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	20Φ Column set
Product Code	X300-002-2420
Lot No.	233006
Expiration Date	May/2025

Tests	Result	Criteria
Blank Values of PCDDs/PCDFs pg-TEQ/column set	< 0.43	< 0.5
Blank values of DL-PCBs pg-TEQ/column set	< 0.019	< 0.05
Blank values of NDL-PCBs pg-congener /column set	The highest isomer (#28) < 1.4	Each isomer < 200
Recovery PCDDs/PCDFs/ DL-PCBs	77 to 98 %	70 to 120 %
Recovery NDL-PCBs	88 to 100 %	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 (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 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, 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	Internal Standard	DF-SS-A	Wellington Laboratories Inc.	10 ng/mL in decane
	Internal Standard	DF-LCS-B		
	Recovery (Surrogate) Standard	DF-IS-J		
DL-PCBs , NDL-PCBs	Internal Standard	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. 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), The sunflower seed oil from Helianthus annuus (S5007-1L, 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	0.26	0.08	0.07	0.08
1,2,3,7,8-PeCDD	ND	0.29	0.09	0.08	0.09
1,2,3,4,7,8-HxCDD	ND	0.8	0.3	0.1	0.03
1,2,3,6,7,8-HxCDD	ND	1.0	0.3	0.1	0.03
1,2,3,7,8,9-HxCDD	ND	0.7	0.2	0.1	0.02
1,2,3,4,6,7,8-HpCDD	ND	0.7	0.2	0.2	0.002
OCDD	ND	1.2	0.4	0.1	0.00012
2,3,7,8-TeCDF	ND	0.8	0.2	0.1	0.02
1,2,3,7,8-PeCDF	ND	0.8	0.2	0.05	0.006
2,3,4,7,8-PeCDF	ND	0.6	0.2	0.04	0.06
1,2,3,4,7,8-HxCDF	ND	0.8	0.2	0.05	0.02
1,2,3,6,7,8-HxCDF	ND	1.0	0.3	0.05	0.03
1,2,3,7,8,9-HxCDF	ND	0.7	0.2	0.06	0.02
2,3,4,6,7,8-HxCDF	ND	0.7	0.2	0.05	0.02
1,2,3,4,6,7,8-HpCDF	ND	0.9	0.3	0.07	0.003
1,2,3,4,7,8,9-HpCDF	ND	1.1	0.3	0.09	0.003
OCDF	ND	2.0	0.6	0.2	0.00018
#81 (3,4,4',5-TeCB)	ND	0.5	0.2	0.05	0.00006
#77 (3,3',4,4'-TeCB)	ND	1.1	0.3	0.05	0.00003
#126 (3,3',4,4',5-PeCB)	ND	0.4	0.1	0.1	0.01
#169 (3,3',4,4',5,5'-HxCB)	ND	1.2	0.3	0.06	0.009
#123 (2',3,4,4',5-PeCB)	ND	1.8	0.5	0.02	0.000015
#118 (2,3',4,4',5-PeCB)	ND	1.4	0.4	0.02	0.000012
#105 (2,3,3',4,4'-PeCB)	ND	1.4	0.4	0.02	0.000012
#114 (2,3,4,4',5-PeCB)	ND	1.2	0.4	0.02	0.000012
#167 (2,3',4,4',5,5'-HxCB)	ND	0.7	0.2	0.02	0.000006
#156 (2,3,3',4,4',5-HxCB)	ND	1.0	0.3	0.02	0.000009
#157 (2,3,3',4,4',5'-HxCB)	ND	0.8	0.2	0.02	0.000006
#189 (2,3,3',4,4',5,5'-HpCB)	ND	1.4	0.4	0.02	0.000012
#28 (2,4,4'-TrCB)	1.4	0.6	0.2	0.02	-
#52 (2,2',5,5'-TeCB)	(0.5)	1.3	0.4	0.03	-
#101 (2,2',4,5,5'-PeCB)	ND	1.7	0.5	0.02	-
#138 (2,2',3,4,4',5'-HxCB)	ND	1.9	0.6	0.02	-
#153 (2,2',4,4',5,5'-HxCB)	ND	1.8	0.5	0.02	-
#180 (2,2',3,4,4',5,5'-HpCB)	ND	1.8	0.6	0.03	-

