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.	243711
Expiration Date	Jul/2026

Tests	Result	Criteria
Blank Values of PCDDs/PCDFs pg-TEQ/column set	< 0.68	< 2.5
Blank values of DL-PCBs pg-TEQ/column set	< 0.085	< 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 (#3) < 13	Each isomer < 50
Recovery PCDDs/PCDFs	88 to 108 %	60 to 120 %
Native Recovery Toxics/LOC/window difining PCB congeners	67 to 113 %	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	0.2	0.2
1,2,3,7,8-PeCDD	ND	0.5	0.1	0.09	0.1
1,2,3,4,7,8-HxCDD	ND	0.8	0.2	0.2	0.02
1,2,3,6,7,8-HxCDD	ND	1.5	0.5	0.2	0.05
1,2,3,7,8,9-HxCDD	ND	0.9	0.3	0.2	0.03
1,2,3,4,6,7,8-HpCDD	ND	1.5	0.4	0.2	0.004
OCDD	ND	2.6	0.8	0.3	0.00024
2,3,7,8-TeCDF	ND	1.4	0.4	0.1	0.04
1,2,3,7,8-PeCDF	ND	1.7	0.5	0.3	0.015
2,3,4,7,8-PeCDF	ND	0.6	0.2	0.2	0.06
1,2,3,4,7,8-HxCDF	ND	1.3	0.4	0.09	0.04
1,2,3,6,7,8-HxCDF	ND	1.3	0.4	0.09	0.04
1,2,3,7,8,9-HxCDF	ND	1.2	0.4	0.1	0.04
2,3,4,6,7,8-HxCDF	ND	1.1	0.3	0.09	0.03
1,2,3,4,6,7,8-HpCDF	ND	2.1	0.6	0.09	0.006
1,2,3,4,7,8,9-HpCDF	ND	1.4	0.4	0.1	0.004
OCDF	ND	3	1	0.3	0.0003
#81 (3,4,4',5-TeCB)	ND	2.0	0.6	0.1	0.00015
#77 (3,3',4,4'-TeCB)	ND	2.4	0.7	0.1	0.0001
#126 (3,3',4,4',5-PeCB)	ND	2.3	0.7	0.1	0.07
#169 (3,3',4,4',5,5'-HxCB)	ND	1.7	0.5	0.1	0.015
#123 (2',3,4,4',5-PeCB)	ND	1.7	0.5	0.08	0.000015
#118 (2,3',4,4',5-PeCB)	ND	2.8	0.8	0.08	0.000024
#105 (2,3,3',4,4'-PeCB)	ND	3.1	0.9	0.08	0.000027
#114 (2,3,4,4',5-PeCB)	ND	3.0	0.9	0.08	0.000027
#167 (2,3',4,4',5,5'-HxCB)	ND	2.9	0.9	0.08	0.000027
#156 (2,3,3',4,4',5-HxCB)	ND	3.0	0.9	0.07	0.000027
#157 (2,3,3',4,4',5'-HxCB)	ND	2.4	0.7	0.07	0.000021
#189 (2,3,3',4,4',5,5'-HpCB)	ND	4	1	0.06	0.00003
#28 (2,4,4'-TrCB)	ND	3	1	0.09	-
#52 (2,2',5,5'-TeCB)	ND	1.5	0.5	0.1	-
#101 (2,2',4,5,5'-PeCB)	ND	2.7	0.8	0.09	-
#138 (2,2',3,4,4',5'-HxCB)	ND	2.0	0.6	0.09	-
#153 (2,2',4,4',5,5'-HxCB)	ND	3	1	0.09	-
#180 (2,2',3,4,4',5,5'-HpCB)	ND	2.0	0.6	0.1	-

* 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	88 %
1,2,3,7,8-PeCDD	96 %
1,2,3,4,7,8-HxCDD	103 %
1,2,3,6,7,8-HxCDD	108 %
1,2,3,7,8,9-HxCDD	99 %
1,2,3,4,6,7,8-HpCDD	97 %
OCDD	102 %
2,3,7,8-TeCDF	96 %
1,2,3,7,8-PeCDF	100 %
2,3,4,7,8-PeCDF	108 %
1,2,3,4,7,8-HxCDF	98 %
1,2,3,6,7,8-HxCDF	103 %
1,2,3,7,8,9-HxCDF	107 %
2,3,4,6,7,8-HxCDF	107 %
1,2,3,4,6,7,8-HpCDF	93 %
1,2,3,4,7,8,9-HpCDF	92 %
OCDF	101 %

