MIURA

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.	263701
Expiration Date	Apr/2028

Tests	Result	Criteria
Blank Values of PCDDs/PCDFs pg-TEQ/column set	< 0.67	< 2.5
Blank values of DL-PCBs pg-TEQ/column set	< 0.056	< 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) < 8.6	Each isomer < 50
Recovery PCDDs/PCDFs	90 to 110 %	60 to 120 %
Native Recovery Toxics/LOC/window defining PCB congeners	73 to 110 %	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 on the label on the column bag is the lot number.
Method: 18D_209PCB

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 n-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 and PCB fraction were 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 measurement. 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 n-decane. 0.02 mL of the diluted standard solution was added in the top of the column. 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 defining 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	0.1	0.1
1,2,3,4,7,8-HxCDD	ND	0.6	0.2	0.2	0.02
1,2,3,6,7,8-HxCDD	ND	1.3	0.4	0.2	0.04
1,2,3,7,8,9-HxCDD	ND	1.3	0.4	0.2	0.04
1,2,3,4,6,7,8-HpCDD	ND	2.4	0.7	0.1	0.007
OCDD	ND	3	1	0.3	0.0003
2,3,7,8-TeCDF	ND	1.1	0.3	0.2	0.03
1,2,3,7,8-PeCDF	ND	1.1	0.3	0.1	0.009
2,3,4,7,8-PeCDF	ND	0.7	0.2	0.09	0.06
1,2,3,4,7,8-HxCDF	ND	1.0	0.3	0.3	0.03
1,2,3,6,7,8-HxCDF	ND	1.1	0.3	0.3	0.03
1,2,3,7,8,9-HxCDF	ND	1.5	0.5	0.3	0.05
2,3,4,6,7,8-HxCDF	ND	1.3	0.4	0.3	0.04
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	2.5	0.7	0.1	0.007
OCDF	ND	3	1	0.2	0.0003
#81 (3,4,4',5-TeCB)	ND	2.4	0.7	0.09	0.00024
#77 (3,3',4,4'-TeCB)	ND	1.5	0.4	0.1	0.00008
#126 (3,3',4,4',5-PeCB)	ND	1.5	0.4	0.1	0.04
#169 (3,3',4,4',5,5'-HxCB)	ND	1.6	0.5	0.1	0.015
#123 (2',3,4,4',5-PeCB)	ND	3.0	0.9	0.08	0.000027
#118 (2,3',4,4',5-PeCB)	ND	2.1	0.6	0.08	0.000018
#105 (2,3,3',4,4'-PeCB)	ND	1.9	0.6	0.08	0.000018
#114 (2,3,4,4',5-PeCB)	ND	2.8	0.8	0.08	0.000024
#167 (2,3',4,4',5,5'-HxCB)	ND	4	1	0.09	0.00003
#156 (2,3,3',4,4',5-HxCB)	ND	4	1	0.09	0.00003
#157 (2,3,3',4,4',5'-HxCB)	ND	2.6	0.8	0.09	0.000024
#189 (2,3,3',4,4',5,5'-HpCB)	ND	1.5	0.5	0.09	0.000015
#28 (2,4,4'-TrCB)	(0.8)	2.6	0.8	0.08	-
#52 (2,2',5,5'-TeCB)	ND	2.8	0.8	0.1	-
#101 (2,2',4,5,5'-PeCB)	ND	2.9	0.9	0.09	-
#138 (2,2',3,4,4',5'-HxCB)	ND	1.9	0.6	0.1	-
#153 (2,2',4,4',5,5'-HxCB)	ND	4	1	0.1	-
#180 (2,2',3,4,4',5,5'-HpCB)	ND	3.2	0.9	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	90 %
1,2,3,7,8-PeCDD	95 %
1,2,3,4,7,8-HxCDD	104 %
1,2,3,6,7,8-HxCDD	103 %
1,2,3,7,8,9-HxCDD	103 %
1,2,3,4,6,7,8-HpCDD	95 %
OCDD	94 %
2,3,7,8-TeCDF	106 %
1,2,3,7,8-PeCDF	107 %
2,3,4,7,8-PeCDF	106 %
1,2,3,4,7,8-HxCDF	99 %
1,2,3,6,7,8-HxCDF	102 %
1,2,3,7,8,9-HxCDF	110 %
2,3,4,6,7,8-HxCDF	104 %
1,2,3,4,6,7,8-HpCDF	94 %
1,2,3,4,7,8,9-HpCDF	100 %
OCDF	98 %

