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), polychlorinated dibenzofurans (PCDFs), and polychlorinated biphenyl (PCB) congeners, in food/feed, environmental matrices, and similar matrices.

Material	18Φ Column set GO-209PCB
Product Code	P10000270467
Lot No.	243708
Expiration Date	Dec/2026

Tests	Result	Criteria
Blank Values of PCDDs/PCDFs pg-TEQ/column set	< 0.92	< 2.5
Blank values of DL-PCBs pg-TEQ/column set	< 0.048	< 0.25
Blank values of NDL-PCBs pg-congener /column set	ND	Each isomer < 50
Blank values of other PCBs pg-congener /column set*	The highest isomer (#11) < 4.8	Each isomer < 50
Recovery PCDDs/PCDFs	84 to 104 %	60 to 120 %
Native Recovery Toxics/LOC/window difining PCB congeners	80 to 114 %	60 to 120 %

*: This congener was detected with the highest level among PCB congeners detected in this analysis.
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, for selected PCB congeners, selected 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 the selected mass labeled PCDD and PCDF congeners, based on the selected mass labeled recovery standards for PCDD and PCDF added before GC-MS measurement, when the blank test was implemented. The recovery values of PCBs, also expressed as percentages, are shown in Table 4 for the native toxics/LOC PCB congeners described in EPA method 1668C and the selected PCB congeners is required by EU regulations, based on a known amount of the native PCB standard solution. The recoveries meet the MIURA criteria for this certification.

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) 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 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 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 1 mL of n-hexane was added to wet the top of the column. Then, a known amount of internal standard solution (containing selected labeled PCB, PCDDs, and PCDFs congeners; as shown in Table 1) was added to the top of the column. 1 mL of n-hexane was added to the column two times 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 to 0.02 mL.

Table 1. Standard solutions used for recovery tests.

Compounds	Standard	Maker Code	Maker	Diluted Concentration
PCDDs and PCDFs	Internal Standard	DF-LCS-B	Wellington Laboratories Inc.	10 ng/mL in decane
	Recovery (Surrogate) Standard	DF-IS-J		
DL-PCBs , NDL-PCBs	Internal Standard (Labeled toxics/LOC)	TPCB-LCS-A500		
	Recovery (Surrogate) Standard	TPCB-IS-A-STK		

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 for dioxin measuremant, after that it was analyzed using GC/HRMS operated in EI mode. A 0.25 mm ID × 60 m fused silica capillary (HT8-PCB, TRAJAN) was used for PCB measurement. All injections were 2 µL using a splitless inlet. The results, blank level values (PCDDs, PCDFs, DL-PCBs, and NDL-PCBs) and the recoveries of labeled compounds (PCDDs and PCDFs), are provided in Table 2 and Table 3, respectively. The chromatograms of each compounds are shown at page 7 and after. Furthermore, the mixed concentrated solution 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.

For the recovery test, several column sets chosen at random per lot production were allowed to reach ambient temperature; the purification columns (upper) and (lower) were assembled. 1 mL of n-hexane was added to wet the top of the column. The native PCB standard solution (model, BP-MS by Wellington Laboratories Inc.) is diluted to 10ng/mL with decane. 0.02 mL of the diluted standard solution was added in the top of the column, subsequently 0.2 mL of toluene was applied. And then, 1 mL of n-hexane was added in the column two times 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 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 recoveries of native toxics/LOC/window difining PCB congeners are displayed in Table 4.

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	0.5	0.2	0.08	0.2
1,2,3,7,8-PeCDD	ND	0.5	0.2	0.1	0.2
1,2,3,4,7,8-HxCDD	ND	1.4	0.4	0.2	0.04
1,2,3,6,7,8-HxCDD	ND	1.7	0.5	0.2	0.05
1,2,3,7,8,9-HxCDD	ND	1.2	0.4	0.2	0.04
1,2,3,4,6,7,8-HpCDD	ND	1.8	0.5	0.2	0.005
OCDD	ND	2.5	0.7	0.1	0.00021
2,3,7,8-TeCDF	ND	1.8	0.5	0.1	0.05
1,2,3,7,8-PeCDF	ND	1.8	0.5	0.09	0.015
2,3,4,7,8-PeCDF	ND	1.4	0.4	0.09	0.12
1,2,3,4,7,8-HxCDF	ND	1.8	0.5	0.09	0.05
1,2,3,6,7,8-HxCDF	ND	1.9	0.6	0.08	0.06
1,2,3,7,8,9-HxCDF	ND	1.4	0.4	0.09	0.04
2,3,4,6,7,8-HxCDF	ND	1.3	0.4	0.08	0.04
1,2,3,4,6,7,8-HpCDF	ND	2.3	0.7	0.09	0.007
1,2,3,4,7,8,9-HpCDF	ND	2.3	0.7	0.1	0.007
OCDF	ND	3.0	0.9	0.2	0.00027
#81 (3,4,4',5-TeCB)	ND	1.3	0.4	0.02	0.00012
#77 (3,3',4,4'-TeCB)	ND	1.3	0.4	0.03	0.00004
#126 (3,3',4,4',5-PeCB)	ND	1.4	0.4	0.03	0.04
#169 (3,3',4,4',5,5'-HxCB)	ND	1.5	0.5	0.02	0.015
#123 (2',3,4,4',5-PeCB)	ND	1.4	0.4	0.02	0.000012
#118 (2,3',4,4',5-PeCB)	ND	0.9	0.3	0.02	0.000009
#105 (2,3,3',4,4'-PeCB)	ND	2.1	0.6	0.02	0.000018
#114 (2,3,4,4',5-PeCB)	ND	1.5	0.5	0.02	0.000015
#167 (2,3',4,4',5,5'-HxCB)	ND	1.3	0.4	0.02	0.000012
#156 (2,3,3',4,4',5-HxCB)	ND	1.2	0.4	0.02	0.000012
#157 (2,3,3',4,4',5'-HxCB)	ND	1.6	0.5	0.02	0.000015
#189 (2,3,3',4,4',5,5'-HpCB)	ND	1.3	0.4	0.01	0.000012
#28 (2,4,4'-TrCB)	(0.6)	1.4	0.4	0.02	-
#52 (2,2',5,5'-TeCB)	ND	1.3	0.4	0.03	-
#101 (2,2',4,5,5'-PeCB)	ND	1.2	0.4	0.02	-
#138 (2,2',3,4,4',5'-HxCB)	ND	1.2	0.4	0.02	-
#153 (2,2',4,4',5,5'-HxCB)	ND	2.7	0.8	0.02	-
#180 (2,2',3,4,4',5,5'-HpCB)	ND	2.3	0.7	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	Recovery
2,3,7,8-TeCDD	84 %
1,2,3,7,8-PeCDD	94 %
1,2,3,4,7,8-HxCDD	94 %
1,2,3,6,7,8-HxCDD	98 %
1,2,3,7,8,9-HxCDD	91 %
1,2,3,4,6,7,8-HpCDD	100 %
OCDD	100 %
2,3,7,8-TeCDF	92 %
1,2,3,7,8-PeCDF	99 %
2,3,4,7,8-PeCDF	101 %
1,2,3,4,7,8-HxCDF	92 %
1,2,3,6,7,8-HxCDF	94 %
1,2,3,7,8,9-HxCDF	101 %
2,3,4,6,7,8-HxCDF	99 %
1,2,3,4,6,7,8-HpCDF	97 %
1,2,3,4,7,8,9-HpCDF	103 %
OCDF	104 %

