Separation of Polychlorinated Biphenyl Congeners 105, 132, and 153 Using GCxGC-ECD with a Selective Column in the Second Dimension

Introduction

Polychlorinated biphenyl congeners 105, 132, and 153 (with chlorine substitutions 234-34, 234-236, and 245-245, respectively) tend to coelute on 100%-dimethyl-polysiloxane and 95%-dimethyl-5%-diphenyl-polysiloxane gas chromatography (GC) columns. This can thwart attempts to quantify them individually in samples, especially when using the non-specific electron capture detector (ECD).

Comprehensive two-dimensional GC (GCxGC) with a GC phase that is selective for PCBs is one way to solve the 105/132/153 (and other) coelution problems. GCxGC is a way to increase peak capacity by applying two independent separations to a sample in one analysis with one detector. GCxGC involves serially connected columns (differing phases) separated by a thermal modulator. A separation is performed on the first column, and then effluent from the first column is continually (and quickly) focused and "injected" onto the second column. By keeping the second column short, a series of high-speed chromatograms are generated, and the first column separation can be maintained. Separation results can be plotted as a retention plane (column 1 time x column 2 time), also known as a contour plot.

Standards

Aroclor 1254 was obtained from AccuStandard (New Haven, Connecticut, USA).

Experimental Conditions

LECO GCxGC-ECD

Agilent 6890 GC-ECD equipped with a LECO Quad Jet—Dual-Stage Thermal Modulator

Column 1:	50 m x 0.18 mm x 0.18 µm Rtx-1 (Restek)
Column 2:	1.5 m x 0.18 mm x 0.10 µm Rtx-PCB (Restek)
Carrier:	Helium at 1.3 mL/min, constant flow
Injection:	1 µL split at 250°C, split ratio 20:1
Oven 1 Program:	160°C (0.2 min), 2°/min to 280°
Oven 2 Program:	20°C offset from oven 1
Modulation Time:	8 sec.
Detector:	ECD, 325°C, N2 makeup gas at 148.7 mL/min, 50 Hz

Results and Discussion

Figure 1 shows the separation of PCBs 105, 132, and 153 in the second dimension by using GCxGC-ECD with the Rtx-PCB column. Note that these PCBs almost line up in the first dimension (a coelution), which represents their retention times on Rtx-1, 100%-dimethyl-polysiloxane. The Rtx-PCB column is particularly retentive for those PCBs that have lower degrees of ortho-chlorine substitution.

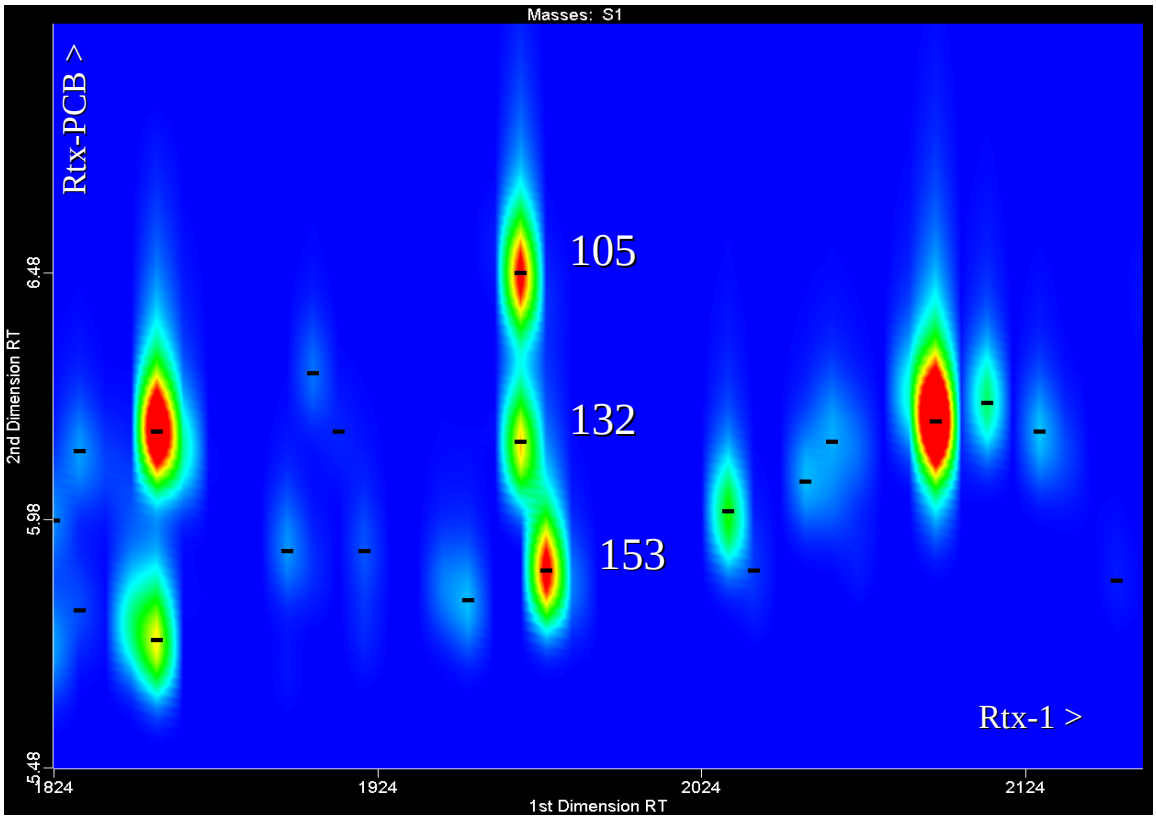


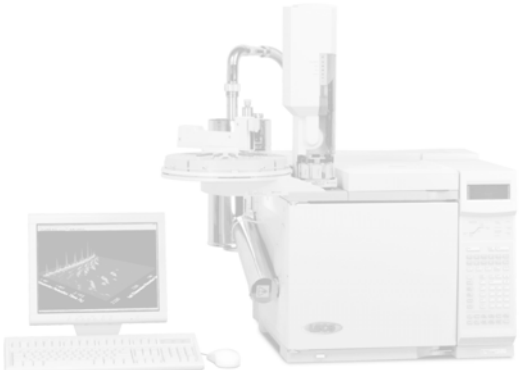
Figure 1. *Contour plot showing GCxGC-ECD separation of PCBs 105, 132, and 153 in the second dimension using the selective Rtx-PCB column. The sample is Aroclor 1254.*

Conclusions

GCxGC-ECD with a selective column in the second dimension offers a way to substantially improve separations for complex halogenated mixtures such as PCBs.

Acknowledgment

Frank Dorman at Restek Corporation provided the GC columns used for this work.



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