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

Congener Name	No.	Recovery
2-MoCB	1	67%
4-MoCB	3	79%
2,2'-DiCB	4	104%
4,4'-DiCB	15	100%
2,2'6-TrCB	19	103%
2,4,4'-TrCB	28	103%
3,4,4'-TrCB	37	94%
2,2',5,5'-TeCB	52	103%
2,2'6,6'-TeCB	54	107%
3,3',4,4'-TeCB	77	86%
3,4,4',5-TeCB	81	97%
2,2',4,5,5'-PeCB	101	105%
2,2',4,6,6'-PeCB	104	100%
2,3,3',4,4'-PeCB	105	113%
2,3,4,4',5-PeCB	114	103%
2,3',4,4',5-PeCB	118	97%
2',3,4,4',5-PeCB	123	103%
3,3',4,4',5-PeCB	126	93%
2,2',3,4,4',5'-HxCB	138	106%
2,2',4,4',5,5'-HxCB	153	105%
2,2',4,4',6,6'-HxCB	155	106%
2,3,3',4,4',5-HxCB	156	106%
2,3,3',4,4',5'-HxCB	157	103%
2,3',4,4',5,5'-HxCB	167	93%
3,3',4,4',5,5'-HxCB	169	86%
2,2',3,4,4',5,5'-HpCB	180	105%
2,2',3,4',5,6,6'-HpCB	188	109%
2,3,3',4,4',5,5'-HpCB	189	103%
2,2',3,3',5,5',6,6'-OcCB	202	107%
2,3,3',4,4',5,5',6-OcCB	205	109%
2,2',3,3',4,4',5,5',6-NoCB	206	105%
2,2',3,3,'4,5,5',6,6'-NoCB	208	103%
DeCB	209	100%

Compound View

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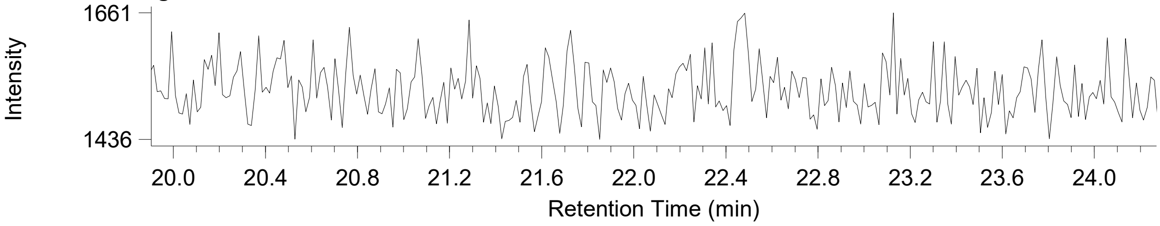
Injection : C5E403001X

T4CDD / Average



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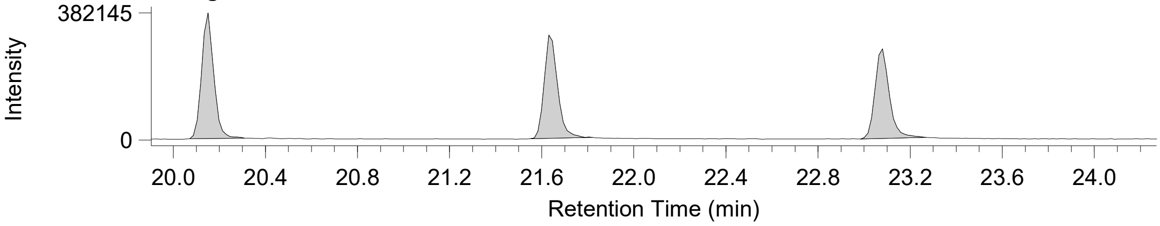
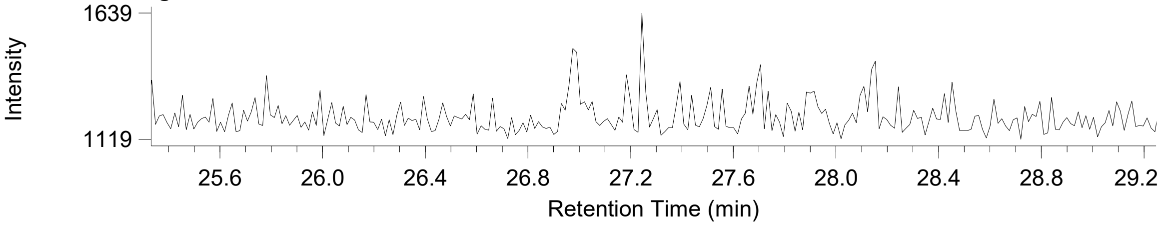


P5CDD / Average



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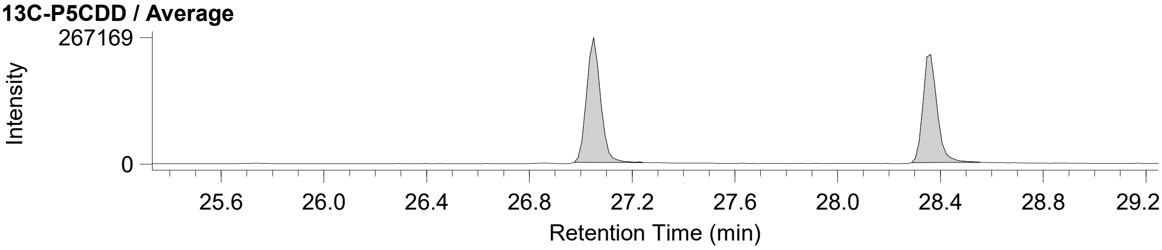