* 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	79 %
1,2,3,7,8-PeCDD	86 %
1,2,3,4,7,8-HxCDD	92 %
1,2,3,6,7,8-HxCDD	90 %
1,2,3,7,8,9-HxCDD	83 %
1,2,3,4,6,7,8-HpCDD	84 %
OCDD	77 %
2,3,7,8-TeCDF	86 %
1,2,3,7,8-PeCDF	92 %
2,3,4,7,8-PeCDF	95 %
1,2,3,4,7,8-HxCDF	86 %
1,2,3,6,7,8-HxCDF	87 %
1,2,3,7,8,9-HxCDF	95 %
2,3,4,6,7,8-HxCDF	87 %
1,2,3,4,6,7,8-HpCDF	83 %
1,2,3,4,7,8,9-HpCDF	84 %
OCDF	84 %
#81 (3,4,4',5-TeCB)	79 %
#77 (3,3',4,4'-TeCB)	88 %
#126 (3,3',4,4',5-PeCB)	86 %
#169 (3,3',4,4',5,5'-HxCB)	84 %
#123 (2',3,4,4',5-PeCB)	98 %
#118 (2,3',4,4',5-PeCB)	92 %
#105 (2,3,3',4,4'-PeCB)	96 %
#114 (2,3,4,4',5-PeCB)	97 %
#167 (2,3',4,4',5,5'-HxCB)	84 %
#156 (2,3,3',4,4',5-HxCB)	83 %
#157 (2,3,3',4,4',5'-HxCB)	78 %
#189 (2,3,3',4,4',5,5'-HpCB)	86 %
#28 (2,4,4'-TrCB)	98 %
#52 (2,2',5,5'-TeCB)	90 %
#101 (2,2',4,5,5'-PeCB)	100 %
#138 (2,2',3,4,4',5'-HxCB)	96 %
#153 (2,2',4,4',5,5'-HxCB)	88 %
#180 (2,2',3,4,4',5,5'-HpCB)	90 %

Compound View

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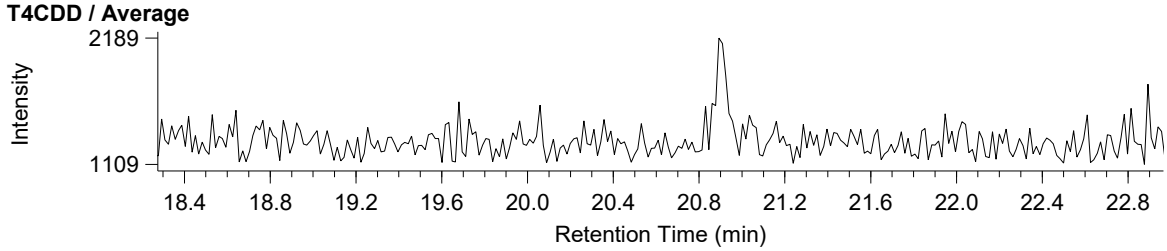

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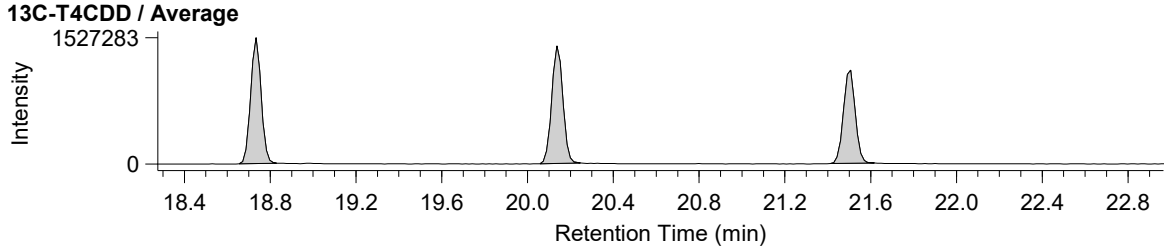

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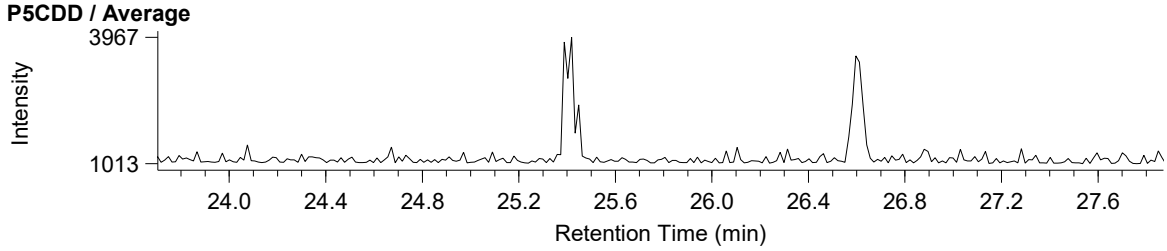

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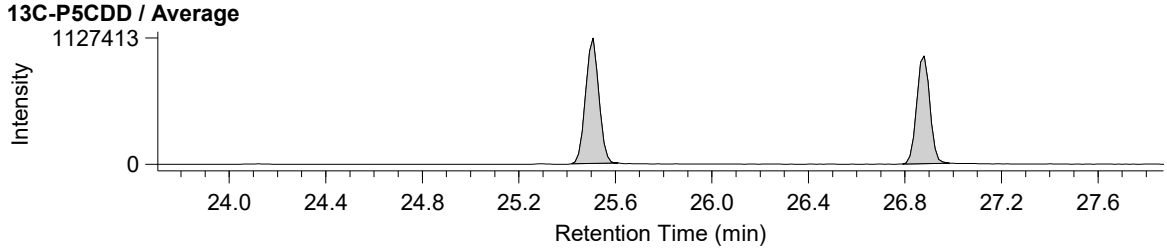

