Fast and Efficient Dioxins Analysis With Optimal GC Column Selectivity

Presented By

Ramkumar Dhandapani PhD
Global Product Manager –GC
Phenomenex

Overview

Introduction

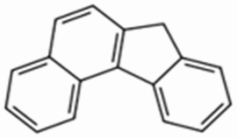
- POPs
- Resolution Improvement
- Current Challenges with Dioxin analysis

ZB-Dioxin Applications

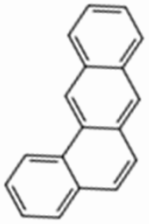
- Exceeding EPA 1613 requirement
- Balance of Resolution and Speed
- Extend lifetime with Guardian
- Dioxins & PCBs
- Robustness with Real Sample Matrix

What Are POP?

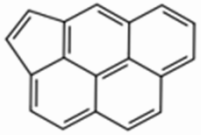
PAH



Benzo[c]fluorene

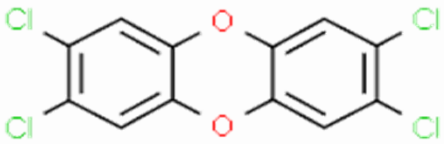


Benz[a] anthracene



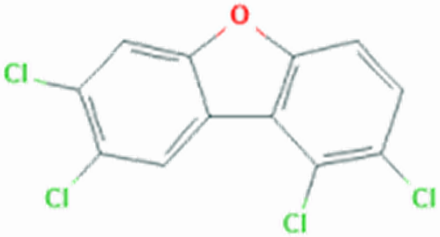
Cyclopenta[cd]pyrene

Dioxins



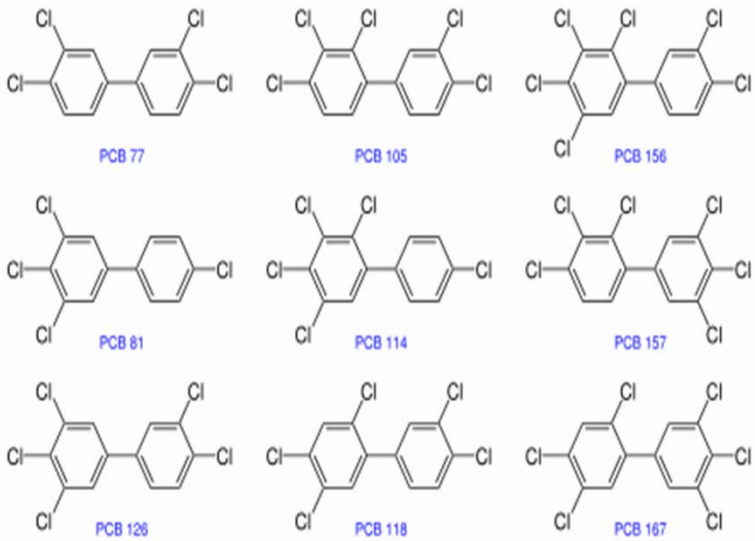
2,3,7,8-TCDD

Furans



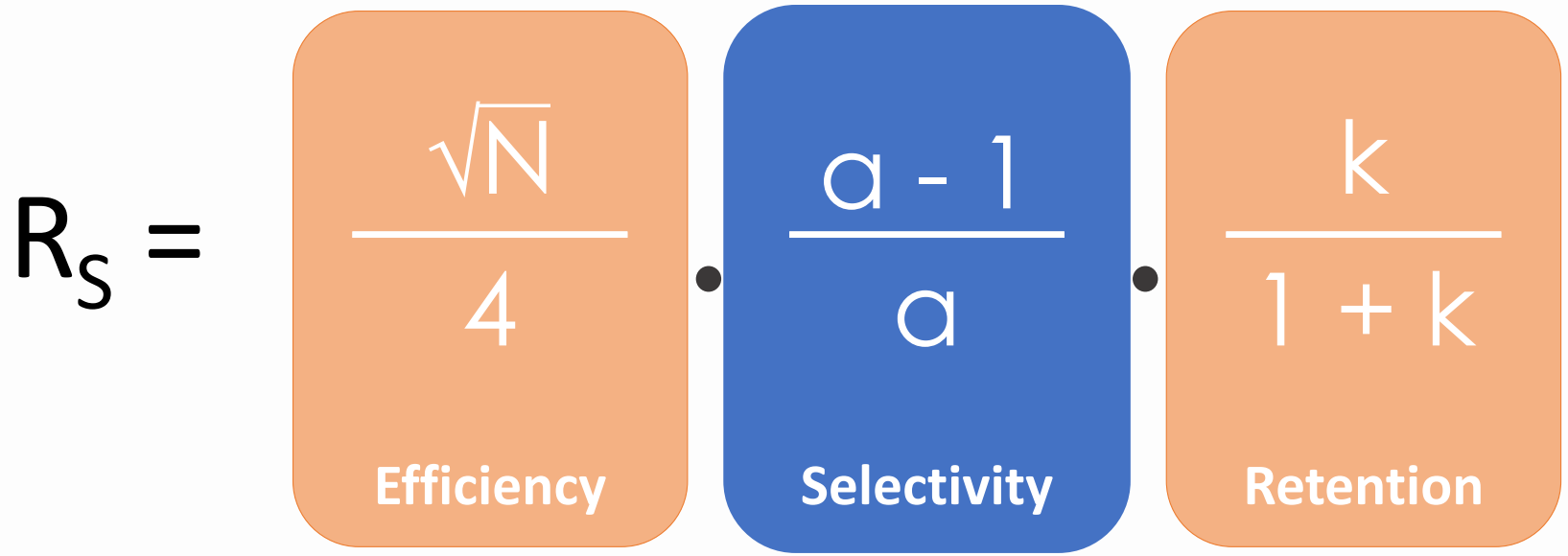
2,3,7,8-TCDF

PCB



Factors Influencing Resolution

Master Resolution Equation

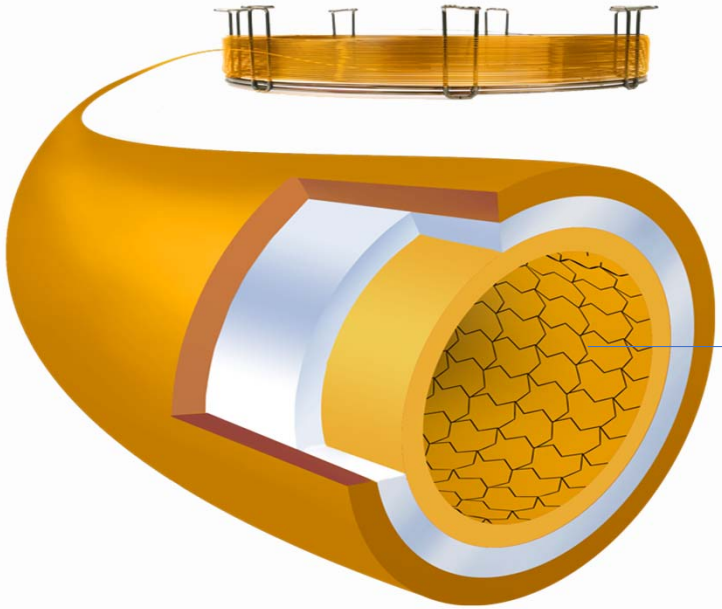


N = theoretical plates

α = k_2/k_1

k = $(t_R - t_o)/t_o$

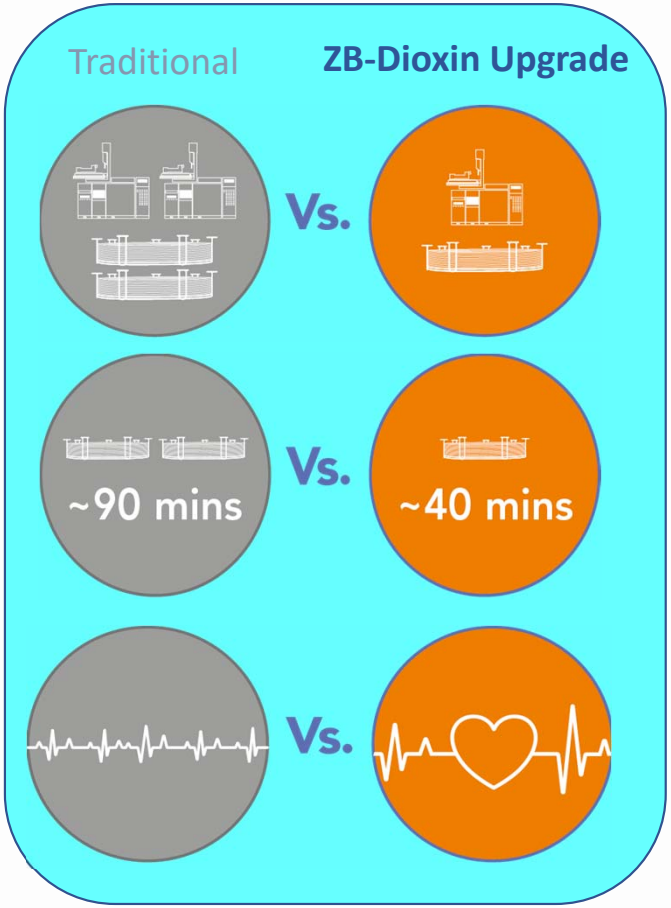
New Zebron ZB-Dioxin GC Column



- Unique selectivity for improved 2,3,7,8-TCDD and 2,3,7,8-TCDF resolution
- Ideal selectivity for Dioxins and PCBs
- Optimal phase volume ratio for shorter run time
- Extensive cross-linking through ESC™ (Engineered Self Cross-Linking™) for low bleed and high temperature stability to minimize GC-MS maintenance and system downtime

