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Improved Determination of Polychlorinated Biphenyl Compounds by US EPA Method 1628

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Introduction

Polychlorinated Biphenyl (PCBs) and the Environment

Polychlorinated Biphenyls (PCBs) are synthesized compounds that belong to the chlorinated hydrocarbon family of compounds. PCBs were used in a variety of industrial applications such as electrical components, plasticizers and pigments/dyes, until they were banned in 1979 by the Toxic Substances Control Act (TSCA)[1]. PCBs are considered to persistent organic pollutants (POPs) as they do not easily degrade in the environment. PCBs can be found in the air, water, and soil and have been known to bioaccumulate in marine life.

The EPA has recently developed a low-resolution mass spectrometry method that can calibrate 65 PCBs congeners and screens for all 209 using labeled PCB compounds for direct and indirect quantitation[2]. The 65 congeners are targeted for calibration and quantitation due to specific factors described in the method[2]. As with most EPA methods, the prescribed methodology utilizes helium as a carrier gas. The recommended column for this analysis makes adequate separation and quantitation more challenging. To achieve separation that meets the method criteria, the run time is over 40 minutes long. By using a column better suited for PCB congener separation and hydrogen as the carrier gas, superior separation can be achieved in under 20 minutes, resolving previously co-eluting analytes.

Experimental

System configuration using hydrogen carrier gas

The Agilent 8890/5977C GC/MSD equipped with the HydroInert Ion source was operated in selective ion monitoring (SIM) mode for this analysis.

Figure 1: Agilent 8890/5977C GC/MSD Instrument with the HydroInert Ion Source

Operational Conditions

Listed below in table 1 are the GC parameters used to carry out the separation of the congeners and the ion source temperature.

Parameters	Values
Injection Volume	1.0µL
Inlet	310°C
Column	DB-XLB 20m X 180µm X 0.18µm
Purge Flow	3mL/min
Column Flow	0.9mL/min
Oven Program	50°C (hold for 0.4min) 30°C/min. to 180°C 10°C/min. to 300°C (hold for 1.257 min.)
Transferline Temperature	300°C
Ion Source Temperature	280°C

Table 1: Operational parameters for the method.

Results and Discussion

DB-XLB column provide superior separation

The chromatogram of the 65 congeners specifically chosen for full quantitation by the EPA 1628 method at a concentration of 160ppb for the native PCBs and 400 ppb for the labeled PCBs is shown below in figure 2 The run time for this method is slightly over 15 minutes, with PCB 209 and PCB 13C-209 eluting at 15.02 minutes. Hydrogen, which has a higher optimal linear velocity than helium, allows for this rapid analysis.

