Characterizing all 136 Tetra- to Octachlorinated Dioxins and Furans

Using the Rtx®-Dioxin2 Column

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- Known elution orders for all tetra- through octachlorinated dioxin and furan congeners.
- Resolve 14 of 17 tetra- through octachlorine 2,3,7,8-substituted dioxins and furans.
- TCDD and TCDF specificity, with a column stable up to 340°C.

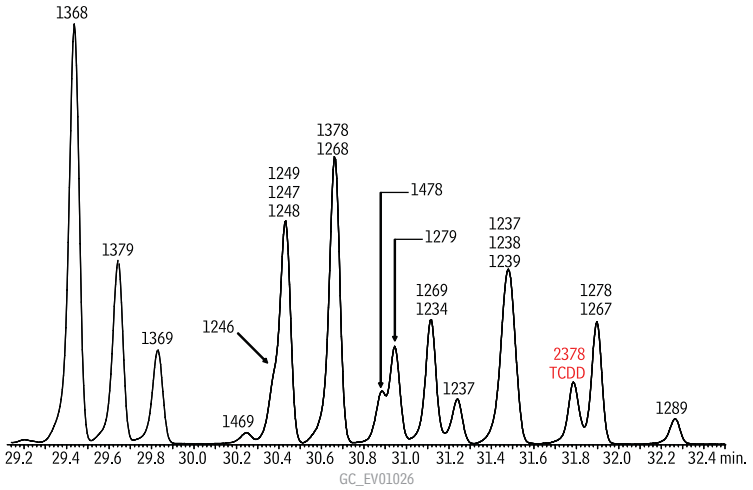
Successful analyses of dioxins and furans are critical because of the extremely toxic nature of these compounds. However, confidently resolving the most toxic congeners, 2,3,7,8-substituted tetrachlorinated dibenzodioxin (TCDD) and tetrachlorinated dibenzofuran (TCDF), is often complicated by the presence of the many other possible congeners. Even with high resolution GC/high resolution MS methods, the proper choice of chromatographic column is essential for separating 2,3,7,8-substituted dioxins and furans from the less toxic congeners and matrix-related compounds.

Complete Column Characterization

It is rare that a column's performance is characterized against all possible 136 tetra- through octachlorinated dioxins and furans. These standards are difficult to obtain, and testing can be time consuming. However, here the Rtx®-Dioxin2 column is characterized against all 136 compounds using standards from Cambridge Isotope Laboratories, Inc. When compared to industry standard stationary phases, a unique selectivity is observed for the Rtx®-Dioxin2 column, and specific resolutions and coelutions are noted. Very few coelutions involving the toxic 2,3,7,8-substituted congeners are observed, making the Rtx®-Dioxin2 column an excellent choice for single column analyses of dioxins and furans (Tables I and II.)

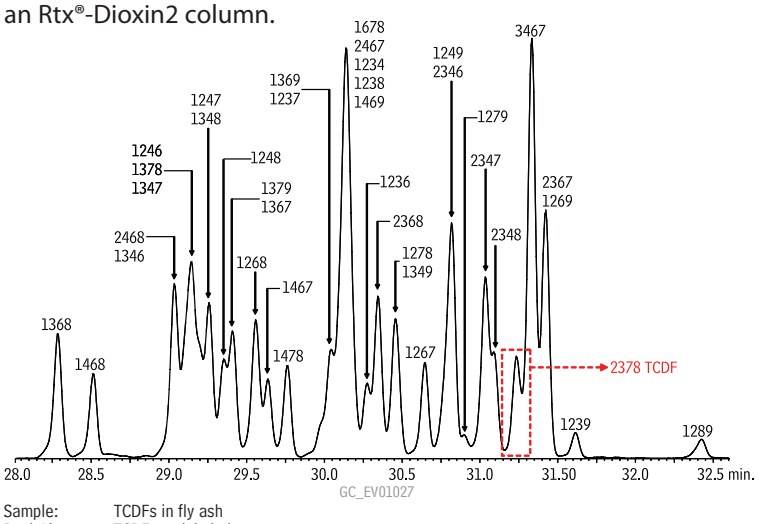
Figure 1 shows fly ash samples, run under the same chromatographic conditions used to characterize the column. 2,3,7,8-tetrachlorodibenzofuran is not resolved under these conditions. However, the characterization study used simple linear temperature programming, and additional work exploring nonlinear oven programs and different flow parameters yielded better resolution between some congeners, especially 2,3,7,8-TCDF (data available upon request). The value in this work is not necessarily to show complete separation of all the congeners on a single column, but to show where all of the 136 compounds of interest elute, making all possible coelutions known.