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

Congener Name	No.	Recovery
2-MoCB	1	91%
4-MoCB	3	76%
2,2'-DiCB	4	110%
4,4'-DiCB	15	89%
2,2'6-TrCB	19	97%
2,4,4'-TrCB	28	97%
3,4,4'-TrCB	37	94%
2,2',5,5'-TeCB	52	103%
2,2'6,6'-TeCB	54	102%
3,3',4,4'-TeCB	77	78%
3,4,4',5-TeCB	81	83%
2,2',4,5,5'-PeCB	101	100%
2,2',4,6,6'-PeCB	104	103%
2,3,3',4,4'-PeCB	105	100%
2,3,4,4',5-PeCB	114	95%
2,3',4,4',5-PeCB	118	98%
2',3,4,4',5-PeCB	123	98%
3,3',4,4',5-PeCB	126	78%
2,2',3,4,4',5'-HxCB	138	95%
2,2',4,4',5,5'-HxCB	153	105%
2,2',4,4',6,6'-HxCB	155	110%
2,3,3',4,4',5-HxCB	156	95%
2,3,3',4,4',5'-HxCB	157	95%
2,3',4,4',5,5'-HxCB	167	93%
3,3',4,4',5,5'-HxCB	169	73%
2,2',3,4,4',5,5'-HpCB	180	98%
2,2',3,4',5,6,6'-HpCB	188	100%
2,3,3',4,4',5,5'-HpCB	189	95%
2,2',3,3',5,5',6,6'-OcCB	202	96%
2,3,3',4,4',5,5',6-OcCB	205	94%
2,2',3,3',4,4',5,5',6-NoCB	206	95%
2,2',3,3,'4,5,5',6,6'-NoCB	208	93%
DeCB	209	100%

Compound View

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Injection : C6E402007X

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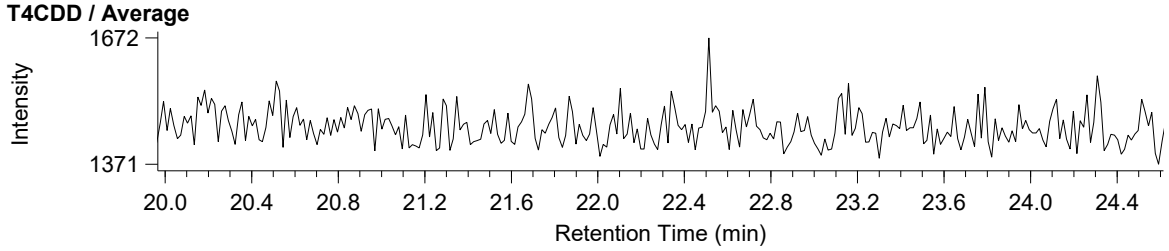

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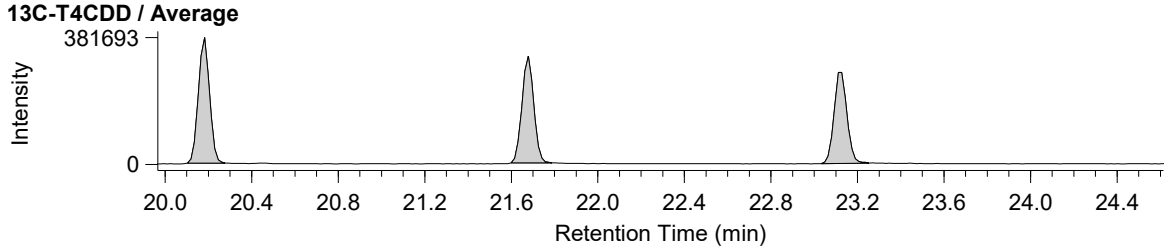

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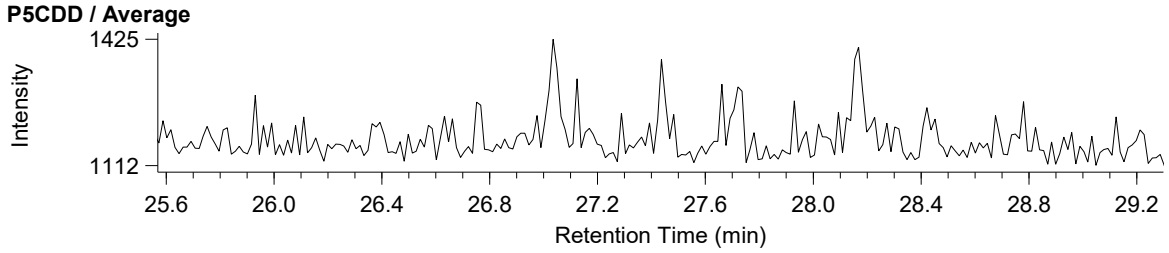

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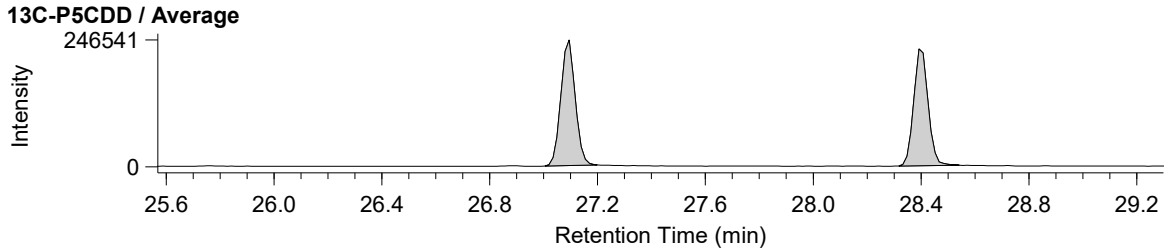

