Analysis of all 136 tetra- through octa- polychlorinated dibenzo-p-dioxins and dibenzofurans using an Agilent J&W FactorFour VF-Xms GC column

Application Note

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Introduction

Out of the 136 polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF) congeners (tetra- through octa-), there are 17 congeners with chlorines in the 2378-positions that are considered to be of toxicological significance and are typically used for Toxic Equivalence (TEQ) calculations.

In order to establish TEQ values, an accurate determination of isomer-specific concentrations of all 17 2378-substituted dioxins and furans is required. Most of the regulatory methods for the PCDD/PCDF analysis have been developed based upon a dual column approach using a combination of non-polar and polar GC columns.

In this note, data is presented for the elution order of all 136 tetra- through octa- polychlorinated dioxins and furans, including the separation of 16 out of 17 2378-substituted dioxins and furans from other close-eluted isomers. We used a high Si-arylene modified liquid phase column, VF-Xms, with no need for a second column.

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Materials and Methods

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Column:	VF-Xms Fused Silica, 60 m x 0.25 mm, df = 0.25 µm (part number CP8809)
Carrier Gas:	He, constant pressure at 290 kPa or 42 psi
Oven Temp:	160 ºC for 1 min
	160-230 ºC at 35 ºC/min, hold for 18 min
	230-250 ºC at 10 ºC/min, hold for 25 min
	250-310 ºC at 10 ºC/min, hold for 6 min
Interface Temp:	290 ºC
Injection:	Splitless, 270 ºC, 1 µL
Detector:	MAT-95XP double focusing high raesolution magnetic sector mass spectrometer, EI 42eV, Ion source 270 °C, Acceleration voltage 4700V, SIM mode at resolution > 10,000 (10% valley)
Sample:	128 qualitative standards (tetra-through hexa-) PCDD/PCDF with an approximate concentration of 25 ng/mL in nonane. (Cambridge Isotope Laboratories Andover, MA, USA). In addition, native HpCDD, HpCDF, OCDD and OCDF were added to the above mixture
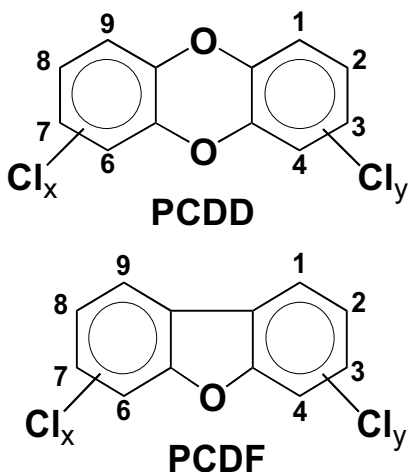
Figure 1. Chemical structures of PCDD and PCDF congeners

Table 1. Number of isomers of the PCDD and PCDF

Chlorines (x+y)	Acronyms	PCDD number of isomers	PCDF number of isomers
4	TCDD/TCDF	22	38
5	PnCDD/PnCDF	14	28
6	HxCDD/HxCDF	10	16
7	HpCDD/HpCDF	2	4
8	OCDD/OCDF	1	1
4-8	Total	49	87

Results

Figures 2 and 3 show the separation of PCCD/PCDF where quantitative results can be easily achieved on 16 out of 17 2378-substituted congeners. Note that critical 2378-TCDD and 2378-TCDF separations can be easily achieved with better than 25% valley as typically required by regulatory methods.