Figure 1 GC/HR-MS analysis of tetrachlorinated dioxins in fly ash on an Rtx®-Dioxin2 column.



Column:	Rtx®-Dioxin2, 40m, 0.18mm ID, 0.18µm (cat.# 10759)
Sample:	TCDDs in fly ash
Inj.:	1µL splitless
Inj. temp.:	280°C
Carrier gas:	helium, constant flow
Flow rate:	1mL/min.
Oven temp.:	120°C (hold 1 min.) to 160°C @ 10°C/min. to 320°C @ 4°C/min. (hold 4 min.)
Det.:	Waters AutoSpec Ultima HRMS
Ionization:	EI
Mode:	SIM
Peak List:	TCDDs as labeled

Figure 2 GC/HR-MS analysis of tetrachlorinated furans in fly ash on an Rtx®-Dioxin2 column.



Sample: TCDFs in fly ash
Peak List: TCDFs as labeled
See Figure 1 for conditions

The Rtx®-Dioxin2 column is an excellent column for the analysis of dioxin and furan congeners. It has a unique selectivity for the toxic congeners, including specificity for 2,3,7,8-TCDD and 2,3,7,8-TCDF. Here we characterized all 136 tetra- through octachlorine dioxins and furans and defined all possible coeutions. While commonly used cyanopropyl columns are limited by a low maximum operating temperature of 240°C, the Rtx®-Dioxin2 column is stable up to 340°C, extending column lifetime and improving analyses of dioxins and furans.

Rtx®-Dioxin2 Columns (fused silica)

ID	df (µm)	temp. limits	length	cat. #
0.18mm	0.18	20°C to 340°C	40-Meter	10759
0.25mm	0.25	20°C to 340°C	60-Meter	10758

Stable up to 340° for extended column lifetime!

Table I Retention times (RT) and relative retention times (RRT) for all tetra- through octachlorinated dioxins on an Rtx®-Dioxin2 column.

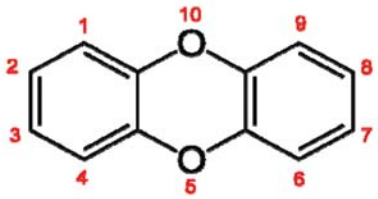
tetrachlorinated			pentachlorinated			hexachlorinated			heptachlorinated			octachlorinated		
Congener	RT (min)	RRT (min)	Congener	RT (min)	RRT (min)	Congener	RT (min)	RRT (min)	Congener	RT (min)	RRT (min)	Congener	RT (min)	RRT (min)
1368	29.43	0.8198	12468	33.75	0.9401	124679	37.89	1.0554	1234679	42.44	1.1822	**12346789**	**46.93**	**1.3069**
1379	29.64	0.8256	12479	33.80	0.9415	124689	37.89	1.0554	**1234678**	**43.34**	**1.2072**			
1369	29.84	0.8312	12469	34.17	0.9515	123468	38.53	1.0730						
1469	30.25	0.8424	12368	34.67	0.9657	123679	38.79	1.0805						
1246	30.38	0.8462	12478	34.83	0.9702	123689	38.82	1.0813						
1249	30.42	0.8474	12379	34.92	0.9727	123469	38.90	1.0833						
1247	30.43	0.8476	12467	35.02	0.9755	**123478**	**39.55**	**1.1017**						
1248	30.44	0.8479	12369	35.08	0.9769	**123678**	**39.66**	**1.1047**						
1378	30.64	0.8535	12489	35.08	0.9772	123467	39.78	1.1081						
1268	30.68	0.8546	12346	35.36	0.9850	**123789**	**39.98**	**1.1136**						
1478	30.88	0.8599	12347	35.40	0.9858									
1279	30.95	0.8621	12367	35.89	0.9997									
1269	31.12	0.8669	**12378**	**35.91**	**1.0003**									
1234	31.15	0.8677	12389	36.21	1.0086									
1236	31.25	0.8705												
1237	31.47	0.8766												
1238	31.50	0.8774												
1239	31.51	0.8777												
2378	**31.79**	**0.8855**												
1278	31.90	0.8883												
1267	31.90	0.8886												
1289	32.27	0.8989												

Blue boxes represent coelutions

Red boxes represent coelutions with 2378-substituted congeners

Bold red text indicates congeners with 2378 substitution

Number Sequence for Polychlorinated Dibenzo Dioxins



RRTs were calculated versus 12378 ^{13}C-labeled dioxin.