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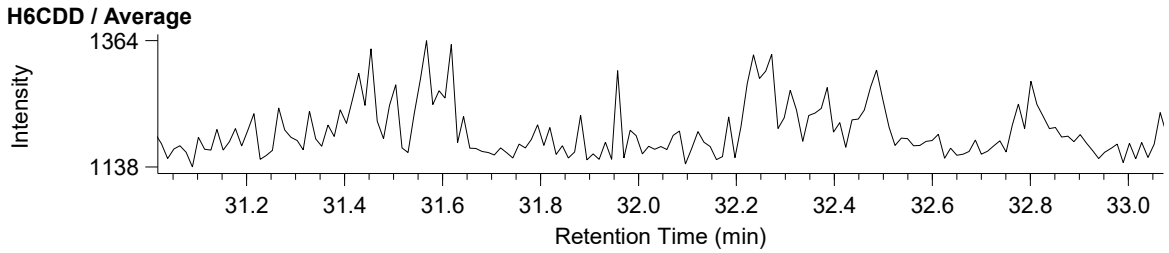

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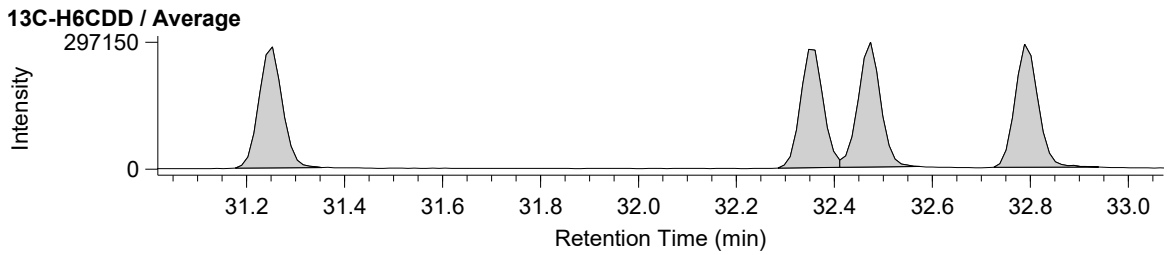

Compound View

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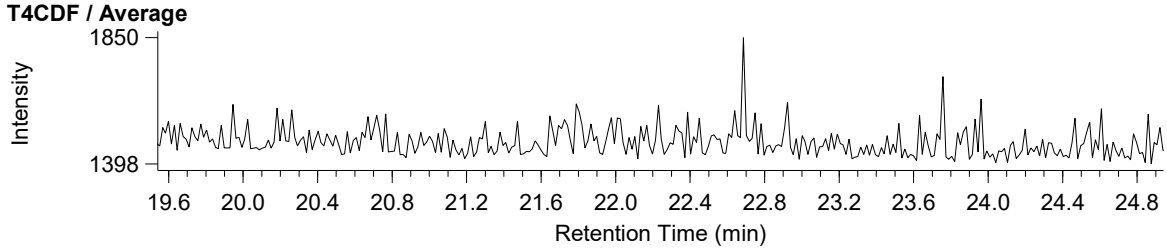

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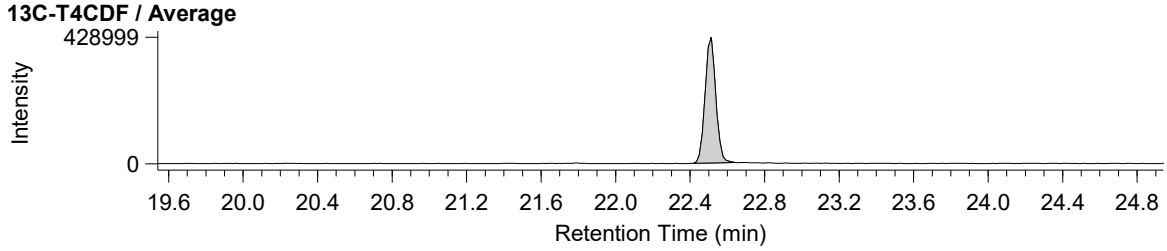

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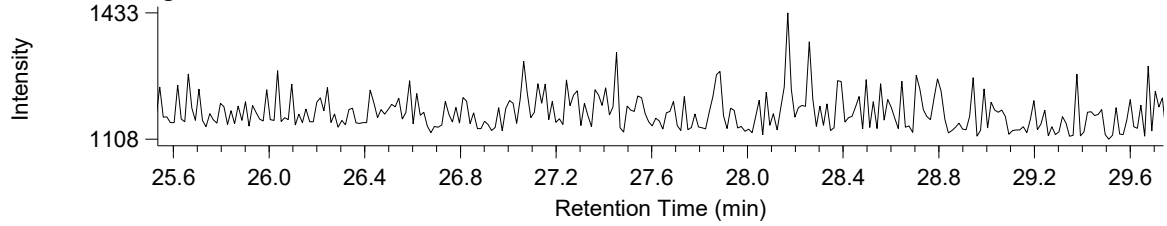

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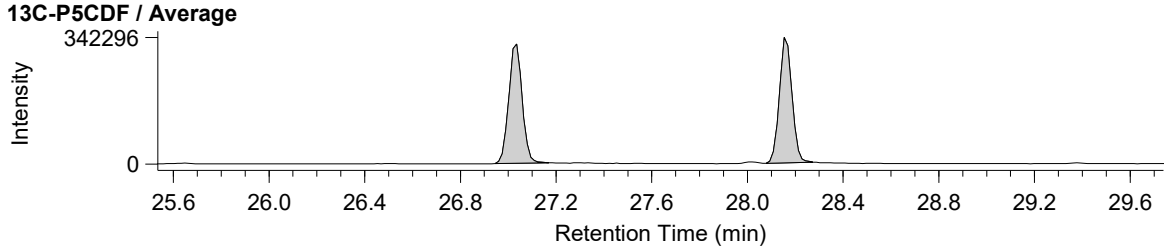