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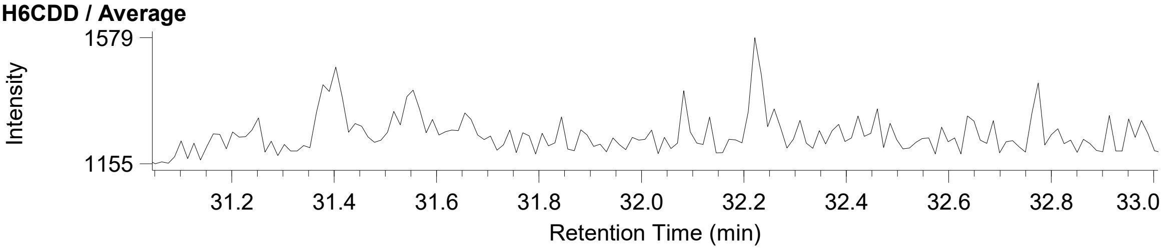

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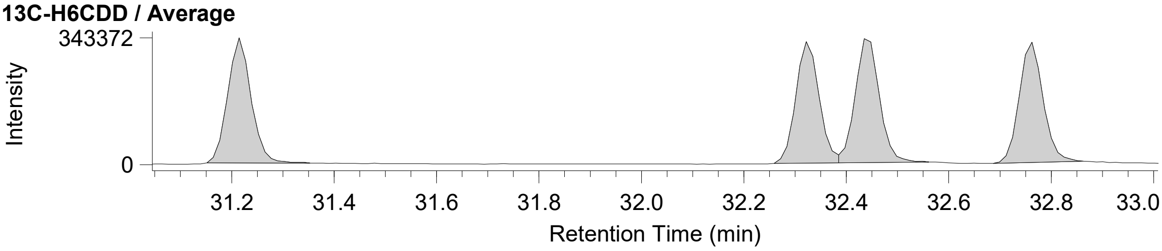

Compound View

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Injection : C5E403001X

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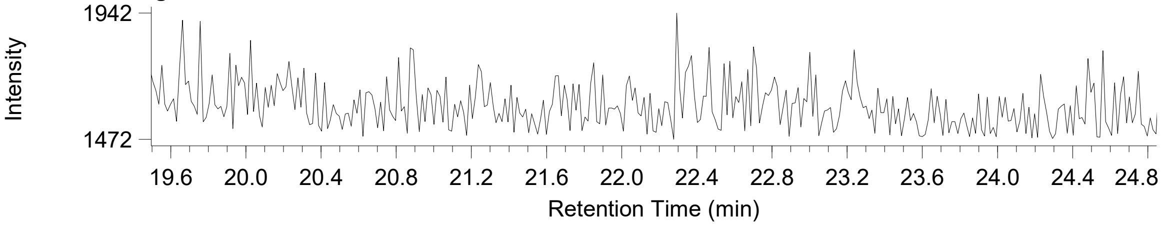

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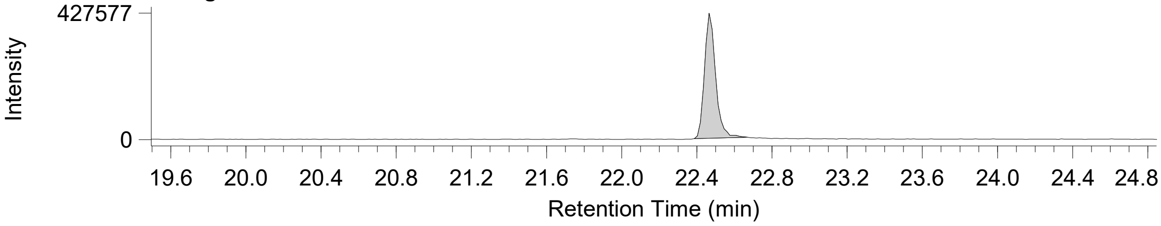

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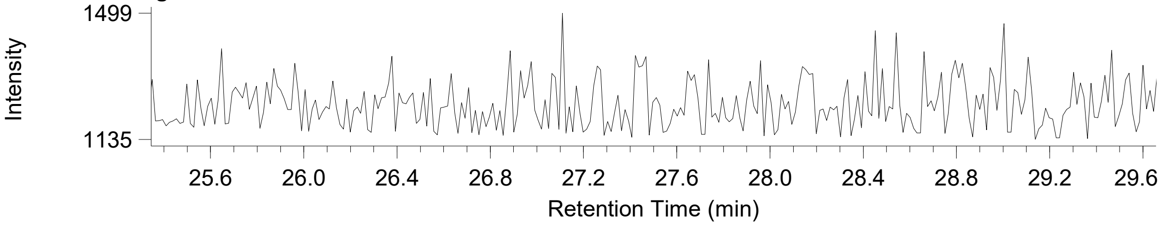

