Application Brief

Environmental

Agilent

Trusted Answers

Improved Determination of Polychlorinated Biphenyl Compounds by US EPA Method 1628

Author

Jennifer Sanderson
Agilent Technologies, Inc.

Abstract

This study refines EPA Method 1628 for the analysis of polychlorinated biphenyl compounds (PCBs) using an Agilent 8890/5977C GC/MSD equipped with an Agilent HydroInert ion source and Agilent J&W DB-XLB column. This approach enables faster run times (< 20 minutes), improves separation of coeluting congeners, and easily meets sensitivity requirements.

Introduction

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

The Environmental Protection Agency (EPA) has recently developed a low-resolution mass spectrometry method (US EPA Method 1628) that calibrates 65 PCB congeners and screens for all 209, using labeled PCB compounds for both 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 procedure uses helium as a carrier gas. Additionally, the column recommended for this analysis makes adequate separation and quantitation more challenging, requiring over 40 minutes of run time to achieve separation that meets the method criteria.

This application brief demonstrates the use of an Agilent J&W DB-XLB column—which is better suited for PCB congener separation—as well as the use of hydrogen as the carrier gas. For EPA Method 1628, this alternative column and carrier gas can provide superior separation in under 20 minutes, resolving analytes that previously coeluted.

Experimental

For this analysis, an 8890/5977C GC/MSD, equipped with a HydroInert ion source, was operated in selected ion monitoring (SIM) mode. The parameters used for this method are listed in Tables 1, 2, and 3.

Table 1. Agilent 8890 GC parameters for EPA Method 1628.

Parameter	Value
Inlet, Mode, Liner	Multimode inlet (MMI), constant flow, Agilent Ultra Inert, low-fritted liner, 4 mm (p/n 5190-5112)
Temperature	300 °C
Carrier Gas	Hydrogen
Septum Purge Flow	2 mL/min
Column	Agilent J&W DB-XLB, 20 m × 180 µm, 0.18 µm (p/n 121-1222)
Flow	0.9 mL/min
Oven Temperature	50 °C (hold for 0.4 min), 30 °C/min to 180 °C, 10 °C/min to 300 °C (hold for 1.257 min)
Equilibrium Time	1 min
GC Transfer Line Temperature	300 °C

Table 2. Agilent 5977C GC/MSD source parameters for EPA Method 1628.

Parameter	Value
Ion Source	Agilent HydroInert source
Ionization Mode	70 eV EI
Acquisition Mode	Selected ion monitoring (SIM)
Scan Speed	N = 2
Ion Source Temperature	280 °C
Quadrupole Temperature	150 °C
Number of SIM Groups	7
Run Time	40 min

Table 3. Agilent 5977C GC/MSD selected ion monitoring (SIM) parameters for EPA Method 1628.

	Start Time (min)	Dwell Time (ms)	SIM Ions (*m/z*)
Group 1	4.25	320	188.00, 190.00, 200.00, 202.00, 222.00, 224.00, 234.00, 236.00
Group 2	5.60	300	222.00, 224.00, 234.00, 236.00, 256.00, 258.00, 268.00, 270.00, 290.00, 291.80, 302.00, 304.00
Group 3	8.00	300	255.90, 258.00, 267.90, 270.00, 290.00, 291.80, 302.00, 304.00, 325.80, 328.00, 338.00, 340.00
Group 4	8.80	300	290.00, 291.80, 302.00, 304.00, 325.80, 328.00, 338.00, 340.00, 359.00, 362.00, 372.00, 374.00
Group 5	10.45	300	325.80, 328.00, 338.00, 340.00, 359.80, 362.00, 372.00, 374.00, 393.60, 396.00, 406.00, 408.00
Group 6	12.00	300	359.80, 362.00, 372.00, 374.00, 393.80, 396.00, 406.00, 408.00, 428.00, 429.70, 440.00, 442.00, 462.00, 463.60, 474.00, 476.00
Group 7	13.70	400	497.00, 500.00, 509.70, 512.00

Results and discussion

Figure 1 shows the chromatogram for the 65 PCB congeners specifically chosen for full quantitation by the EPA 1628 method, analyzed at concentrations of 160 ppb for native PCBs and 400 ppb for labeled PCBs. The run time for this method is just 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.