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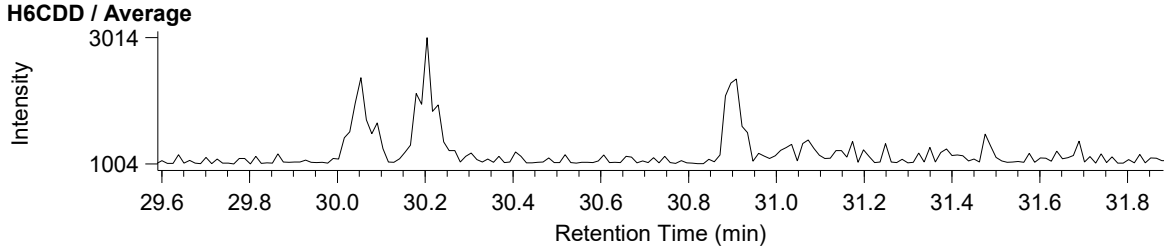

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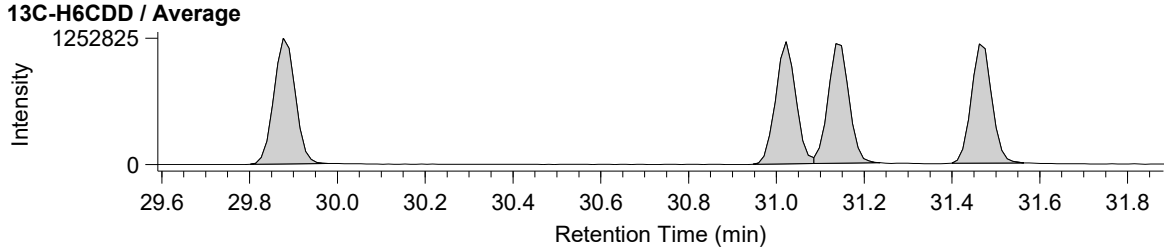

Compound View

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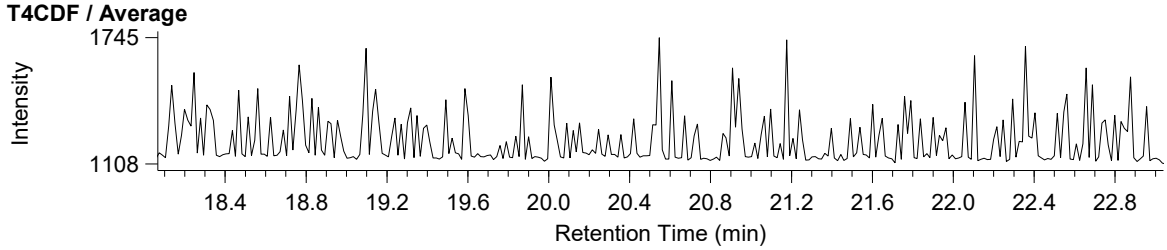

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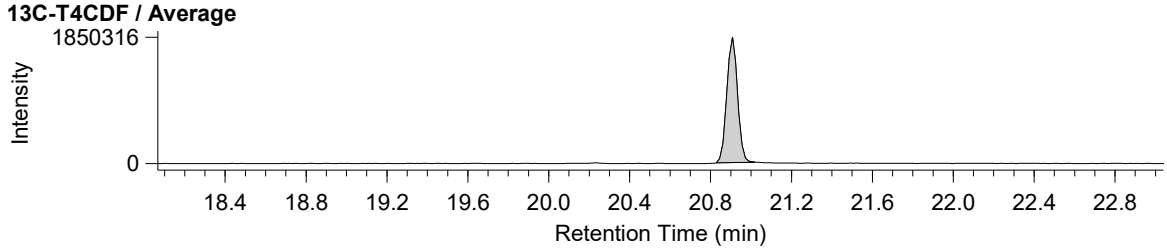

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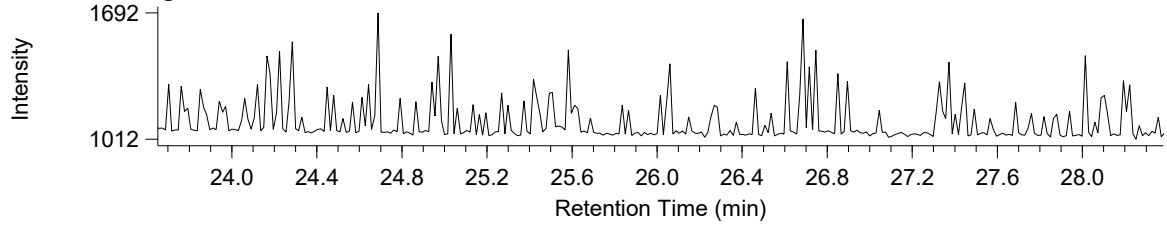

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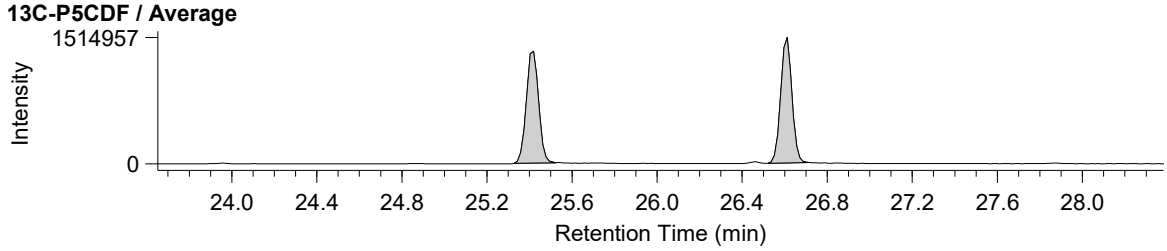