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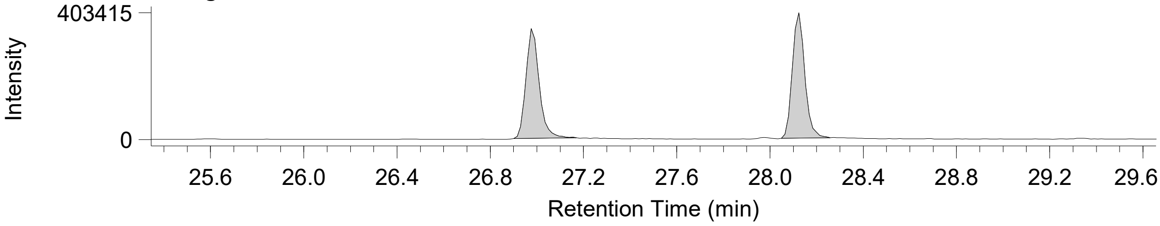

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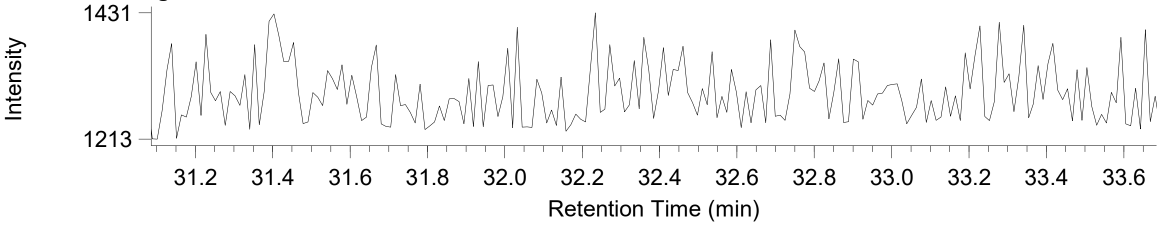

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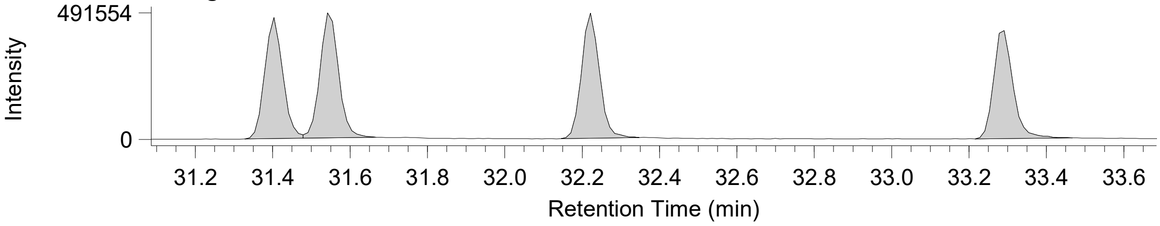

Compound View

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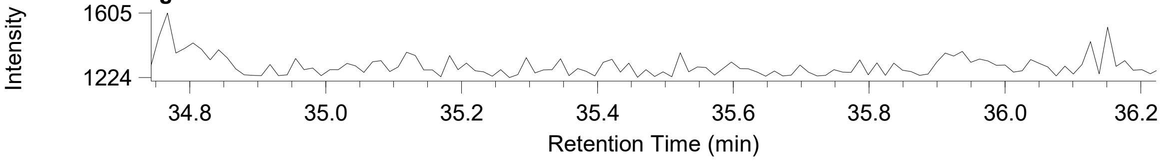

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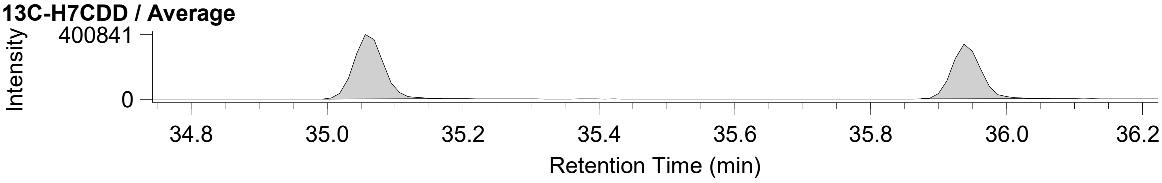
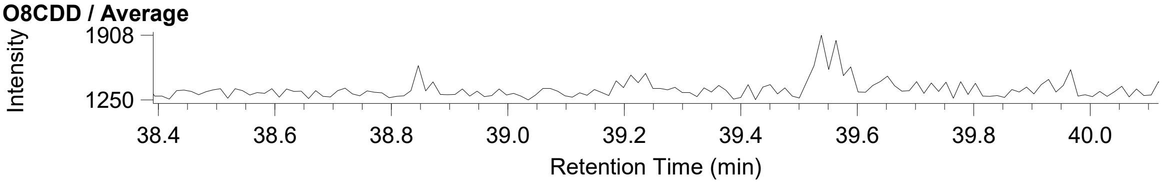


O8CDD / Average



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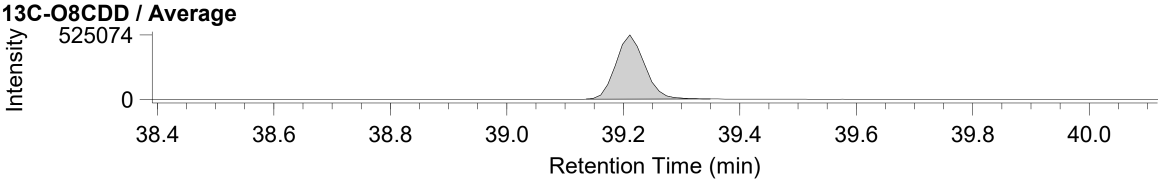