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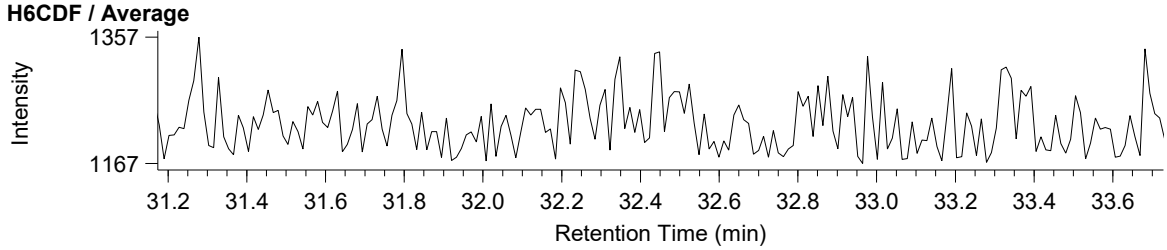

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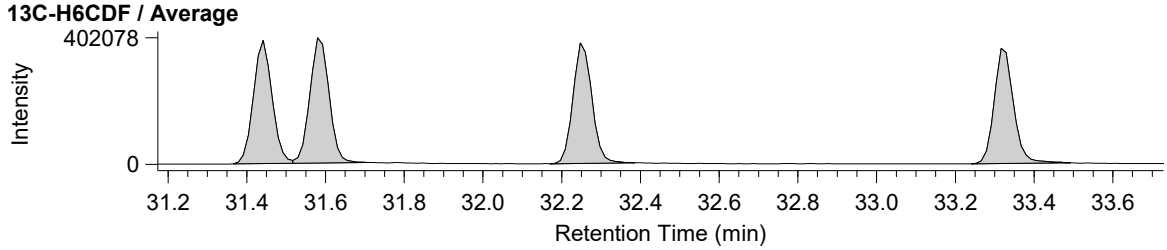

Compound View

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Injection : C6E402007X

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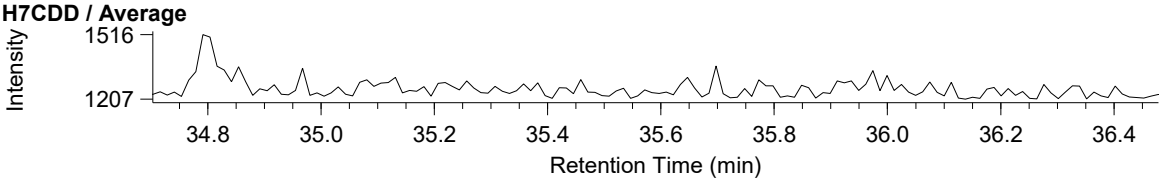

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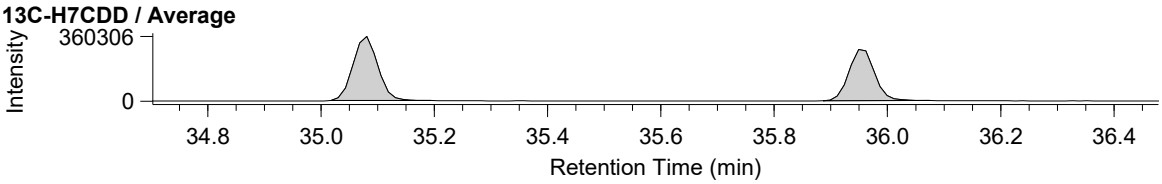
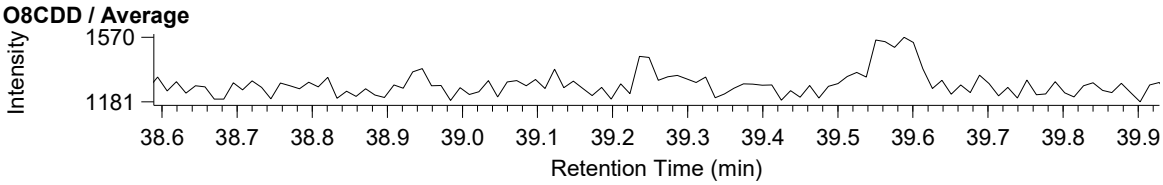


O8CDD / Average



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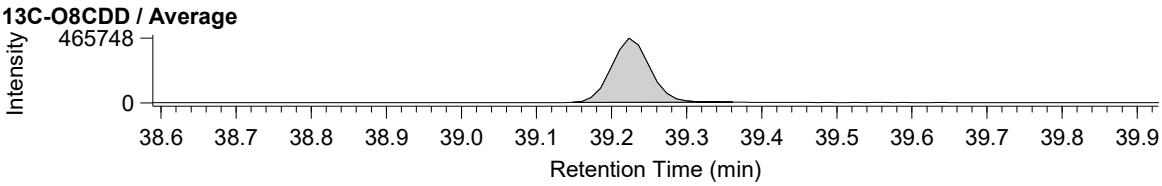