Table II Retention times (RT) and relative retention times (RRT) for all tetra- through octachlorinated furans on an Rtx®-Dioxin2 column.

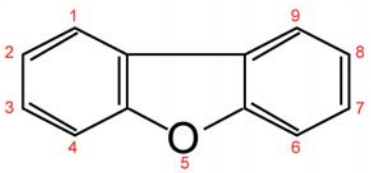
tetrachlorinated			pentachlorinated			hexachlorinated			heptachlorinated			octachlorinated		
Congener	RT (min)	RRT (min)	Congener	RT (min)	RRT (min)	Congener	RT (min)	RRT (min)	Congener	RT (min)	RRT (min)	Congener	RT (min)	RRT (min)
1368	28.29	0.8181	13468	32.38	0.9364	123468	37.23	1.0766	**1234678**	**41.99**	**1.2143**	**12346789**	**47.07**	**1.3604**
1468	28.52	0.8243	12468	32.44	0.9378	134678	37.38	1.0807	1234679	42.36	1.2243			
2468	29.03	0.8393	13678	33.53	0.9694	124678	37.40	1.0812	1234689	42.60	1.2319			
1346	29.03	0.8393	13467	33.58	0.9705	134679	37.62	1.0873	**1234789**	**43.92**	**1.2697**			
1246	29.11	0.8413	12467	33.61	0.9717	124679	37.83	1.0876						
1378	29.15	0.8427	14678	33.70	0.9717	124689	38.08	1.1009						
1347	29.19	0.8441	13478	33.69	0.9743	123467	38.45	1.1116						
1247	29.26	0.8459	12368	33.71	0.9746	**123478**	**38.58**	**1.1154**						
1348	29.27	0.8459	12478	33.76	0.9760	**123678**	**38.70**	**1.1191**						
1248	29.35	0.8485	13479	33.85	0.9783	123479	38.86	1.1234						
1379	29.40	0.8497	13469	34.00	0.9829	123469	38.96	1.1263						
1367	29.42	0.8503	12479	34.09	0.9858	123679	39.14	1.1315						
1268	29.56	0.8546	12346	34.14	0.9870	123689	39.40	1.1387						
1467	29.64	0.8569	12469	34.25	0.9902	**234678**	**39.42**	**1.1400**						
1478	29.76	0.8604	23468	34.35	0.9928	123489	40.29	1.1651						
1369	29.97	0.8664	12347	34.36	0.9931	**123789**	**40.31**	**1.1654**						
1237	30.03	0.8684	12348	34.39	0.9945									
1678	30.10	0.8702	**12378**	**34.61**	**1.0006**									
2467	30.14	0.8714	12678	34.85	1.0075									
1234	30.16	0.8719	12367	34.86	1.0075									
1238	30.18	0.8725	12379	34.99	1.0116									
1469	30.19	0.8725	12679	35.27	1.0197									
1236	30.27	0.8754	23467	35.48	1.0257									
2368	30.35	0.8772	12369	35.51	1.0266									
1278	30.45	0.8803	12489	35.56	1.0277									
1349	30.48	0.8812	**23478**	**35.68**	**1.0318**									
1267	30.66	0.8864	12349	35.74	1.0335									
1249	30.78	0.8864	12389	36.47	1.0544									
2346	30.83	0.8910												
1279	30.89	0.8930												
2347	31.03	0.8968												
2348	31.10	0.8991												
2378	**31.22**	**0.9028**												
3467	31.33	0.9058												
2367	31.41	0.9081												
1269	31.44	0.9089												
1239	31.61	0.9141												
1289*	32.43	0.9376												

Blue boxes represent coelutions

Red boxes represent coelutions with 2378-substituted congeners

Bold red text indicates congeners with 2378 substitution

Number Sequence for Polychlorinated Dibenzo Furans



RRTs were calculated versus 12378 ^{13}C-labeled furan.

*Note that the 1289 tetra chlorinated congener elutes after the 13468 penta chlorinated congener.