ZB-Dioxin is a Single column solution for Dioxins & PCBs

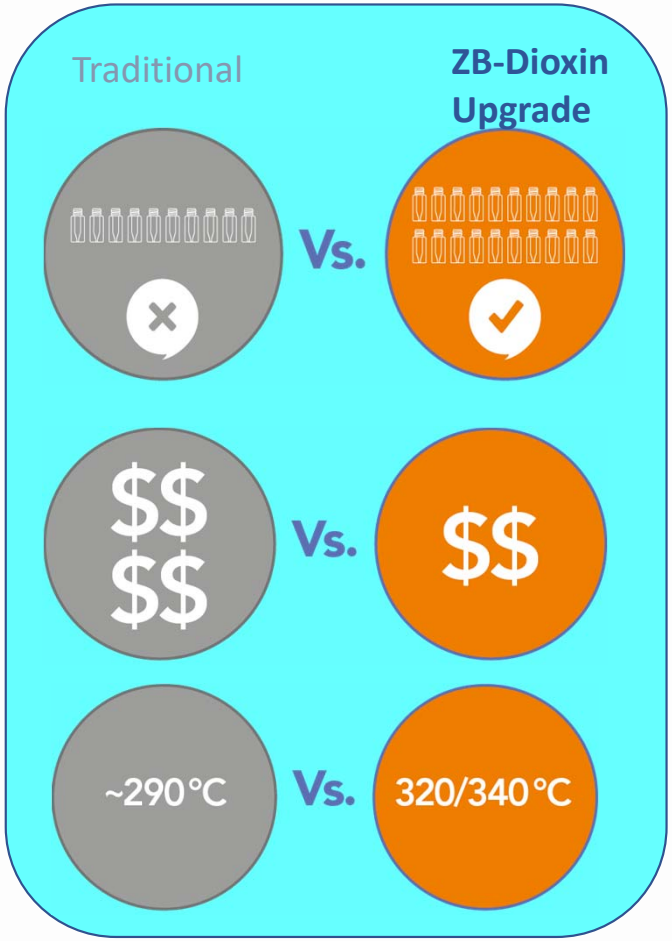
Current Challenges with Dioxin Analysis



Single Column
Single GC-HRMS

Fast Analysis

Extended lifetime
with Guardian



High Throughput

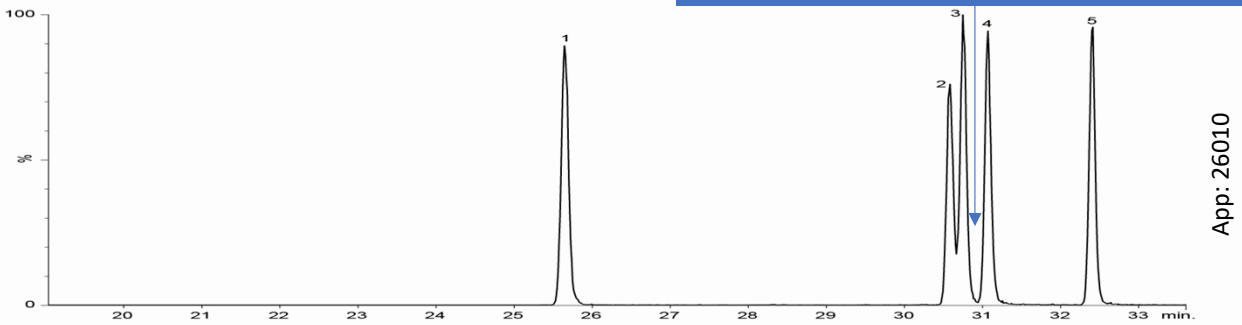
Single Column for
PCB & Dioxin

High Temperature
Resistance

TCDD Analysis- Exceeding EPA-1613 Requirement

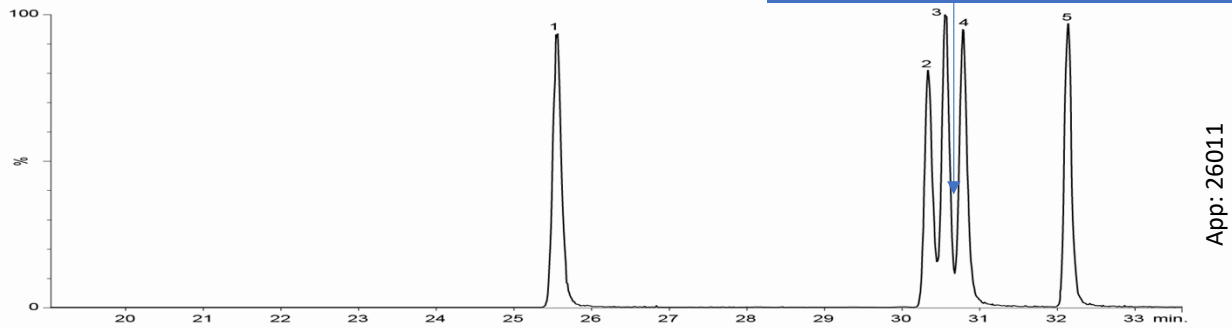
2,3,7,8-TCDD on Zebron ZB-Dioxin GC column
60 meter x 0.25 mm x 0.20 µm

High Resolution of 2,3,7,8-TCDD, exceeds 25% valley EPA-1613 method requirement. This extends column lifetime



2,3,7,8-TCDD on Brand A Premium 5MS
60 meter x 0.25 mm x 0.25 µm

2,3,7,8-TCDD is not completely resolved and reduces lifetime



GC-HRMS Method Conditions

Column1: Zebron™ ZB-Dioxin
Dimensions: 60 meter x 0.25 mm x 0.20 µm
Part No.: 7KG-G045-10
Column2: Brand A Premium 5ms
Dimensions: 60 meter x 0.25 mm x 0.25 µm
Injection: Pulse Splitless (2.0 min, 60 psi) @ 280 °C, 1 µL

Recommended Liner: Zebron PLUS 4 mm ID Single Taper
Liner Part No.: AG2-0A10-05

Carrier Gas: Helium @ 1.25 mL/min (constant flow)

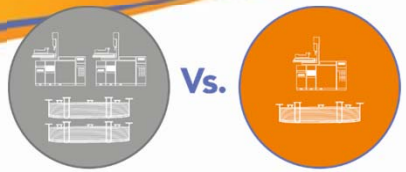
Oven Program: 160 °C for 2.4 min to 200 °C @ 25 °C/min to 220 °C @ 5 °C/min for 19 min to 288 °C @ 4 °C/min to 300 °C @ 5 °C/min for 7.6 min

Detector: HRMS
Transfer line Temperature: 300 °C

Analyte:

1. 1,3,6,8-TCDD
2. 2. 1,2,3,7-TCDD
3. 3. 1,2,3,8-TCDD
4. 4. 2,3,7,8-TCDD
5. 5. 1,2,8,9-TCDD

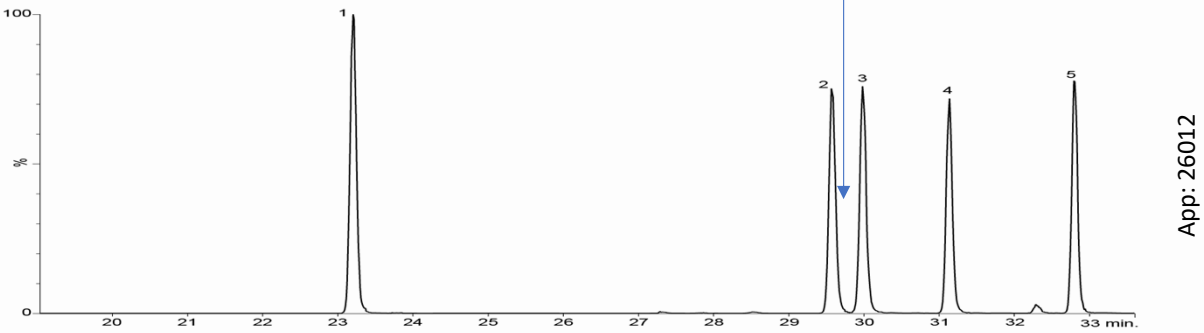
Single Column Solution for EPA-1613



Zebron ZB-Dioxin GC column

60 meter x 0.25 mm x 0.20 µm

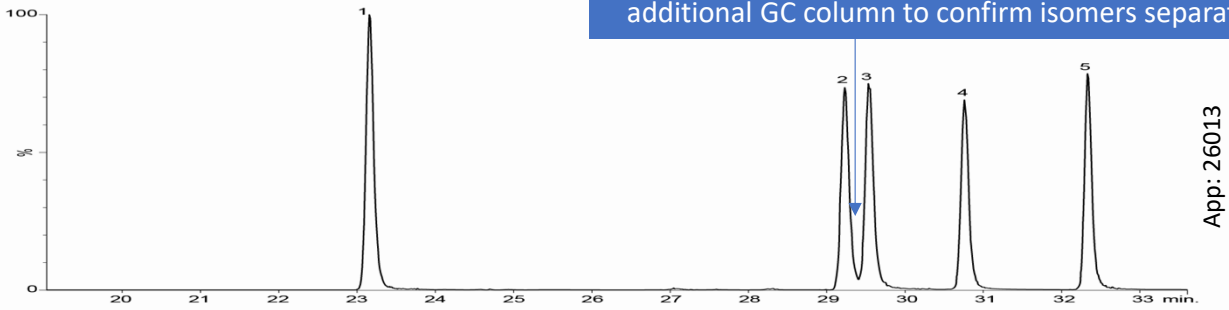
Complete resolution of 2,3,7,8-TCDF on a single column ZB-Dioxin



Brand A Premium 5MS

60 meter x 0.25 mm x 0.25 µm

2,3,7,8-TCDF are not completely resolved and need an additional GC column to confirm isomers separation



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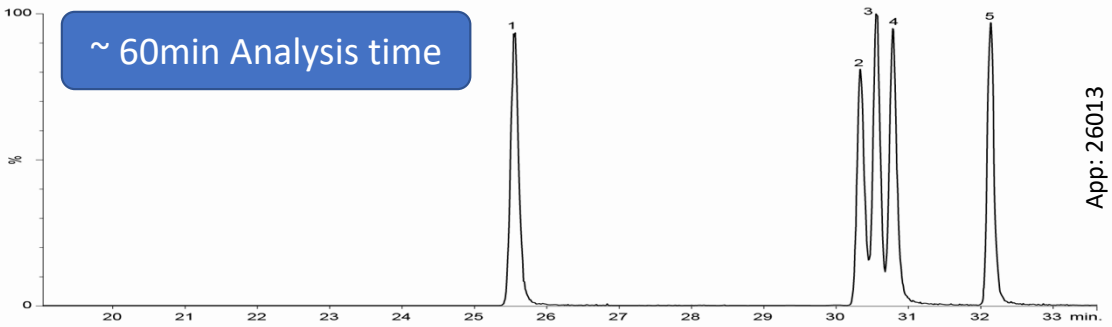
Detector: HRMS
Transfer line Temperature: 300 °C

Analyte:

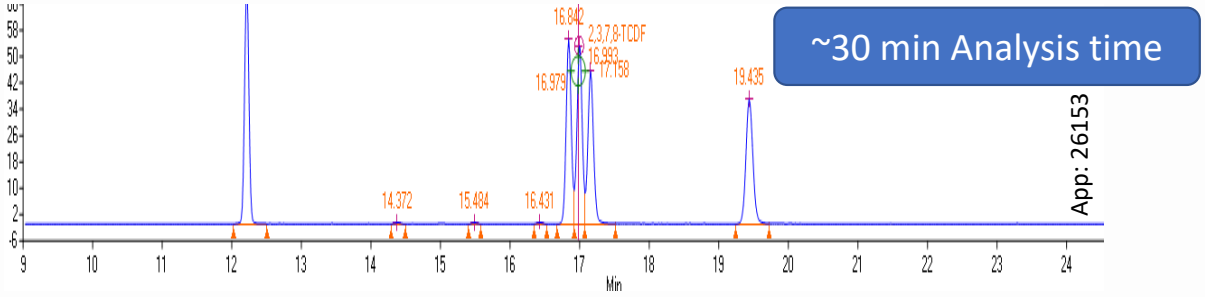
1. 1,3,6,8-TCDF
2. 1,3,4,7-TCDF
3. 2,3,7,8-TCDF
4. 1,2,3,9-TCDF
5. 1,2,8,9-TCDF

Traditional Solution for EPA-1613

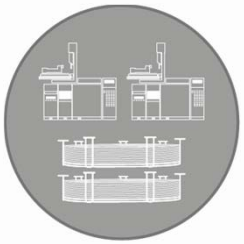
Incomplete TCDF separation on a traditional **5MS Phase**



TCDF Isomer separation on **225 Phase**

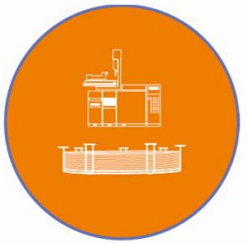


Traditional



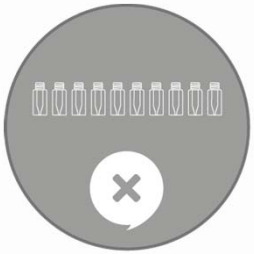
Vs.

ZB-Dioxin Upgrade



- 5MS phase does not completely resolve TCDF
- Isomer specific separation to be confirmed on a 225 phase

Traditional



Vs.