A six-point calibration curve with concentrations ranging from 10 to 2,000 ppb was prepared in iso-octane. Each calibration level was analyzed in triplicate. Agilent MassHunter Quantitative Analysis software calculates R^2 values as well as the average of response factors (RFs), as shown in two sample calibration curves from this data set (Figures 2A and 2B). The average RF values were less than 20 for all calibrated congeners, and R^2 values were greater than 0.97 across the calibration set.

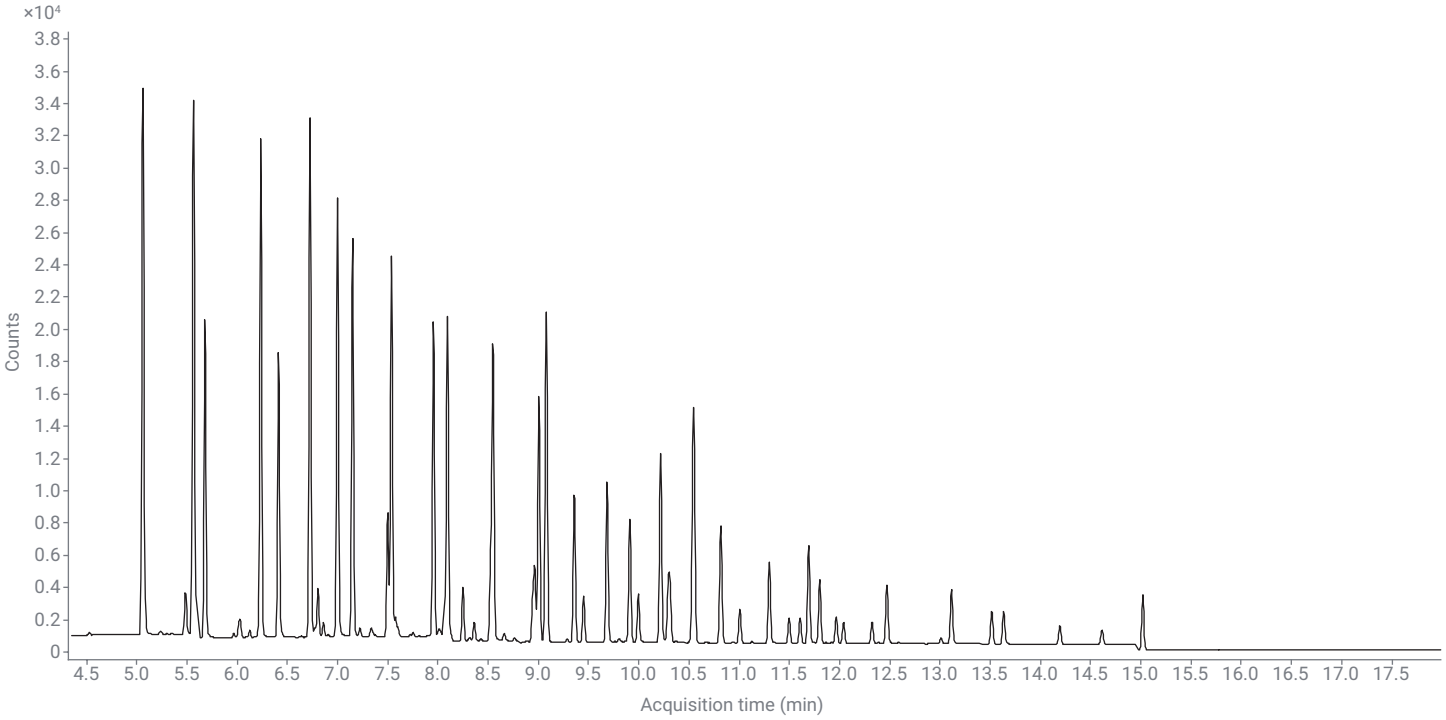

Figure 1. Chromatogram of calibrated PCB congeners.