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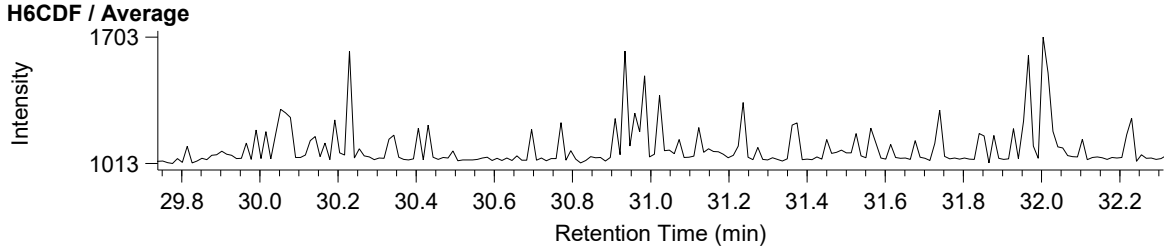

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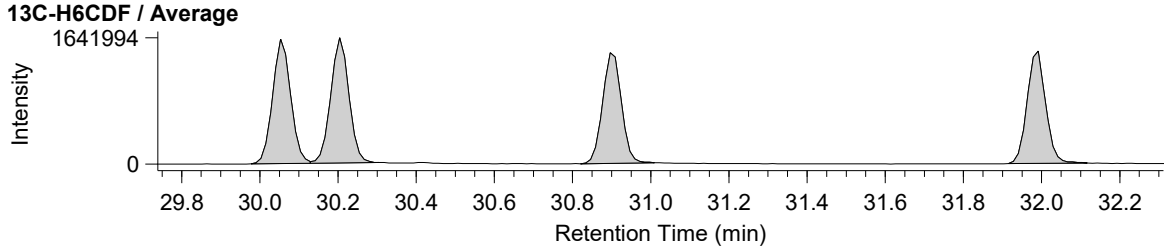

Compound View

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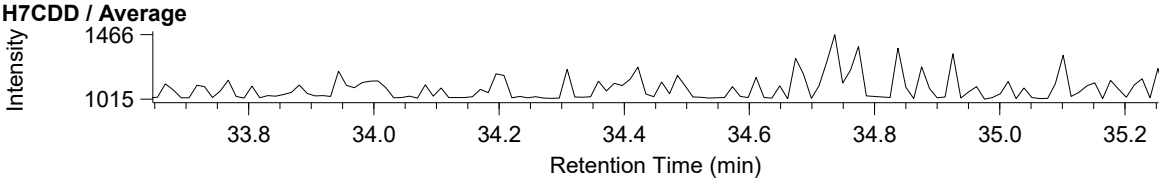

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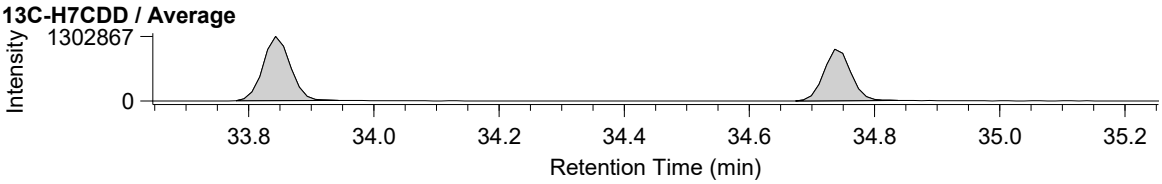
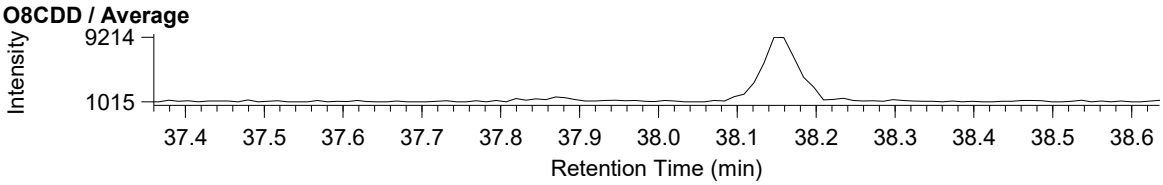


O8CDD / Average



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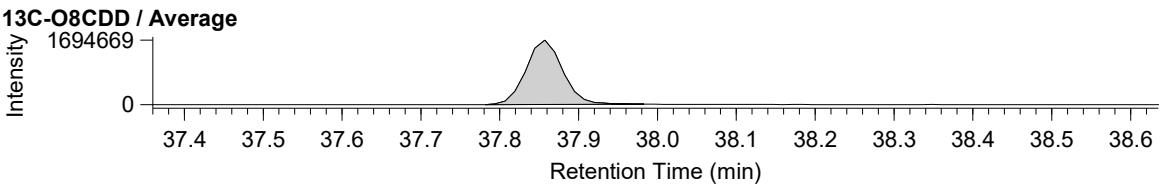