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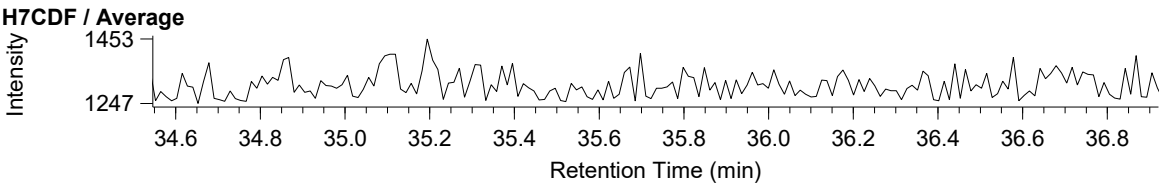

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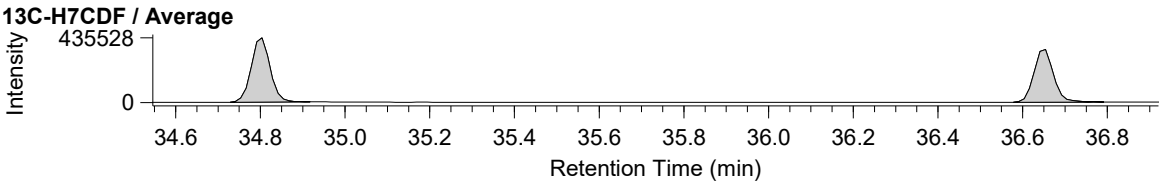

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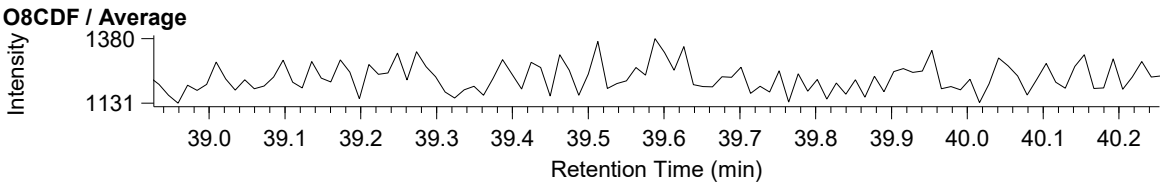

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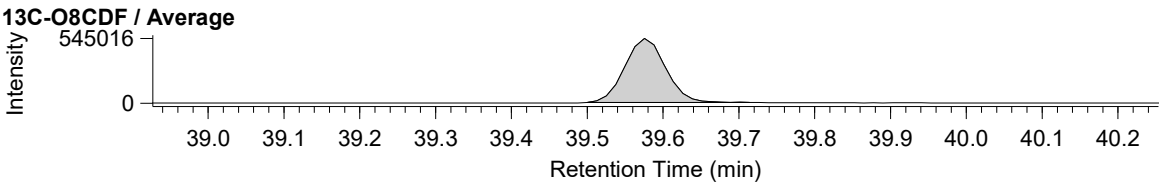

Compound View

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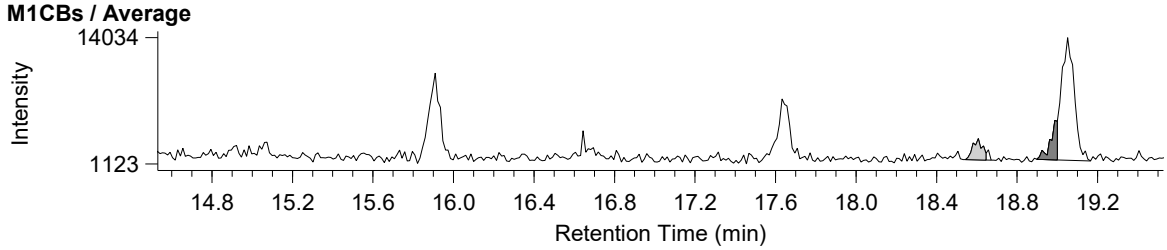
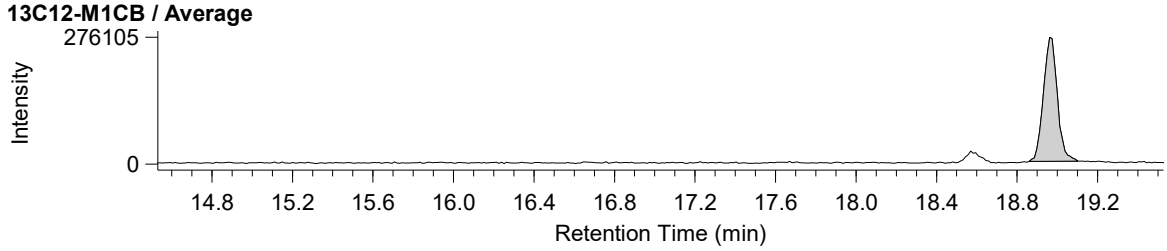
Injection : C6E402008X

