

APPLICATIONS

A Single Column Solution for Improved Resolution of Tetrachlorodibenzodioxins (TCDD) and Tetrachlorodibenzofurans (TCDF) using Zebron™ ZB-Dioxin by GC-HRMS

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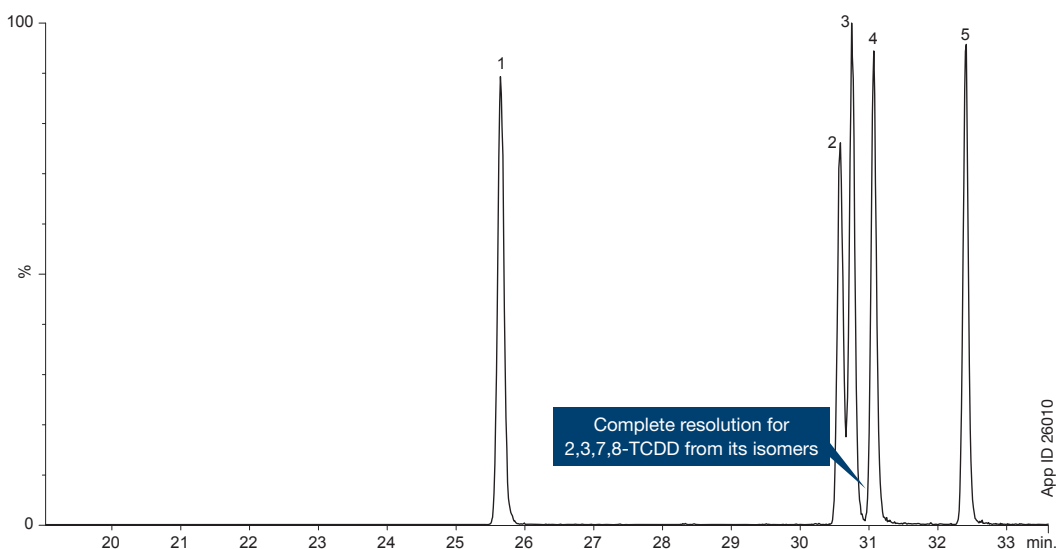
Introduction

Dioxins are Persistent Organic Pollutants (POPs) generated from industrial exhaust and from combustion of electronic components that can remain stable in the atmosphere for a long time. The presence of chlorine atom in these molecules stabilizes their structure and makes them persistent. According to World Health Organization (WHO), dioxins can cause reproductive and developmental problems, damage the immune system, interfere with hormones and also cause cancer. In addition to environmental samples, these are continuously monitored in food as well. Environmental Protection Agency (EPA) has several methods including EPA-1613 and EPA-8290 that can be used for monitoring dioxins. These methods specify that if a 5% Phenyl GC

column (5ms column) is used for the analysis by GC-HRMS as the primary column for testing and if the samples show presence of 2,3,7,8-TCDD, it recommends a 50%-Cyanopropylphenyl-dimethylpolysiloxane (225 column) confirmation column to resolve the TCDD isomers from toxic 2,3,7,8-TCDD. Hence, 5% Phenyl column methods will need two GC-HRMS instruments and two GC columns to complete the analysis. This reduces lab productivity and increases overall cost of dioxin analysis. In this tech note, we present separation of Tetra dioxins & furans using a single GC column, ZB-Dioxin. The experiment shows that ZB-Dioxin provides the highest resolution for 2,3,7,8-TCDD and 2,3,7,8-TCDF from its isomers on a single column, thereby exceeding EPA-1613 method requirement.

