5,5'-HxCB)	ND	0.6	0.2	0.01	-
#180 (2,2',3,4,4',5,5'-HpCB)	ND	0.7	0.2	0.02	-

* 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 are 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 are calculated with the LOD.

Table 3. Recoveries of labeled internal standards.

Congener	Sample
2,3,7,8-TeCDD	71 %
1,2,3,7,8-PeCDD	74 %
1,2,3,4,7,8-HxCDD	81 %
1,2,3,6,7,8-HxCDD	85 %
1,2,3,7,8,9-HxCDD	75 %
1,2,3,4,6,7,8-HpCDD	88 %
OCDD	86 %
2,3,7,8-TeCDF	75 %
1,2,3,7,8-PeCDF	83 %
2,3,4,7,8-PeCDF	76 %
1,2,3,4,7,8-HxCDF	88 %
1,2,3,6,7,8-HxCDF	91 %
1,2,3,7,8,9-HxCDF	80 %
2,3,4,6,7,8-HxCDF	78 %
1,2,3,4,6,7,8-HpCDF	90 %
1,2,3,4,7,8,9-HpCDF	91 %
OCDF	91 %
#81 (3,4,4',5-TeCB)	86 %
#77 (3,3',4,4'-TeCB)	89 %
#126 (3,3',4,4',5-PeCB)	77 %
#169 (3,3',4,4',5,5'-HxCB)	82 %
#123 (2',3,4,4',5-PeCB)	84%
#118 (2,3',4,4',5-PeCB)	92%
#105 (2,3,3',4,4'-PeCB)	80%
#114 (2,3,4,4',5-PeCB)	85%
#167 (2,3',4,4',5,5'-HxCB)	90%
#156 (2,3,3',4,4',5-HxCB)	80%
#157 (2,3,3',4,4',5'-HxCB)	74%
#189 (2,3,3',4,4',5,5'-HpCB)	71%
#28 (2,4,4'-TrCB)	90%
#52 (2,2',5,5'-TeCB)	92%
#101 (2,2',4,5,5'-PeCB)	92%
#138 (2,2',3,4,4',5'-HxCB)	89%
#153 (2,2',4,4',5,5'-HxCB)	96%
#180 (2,2',3,4,4',5,5'-HpCB)	98%

Compound View

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Injection : B9E237001X

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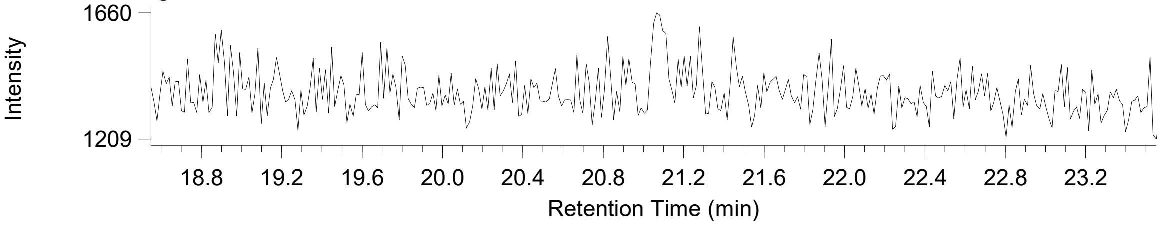

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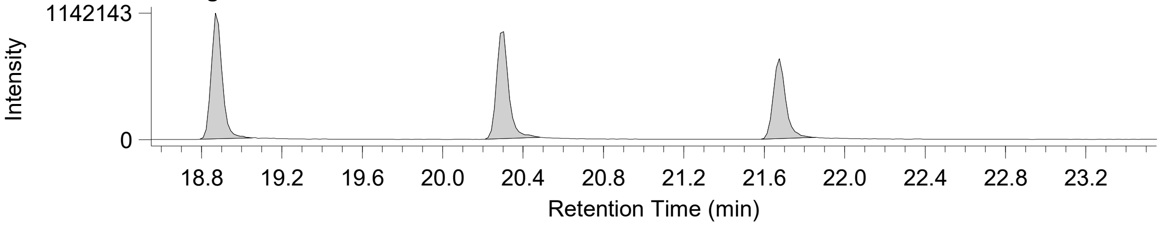