Table 4. Recoveries of native toxics/LOC/window difining PCB congeners.

Congener Name	No.	Recovery
2-MoCB	1	89%
4-MoCB	3	87%
2,2'-DiCB	4	108%
4,4'-DiCB	15	103%
2,2'6-TrCB	19	110%
2,4,4'-TrCB	28	105%
3,4,4'-TrCB	37	94%
2,2',5,5'-TeCB	52	110%
2,2'6,6'-TeCB	54	111%
3,3',4,4'-TeCB	77	89%
3,4,4',5-TeCB	81	93%
2,2',4,5,5'-PeCB	101	108%
2,2',4,6,6'-PeCB	104	108%
2,3,3',4,4'-PeCB	105	108%
2,3,4,4',5-PeCB	114	111%
2,3',4,4',5-PeCB	118	113%
2',3,4,4',5-PeCB	123	105%
3,3',4,4',5-PeCB	126	90%
2,2',3,4,4',5'-HxCB	138	110%
2,2',4,4',5,5'-HxCB	153	105%
2,2',4,4',6,6'-HxCB	155	114%
2,3,3',4,4',5-HxCB	156	105%
2,3,3',4,4',5'-HxCB	157	107%
2,3',4,4',5,5'-HxCB	167	103%
3,3',4,4',5,5'-HxCB	169	80%
2,2',3,4,4',5,5'-HpCB	180	111%
2,2',3,4',5,6,6'-HpCB	188	111%
2,3,3',4,4',5,5'-HpCB	189	111%
2,2',3,3',5,5',6,6'-OcCB	202	108%
2,3,3',4,4',5,5',6-OcCB	205	106%
2,2',3,3',4,4',5,5',6-NoCB	206	110%
2,2',3,3,'4,5,5',6,6'-NoCB	208	106%
DeCB	209	108%

Compound View

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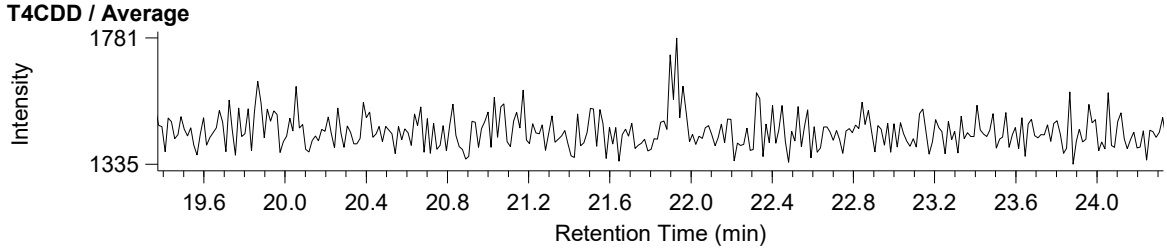

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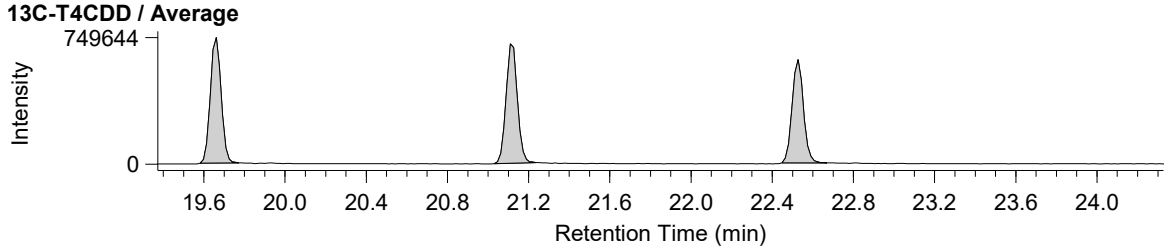