Figure 1a

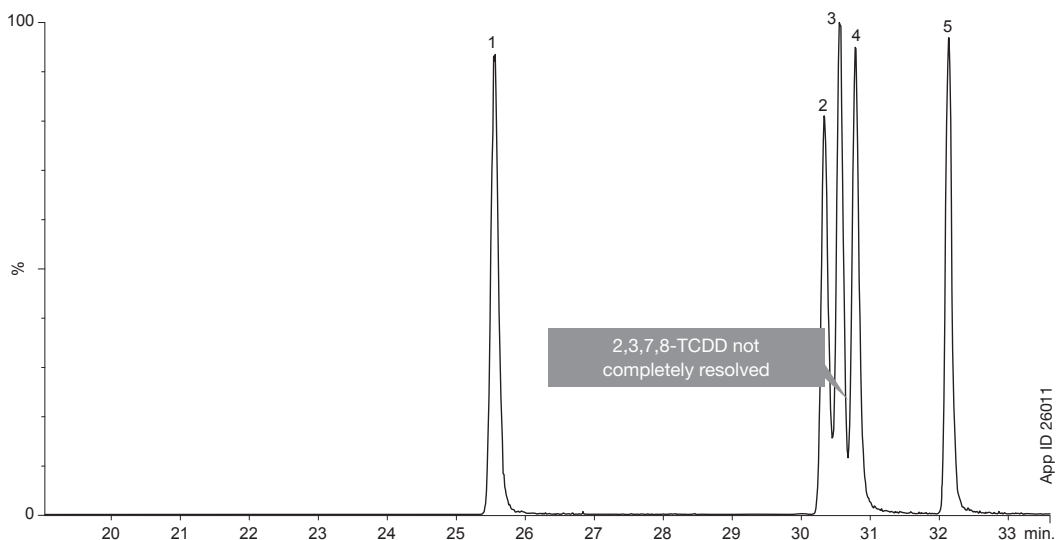
Separation of 2,3,7,8-TCDD and its isomers on a 60 meter Zebron ZB-Dioxin GC Column.



Analyte Name	RT (min)
1	1,3,6,8-TCDD 25.65
2	1,2,3,7-TCDD 30.58
3	1,2,3,8-TCDD 30.75
4	2,3,7,8-TCDD 31.07
5	1,2,8,9-TCDD 32.41

Figure 1b

Separation of 2,3,7,8-TCDD and its isomers on a 60 meter Brand A premium 5ms phase.



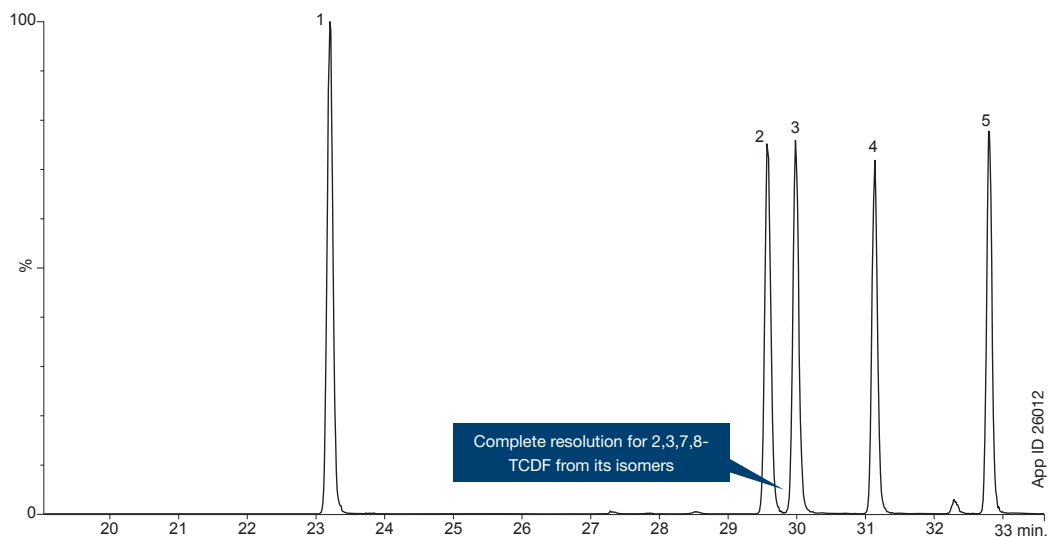
Analyte Name	RT (min)
1	1,3,6,8-TCDD 23.20
2	1,2,3,7-TCDD 30.33
3	1,2,3,8-TCDD 30.55
4	2,3,7,8-TCDD 30.78
5	1,2,8,9-TCDD 32.13

Comparative separations may not be representative of all applications.