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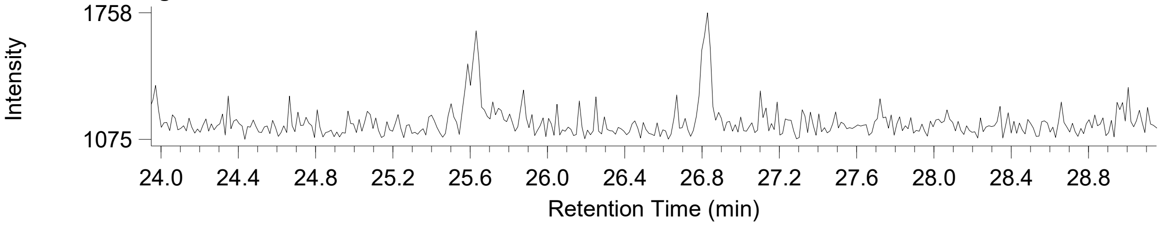

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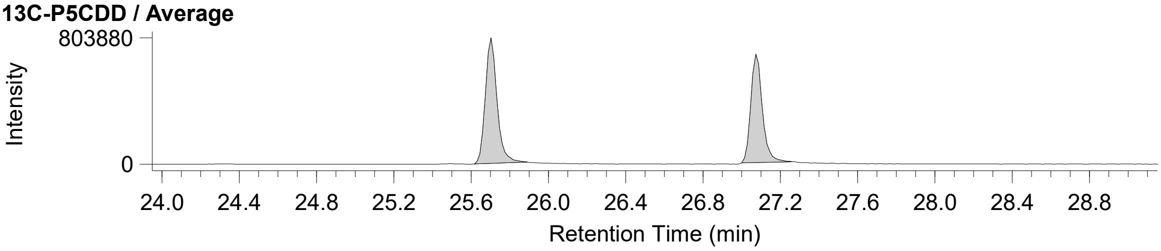

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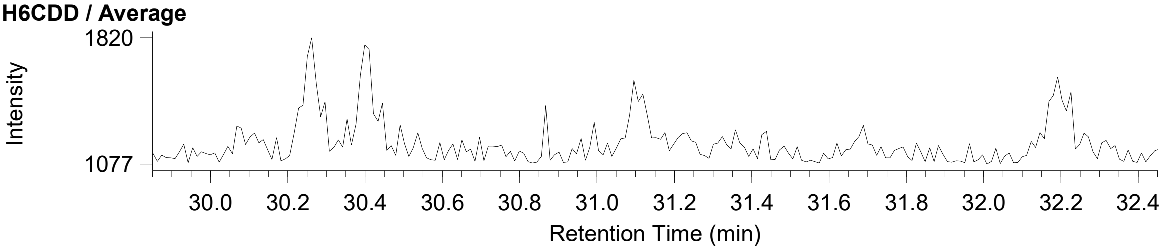

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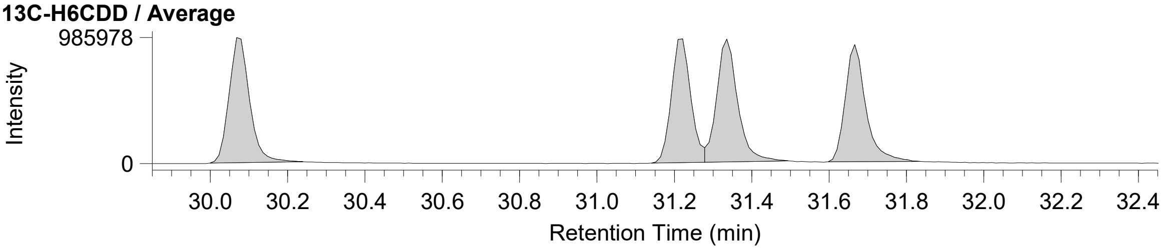

Compound View

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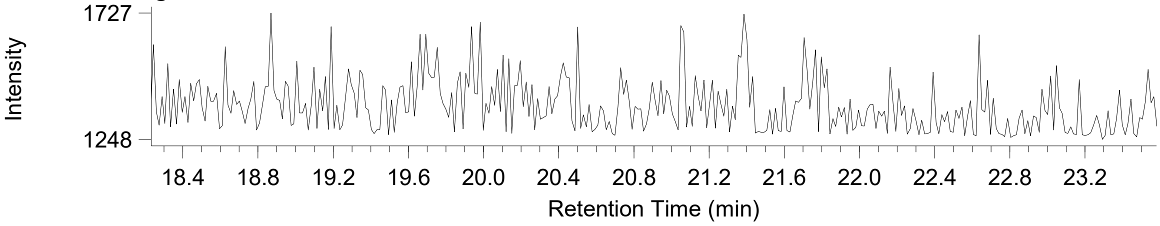