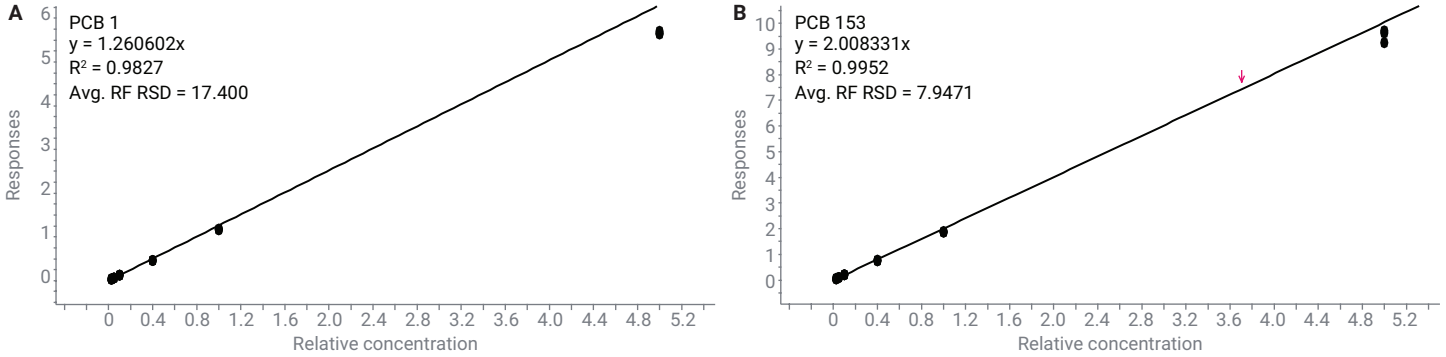

Figure 2. Six-point calibration curves for (A) PCB 1 and (B) PCB 153.

EPA Method 1628 stipulates that closely eluting PCB congeners 28 and 31 have a valley between the two peaks that is less than 80% of the height of the smaller peak. This requirement ensures that each of the congeners can be quantitated without interference. As shown in Figure 3, the two congeners meet this criterion and yield two peaks that can be quantitated individually.

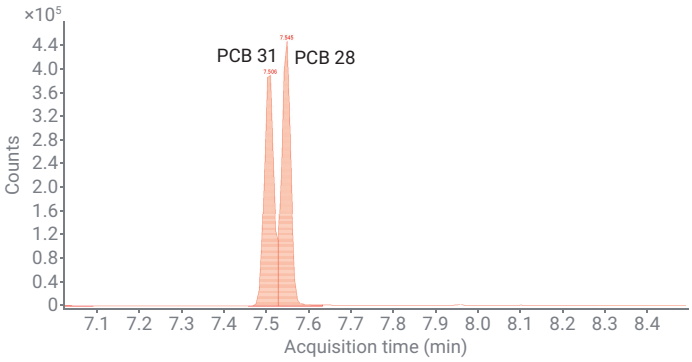

Figure 3. Chromatogram for PCB congeners 28 and 31.

In addition to PCB congeners 28 and 31, most congeners that previously coeluted were able to be resolved, enabling easier quantitation of samples with complex PCB compositions. Some examples include tetrachlorobiphenyl congeners 41 and 64, hexachlorobiphenyl congeners 139 and 149, and pentachlorobiphenyl congeners 85 and 120 (Figures 4A to 4C).