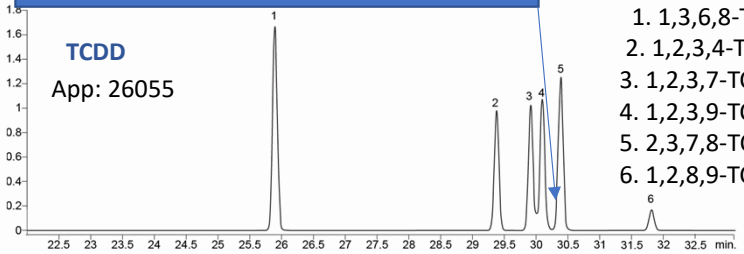
ZB-Dioxin Upgrade

GC-MS/MS analysis of Dioxins on Zebron ZB-Dioxin GC Columns

Complete Baseline separation of 2,3,7,8-TCDD using SINGLE column ZB-Dioxin

TCDD

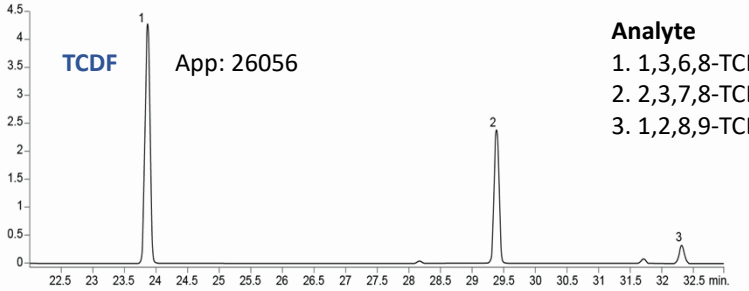
App: 26055



Analyte

1. 1,3,6,8-TCDD
2. 1,2,3,4-TCDD
3. 1,2,3,7-TCDD/ 1,2,3,8-TCDD
4. 1,2,3,9-TCDD
5. 2,3,7,8-TCDD
6. 1,2,8,9-TCDD

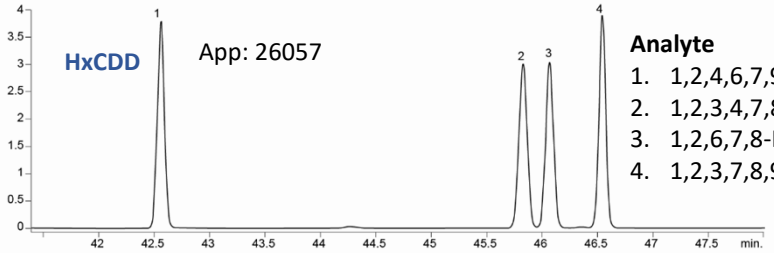
TCDF App: 26056



Analyte

1. 1,3,6,8-TCDF
2. 2,3,7,8-TCDF
3. 1,2,8,9-TCDF

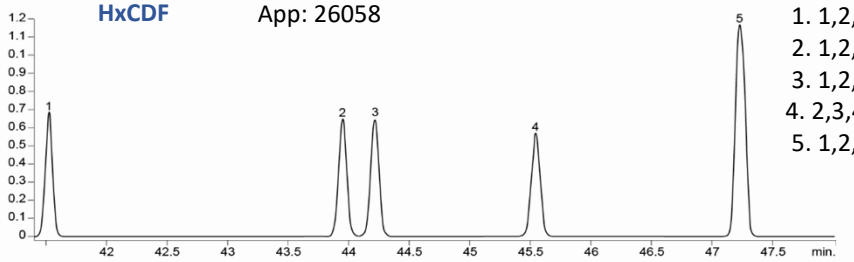
HxCDD App: 26057



Analyte

1. 1,2,4,6,7,9-HxCDD
2. 1,2,3,4,7,8-HxCDD
3. 1,2,6,7,8-HxCDD
4. 1,2,3,7,8,9-HxCDD

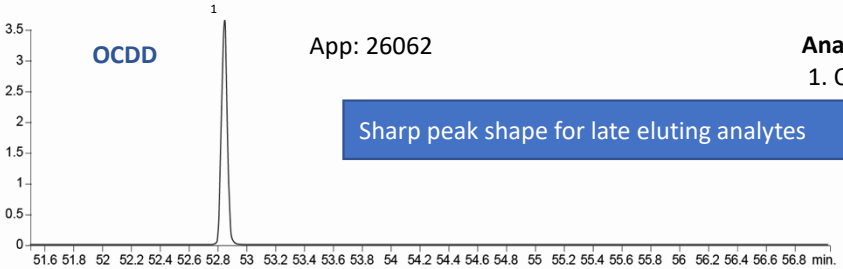
HxCDF App: 26058



Analyte

1. 1,2,3,4,7,8-HxCDF
2. 1,2,3,4,6,8-HxCDF
3. 1,2,3,6,7,8-HxCDF
4. 2,3,4,6,7,8-HxCDF
5. 1,2,3,7,8,9-HxCDF

OCDD App: 26062

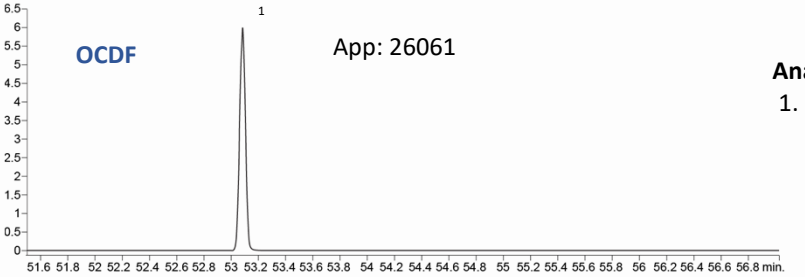


Analyte

1. OCDD

Sharp peak shape for late eluting analytes

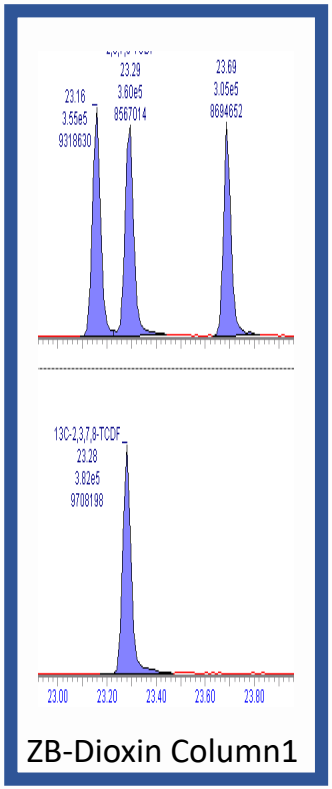
OCDF App: 26061



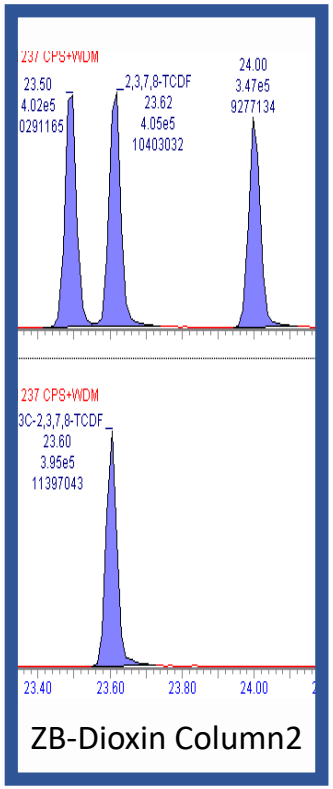
Analyte

1. OCDF

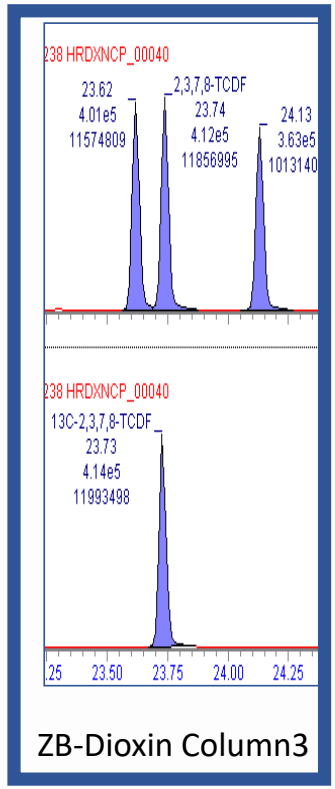
ZB-Dioxin Column to Column Reproducibility



ZB-Dioxin Column1



ZB-Dioxin Column2



ZB-Dioxin Column3

App: 26076

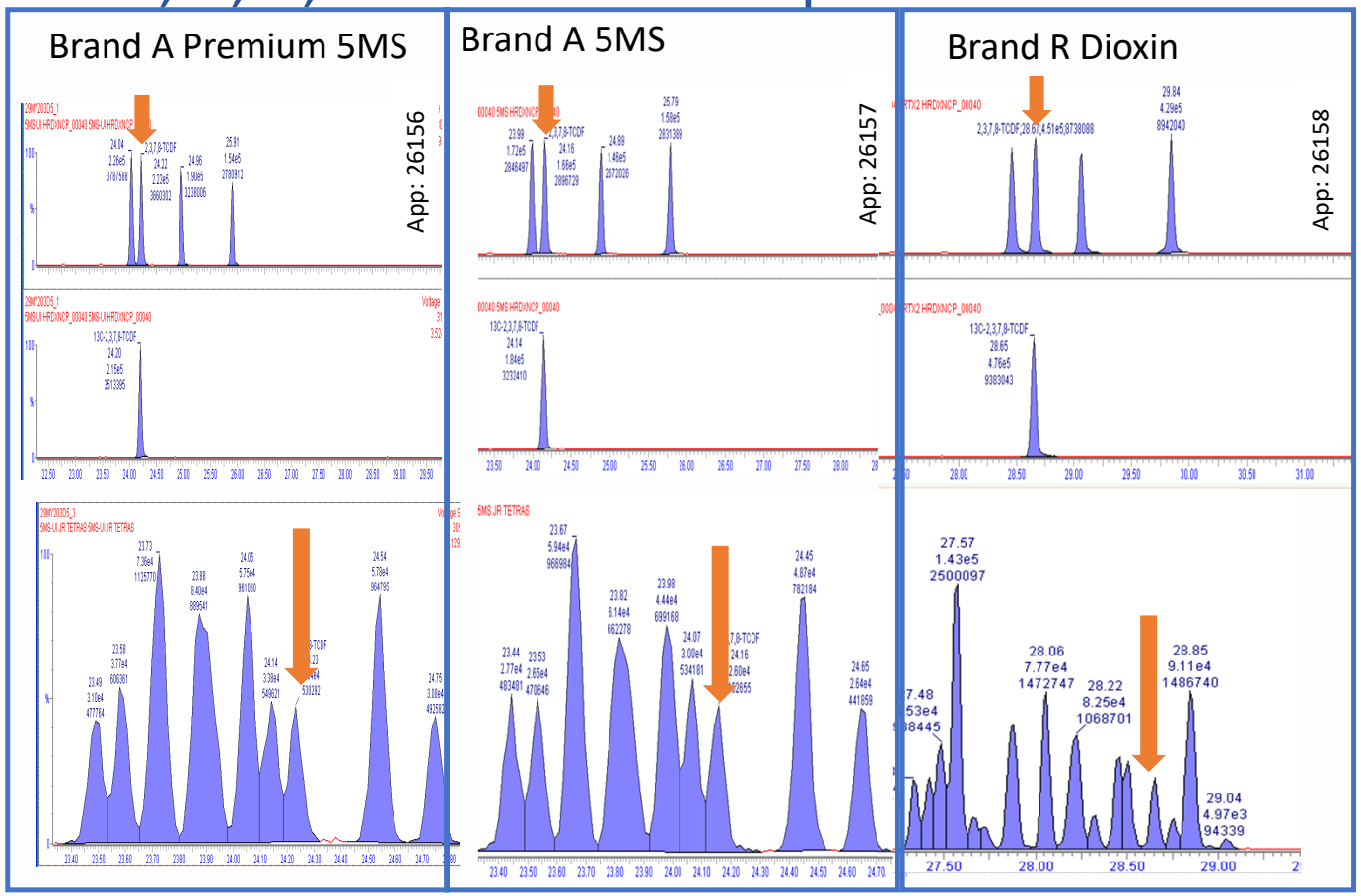
TCDF Separation

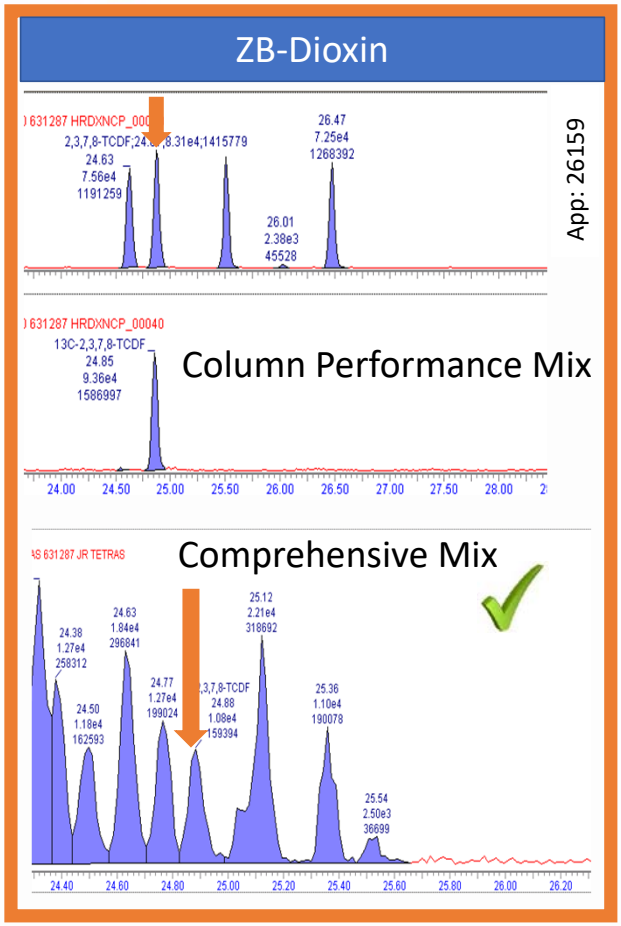
Compound	Relative Retention Time (RRT)			% RSD
	Column1	Column2	Column3	
2,3,7,8-TCDF	1.000	1.000	1.000	-
1,2,3,7,8-PeCDF	1.098	1.097	1.097	**0.05%**
2,3,4,7,8-PeCDF	1.127	1.127	1.128	**0.05%**
1,2,3,4,7,8-HxCDF	1.220	1.223	1.223	**0.14%**
1,2,3,6,7,8-HxCDF	1.225	1.227	1.228	**0.12%**
2,3,4,6,7,8-HxCDF	1.248	1.249	1.248	**0.05%**
1,2,3,7,8,9-HxCDF	1.280	1.277	1.277	**0.14%**
1,2,3,4,6,7,8-HpCDF	1.321	1.319	1.319	**0.09%**
1,2,3,4,7,8,9-HpCDF	1.382	1.381	1.381	**0.04%**
OCDF	1.482	1.485	1.485	**0.12%**

Low % RSD = Reproducible results

Why compromise your Dioxin analysis quality with an average separation on a 5% Phenyl GC phase when you can upgrade to the Zebron ZB-Dioxin that is specially tailored for reproducible Dioxin analysis.