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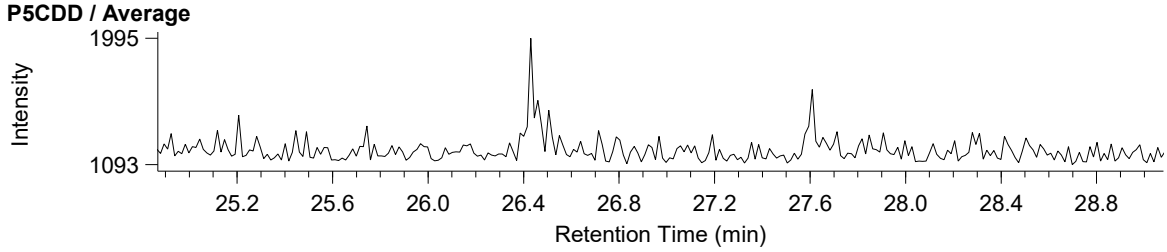

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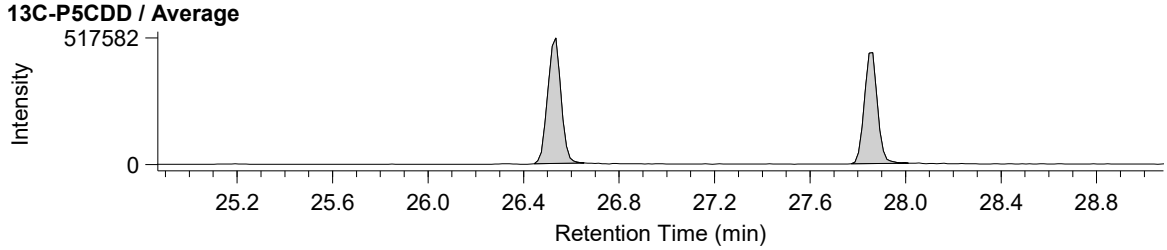

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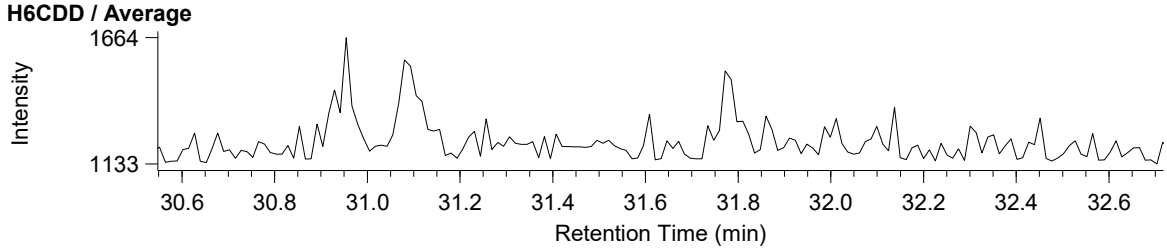

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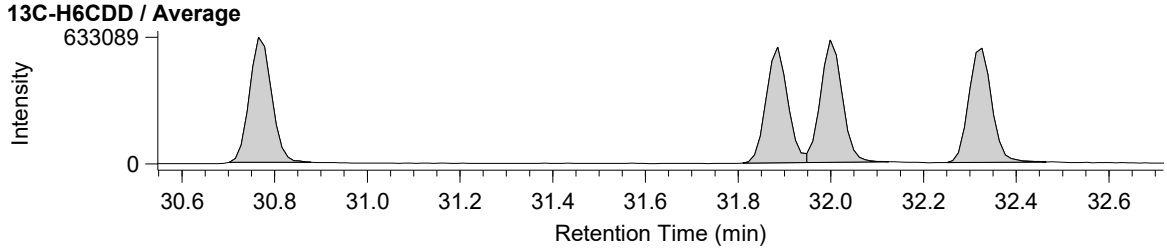

Compound View

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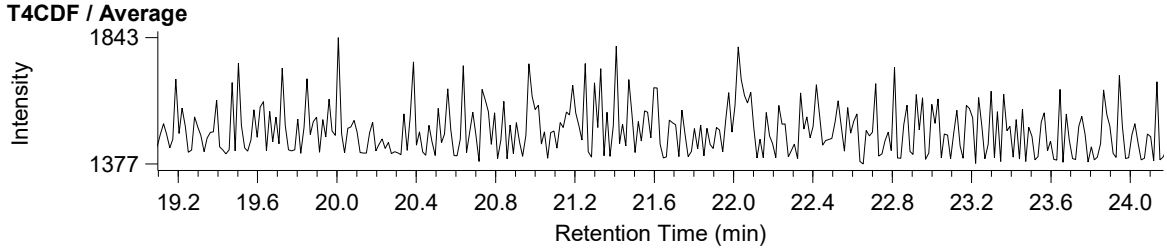