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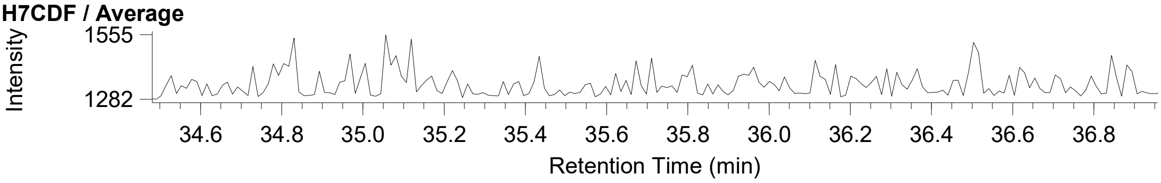

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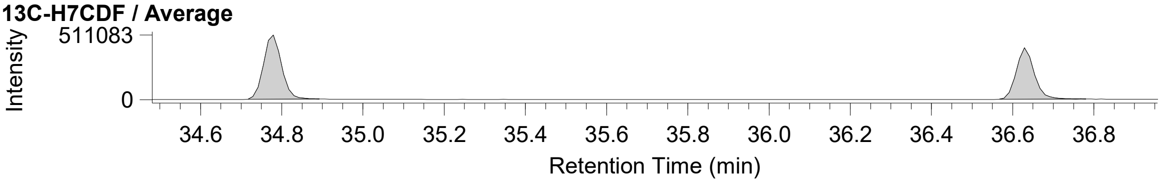

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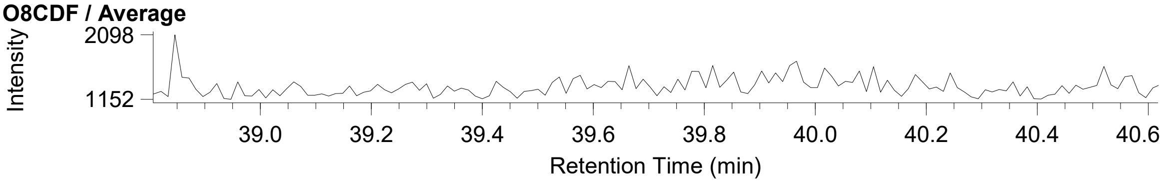

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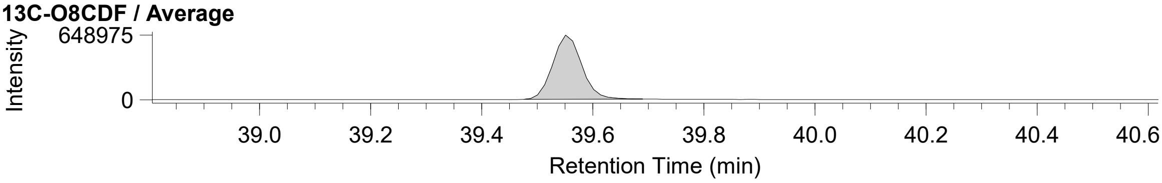

Compound View

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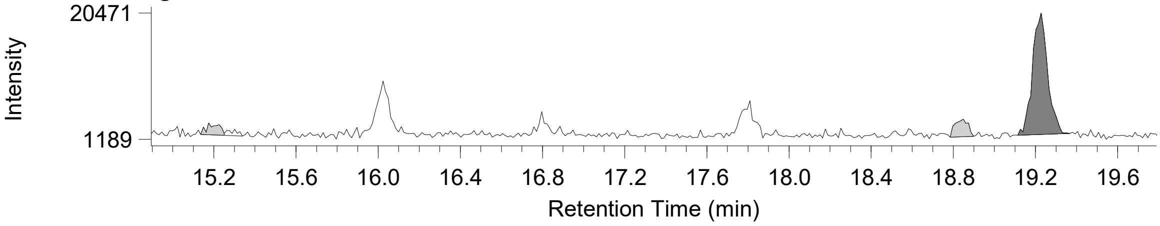
Injection : C5E403002X

M1CBs / Average



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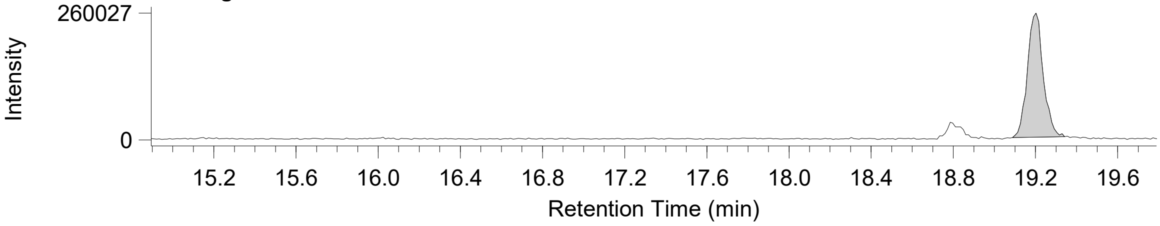