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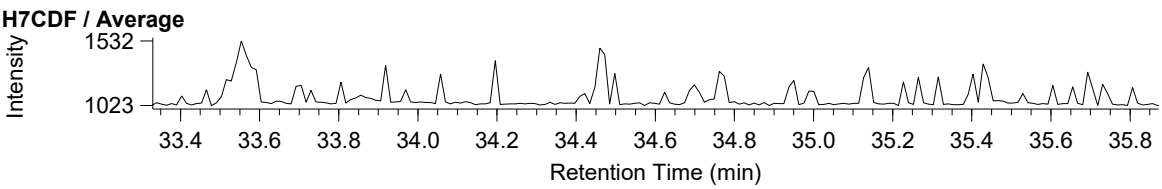

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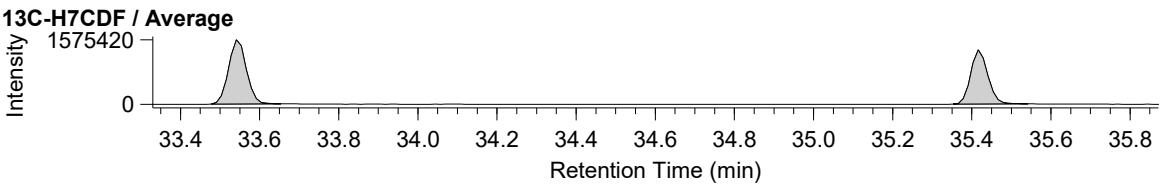

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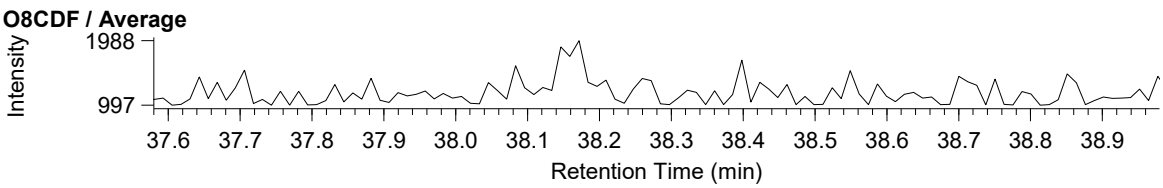

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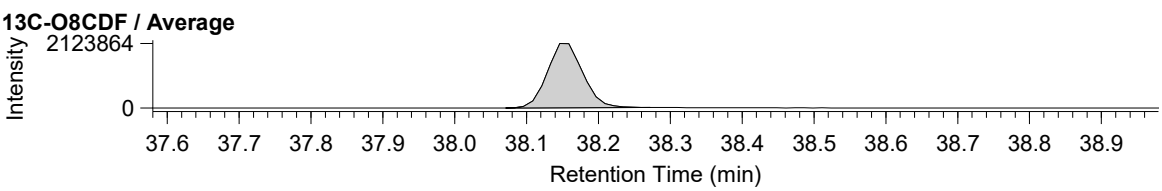

Compound View

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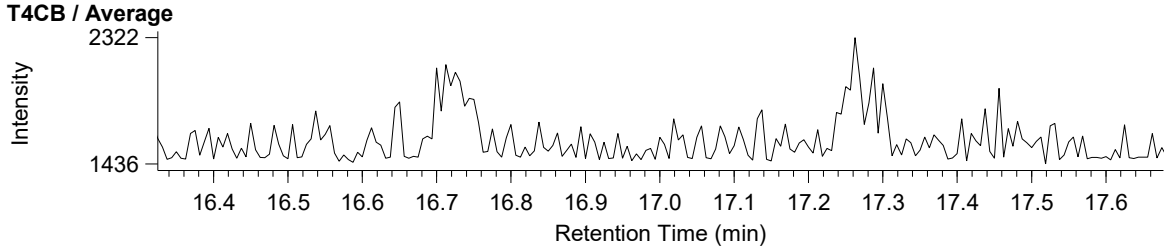

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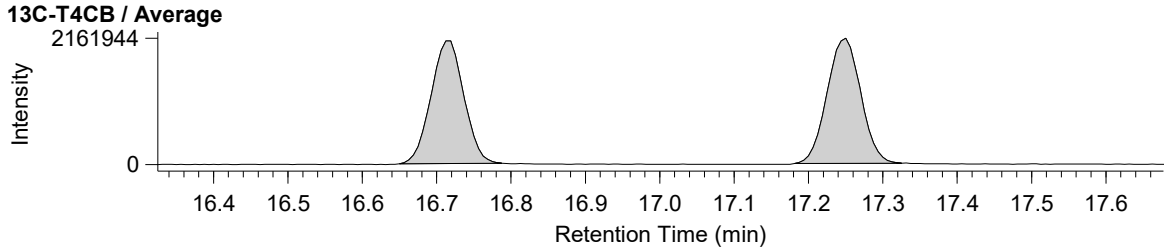
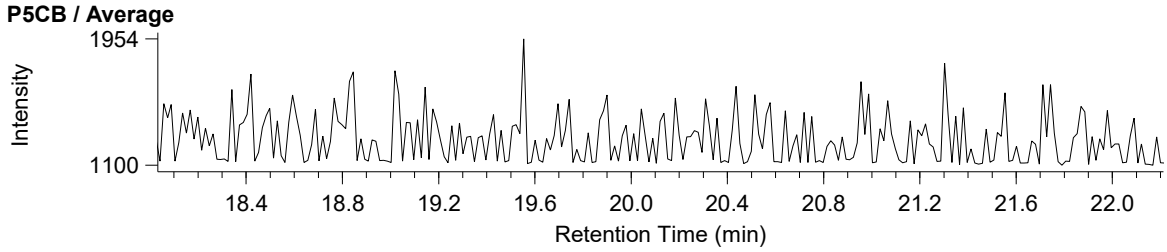