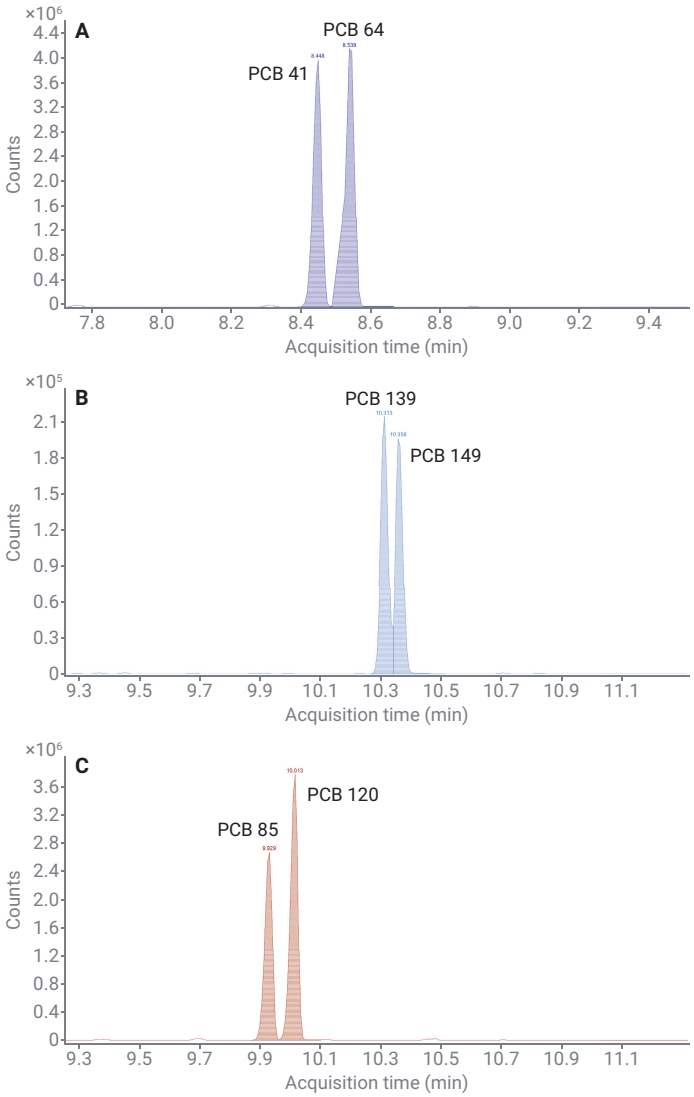

Figure 4. Chromatographically resolved congeners: (A) PCBs 41 and 64, (B) PCBs 139 and 149, and (C) PCBs 85 and 120.

EPA Method 1628 requires that PCB congener 118 have a signal-to-noise ratio of 3:1 or greater at a concentration of 10 ppb.[2] Due to the combination of very low column bleed and SIM detection, the sensitivity requirement is easily satisfied (Figure 5).

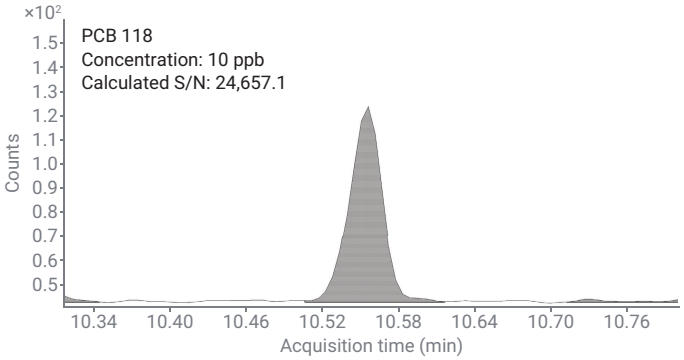

Figure 5. Extracted ion chromatogram (EIC) of PCB 118 at 10 ppb, showing the calculated signal-to-noise ratio for quantifier ion *m/z* 325.8.

Conclusion

This application brief describes the refinement of EPA Method 1628 using an Agilent 8890/5977C GC/MSD, equipped with an Agilent J&W DB-XLB column and an Agilent HydroInert ion source. When operated in SIM mode, the system easily delivers improved selectivity for PCBs and meets the 10 ppb detection limit for all quantified compounds. Furthermore, the use of hydrogen as the carrier gas facilitates faster analysis. As a result of these method enhancements, superior separation of previously coeluting congeners is achieved.

References

1. U.S. Environmental Protection Agency. Learn About Polychlorinated Biphenyls (PCBs). https://www.epa.gov/pcbs/learn-about-polychlorinated-biphenyls.
2. U.S. Environmental Protection Agency. Method 1628: Polychlorinated Biphenyl (PCB) Congeners in Water, Soil, Sediment, Biosolids, and Tissue by Low-Resolution GC/MS Using Selected Ion Monitoring; EPA 821-R-21-002, July 2021. https://www.epa.gov/system/files/documents/2021-07/method-1628_pcb-congeners-by-low-resolution-gc-ms_july-2021.pdf.

www.agilent.com

DE-008323

Printed in the USA, August 22, 2025
5994-8541EN

Agilent
Trusted Answers