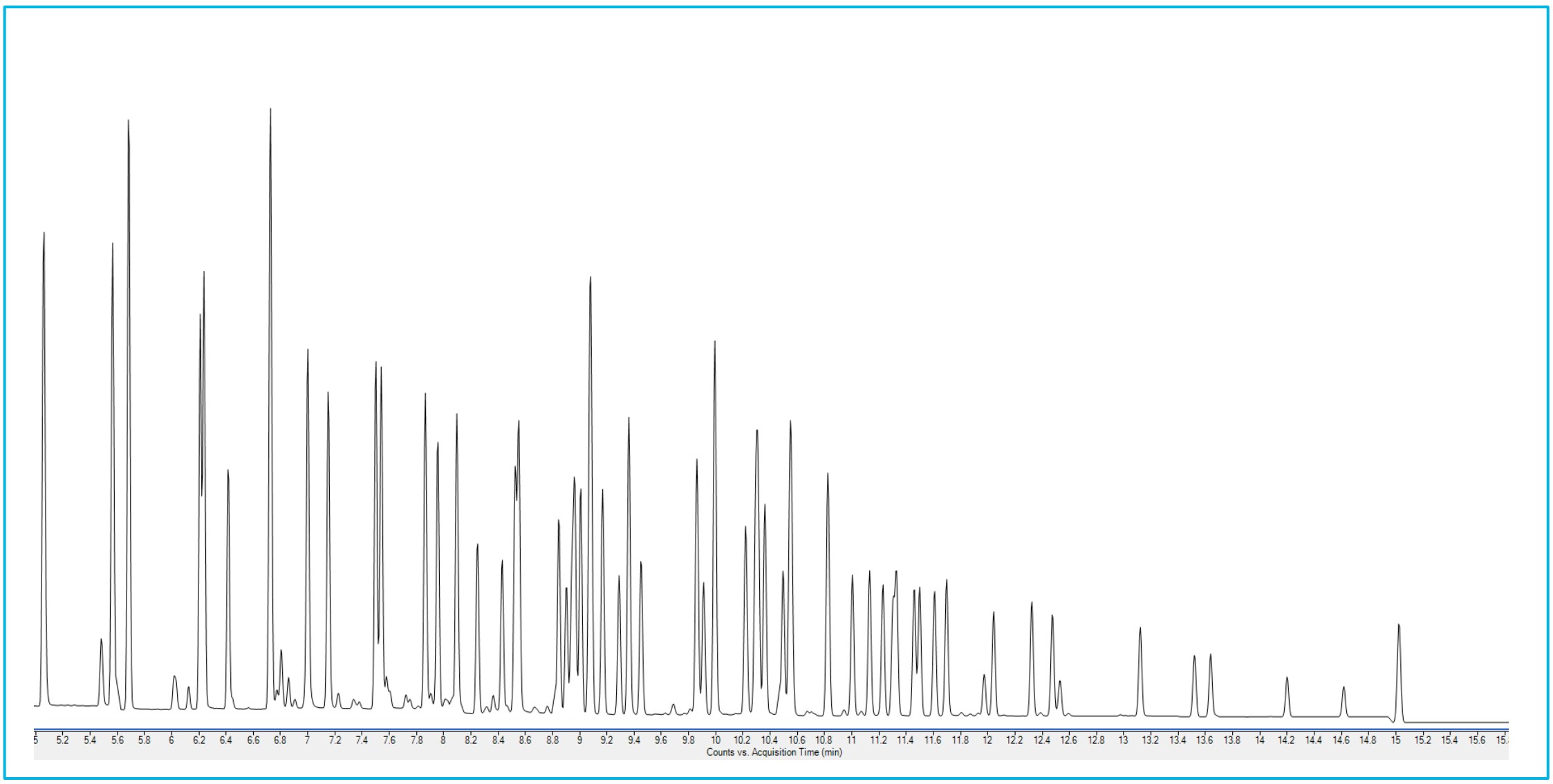
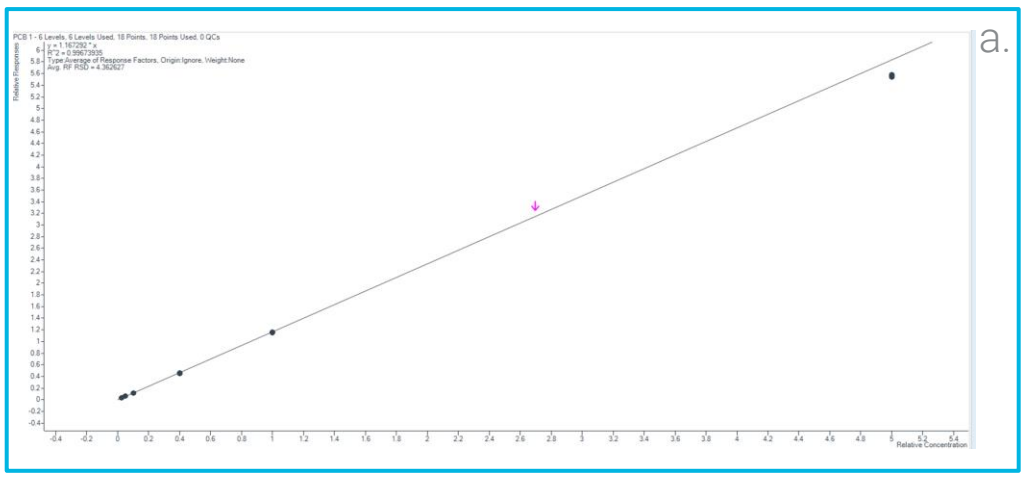