2,3,7,8-TCDF Comparison

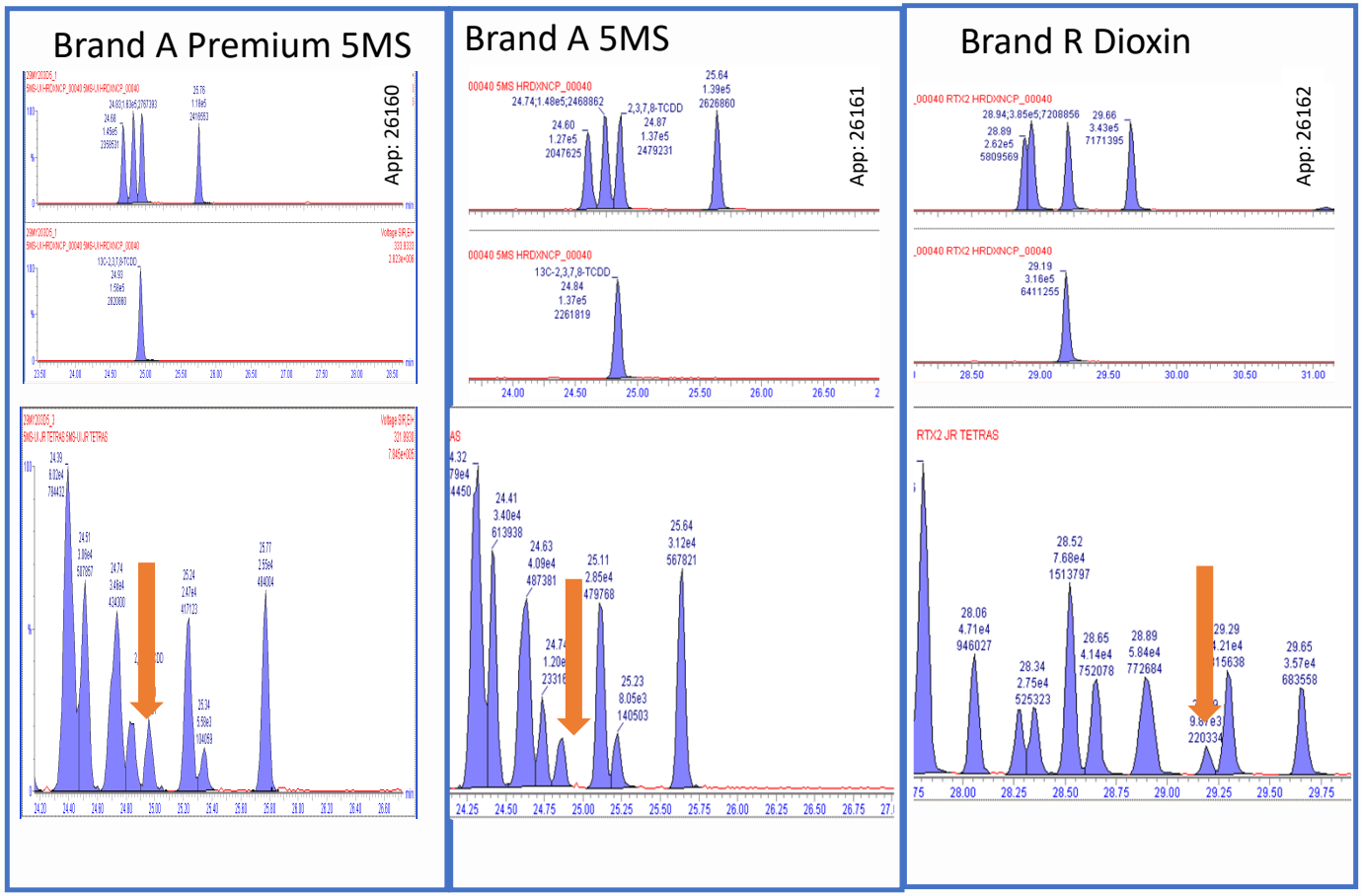


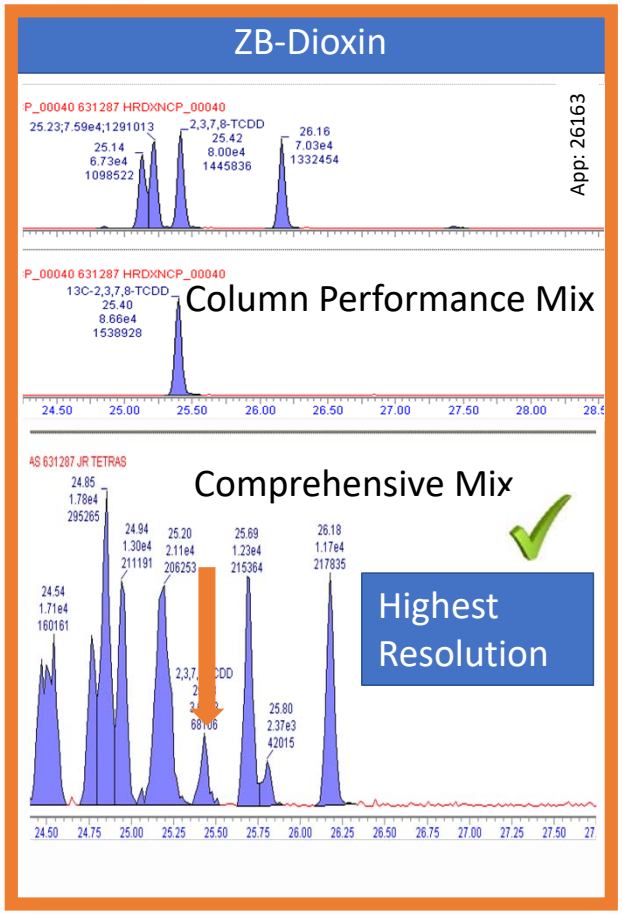


→ 2,3,7,8-TCDF

Comparative separations may not be representative of all applications.

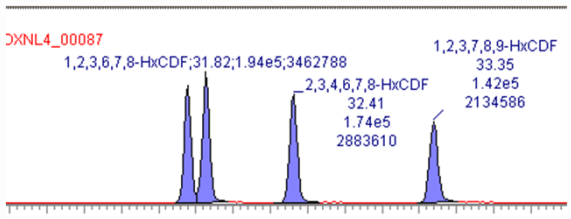
2,3,7,8-TCDD Comparison



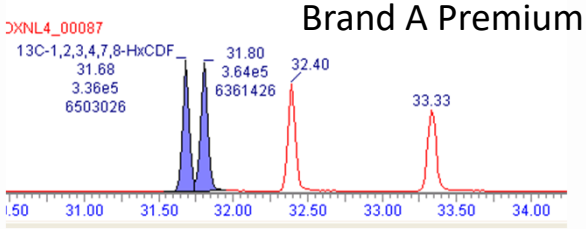


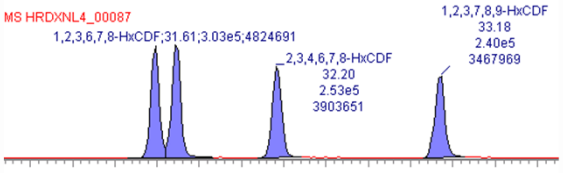
Comparative separations may not be representative of all applications.

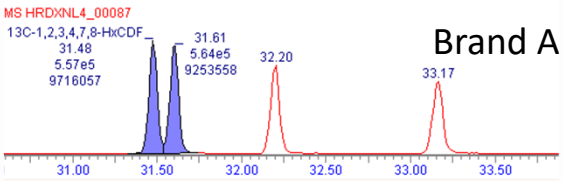
Hexa Furan Separation



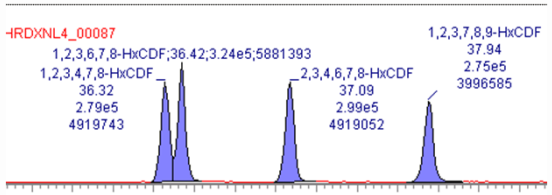
Brand A Premium 5MS

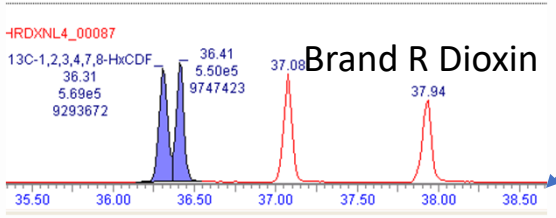






Brand A 5MS

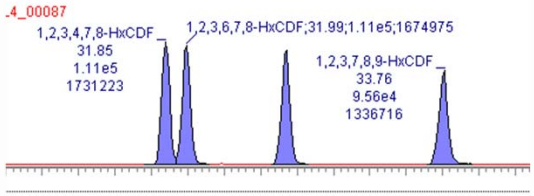


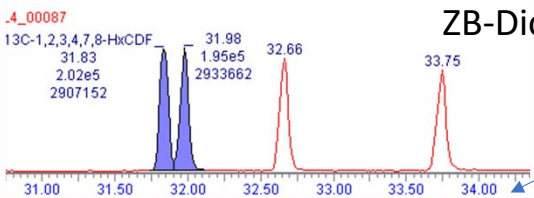


Brand R Dioxin

Long Run time

App: 26165





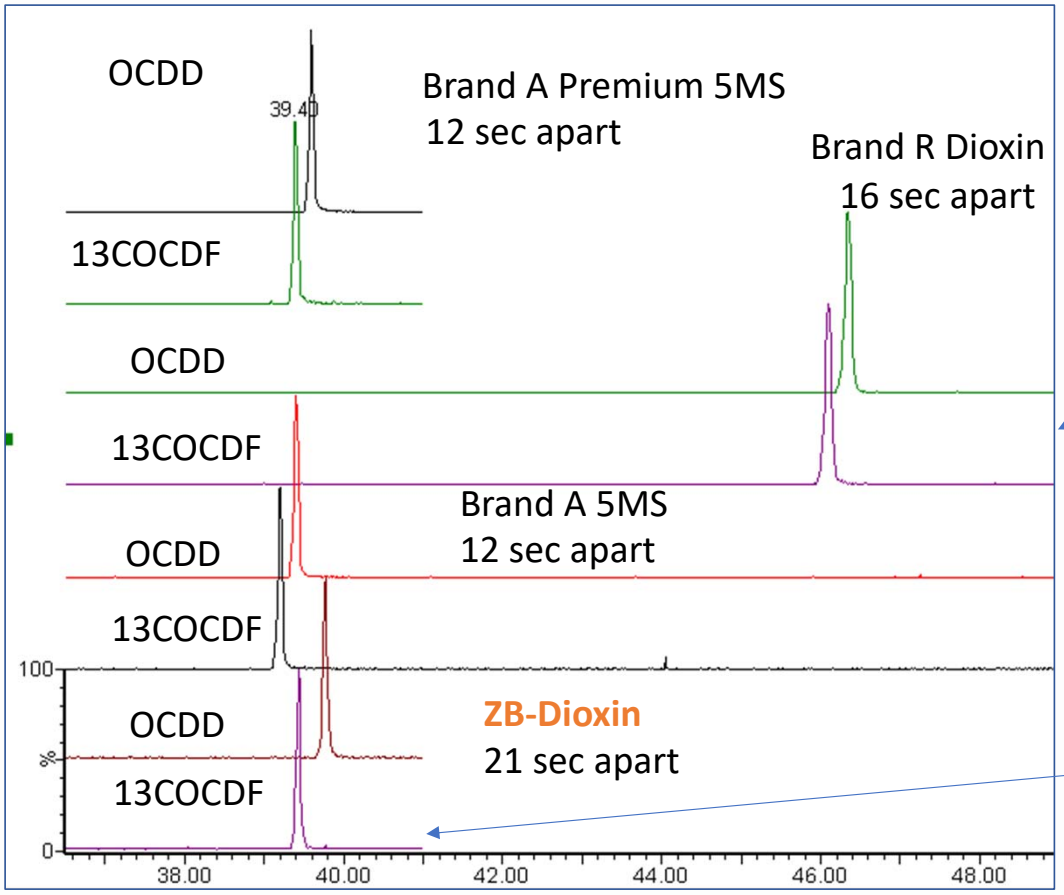
ZB-Dioxin

Short Run time and better separation

Comparative separations may not be representative of all applications.