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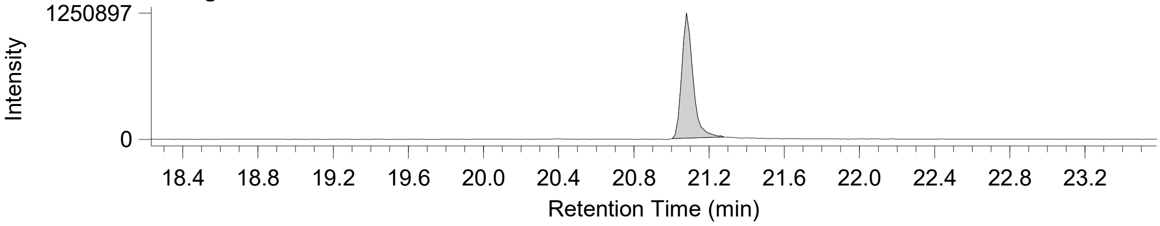

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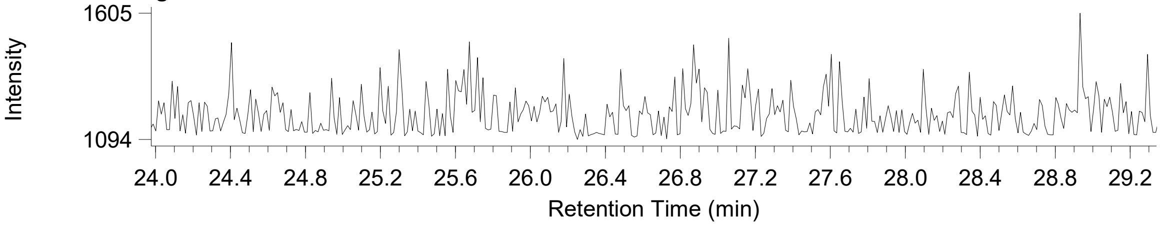

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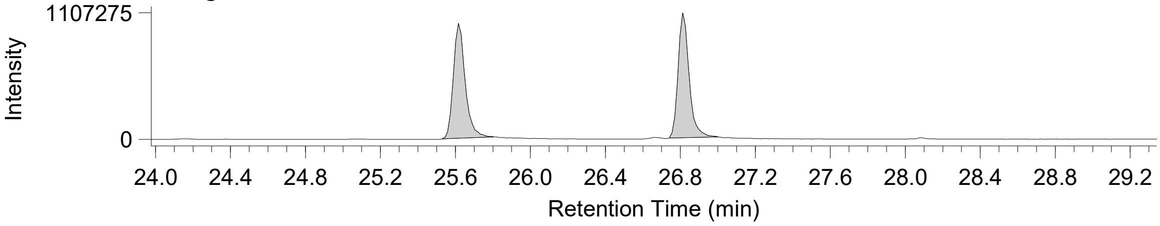

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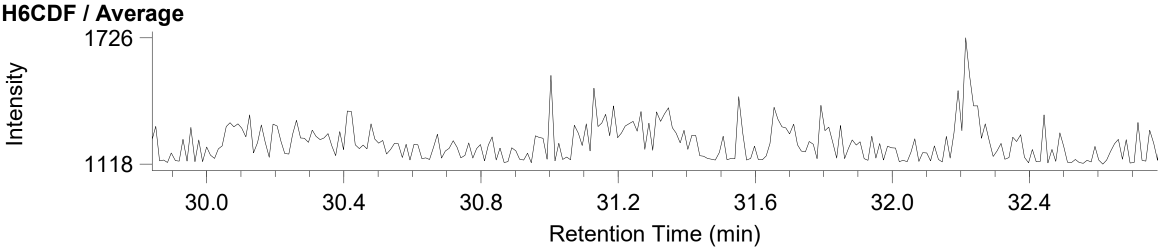

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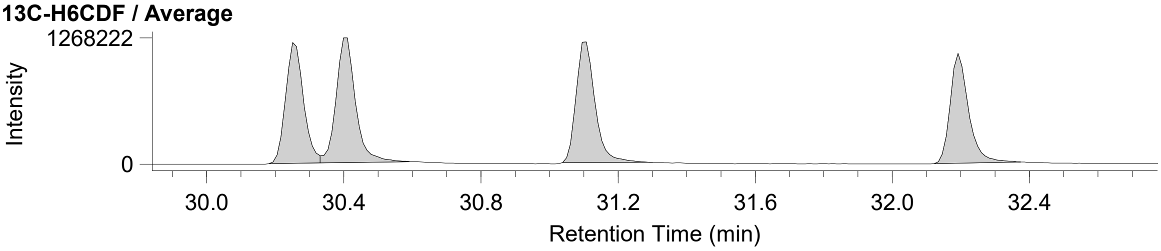