Figure 2: Chromatogram of Calibrated PCB Congeners

Calibration of the 65 native PCB congeners

A six-point calibration curve with concentrations from 10-2000ppb was prepared in iso-octane Each calibration level was analyzed in triplicate. Quantitation in Agilent MassHunter Quantitative Analysis software package will calculate both the r^2 values as well as the average of response factors, which can be see in two sample calibration curves from this data set in figures 3a and 3b. The average RF were less that 20 for all the calibrated congeners. The r^2 values were greater that 0.97 for the calibration set.



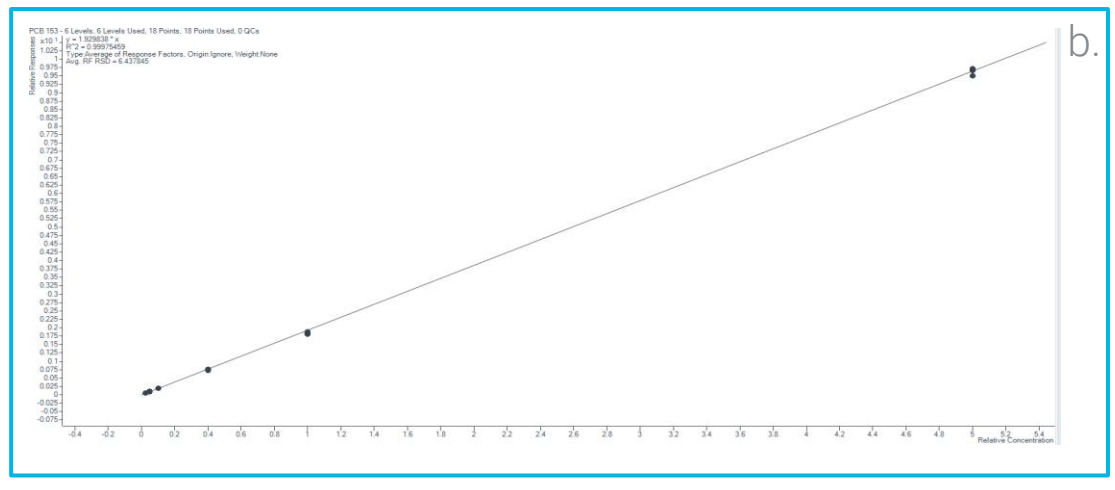


Figure 3: The six-point calibration curve from a. PCB 1 and b. PCB 153

Results and Discussion

Calibration of Select PCB Congeners

The 65 native PCB congeners and 31 ^{13}C isotopically labeled congeners were analyzed in solvent go beyond the required chromatographic resolution of PCB congeners 28 and 31, which requires the valley between the two congeners to be less than 80% of smallest peak.