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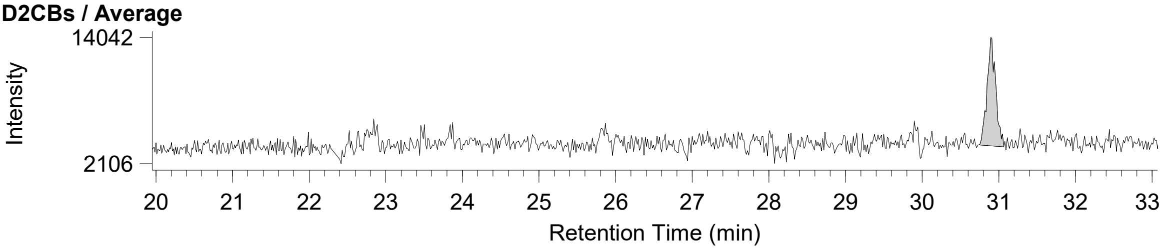

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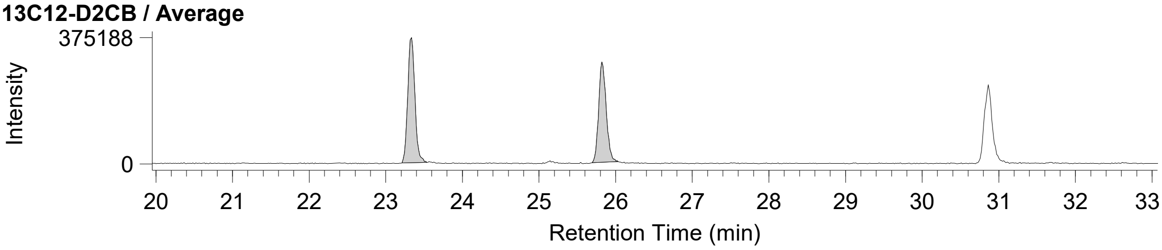

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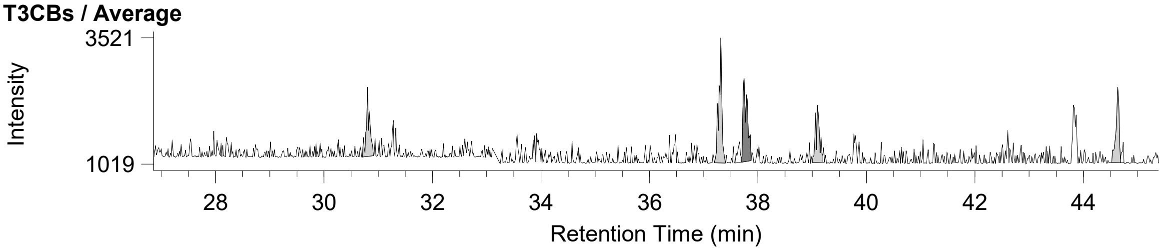

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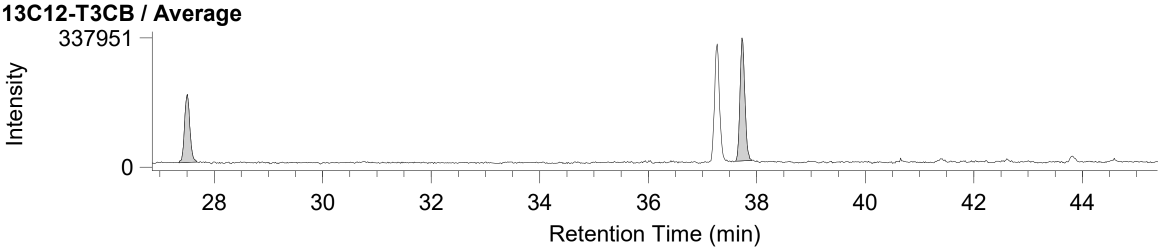

