

APPLICATIONS

Reproducible Dioxin Analysis of Fly Ash and Soil Samples Using Zebron™ ZB-Dioxin GC Columns

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Overview

Poly Chlorinated Dibenzo Dioxins (PCDD) and Poly Chlorinated Dibenzo Furans (PCDF) are stable chemical structures classified as Persistent Organic Pollutants. These compounds are monitored for safe levels in environmental samples like water, soil, sediments, tissue, and air by GC-HRMS. Presented in this application is the reproducible retention profile of tetra through octa dioxins and furans spiked into ash and soil samples. The goal of this work was to demonstrate the robustness of the analysis using harsh matrix like fly ash, and soil sample extracts. The enhanced resolution and reproducible retention times were observed in this analysis, which is possible due to a novel, Engineered Self Crosslinking (ESCTM) process which is employed in the manufacturing of Zebron ZB-Dioxin GC Columns.

Sample Preparation

The sample extraction and clean up was performed using a modified version of EPA 1613B. The 13C labelled compounds were spiked into a sample containing 10g (dry weight) of solid sample. Samples containing multiple phases are pressure filtered and any aqueous liquid is discarded and coarse solids were ground or homogenized. Samples were extracted in either an accelerated solvent extractor (ASE) or in a Soxhlet extractor. The extract was concentrated for cleanup and sensitivity. After extraction, 37Cl - labelled 2,3,7,8-TCDD was added to each extract to measure the accuracy of the cleanup process. Sample cleanup was performed using pre-packed acidic silica gel columns and activated carbon chromatography (CAPE Technologies). Samples were further concentrated and recovery standard was added prior to injection. Ash samples were pre-treated with 3N Hydrochloric acid before analysis.

GC-HRMS Conditions

Column: Zebron ZB-Dioxin
Dimension: 60 meter x 0.25 mm x 0.20 µm
Part No.: [7KG-G045-10](#)
Recommended Z-Guard™: [7AG-G000-00-GZK](#)
Injection: Splitless (1.0 min) @ 240 °C, 1 µL
Recommended Liner: Zebron PLUS Liner Compatible with Thermo GC 105 mm, 3 mm ID Single Taper Wool on Bottom
Liner Part No.: [AG2-5K11-05](#)
Carrier Gas: Helium @ 1.3 mL/min (constant flow)
Oven Program: 80 °C for 3 min, 235 °C @ 40 °C/min, 260 °C @ 1 °C/min, 275 °C @ 5 °C/min, 338°C @ 20°C/min for 10 min
Detector: GC-HRMS
Transfer Line Temperature: 230 °C

Figure 1: Reproducible Retention Time of Mid-Level Calibration Standards of Tetra through Octa Dioxins and Furans During the Robustness Experiment

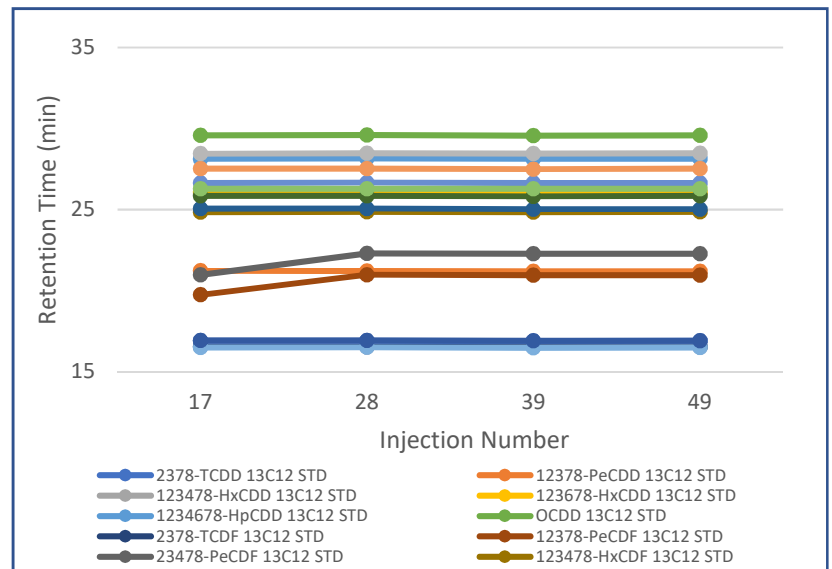
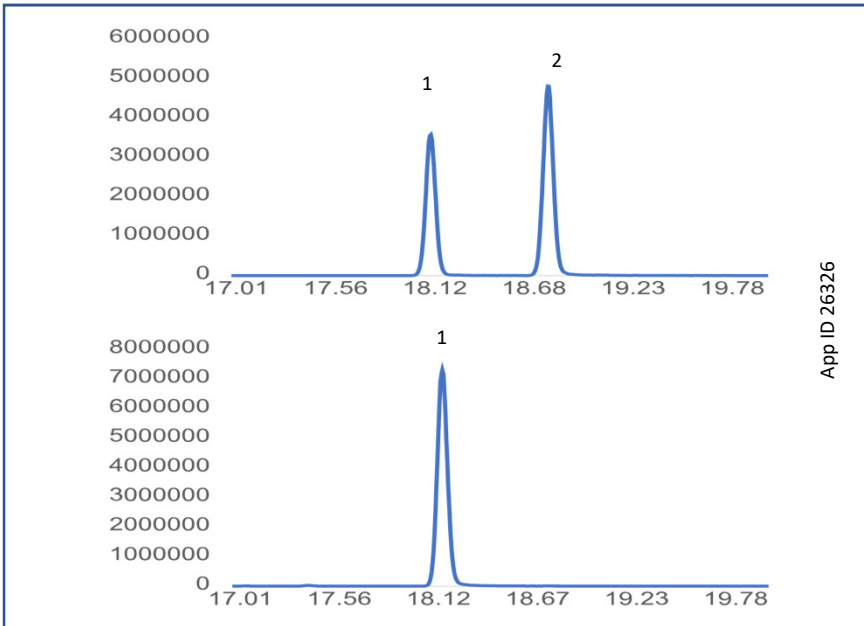


Figure 2: Separation of TCDD on a ZB-Dioxin GC Column by HRGC-HRMS



Our Customer Says YES!