P5CB / Average



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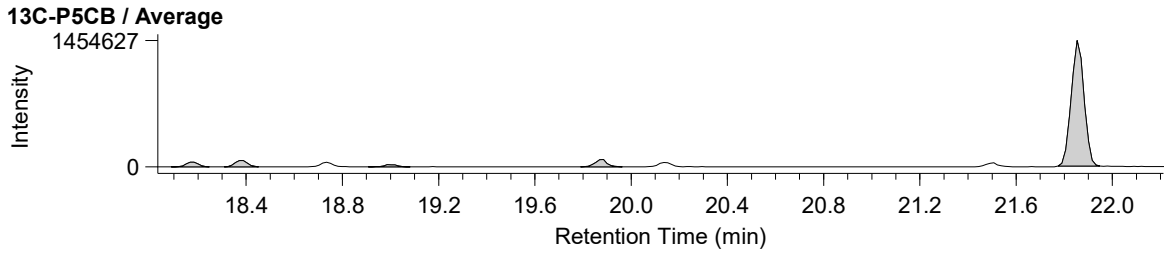
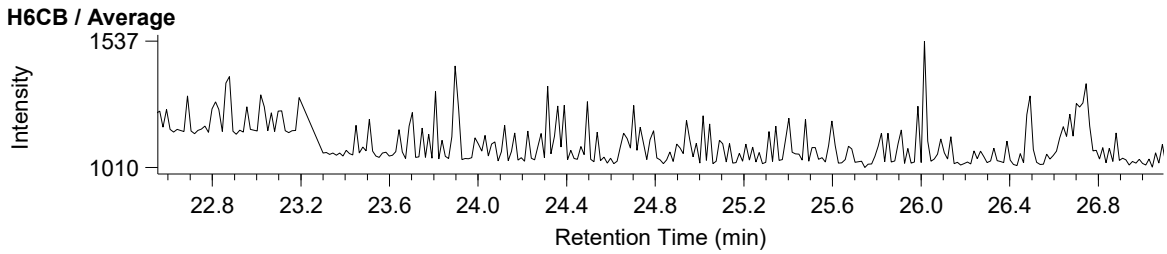
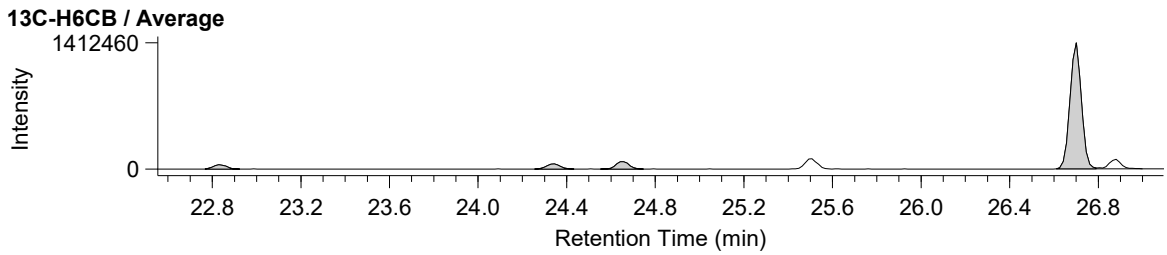


H6CB / Average



13C-H6CB / Average



Compound View

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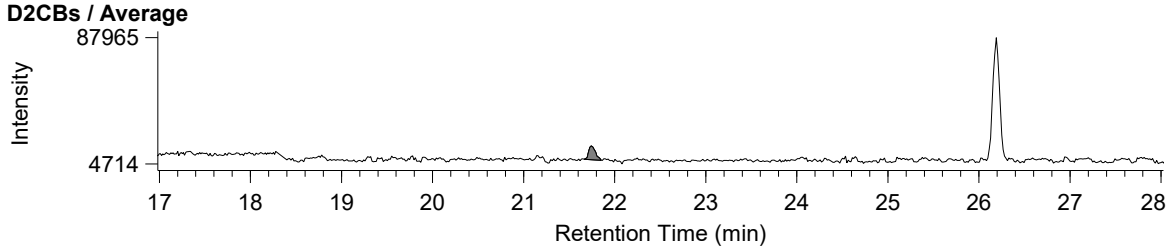
D2CBs / Average