Better Separation of Octa Dioxin/Furan on ZB-Dioxin

~90 mins Vs. ~40 mins



Long Analysis Time

App: 26166

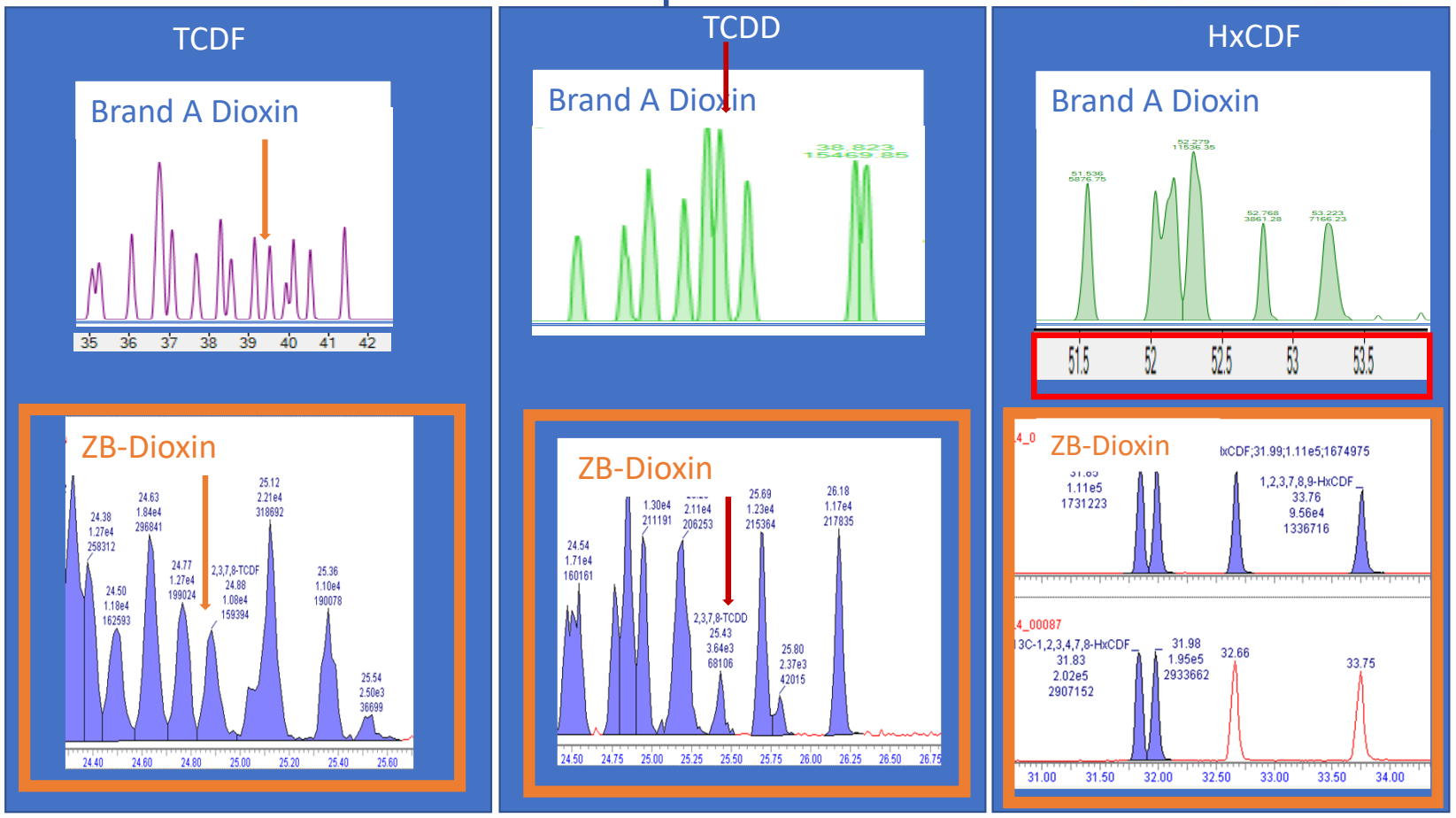
Highest Resolution= Accurate quantification

Short Analysis Time

Comparative separations may not be representative of all applications.

ZB-Dioxin Comparison

Zebron™ GC Columns



2,3,7,8-TCDF

2,3,7,8-TCDD

Brand A Dioxin **ZB-Dioxin Upgrade**

~290°C Vs. 320/340°C

Brand A Dioxin

- Poor resolution
- Long Run time
- 290^{0}C
- OCDF did not elute till 60min

ZB-Dioxin Upgrade

- Better Resolution
- Shorter Run time
- 320/340^{0}C
- Up to 35% faster analysis

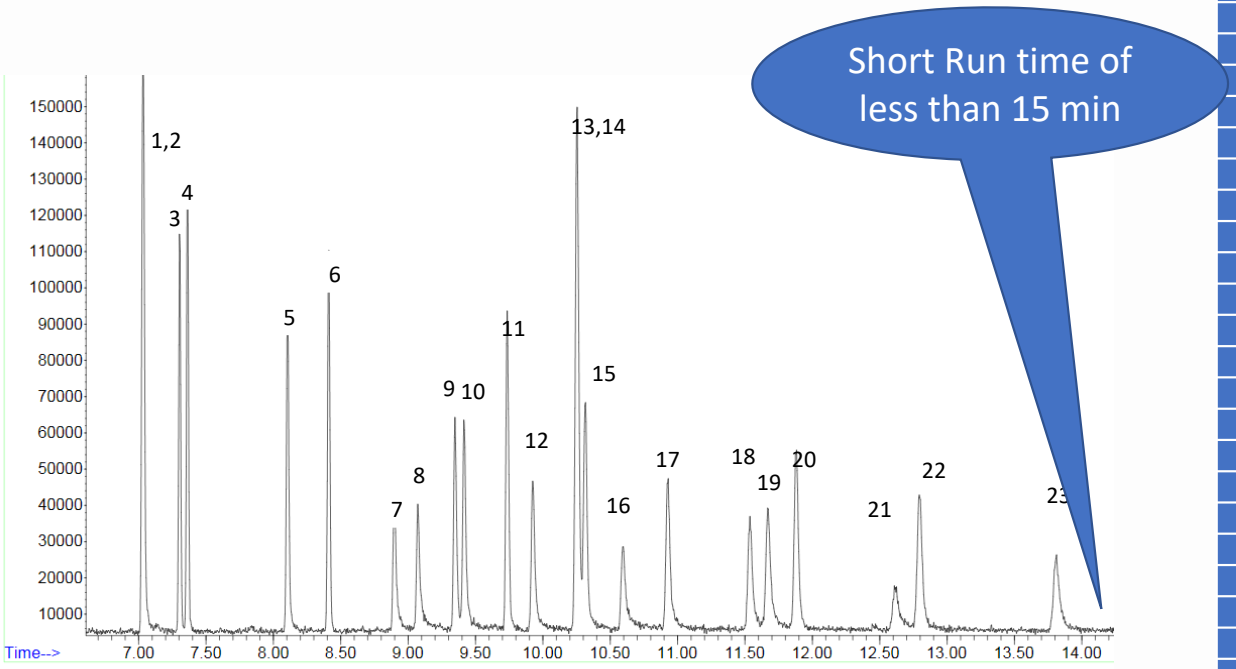
Comparative separations may not be representative of all applications.

Zebron™ GC Columns

ZB-Dioxin : Combination of Better Resolution and Speed

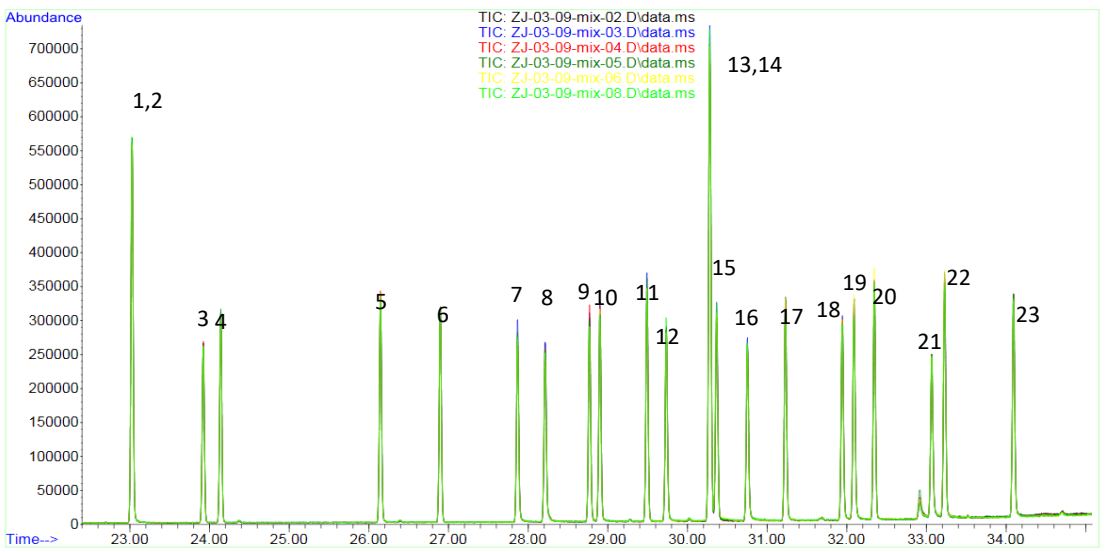
Separation Criteria	Brand A Premium 5MS	Brand A 5MS	Brand A Dioxin	Brand R Dioxin	ZB-Dioxin
2,3,7,8-TCDF Column Performance Test Mix	Requires additional column	Requires additional column	Requires additional column	Passes	Exceeds Requirement/ Single column solution ✓
2,3,7,8-TCDF Comprehensive Test Mix	Fail	Fail	Fail	Passes	Exceeds Requirement/ Single column solution ✓
2,3,7,8-TCDD Column Performance Test Mix	Pass	Pass	Pass	Pass	Exceeds 25% valley resolution ✓
2,3,7,8- TCDD Comprehensive Test Mix	Close to 25%	Close to 25%	Close to 25%	Pass	Exceeds Requirement ✓
OCDD/13C-OCDF Separation	12 sec	16 sec	Not eluted within time window	12 sec	21 sec ✓
Run time	40 min	40 min	60+ min	48 min	40 min ✓
Temperature Limit	325/350 °C	325/350 °C	~290 ^{0}C	320/340 ^{0}C	320/340 ^{0}C ✓