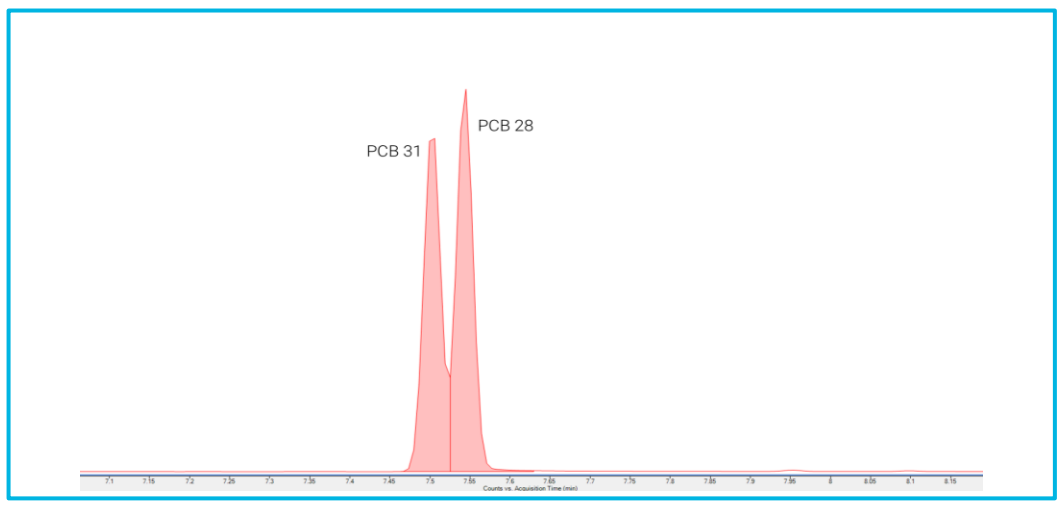


Figure 4: PCB congeners 28 and 31

The enhanced selectivity of the XLB phase allows for most congeners that previously co-eluted to separate out for easier quantitation of samples with complex PCB composition. Some examples include tetrachlorobiphenyl congeners 41 and 64, pentachlorobiphenyl congeners 85 and 120 have and hexachlorobiphenyl congeners 139 and 149, which are below in figure 5 (a-c).