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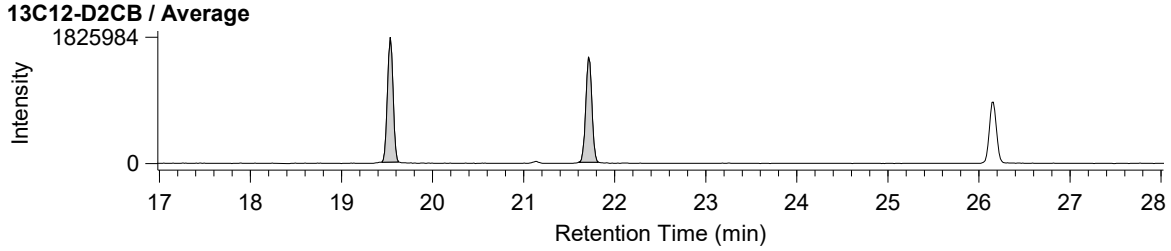
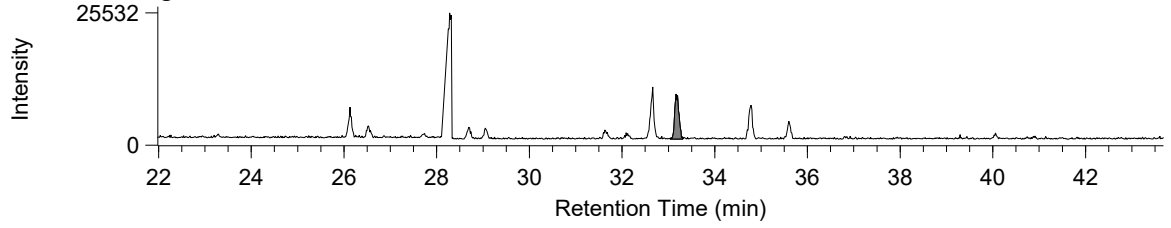
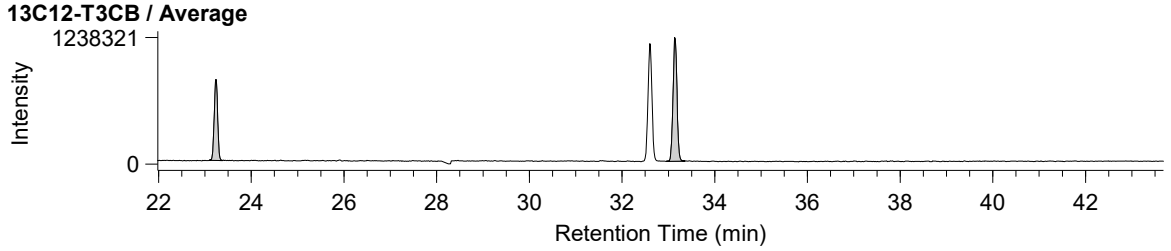


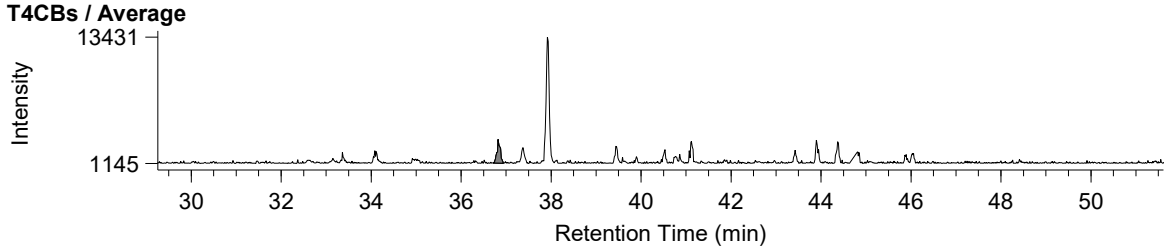
T3CBs / Average



13C12-T3CB / Average



T4CBs / Average



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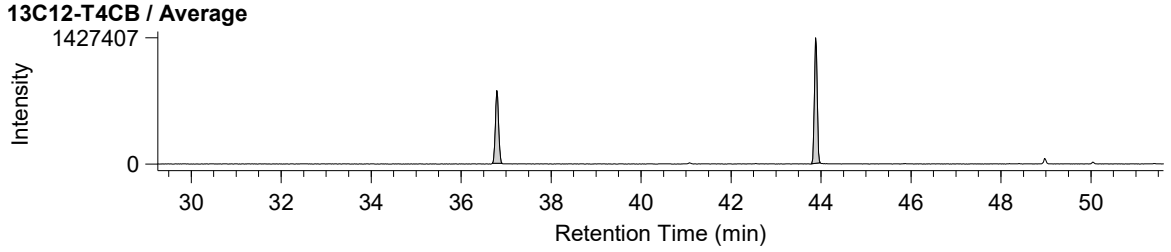
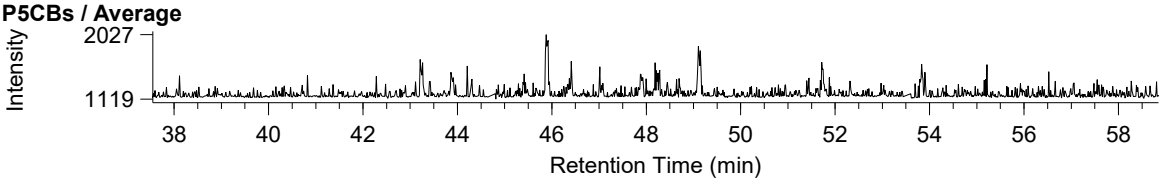