APPLICATIONS

Figure 2a

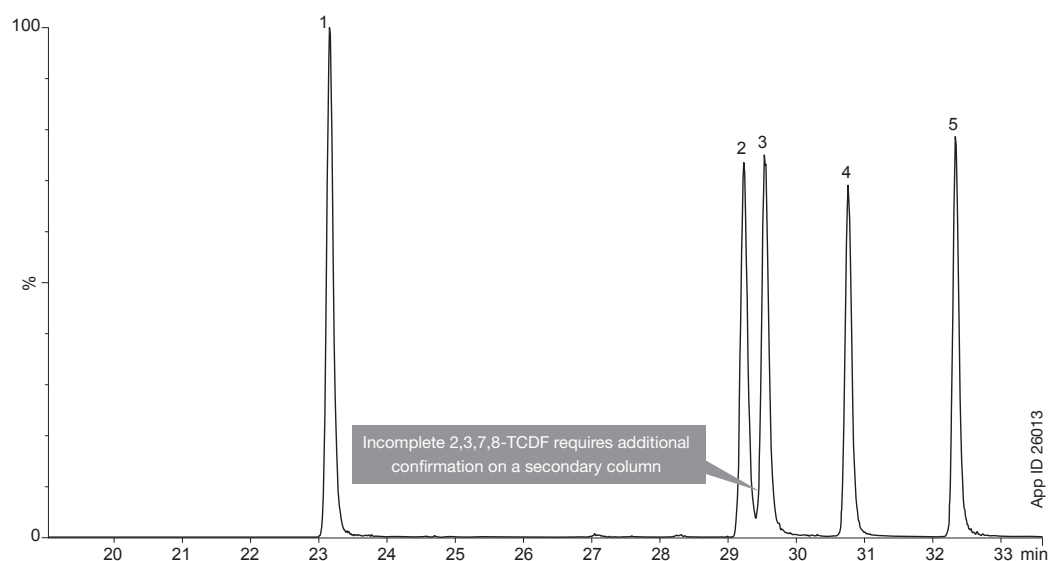
Separation of 2,3,7,8-TCDF and its isomers on a 60 meter Zebtron ZB-Dioxin GC Column.



Analyte Name	RT (min)	
1	1,3,6,8-TCDF	23.20
2	1,3,4,7-TCDF	29.57
3	2,3,7,8-TCDF	29.98
4	1,2,3,9-TCDF	31.14
5	1,2,8,9-TCDF	32.79

Figure 2b

Separation of 2,3,7,8-TCDF and its isomers on a 60 meter Brand A premium 5ms phase.



Analyte Name	RT (min)	
1	1,3,6,8-TCDF	23.16
2	1,3,4,7-TCDF	29.23
3	2,3,7,8-TCDF	29.53
4	1,2,3,9-TCDF	30.76
5	1,2,8,9-TCDF	32.33

GC-HRMS Method Parameters

Column (Figure 1a and 2a): Zebtron™ ZB-Dioxin
Dimensions: 60 meter x 0.25 mm x 0.20 µm
Part No.: [ZKG-G045-10](#)
Column (Figure 1b and 2b): Brand A Premium 5ms
Dimensions: 60 meter x 0.25 mm x 0.25 µm
Recommended Guard Column: 5 meter Z-Guard™ Kit
Guard Kit Part No: [ZAG-G000-00-GZK](#)
Injection: Pulse Splitless (2.0 min, 60 psi) @ 280 °C, 1 µL
Recommended Liner: Zebtron PLUS 4 mm ID Single Taper Liner
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

Comparative separations may not be representative of all applications.

Results and Discussion

The Zebon™ ZB-Dioxin GC column offers proprietary selectivity for the separation of closely related Dioxins and Furans. This GC column is designed to provide enhanced resolution of 2,3,7,8-TCDD and 2,3,7,8-TCDF from their isomeric compounds. EPA-1613 and EPA-8290 methods require the percent valley separation between the GC peaks that elute most closely to the 2,3,7,8-TCDD and TCDF isomers to be less than 25%. This is one of the system suitability requirements. **Figure 1a** represents the separation of 2,3,7,8-TCDD from its closely eluting isomer compounds on ZB-Dioxin GC column. As seen in the chromatogram, 2,3,7,8-TCDD shows less than 15% valley separation from its closely eluting isomer 1,2,3,8-TCDD which is difficult separation to achieve on a 5% Phenyl phase. On a traditional 5% phenyl phase, when the resolution of this critical pair is more than 25% valley, the columns are often replaced. With ZB-Dioxin GC column, the initial resolution exceeds method requirement thereby providing the opportunity to run more samples before the failure. **Figure 1b** shows the separation of the same mixture under identical conditions on Brand A premium 5MS column. Comparing the chromatograms in **Figure 1a** and **1b**, ZB-Dioxin GC column provides the highest resolution for 2,3,7,8-TCDD from its closely eluting isomer.