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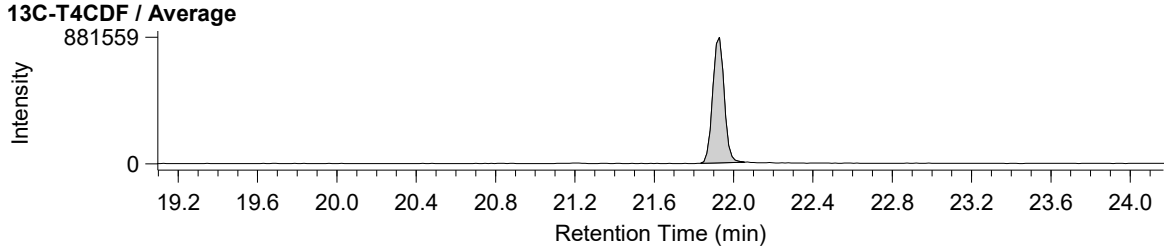

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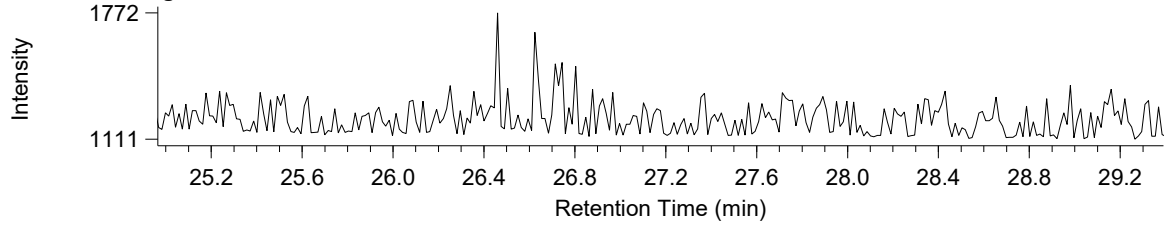

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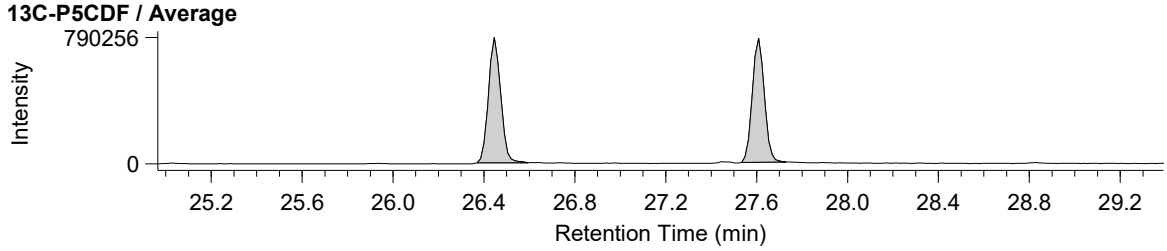

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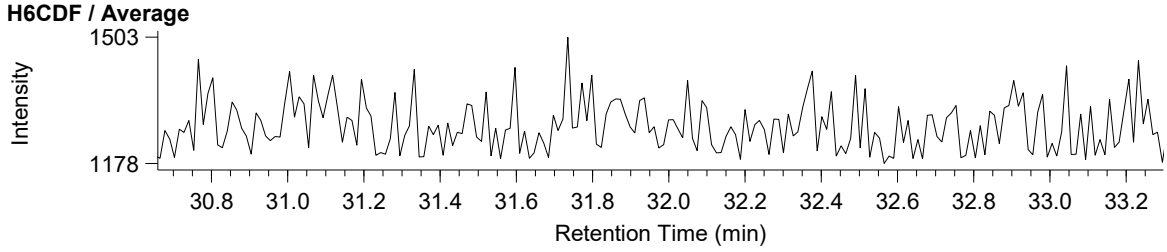

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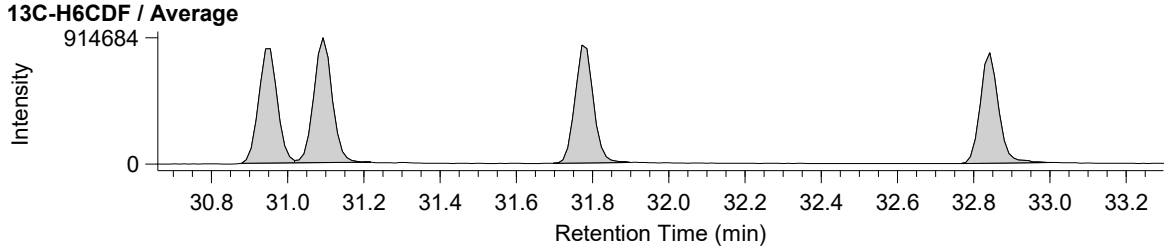

Compound View

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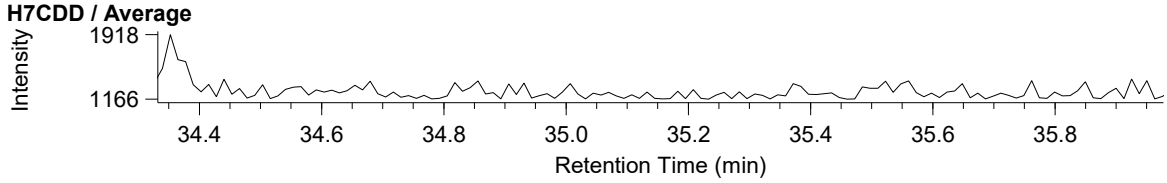