Compound View

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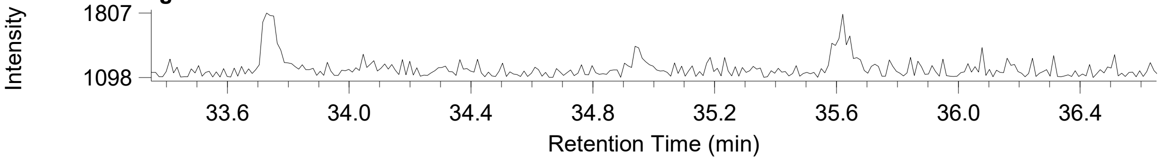
H7CDD / Average



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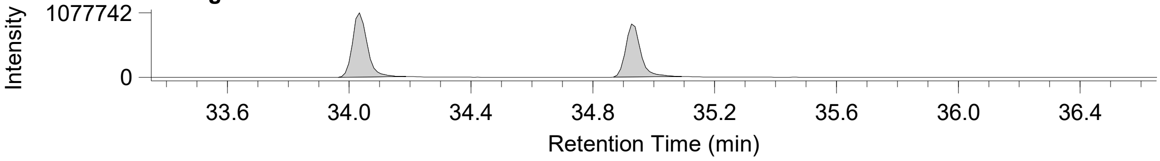
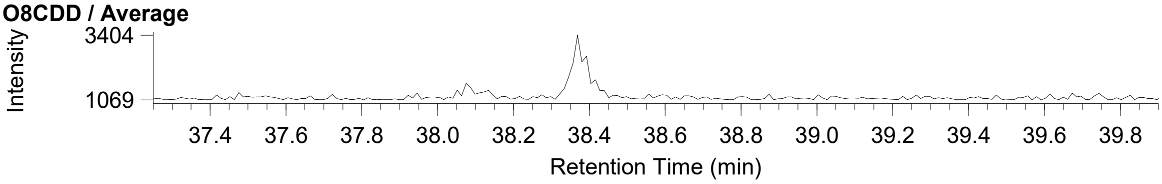


O8CDD / Average



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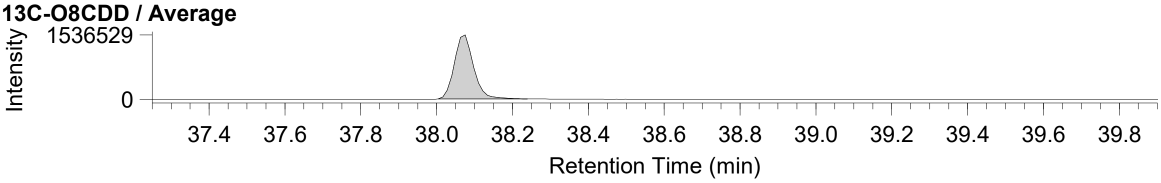

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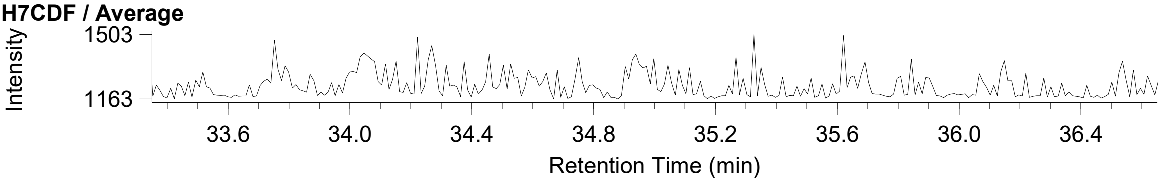

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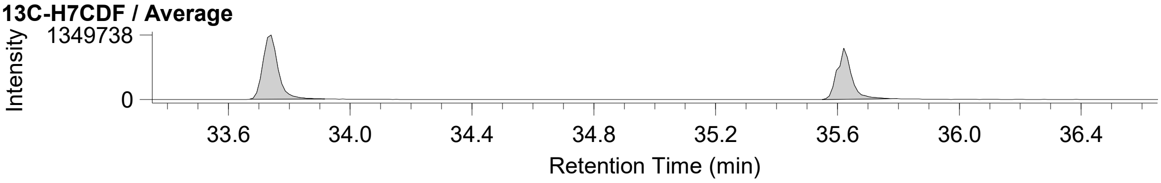