Fast PCB Analysis on a 40m ZB-Dioxin GC Column



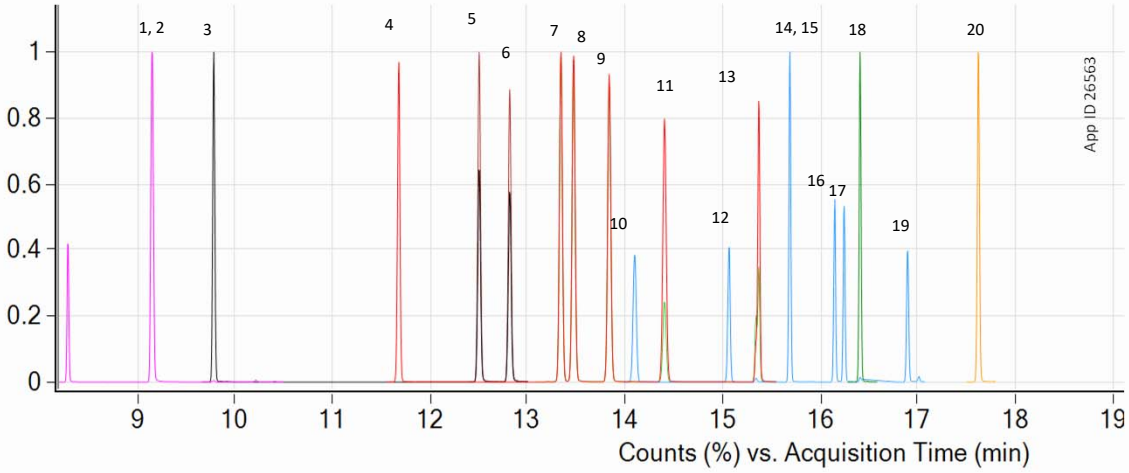
Number	PCB	Description
1	PCB 31	2,4',5-Trichlorobiphenyl
2	PCB 28	2,4,4'-Trichlorobiphenyl
3	PCB 69	2,3',4,6-Tetrachlorobiphenyl
4	PCB 52	2,2',5,5'-Tetrachlorobipheny
5	PCB 70	2,3',4',5-Tetrachlorobiphenyl
6	PCB101	2,2',4,5,5'-Pentachlorobiphenyl
7	PCB 81	3,4,4',5-Tetrachlorobiphenyl
8	PCB 77	3,3',4,4'-Tetrachlorobiphenyl
9	PCB 123	2',3,4,4',5-Pentachlorobiphenyl
10	PCB 118	2,3',4,4',5-Pentachlorobiphenyl
11	PCB 153	2,2',4,4',5,5'-Hexachlorobiphenyl
12	PCB 105	2,3,3',4,4'-Pentachlorobiphenyl
13	PCB 164	2,3,3',4',5',6-Hexachlorobiphenyl
14	PCB 163	2,3,3',4',5,6-Hexachlorobiphenyl
15	PCB 138	2,2',3,4,4',5'-Hexachlorobiphenyl
16	PCB 126	3,3',4,4',5-Pentachlorobiphenyl
17	PCB 167	2,3',4,4',5,5'-Hexachlorobiphenyl
18	PCB 156	2,3,3',4,4',5-Hexachlorobiphenyl
19	PCB 157	2,3,3',4,4',5'-Hexachlorobiphenyl
20	PCB 180	2,2',3,4,4',5,5'-Heptachlorobiphenyl
21	PCB 169	3,3',4,4',5,5'-Hexachlorobiphenyl
22	PCB 170	2,2',3,3',4,4',5-Heptachlorobiphenyl
23	PCB 189	2,3,3',4,4',5,5'-Heptachlorobiphenyl

Reproducible PCB Analysis on a 60m ZB-Dioxin GC Column



Number	Compound	Description	RT (min)	Area-RSD%
1	PCB 31	2,4',5-Trichlorobiphenyl	23.026	0.666
2	PCB 28	2,4,4'-Trichlorobiphenyl		
3	PCB 69	2,3',4,6-Tetrachlorobiphenyl	23.922	0.763
4	PCB 52	2,2',5,5'-Tetrachlorobipheny	24.140	0.962
5	PCB 70	2,3',4',5-Tetrachlorobiphenyl	26.145	0.472
6	PCB101	2,2',4,5,5'-Pentachlorobiphenyl	26.897	0.976
7	PCB 81	3,4,4',5-Tetrachlorobiphenyl	27.867	2.696
8	PCB 77	3,3',4,4'-Tetrachlorobiphenyl	28.212	2.228
9	PCB 123	2',3,4,4',5-Pentachlorobiphenyl	28.770	2.660
10	PCB 118	2,3',4,4',5-Pentachlorobiphenyl	28.901	2.626
11	PCB 153	2,2',4,4',5,5'-Hexachlorobiphenyl	29.492	1.652
12	PCB 105	2,3,3',4,4'-Pentachlorobiphenyl	29.734	2.262
13	PCB 164	2,3,3',4',5',6-Hexachlorobiphenyl	30.280	2.302
14	PCB 163	2,3,3',4',5,6-Hexachlorobiphenyl		
15	PCB 138	2,2',3,4,4',5'-Hexachlorobiphenyl	30.365	3.382
16	PCB 126	3,3',4,4',5-Pentachlorobiphenyl	30.752	3.198
17	PCB 167	2,3',4,4',5,5'-Hexachlorobiphenyl	31.231	0.605
18	PCB 156	2,3,3',4,4',5-Hexachlorobiphenyl	31.943	0.670
19	PCB 157	2,3,3',4,4',5'-Hexachlorobiphenyl	32.089	2.127
20	PCB 180	2,2',3,4,4',5,5'-Heptachlorobiphenyl	32.346	1.292
21	PCB 169	3,3',4,4',5,5'-Hexachlorobiphenyl	33.065	1.653
22	PCB 170	2,2',3,3',4,4',5-Heptachlorobiphenyl	33.228	2.432
23	PCB 189	2,3,3',4,4',5,5'-Heptachlorobiphenyl	34.093	1.510

Fast GC-MS/MS Analysis of 20 PCBs



- 20 PCBs in less than 18 min
- Critical Pair separation
- Dioxins and PCB Spectral resolution

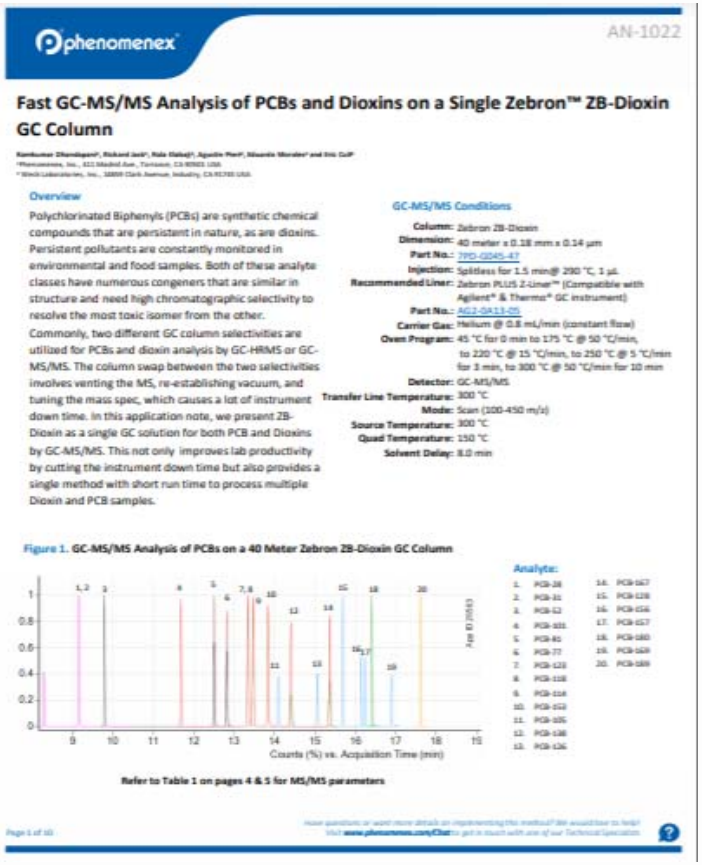
GC-MS Conditions

Column:	Zebron ™ ZB-Dioxin
Dimension:	40 meter x 0.18 mm x 0.14 µm
Part No.:	7PD-G045-47
Injection:	1 µL, Splitless for 1.5 min@ 290 °C
Recommended Liner:	Zebron™ PLUS Liner compatible with Agilent & Thermo GC instrument
Part No.:	AG2-0A13-05
Carrier Gas:	Helium @ 0.8 mL/min (constant flow)
Oven Program:	45 °C for 0 min to 175 °C @ 50 °C/min, to 220 °C @ 15 °C/min, to 250 °C @ 5 °C/min for 3 min, to 300 °C @ 50 °C/min for 10 min
Detector:	GC-MS
Transfer Line Temperature:	300 °C
Mode	Scan (100-450 m/z)
Source Temperature:	300 °C
Quad Temperature:	150 °C
Solvent delay:	8.0 min

Analyte:

1. PCB-28
2. PCB-31
3. PCB-52
4. PCB-101
5. PCB-81
6. PCB-77
7. PCB-123
8. PCB-118
9. PCB-114
10. PCB-153
11. PCB-105
12. PCB-138
13. PCB-126
14. PCB-167
15. PCB-128
16. PCB-156
17. PCB-157
18. PCB-180
19. PCB-169
20 PCB-189

Fast GC-MS/MS Analysis of PCBs



phenomenex AN-1022

Fast GC-MS/MS Analysis of PCBs and Dioxins on a Single Zebron™ ZB-Dioxin GC Column

Overview

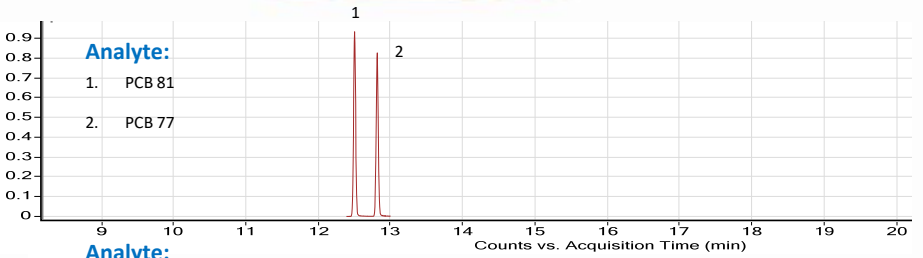
Polychlorinated Biphenyls (PCBs) are synthetic chemical compounds that are persistent in nature, as are dioxins. Persistent pollutants are constantly monitored in environmental and food samples. Both of these analyte classes have numerous congeners that are similar in structure and need high chromatographic selectivity to resolve the most toxic isomer from the other. Commonly, two different GC column selectivities are utilized for PCBs and dioxin analysis by GC-HRMS or GC-MS/MS. The column swap between the two selectivities involves venting the MS, re-establishing vacuum, and tuning the mass spec, which causes a lot of instrument down time. In this application note, we present ZB-Dioxin as a single GC solution for both PCB and Dioxins by GC-MS/MS. This not only improves lab productivity by cutting the instrument down time but also provides a single method with short run time to process multiple Dioxin and PCB samples.