Compound View

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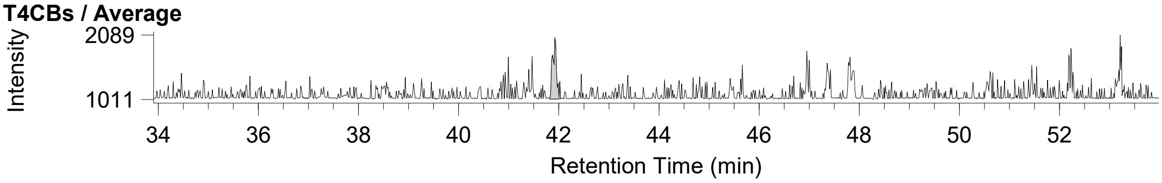
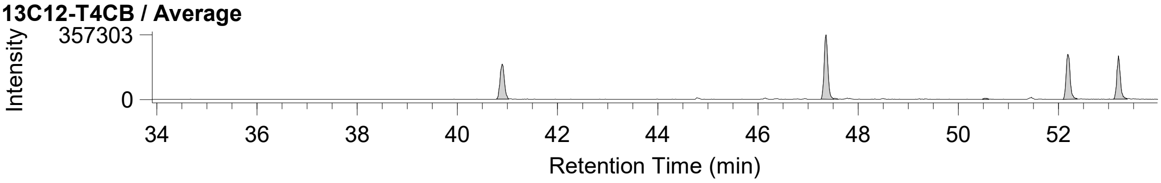
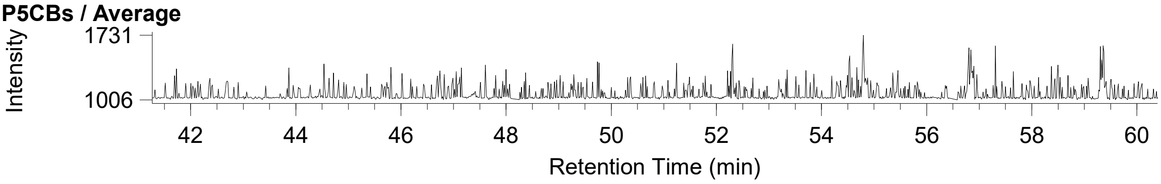