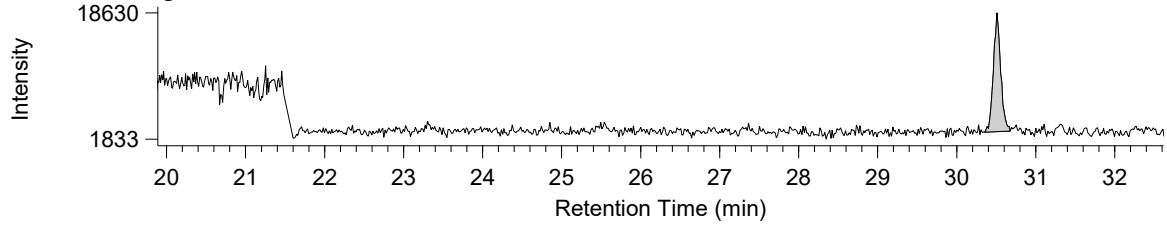
M1CBs / Average



13C12-M1CB / Average



D2CBs / Average



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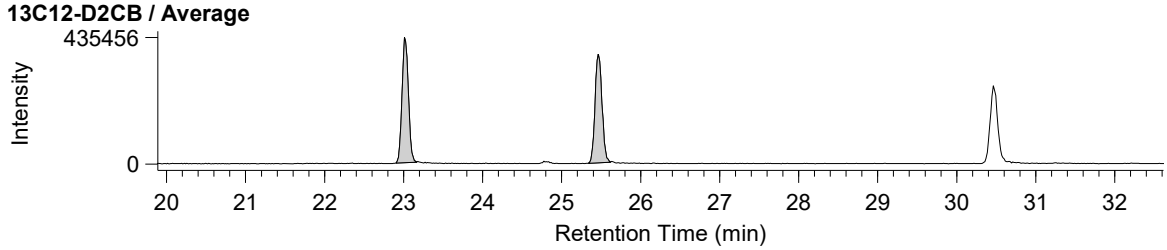

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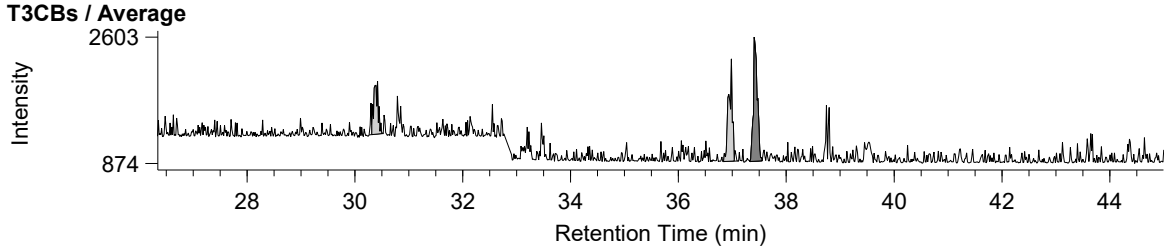
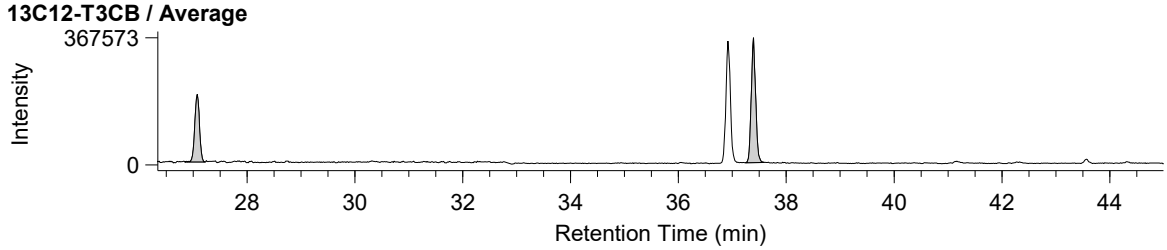


13C12-T3CB / Average



Compound View

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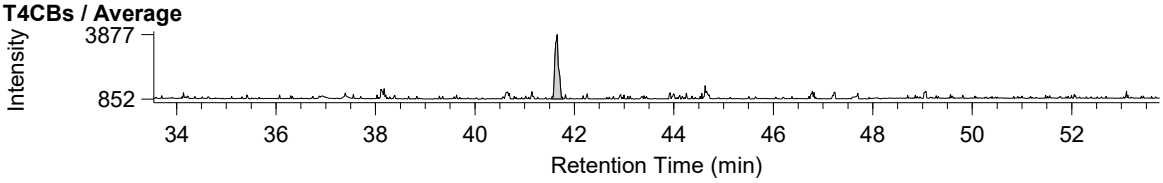
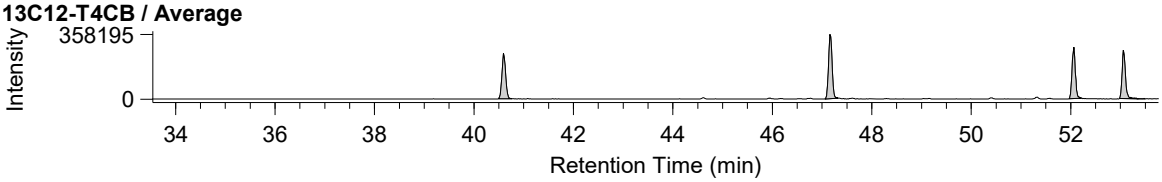
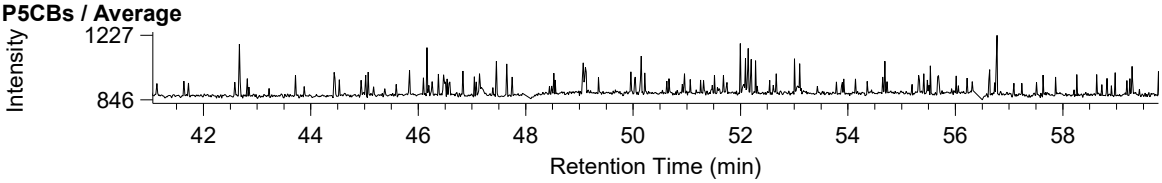
T4CBs / Average