Conclusion

The VF-Xms liquid phase provides superior performance towards dioxins and furans separations compared to other GC columns, delivering a high degree of quantification accuracy for 2378-substituted congeners. The ability to separate 2378-TCDF at the baseline from other closely eluting isomers means there is no need for confirmatory analysis by a complementary GC column when strictly following US and Canadian regulatory methods.
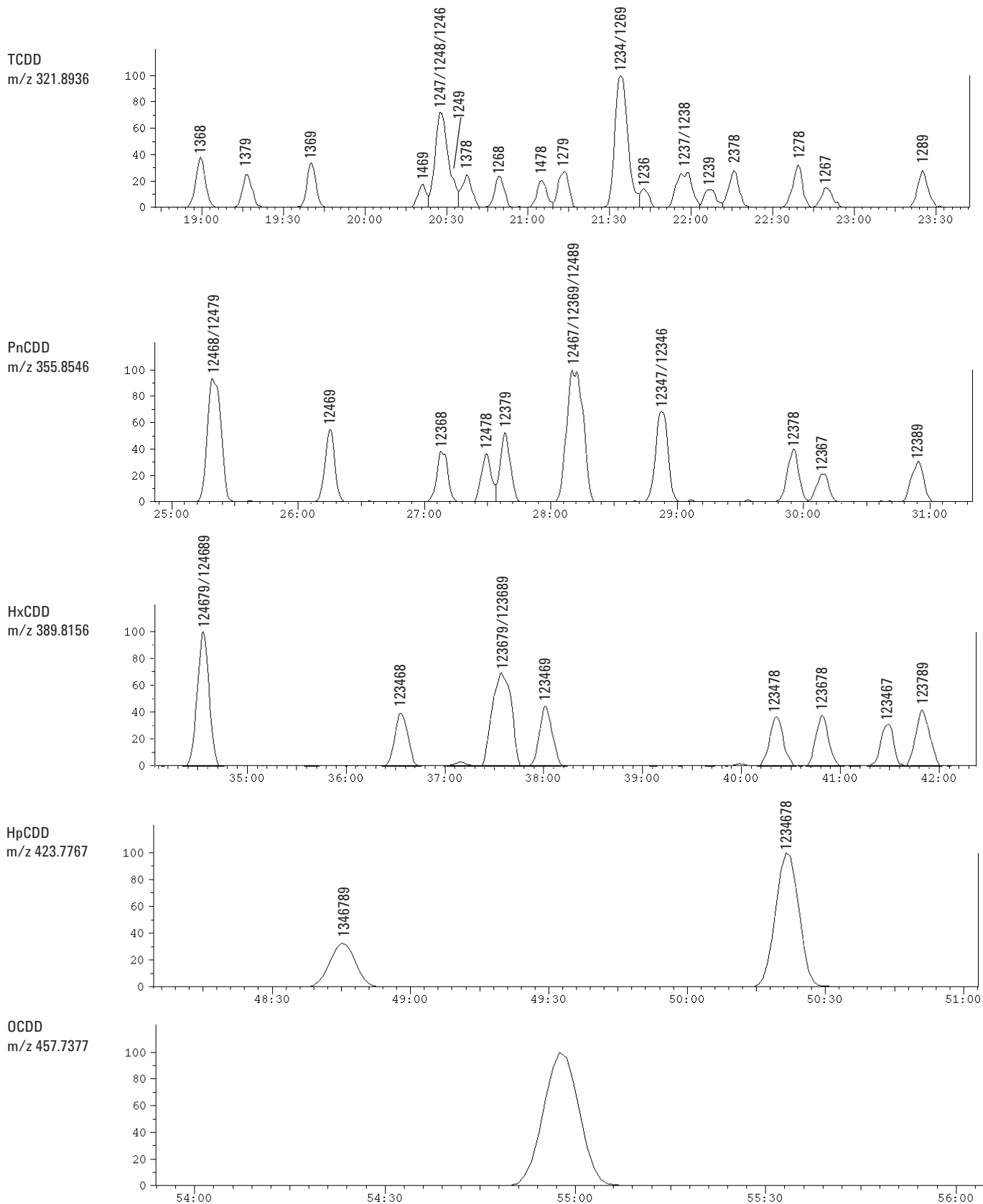
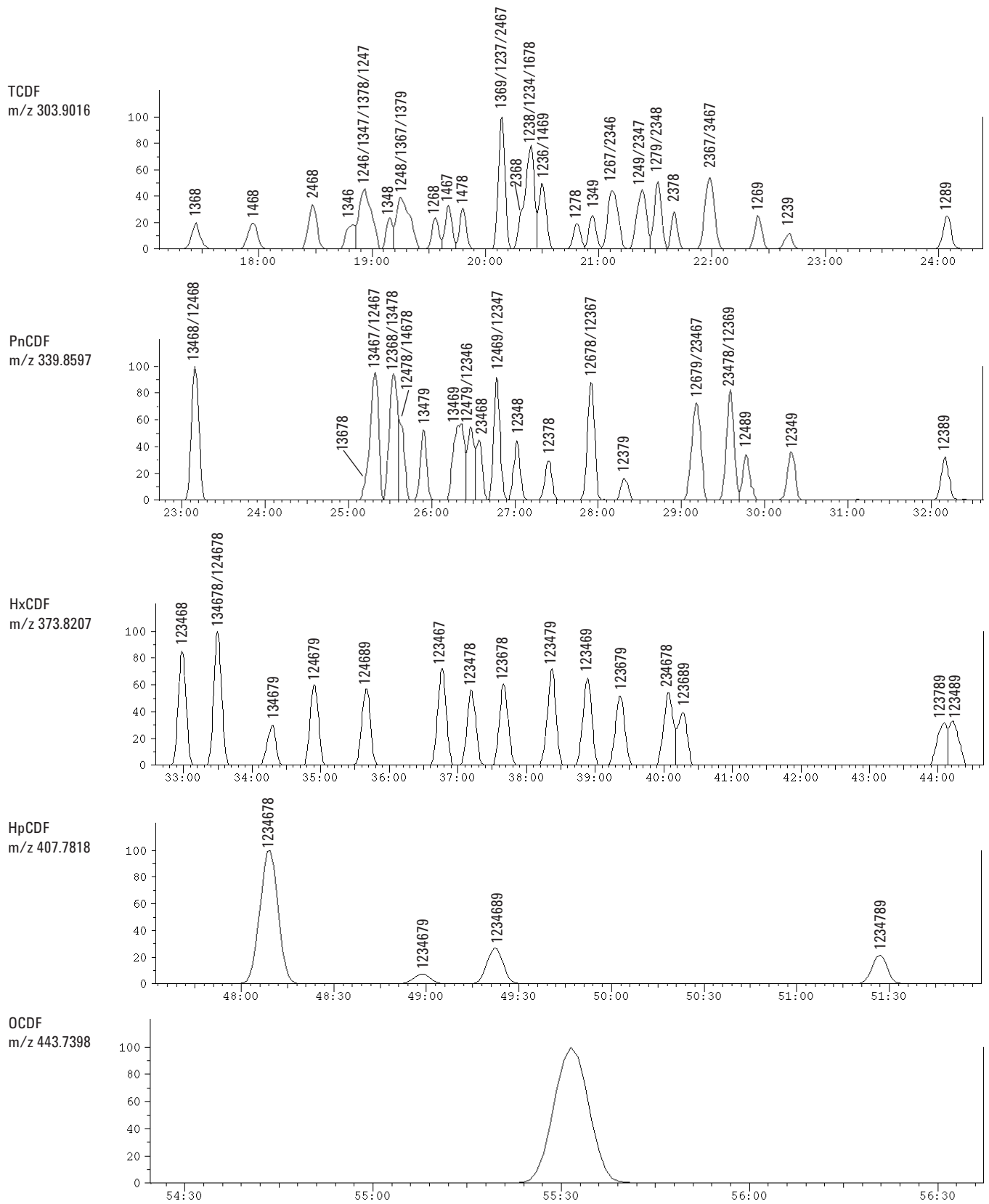*Figure 2. Separations of polychlorinated dibenzo-p-dioxins on a VF-Xms GC column*

Figure 3. Separations of polychlorinated dibenzofurans on a VF-Xms GC column

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