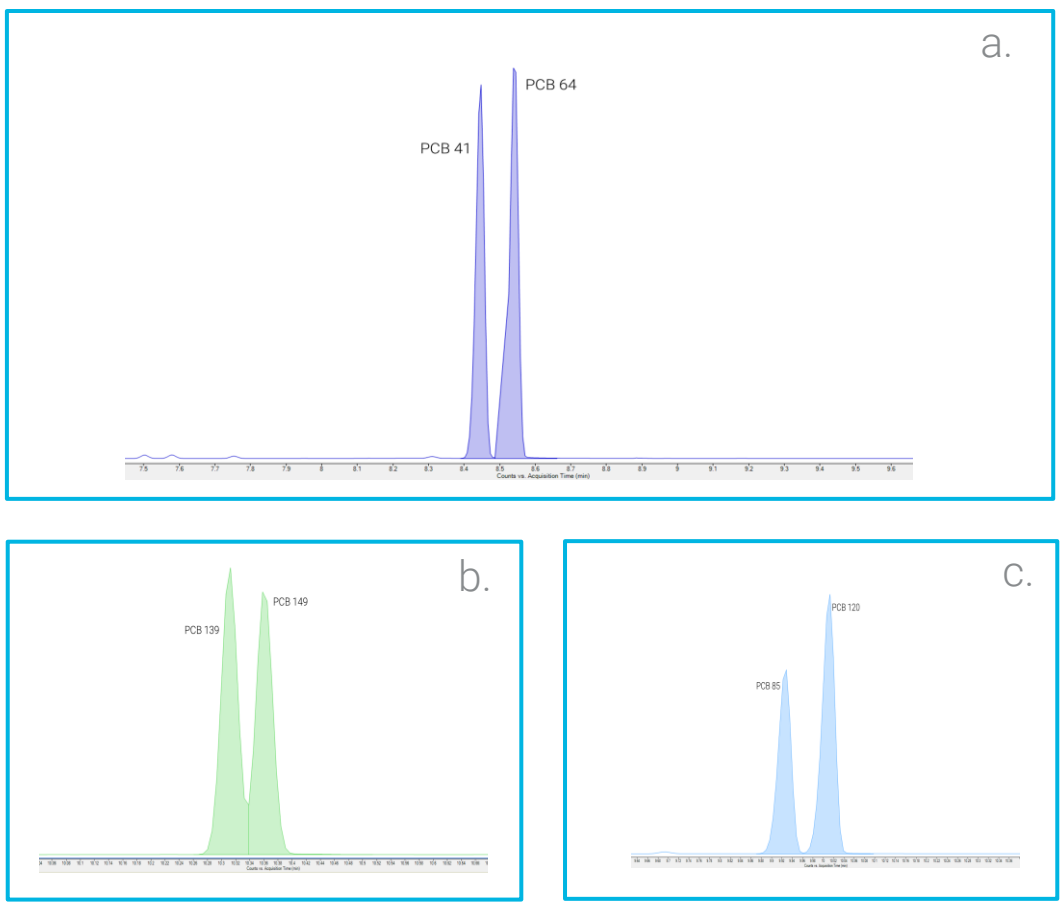


Figure 5: Chromatographically resolved congeners a. PCBs 41-64 b. PCBs139-149 c. PCBs 85-120

MSD Sensitivity

The method calls for PCB congener 118 to have a signal to noise ratio of 3:1 at a concentration of 10ppb. The method easily meets this requirement with the very low bleed of the column combined with SIM detection.

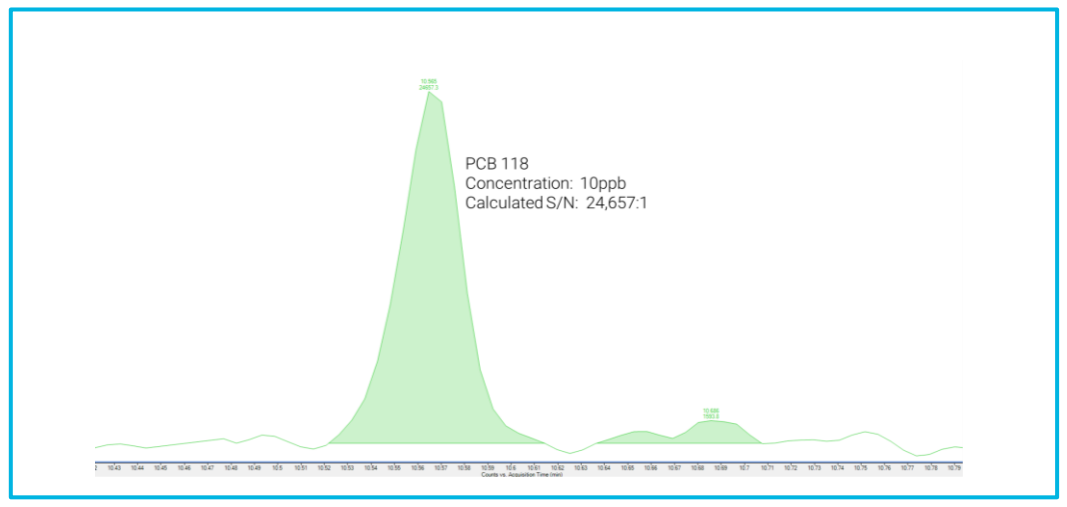


Figure 5: EIC of PCB 118 at 10ppb with the calculated signal to noise on quant ion m/z 325.8

Conclusions

Faster Analysis, Superior Separation Achieved

- The DB-XLB column offers enhanced selectivity for PCB congeners, resulting in previously co-eluting congeners to separate.
- Selectivity is easily achieved operating the MSD in SIM mode.
- The method meets the 10ppb detection limit for all the quantified compounds.
- The HydroInert ion source allows hydrogen carrier gas to be used for more rapid analysis.

References

[1]https://www.epa.gov/pcbs/learn-about-polychlorinated-biphenyls.

[2] Method 1628 Polychlorinated Biphenyl (PCB) Congeners in Water, Soil, Sediment, Biosolids, and Tissue by Low-resolution GC/MC using Selected Ion Monitoring (2021) Retrieved May 1, 2024 from https://www.epa.gov/system/files/documents/2021-07/method-1628_pcb-congeners-by-low-resolution-gc-ms_july-2021.pdf.

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