As per EPA-1613, isomer specificity for 2,3,7,8-TCDF cannot be achieved on the a 5% phenyl phase hence, the method recommends using a 225 phase, 2330 or equivalent phase for confirmation, if the sample shows presence of 2,3,7,8-TCDF. The isomers to be confirmed on the secondary column are 2,3,4,7-TCDF, 2,3,7,8-TCDF, 1,2,3,9-TCDF. Represented in **Figure 2a** is the separation of 2,3,7,8-TCDF from its isomers. As seen in the chromatogram, all the isomers 2,3,4,7-TCDF, 2,3,7,8-TCDF, 1,2,3,9-TCDF are baseline resolved from 2,3,7,8-TCDF on ZB-Dioxin GC column. When comparing TCDF separation on ZB-Dioxin against Brand A premium 5MS column (**Figure 2a** and **2b**), ZB-Dioxin provides complete baseline resolution of 2,3,7,8-TCDF from its isomers. The superior resolution and selectivity of TCDF isomers on ZB-Dioxin GC column eliminates the need for a secondary column confirmation. Thus the Dioxin analysis is made cost efficient with Single GC column ZB-Dioxin and single GC-HRMS, thereby improving lab productivity.

In addition to providing enhanced resolution of TCDD and TCDF isomers, Zebon ZB-Dioxin stationary phase undergoes extensive cross-linking through Engineered Self Cross-Linking™ (ESC™) process to provide mass spec compatible low bleed column. This reduces potential bleed and reduces instrument down time and maintenance. Thus, the proprietary selectivity of ZB-Dioxin, enhanced resolution of TCDD and TCDF, ESC and high efficiency makes it a single column solution for the separation of Dioxin compounds by GC-HRMS.

Conclusion

The Zebon ZB-Dioxin GC column provides the highest resolution for 2,3,7,8-TCDD and 2,3,7,8-TCDF from its isomer compounds and serves as a single GC column solution for dioxin separation.

Acknowledgement

This study was performed by McCampbell Analytical, Inc. and we thank them for their collaboration on this project. Phenomenex is not affiliated with McCampbell Analytical, Inc.,.

Ordering Information

Zebron™ ZB-Dioxin GC Column

ID(mm)	df(μm)	Temp. Limits °C	Part No.
60-Meter			
0.25	0.2	40 to 320/340	7KG-G045-10
60-Meter with 5-Meter Guardian™			
0.25	0.2	40 to 320/340	7KG-G045-10-GGA
40-Meter			
0.18	0.14	40 to 320/340	7PD-G045-47

Zebron PLUS Liners

Description	Inlet Style	Dimensions ID x L (mm)	Deactivation	Part No.	Unit
For Agilent® 5890, 6890 and 7890 Models					
Single taper	S/SL	4 x 78.5	PLUS Inert	AG2-0A10-05	5/pk
For Agilent® and Thermo Scientific®					
Single taper	S/SL	2 x 78.5	PLUS Inert	AG2-0E00-05	5/pk

Standard Z-Guard™ Columns and Kits

ID (mm)	Description	Part No.	
		5-Meter	10-Meter
0.25	Guard Column	7AG-G000-00-GZ0	7CG-G000-00-GZ0
	Guard Column Kit	7AG-G000-00-GZK	7CG-G000-00-GZK

Easy Seals™ Inlet Base Seals

Description	Injection Type	Groove Style	Inlet Hole Diameter (mm)	10/pk
				Part No.
Easy Seals Gold Inlet Seal	Splitless	Single	0.8	AG0-8620



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