GC-MS/MS Conditions

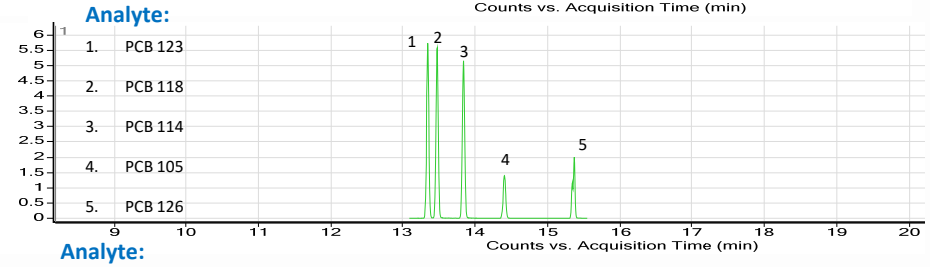
Column: Zebron ZB-Dioxin
Dimension: 40 meter x 0.18 mm x 0.14 µm
Part No.: 7PD-GD45-47
Injection: Splitless for 1.5 min@ 290 °C, 1 µL
Recommended Liner: Zebron PLUS Z-Liner™ (Compatible with Agilent® & Thermo® GC instrument)
Part No.: AG2-SA13-05
Carrier Gas: Helium @ 0.8 mL/min (constant flow)
Oven Program: 45 °C for 0 min to 175 °C @ 50 °C/min, to 220 °C @ 15 °C/min, to 250 °C @ 5 °C/min for 3 min, to 300 °C @ 50 °C/min for 10 min
Detector: GC-MS/MS
Transfer Line Temperature: 300 °C
Mode: Scan (100-450 m/z)
Source Temperature: 300 °C
Quad Temperature: 150 °C
Solvent Delay: 8.0 min

Figure 1. GC-MS/MS Analysis of PCBs on a 40 Meter Zebron ZB-Dioxin GC Column

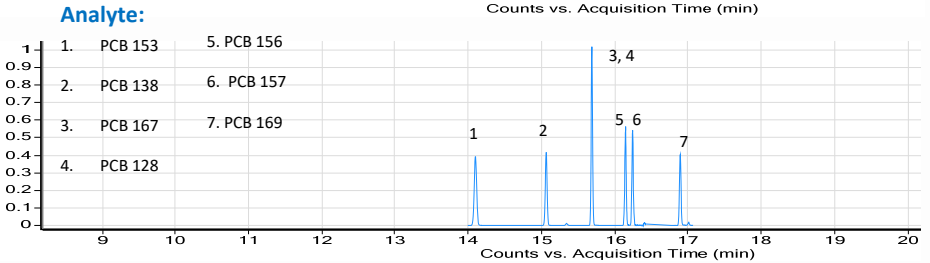
Refer to Table 1 on pages 4 & 5 for MS/MS parameters



App ID 26566



App ID 26564



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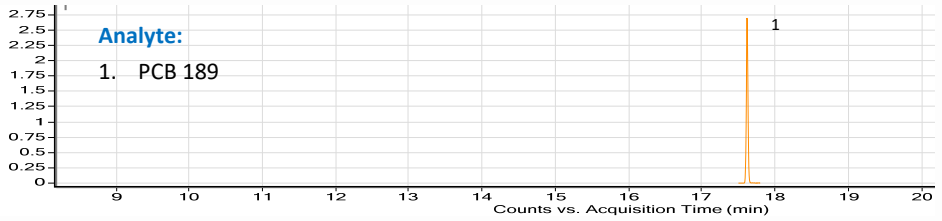
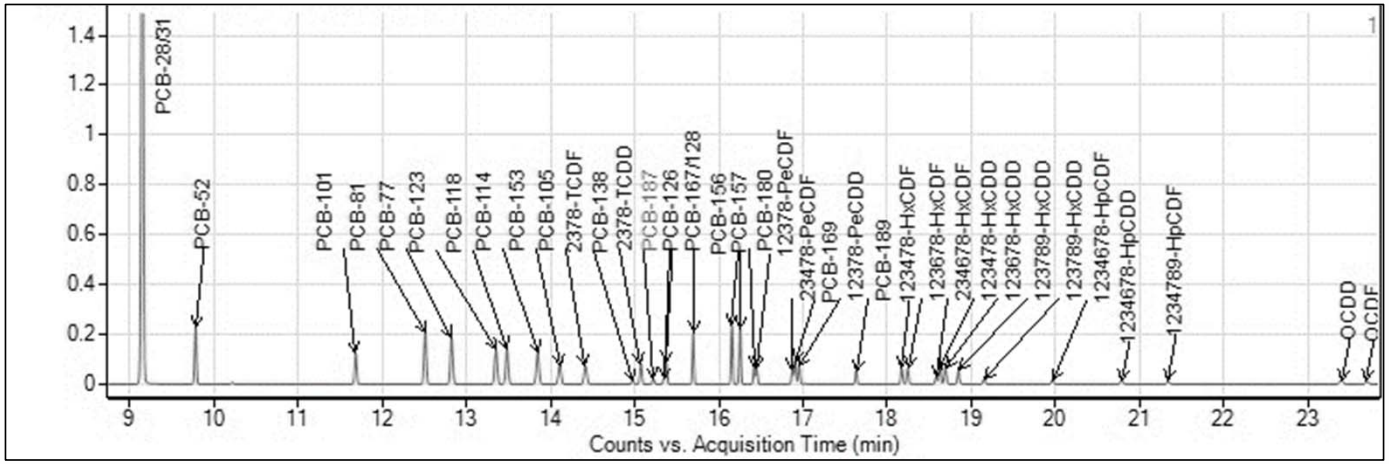


App ID 26573

Fast GC-MS/MS Analysis of 36 Dioxins & PCBs

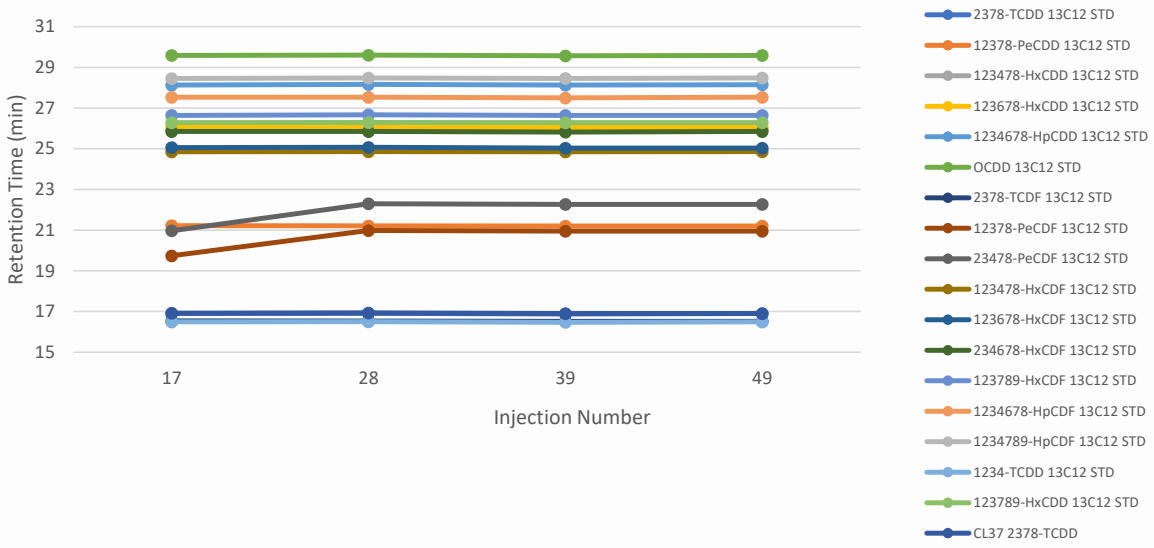


- Optimal 40m column for short run time
- 36 Dioxins & PCBs in less than 24 min
- Critical PCBs resolved
- Dioxins, Furans and PCBs spectrally resolved

Analyte:

1. PCB-28
2. PCB-31
3. PCB-52
4. PCB-101
5. PCB-81
6. PCB-77
7. PCB-123
8. PCB-118
9. PCB-114
10. PCB-153
11. PCB-105
12. 2,3,7,8-TCDF
13. PCB-138
14. 2,3,7,8-TCDD
15. PCB-187
16. PCB-126
17. PCB-167
18. PCB-128
19. PCB-156
20. PCB-157
21. PCB-180
22. 1,2,3,7,8-PeCDF
23. 2,3,4,7,8-PeCDF
24. PCB-169
25. 1,2,3,7,8-PeCDD
26. PCB-189
27. 1,2,3,4,7,8-HxCDF
28. 1,2,3,6,7,8-HxCDF
29. 2,3,4,7,8-HxCDF
30. 1,2,3,4,7,8-HxCDD
31. 1,2,3,6,7,8-HxCDD
32. 2,3,4,7,8-HxCDD
33. 1,2,3,4,6,7,8-HpCDD
34. 1,2,3,4,6,7,8-HpCDF
35. OCDD
36. OCDF

Robust Dioxin Analysis with Real Sample- Fly Ash

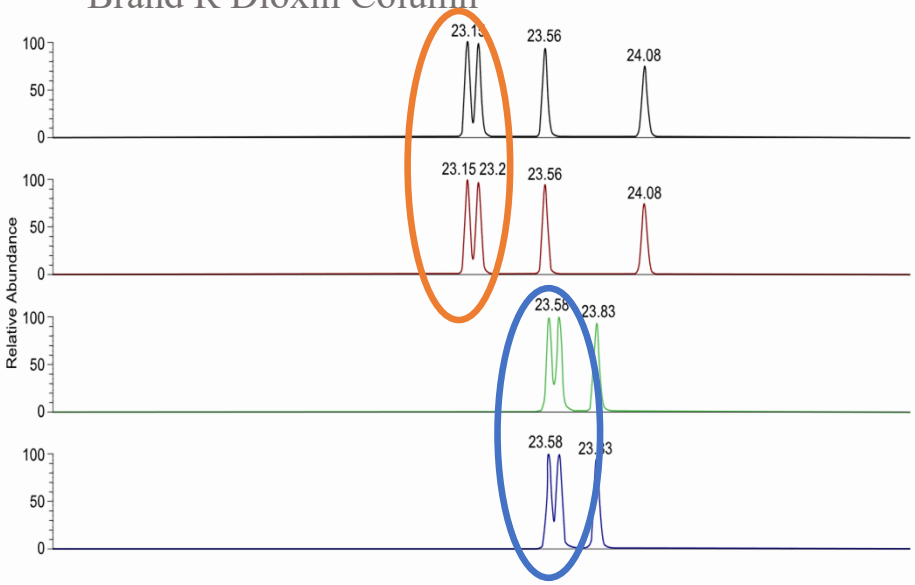


Reproducible Retention Time of Mid-Level Calibration Standards of Tetra through Octa Dioxins and Furans During the Robustness Experiment

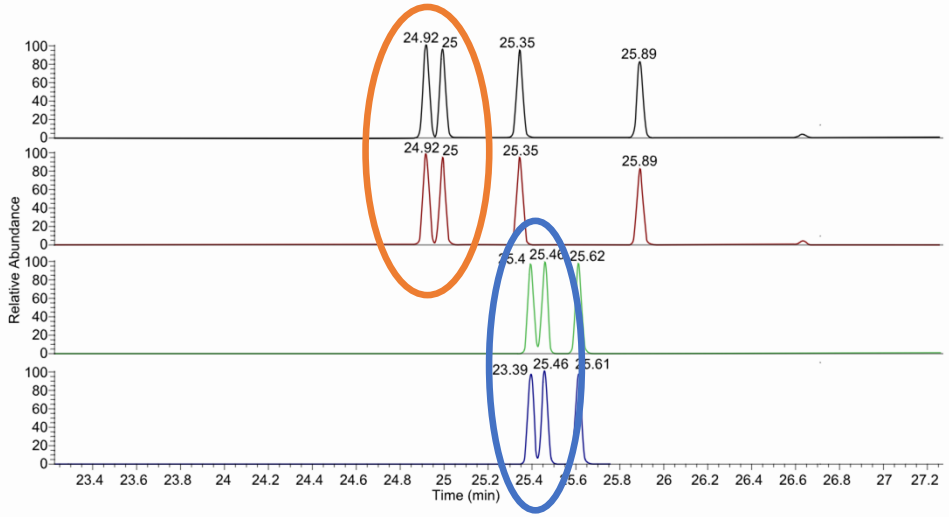
Retention Time (min)	Analyte Name	Internal Standard
15.18	1,3,6,8-TCDD	
16.95	2,3,7,8-TCDD	13C 2,3,7,8-TCDD
17.53	1,2,8,9-TCDD	
16.52	2,3,7,8-TCDF	13C 2,3,7,8-TCDF
17.83	1,2,8,9-TCDF	
18.67	1,2,4,7,9- PeCDD	
21.25	1,2,3,7,8-PeCDD	13C 1,2,3,7,8-PeCDD
21.76	1,2,3,8,9-PeCDD	
17.43	1,3,4,6,8-PeCDF	
19.78	1,2,3,7,8-PeCDF	13C 1,2,3,7,8-PeCDF
22.32	1,2,3,8,9-PeCDF	
20.79	2,3,4,7,8-PeCDF	13C 2,3,4,7,8-PeCDF
25.97	1,2,3,4,7,8-HxCDD	13C 1,2,3,4,7,8-HxCDD
23.81	1,2,4,6,7,9-HxCDD	
26.30	1,2,3,4,6,7-HxCDD	
26.08	1,2,3,6,7,8-HxCDD	13C 1,2,3,6,7,8-HxCDD
26.20	1,2,3,7,8,9-HxCDD	
24.90	1,2,3,4,7,8-HxCDF	13C 123478-HxCDF
25.08	1,2,3,6,7,8-HxCDF	13C 123678-HxCDF
26.69	1,2,3,7,8,9-HxCDF	13C 123789-HxCDF
26.69	1,2,3,4,8,9-HxCDF	
25.87	2,3,4,6,7,8-HxCDF	13C 2,3,4,6,7,8-HxCDF
28.15	1,2,3,4,6,7,9-HpCDD	13C 1,2,3,4,6,7,8-HpCDD
27.70	1,2,3,4,6,7,8-HpCDD	
27.55	1,2,3,4,6,7,8-HpCDF	13C 1,2,3,4,6,7,8-HpCDF
28.48	1,2,3,4,7,8,9-HpCDF	13C 1,2,3,4,7,8,9-HpCDF
29.59	OCDD	13C OCDD
29.74	OCDF	