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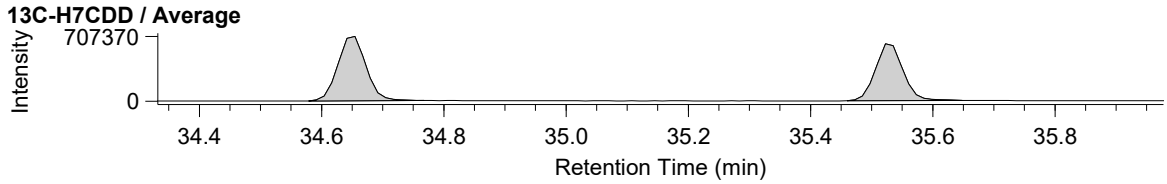

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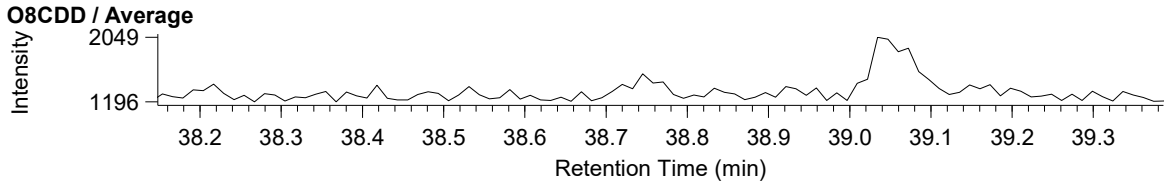

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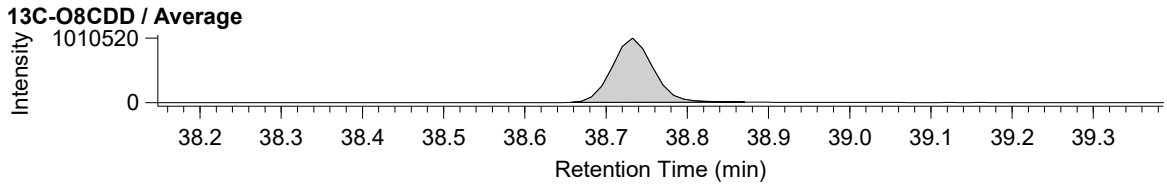

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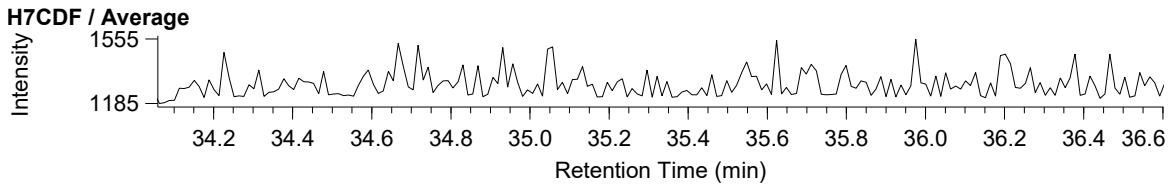

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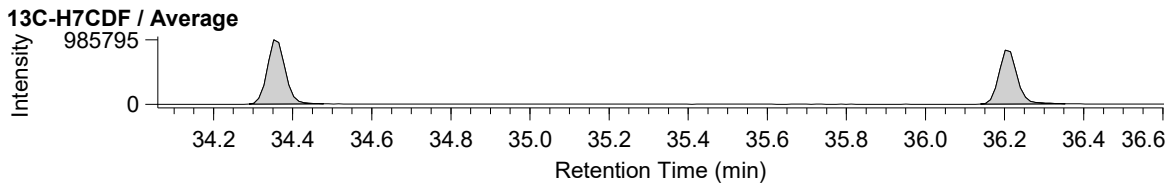

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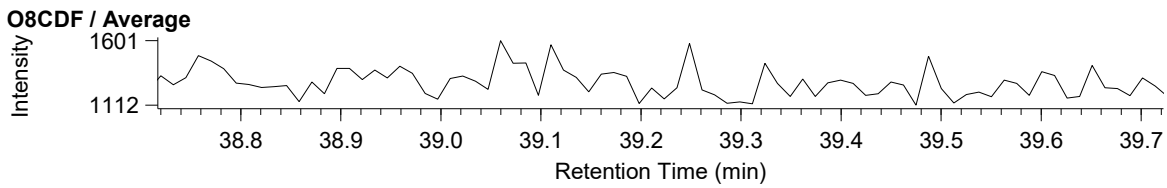

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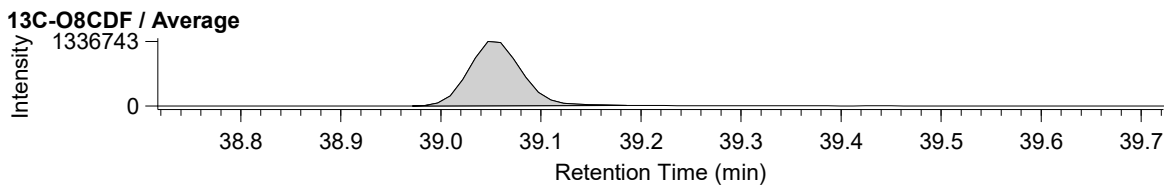

Compound View

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Injection : C4E409002X

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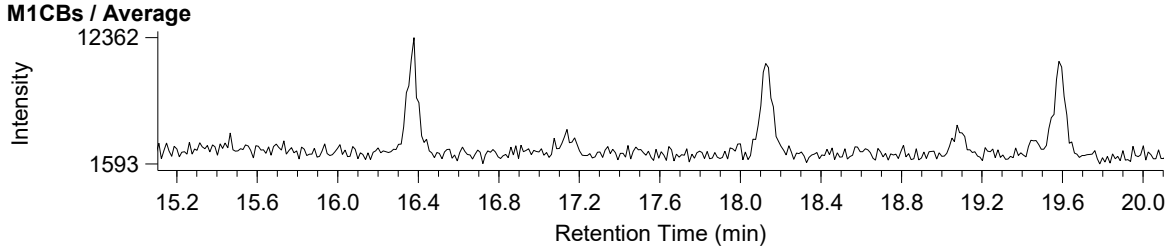