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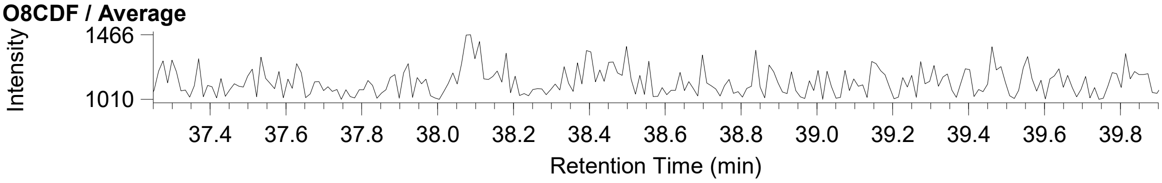

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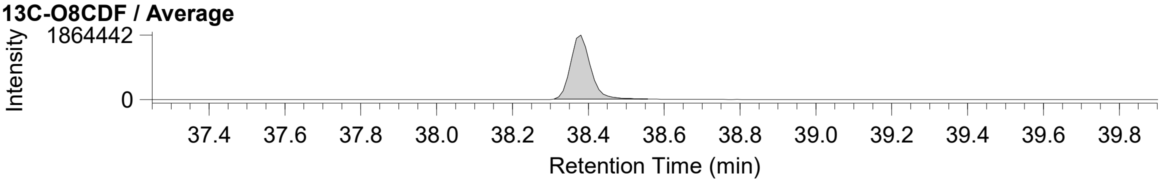

Compound View

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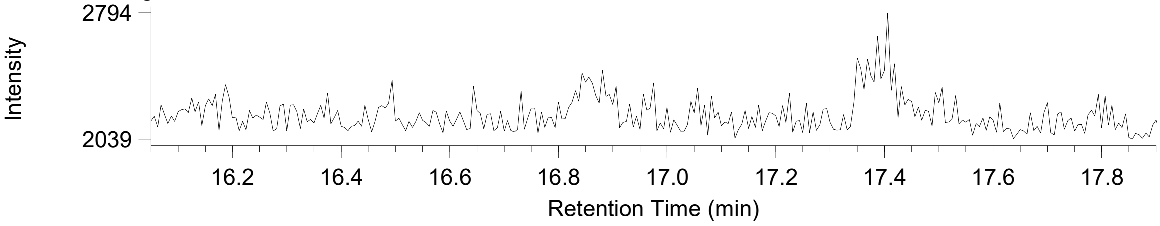
T4CB / Average



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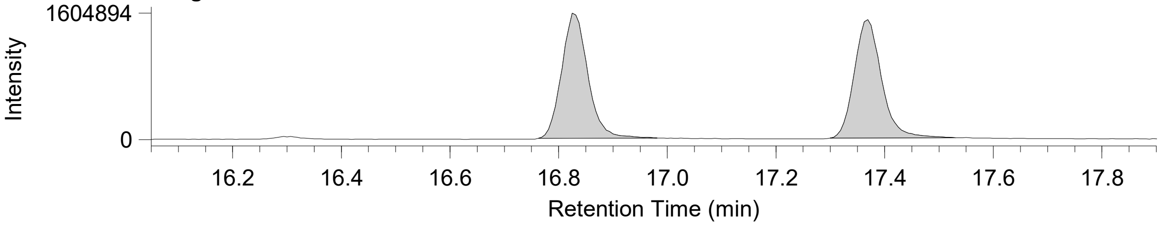
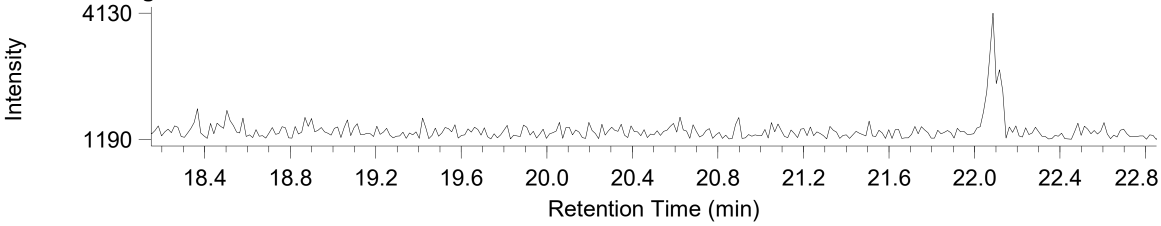