“The new ZB-Dioxin column we tested far exceeded our expectations with respect to reliability and robustness. We would love to continue to use this product in future analysis!”

Patrick Pond, Pacific Rim, Canada

Sample:

1. 1,2,3,4-TCDD
2. 2,3,7,8-TCDD

Figure 3: Reproducible Retention Times of Fly Ash and Soil Sample Spiked with Tetra through Octa Dioxins and Furans on a ZB-Dioxin GC Column on HRGC-HRMS

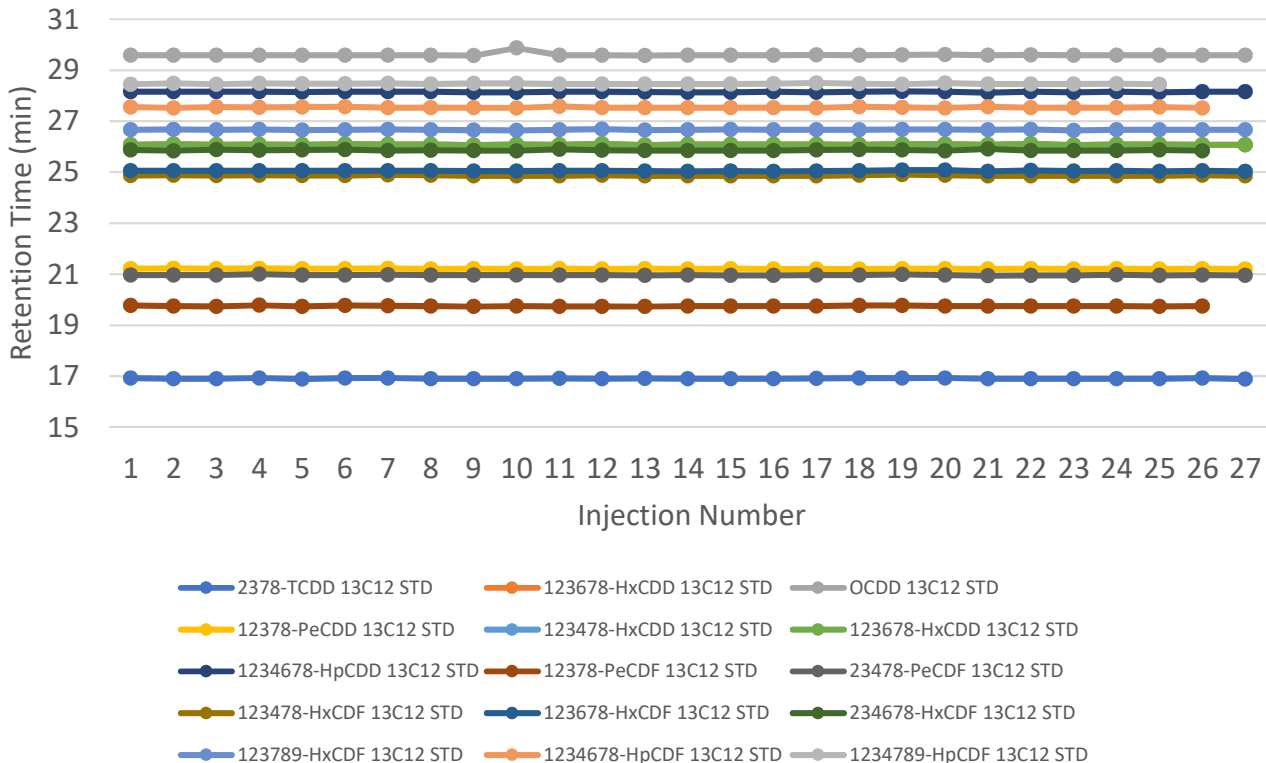


Figure 3: Retention Identification of Tetra through Octa Dioxins and Furans

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	
29.74	OCDF	13C OCDD

Summary of Results

This application note demonstrates reproducible retention of Tetra through Octa Dioxins and Furans on ZB-Dioxin GC Column. The retention times are presented in **Table 1**. **Figure 1** represents a plot of retention time of Tetra through Octa Dioxin at mid-level calibration (10-100 ng/mL). The results demonstrates reproducible retention of mid-level calibration standards of Tetra through Octa Dioxins and Furans. **Figure 2** shows the separation of TCDD. **Figure 3** is a plot of retention time vs injection of fly ash and soil samples spiked with Tetra through Octa Dioxins and Furans. The consistent retention times over several matrix injections, demonstrates the intact stationary phase chemistry present in ZB-Dioxin GC column.

Conclusion

Zebtron™ ZB-Dioxin GC column provides optimal separation of Tetra through Octa Dioxins and Furans as well demonstrates reproducible retention for harsh environmental samples.

Data Courtesy: Pacific Rim Laboratories Inc, Vancouver, Canada. Phenomenex is not associated with Pacific Rim Laboratories Inc

APPLICATIONS

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