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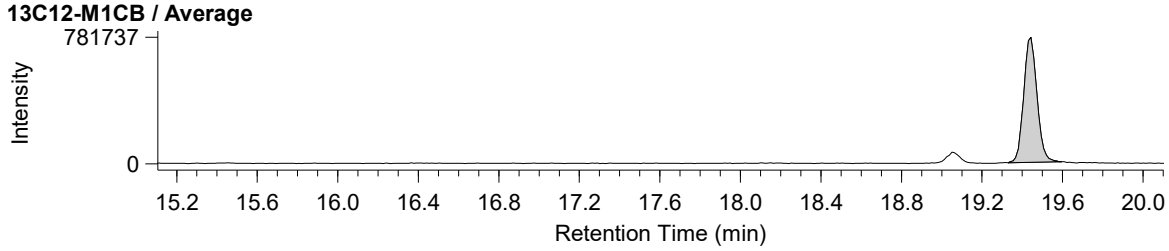

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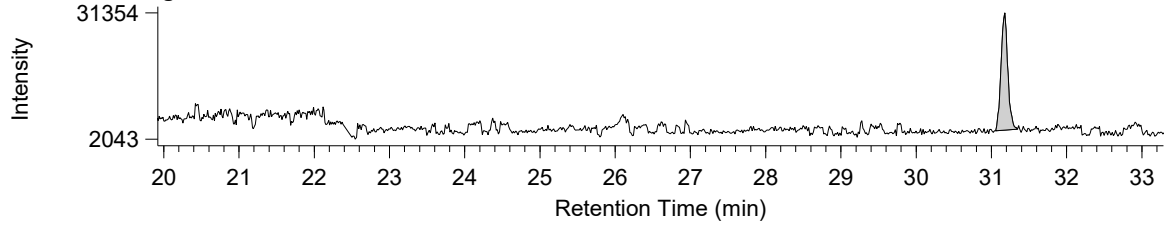

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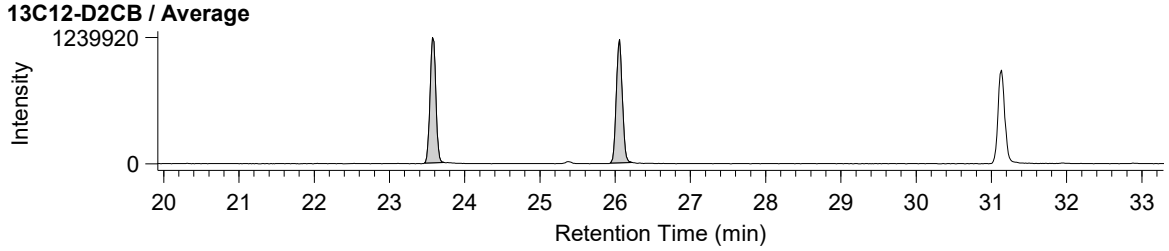
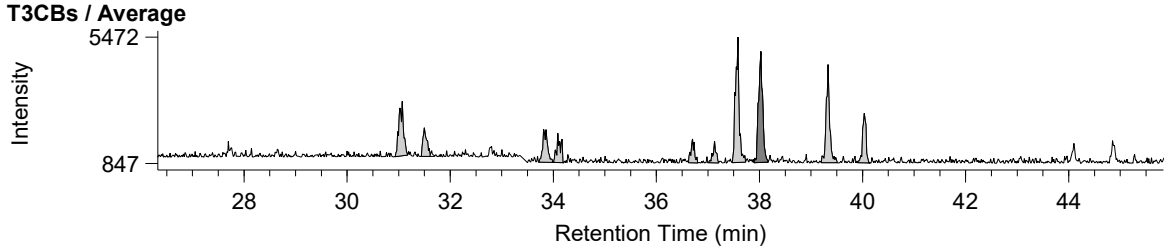
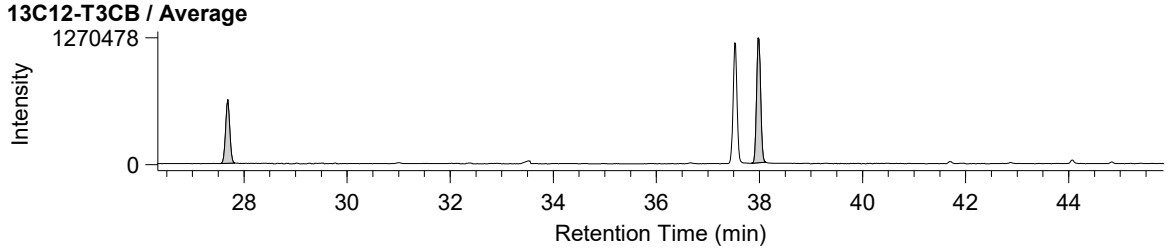