P5CB / Average



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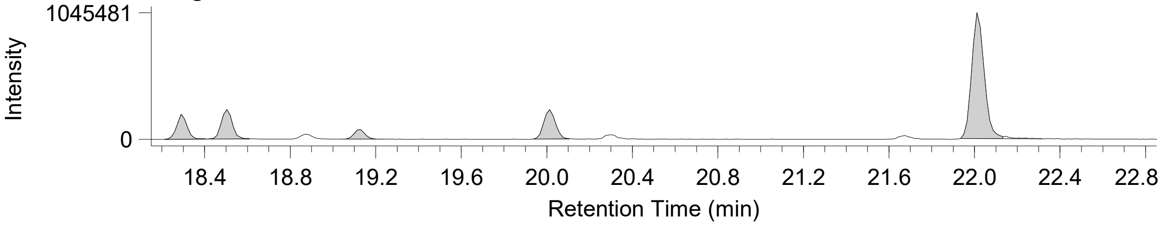
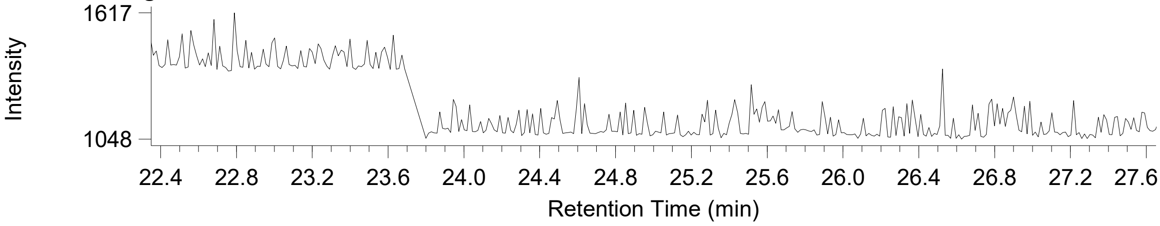


H6CB / Average



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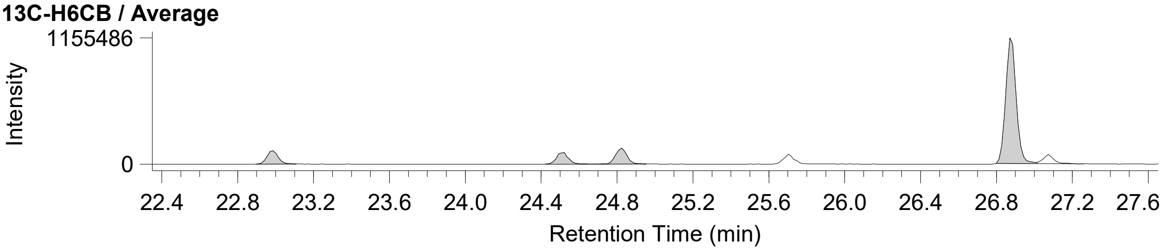

Compound View

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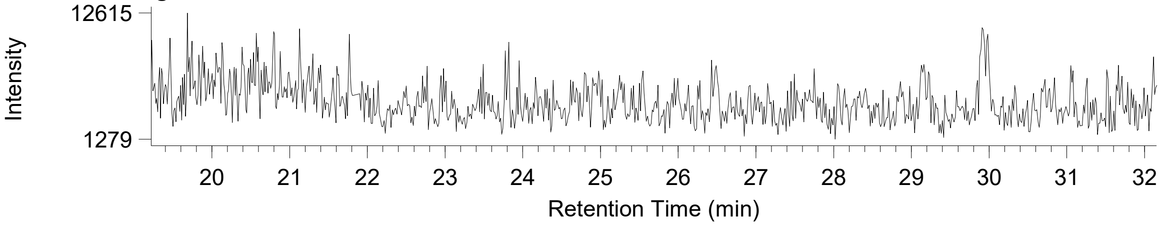
Injection : B9E237002X