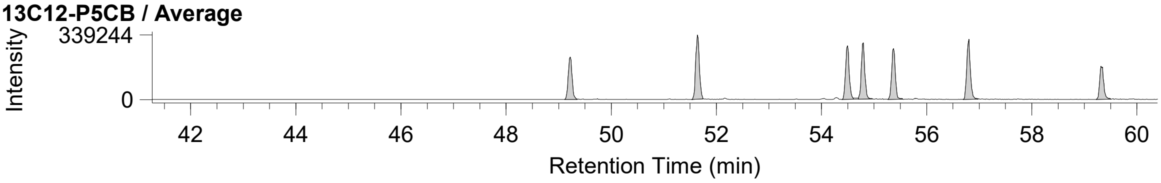
13C12-T4CB / Average



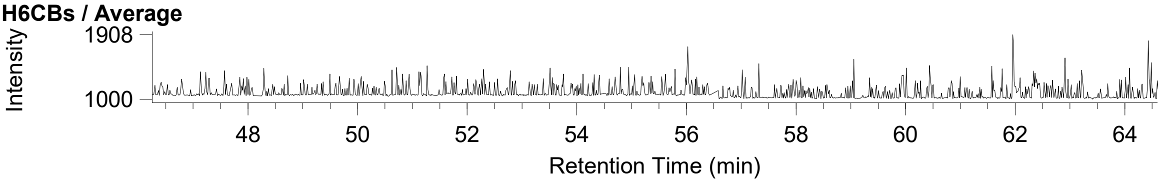
P5CBs / Average



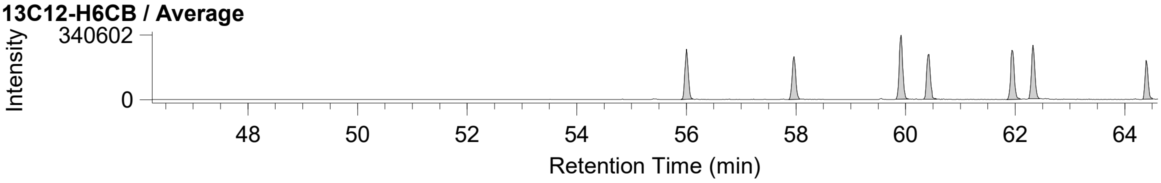
13C12-P5CB / Average



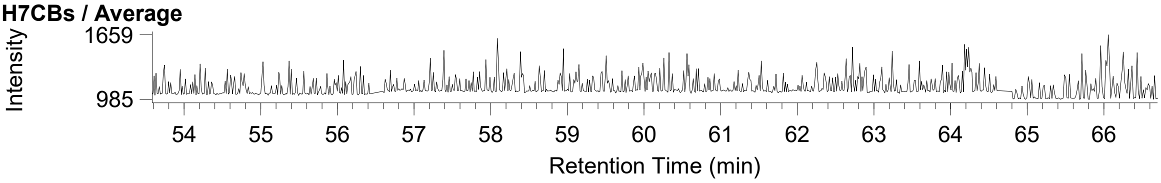
H6CBs / Average



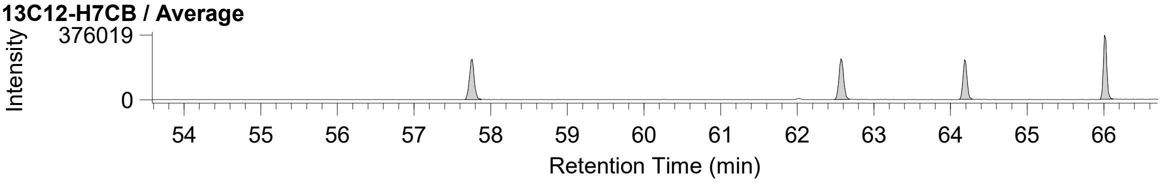
13C12-H6CB / Average



H7CBs / Average



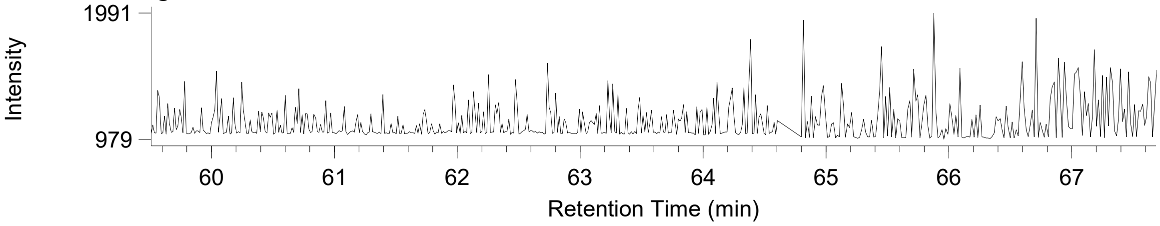
13C12-H7CB / Average



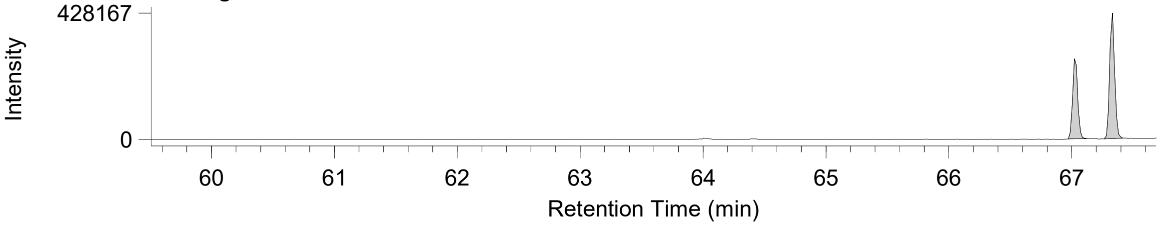
Compound View

DqData : M:\Diok\DqData\2025\C5E403\HT8-PCB(1-10)-101-1
Injection : C5E403002X

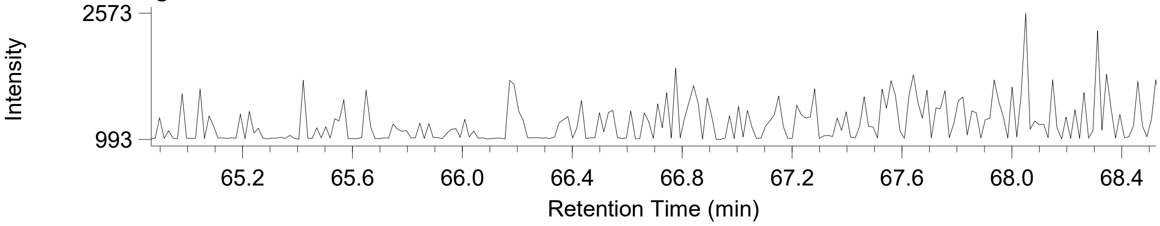
O8CBs / Average



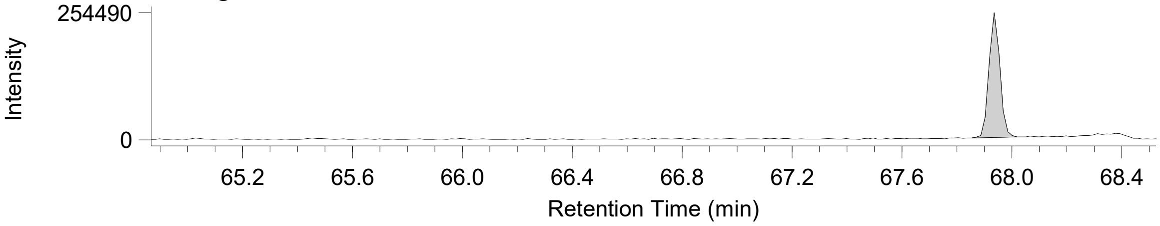
13C12-O8CB / Average



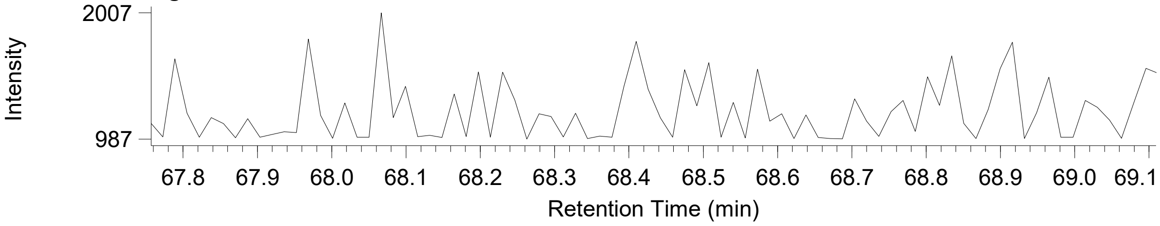
N9CBs / Average



13C12-N9CB / Average



D10CBs / Average



13C12-D10CB / Average