Compound View

DqData : M:\Diok\DqData\2023\C3E115\HT8-PCB(1-10)-102-2
Injection : C3E115006X

P5CBs / Average



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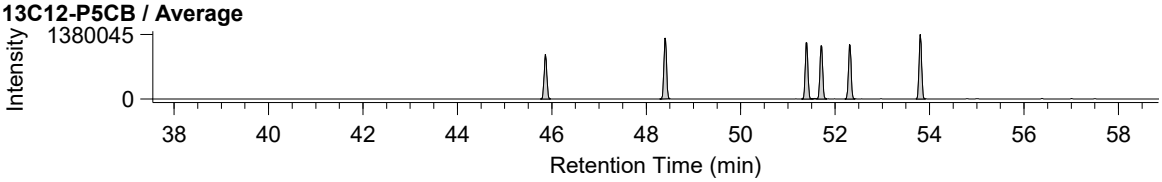

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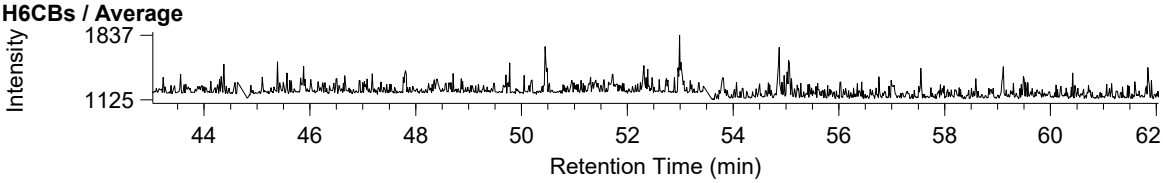

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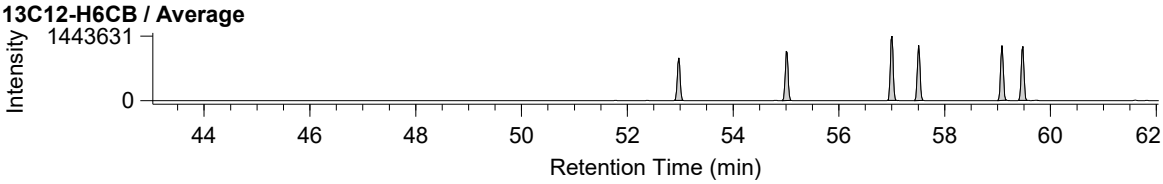

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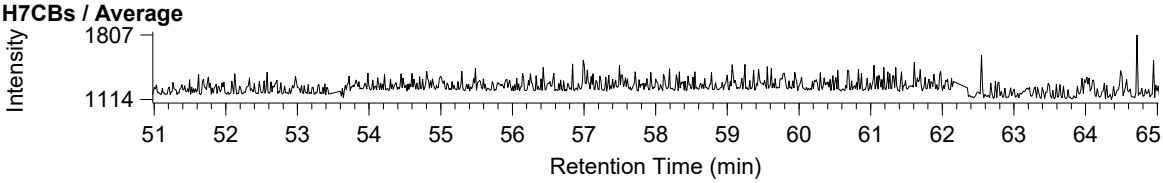

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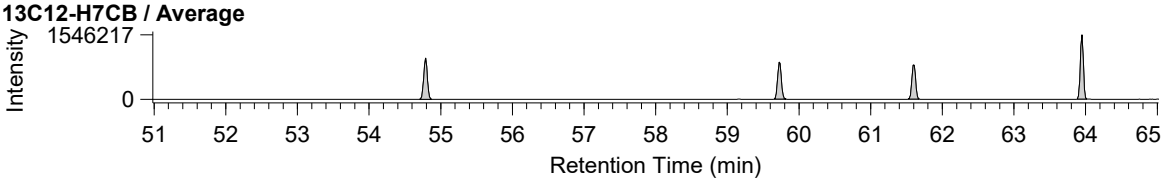
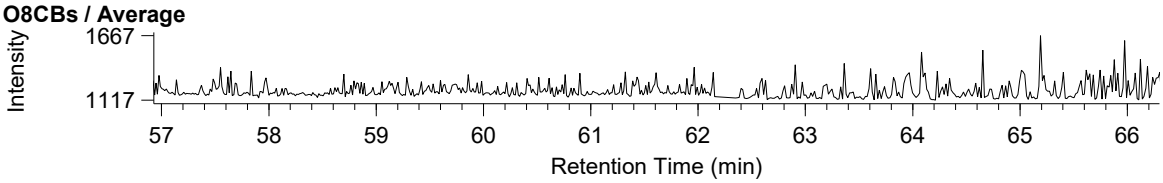


O8CBs / Average



13C12-O8CB / Average