D2CBs / Average



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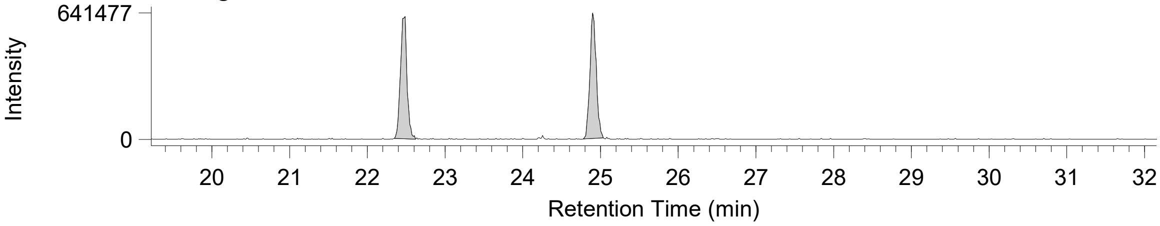
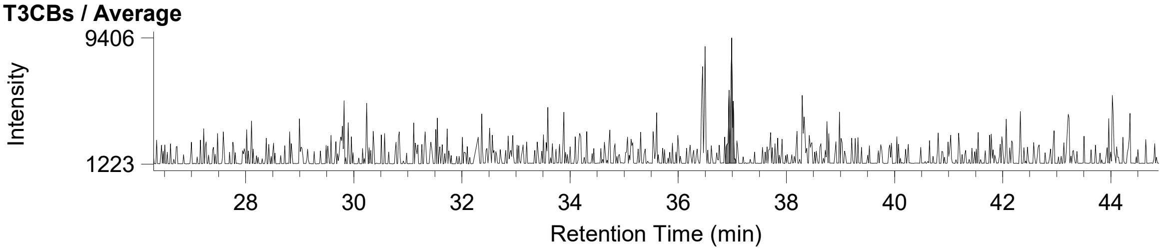
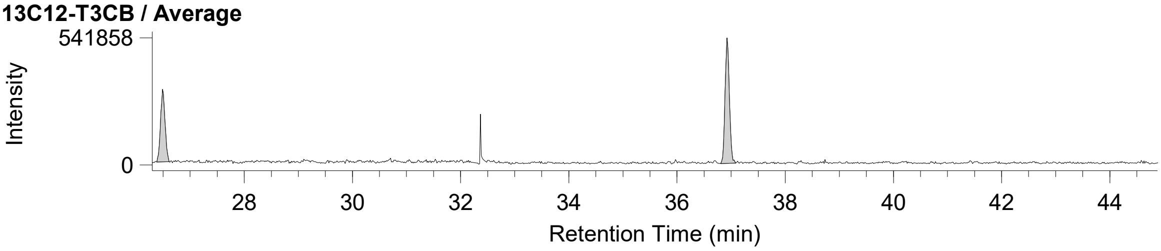


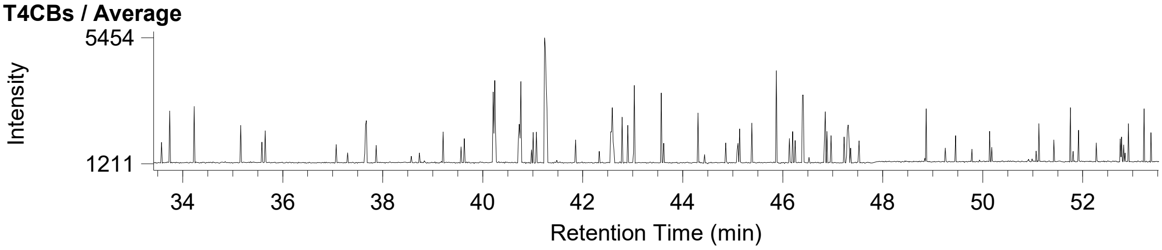
T3CBs / Average



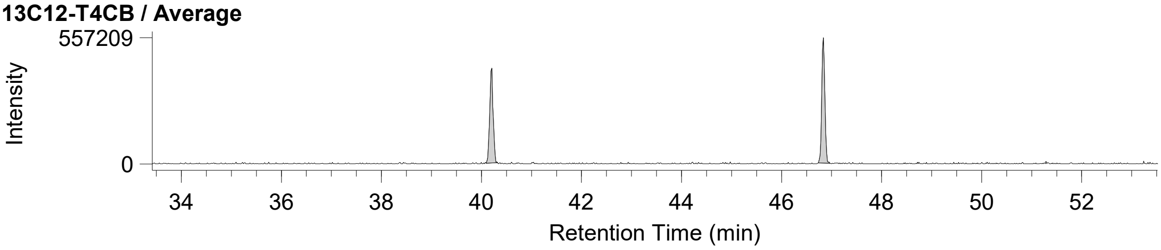
13C12-T3CB / Average



T4CBs / Average



13C12-T4CB / Average



Compound View

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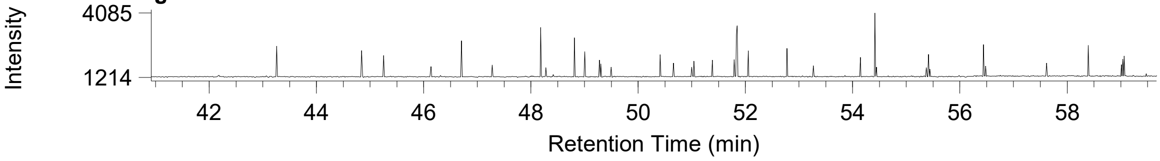
P5CBs / Average