13C12-T4CB / Average



P5CBs / Average



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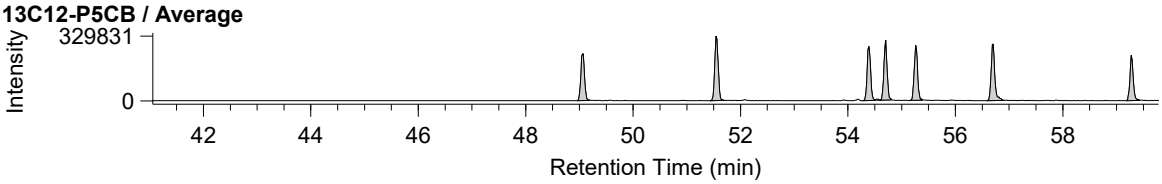
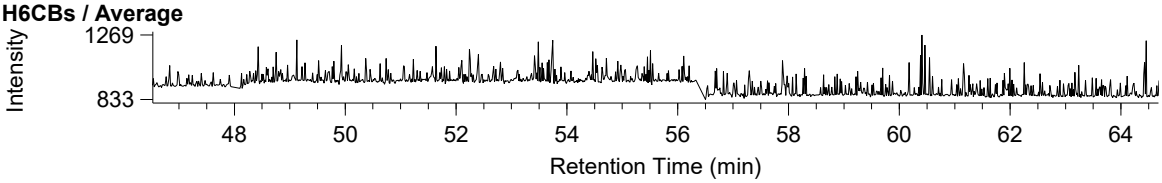


H6CBs / Average



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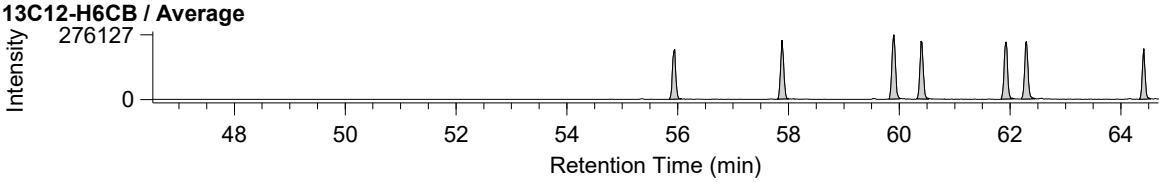
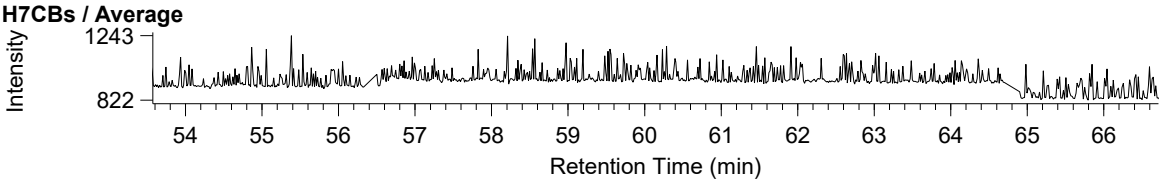


H7CBs / Average



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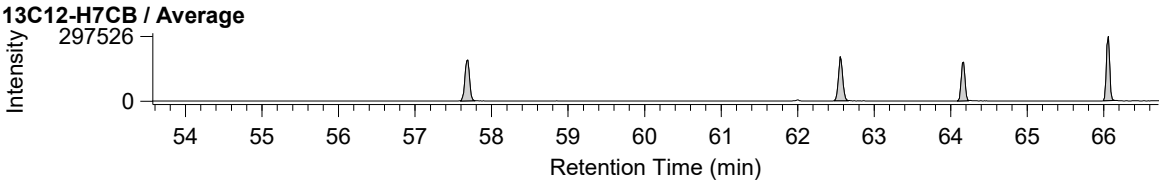