T3CBs / Average



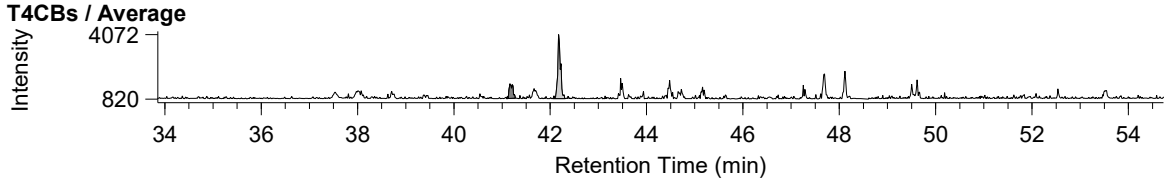
13C12-T3CB / Average



Compound View

DqData : m:\DioK\DqData\2024\C4E409\HT8-PCB(1-10)-105-1
Injection : C4E409002X

T4CBs / Average



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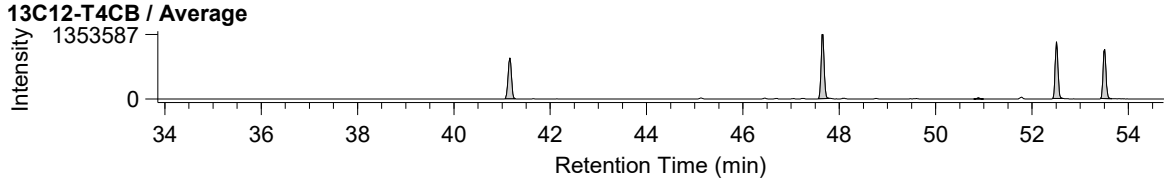
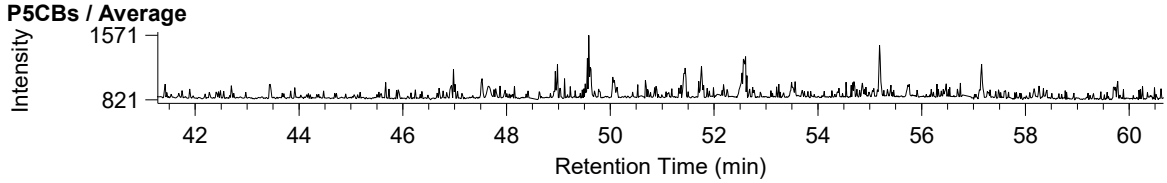
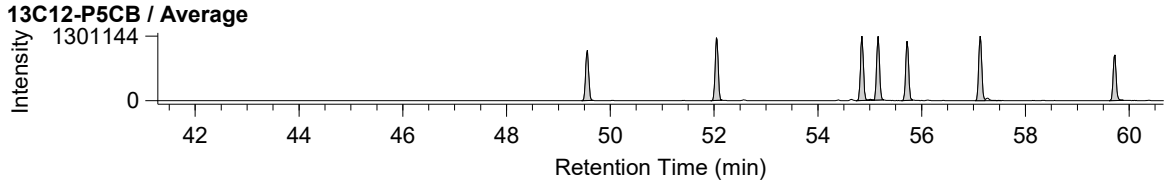