Improved Resolution of Hexachlorinated Dioxins in Real Sample- Fish Oil

Brand R Dioxin Column

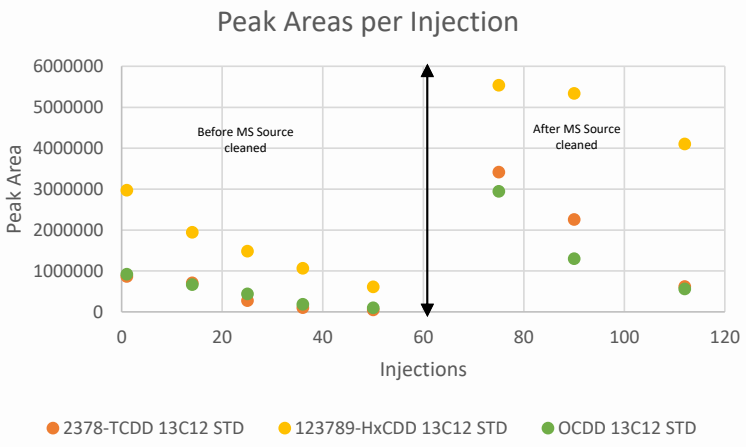


ZB-Dioxin GC Column

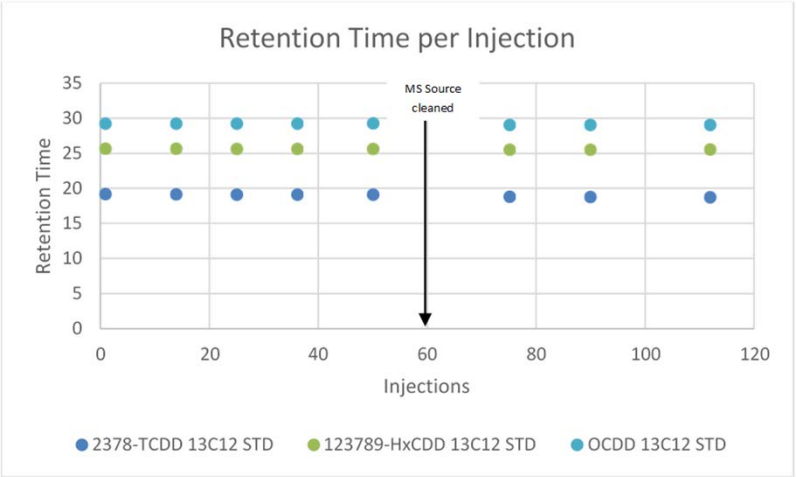


Improved Resolution of HxCDD

Robust Dioxin Analysis with Real Sample- Fish Oil



Peak areas for 2378-TCDD, 123789 HxCDD and OCDD standards after 112 injections of fish oil samples.

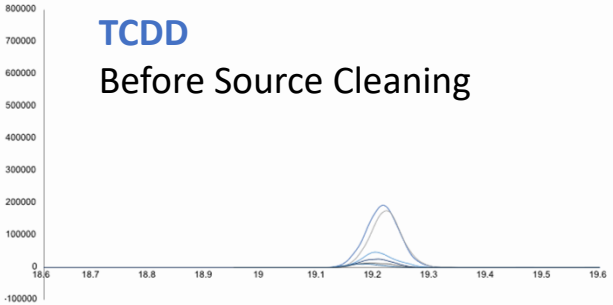


Retention times for 2378 TCDD, 123789 HxCDD and OCDD after 112 injections of fish oil samples.

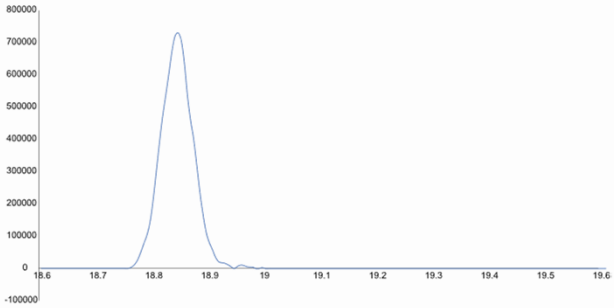
Robust Dioxin Analysis with Real Sample- Fish Oil

TCDD

Before Source Cleaning

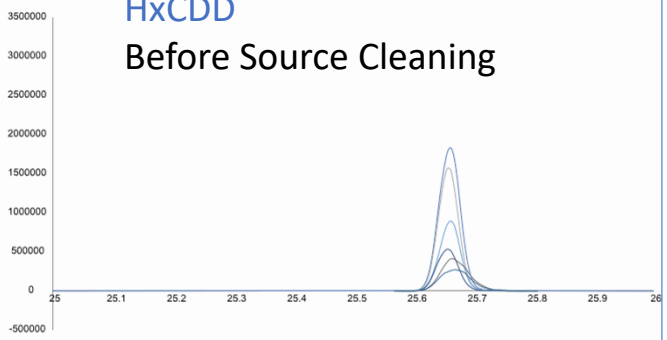


After Source Cleaning

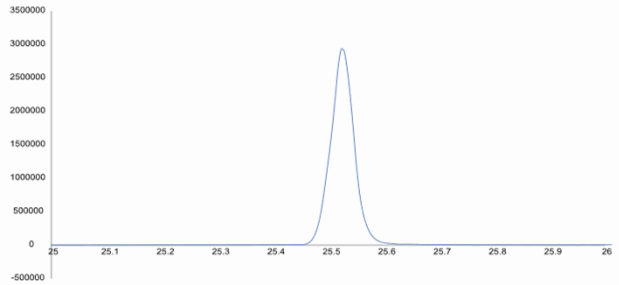


HxCDD

Before Source Cleaning

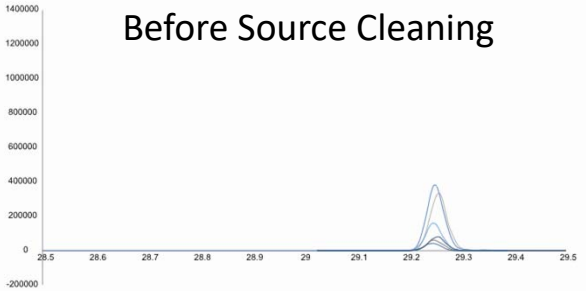


After Source Cleaning

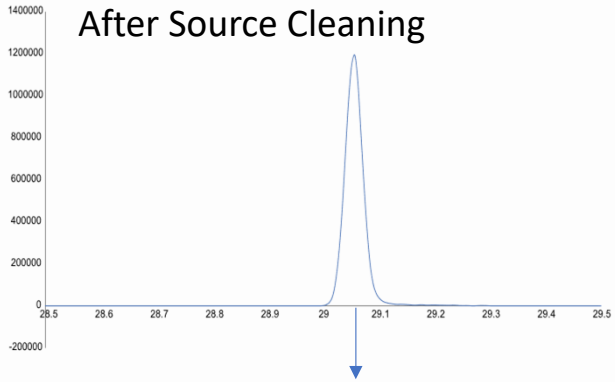


OCDD

Before Source Cleaning



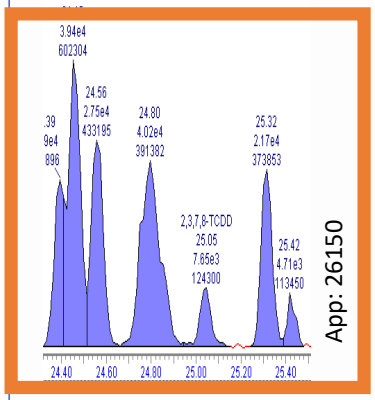
After Source Cleaning



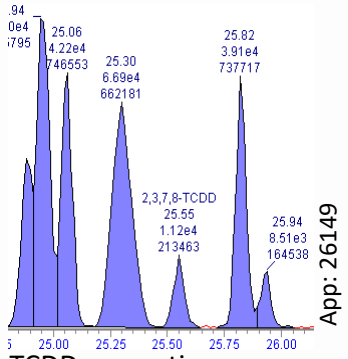
Last Eluting Dioxin within 30 min

Zebron Guardian™ Benefits

With Guardian

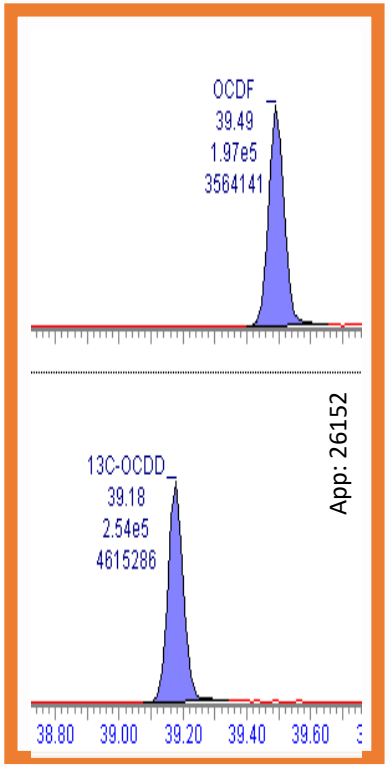


Without Guardian



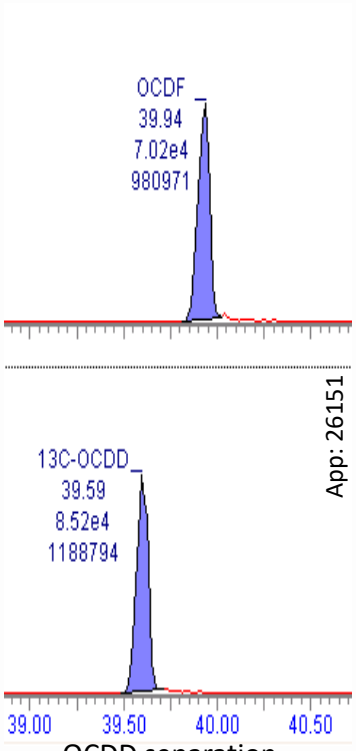
TCDD separation

With Guardian



OCDD separation

Without Guardian

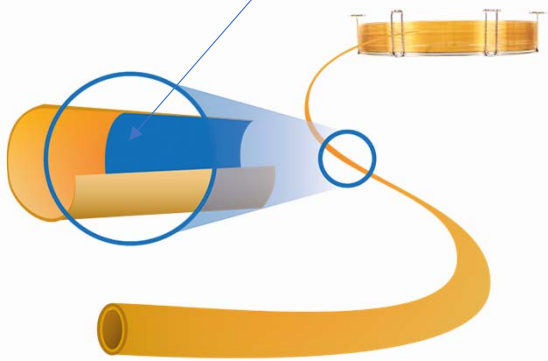


OCDD separation

ZB-Dioxin with Guardian™

- Negligible change in retention time
- Prevent Excessive trimming
- Extend life time

Seamless union of guard and analytical column for leak free connection



Summary

Introduction

- POPs
- Resolution Improvement
- Current Challenges with Dioxin analysis

ZB-Dioxin Applications

- Exceeding EPA 1613 requirement
- Balance of Resolution and Speed
- Extend lifetime with Guardian
- Dioxins & PCBs
- Robustness with Real Sample Matrix

WWW. Phenomenex.com/gcdioxin

Thank You