Compound View

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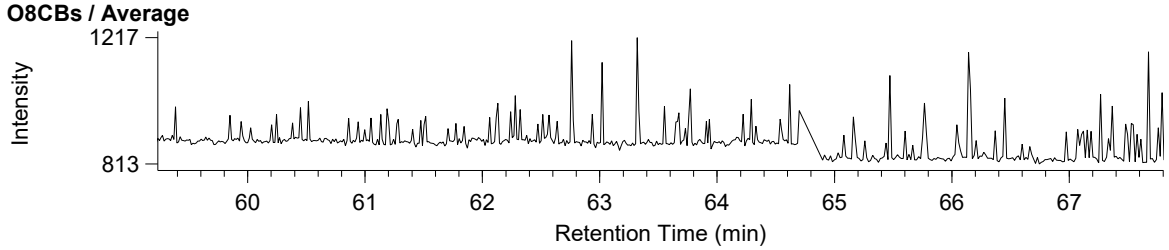
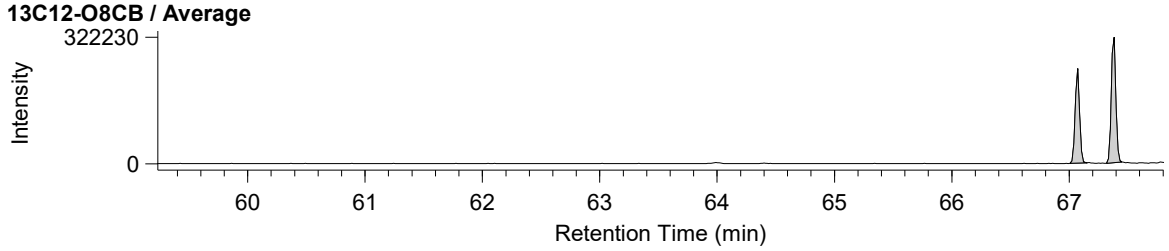
Injection : C6E402008X

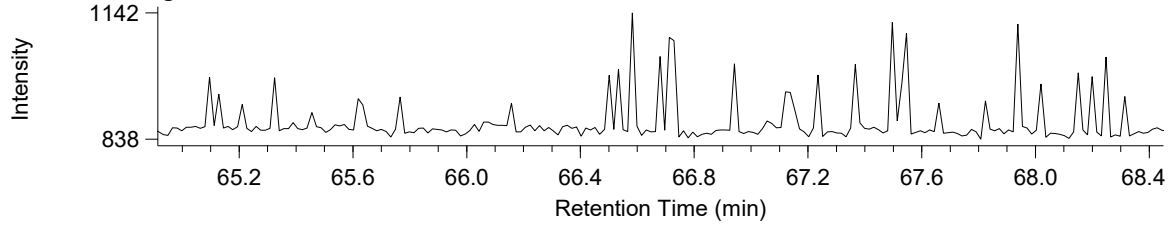
O8CBs / Average



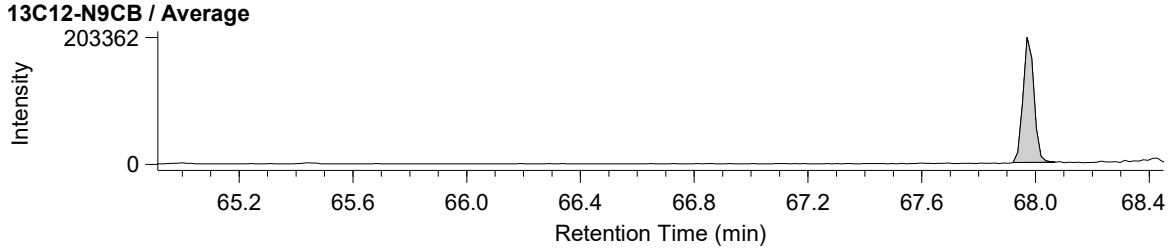
13C12-O8CB / Average



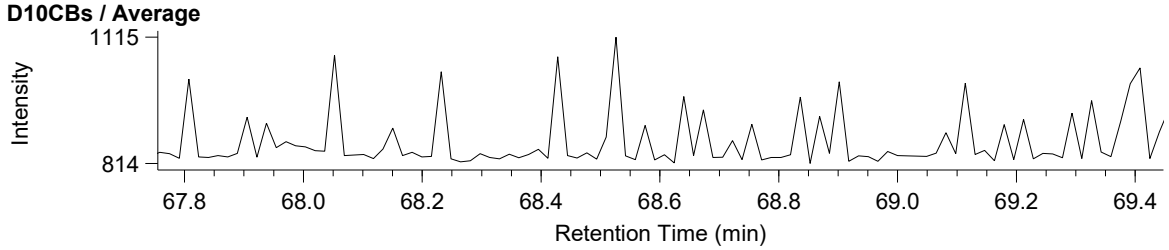
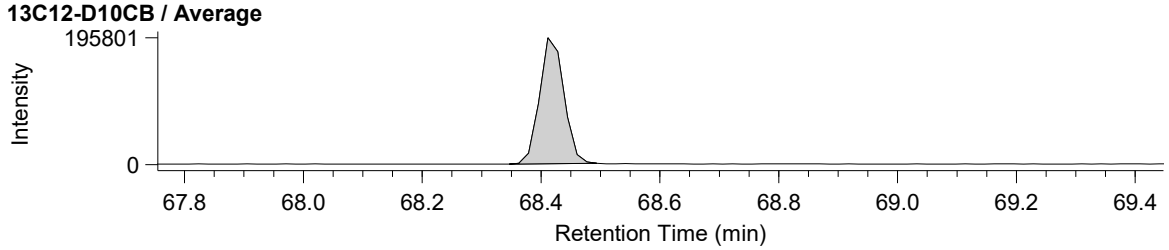
N9CBs / Average



13C12-N9CB / Average



D10CBs / Average



13C12-D10CB / Average