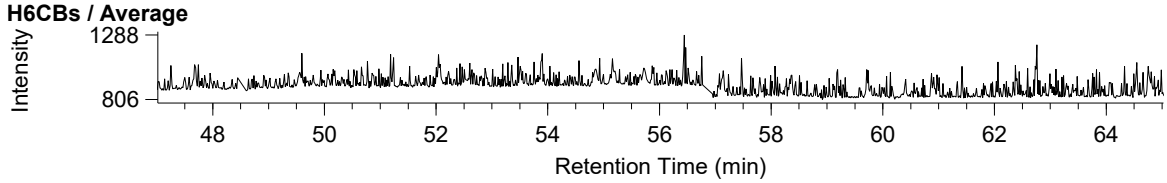
P5CBs / Average



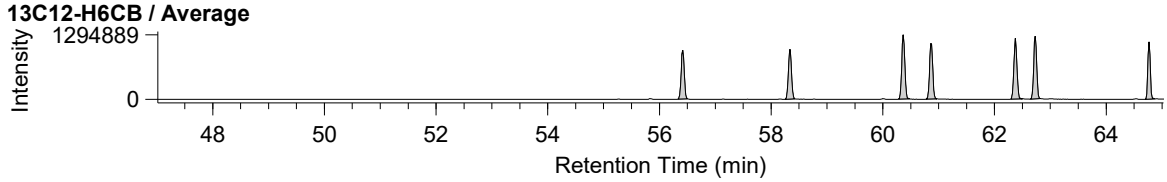
13C12-P5CB / Average



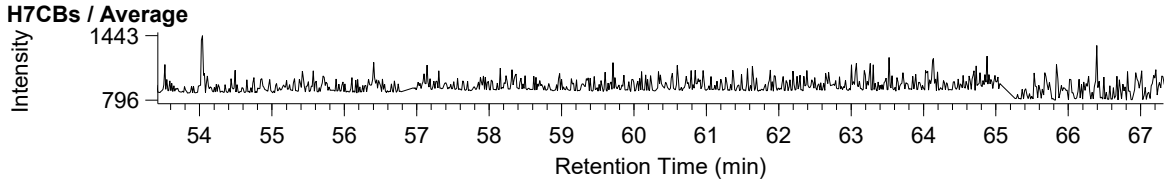
H6CBs / Average



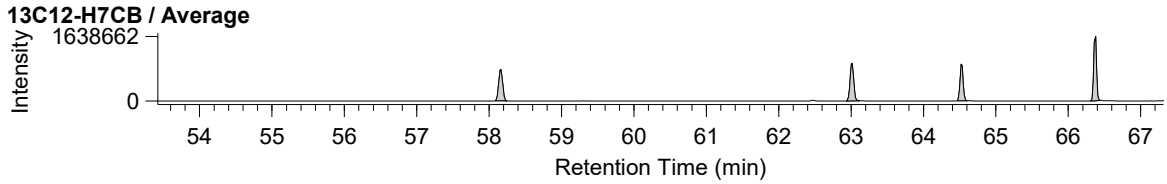
13C12-H6CB / Average



H7CBs / Average



13C12-H7CB / Average



Compound View

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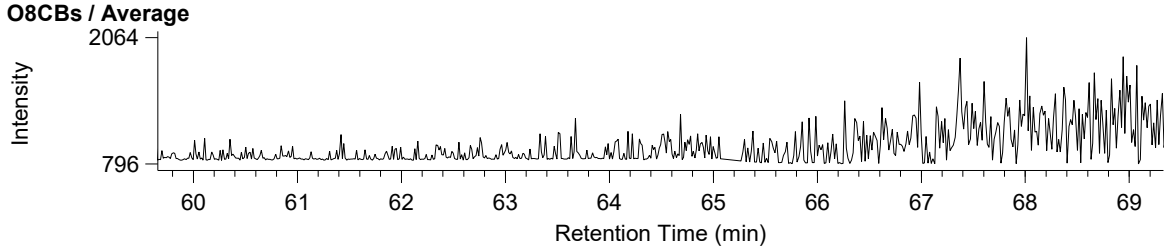
Injection : C4E409002X

O8CBs / Average



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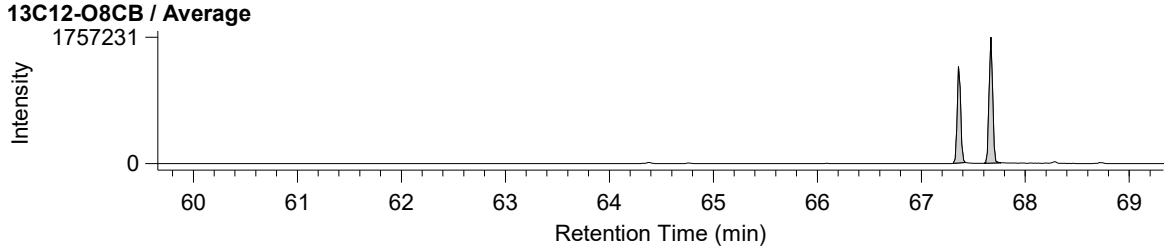
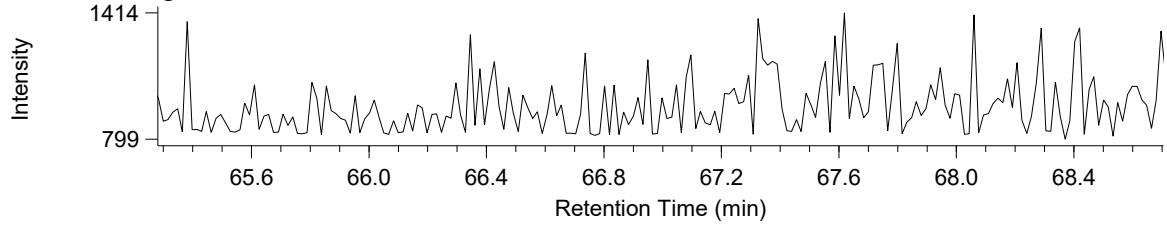


N9CBs / Average



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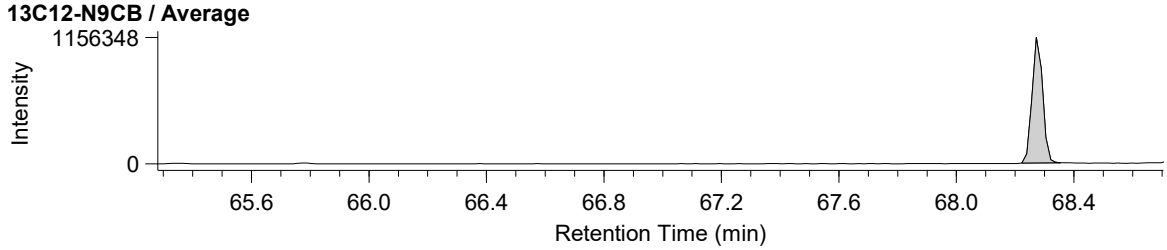
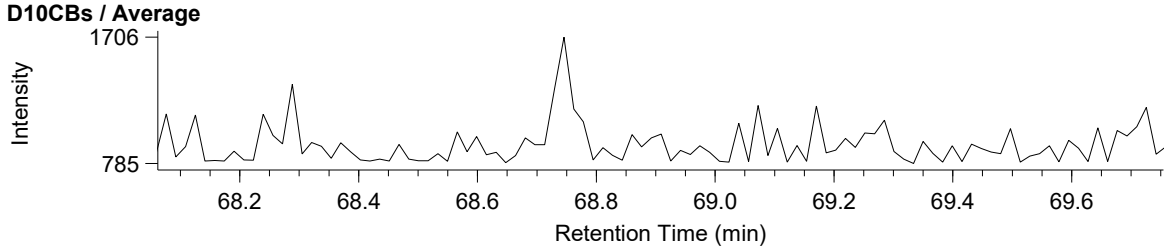


D10CBs / Average



13C12-D10CB / Average