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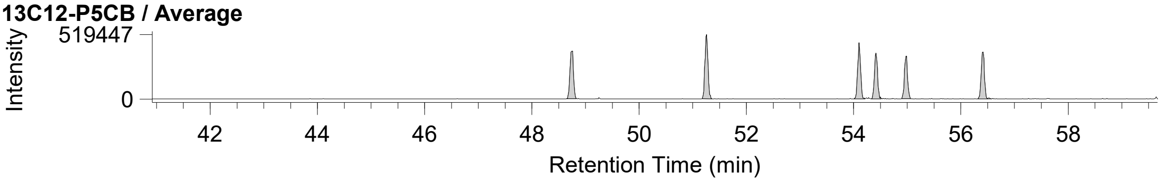
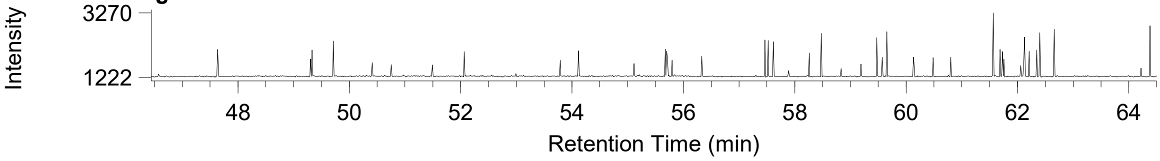


H6CBs / Average



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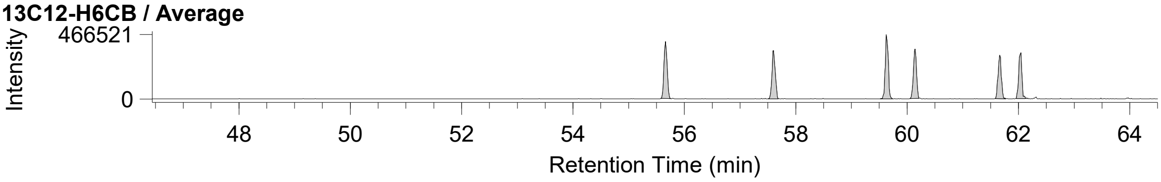

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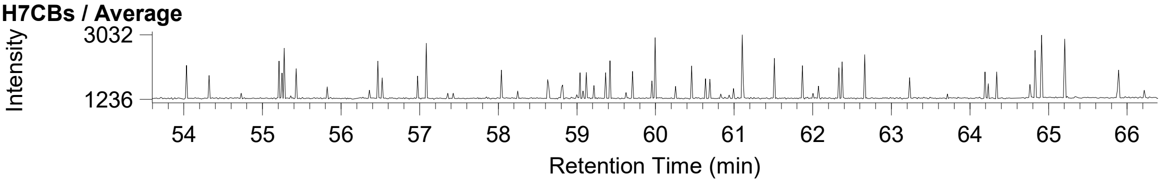

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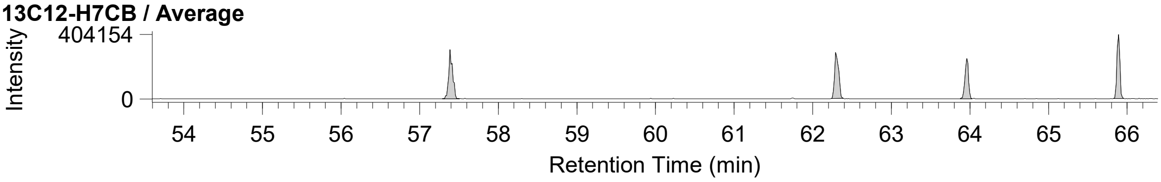

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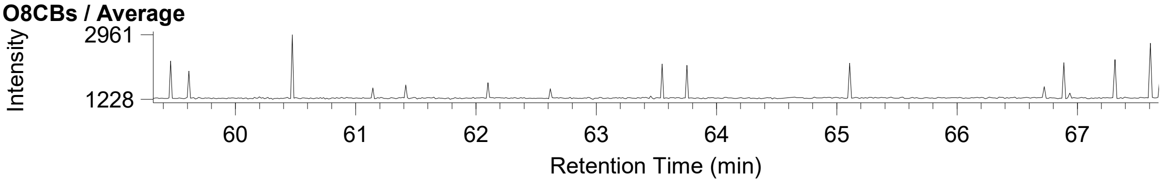

13C12-O8CB / Average