Application Note

Environmental

Determination of Polychlorinated Biphenyl (PCB) by GC/MS According to EPA Method 1628

Using an Agilent 8890 GC with an Agilent 5977C GC/MSD in selected ion monitoring (SIM) mode

Author

Jennifer Sanderson
Agilent Technologies, Inc.

Abstract

Polychlorinated biphenyls (PCBs), once widely used in industrial applications, have been banned and classified as persistent organic pollutants (POPs) by the U.S. Environmental Protection Agency (EPA).[1] EPA method 1628 employs low-resolution mass spectrometry in selected ion monitoring (SIM) mode to measure all 209 PCB congeners, calibrating 65 and screening 144. This study uses an Agilent 8890 GC with an Agilent 5977C GC/MSD to analyze PCBs in sand and soil, meeting all method requirements for linearity, resolution, and sensitivity.

Introduction

PCBs are synthetic compounds belonging to the chlorinated hydrocarbon family. Historically, PCBs were extensively used in various industrial applications, including electrical components, plasticizers, and pigments and dyes. However, due to their environmental persistence and potential health risks, their use was banned in 1979 under the Toxic Substances Control Act (TSCA). Classified as POPs, PCBs resist degradation and are known to bioaccumulate in aquatic life according to the EPA.[1]

In response to the need for effective monitoring of PCBs, the EPA has developed method 1628, which employs low-resolution mass spectrometry in SIM mode.[2] This method is the first to measure all 209 individual PCB congeners, calibrating for 65 PCBs and screening for the remaining 144 congeners. The method includes 29 labeled PCB surrogates and three labeled PCBs as internal standards for analytical accuracy. In this application note, an 8890 GC with a 5977C GC/MSD was used to analyze PCBs in sand and soil, meeting all requirements for EPA method 1628.

Experimental

An 8890 GC with a 5977C GC/MSD was used for this work. The 8890 GC was configured with mid-column backflush to prolong column lifetime. Detailed method parameters were set according to EPA method 1628 and are shown in Tables 1 to 3.

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

Parameter	Value	
Inlet, Mode, and Temperature	Multimode inlet (MMI), constant flow, 300 °C	
Carrier Gas	Helium	
Septum Purge Flow	2 mL/min	
Backflush	Midcolumn	
Column	1. Agilent J&W DB-5ms Ultra Inert, 30 m × 250 µm × 0.5 µm (p/n 122-5536UI)	2. Agilent J&W DB-5ms Ultra Inert, 30 m × 250 µm × 0.5 µm (p/n 122-5536UI)
Flow	1.2 mL/min	1.0 mL/min
Inlet	MMI	PSD 4 helium
Outlet	PSD 4 helium	GC/MSD
PSD Purge Flow		2 mL/min
Oven Temperature	50 °C (1 min), 12 °C/min to 240 °C (1.167 min), 5 °C/min to 320 °C (6 min)	
Post Run Flow	–5.7798 mL/min	6.0 mL/min
Post Time	3 min	
Equilibrium Time	1 min	
GC Transfer Line Temperature	325 °C	

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

Parameter	Value
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 parameters for EPA method 1628.

	Start Time (min)	Dwell Time (ms)	SIM Ions (*m/z*)
Group 1	13	50	188.00, 190.00, 200.00, 202.00, 222.00, 224.00, 234.00, 236.00
Group 2	19.2	25	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	24	25	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	25.3325	25	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	28.8	25	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	31.5	25	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	38	100	497.00, 500.00, 509.70, 512.00

For the 65 specified PCB congeners in EPA method 1628, a nine-point calibration curve ranging from 5 to 1,000 ppb was analyzed in triplicate to ensure accuracy. Method blanks and system blanks were analyzed to check for any potential contamination. Also, the method detection limit (MDL) was determined using eight replicates in a sand matrix to represent a high solid matrix. Soil samples, both with and without spiking with the 65 calibrated PCBs, were also analyzed to assess the method performance in real-world conditions. The remaining 144 congeners were evaluated in sand and soil matrix and were not quantified in this study. Sand was used as a control for the solid matrices.

Results and discussion

Example chromatograms are shown in Figures 1 and 2. Figure 1 shows the separation of the 65 calibrated PCB congeners and the 32 labeled PCB congeners at 50 and 40 ppb, respectively. The analytical separation of all 209 PCB congeners and 32 labeled PCB congeners at 50 and 40 ppb is shown in Figure 2.

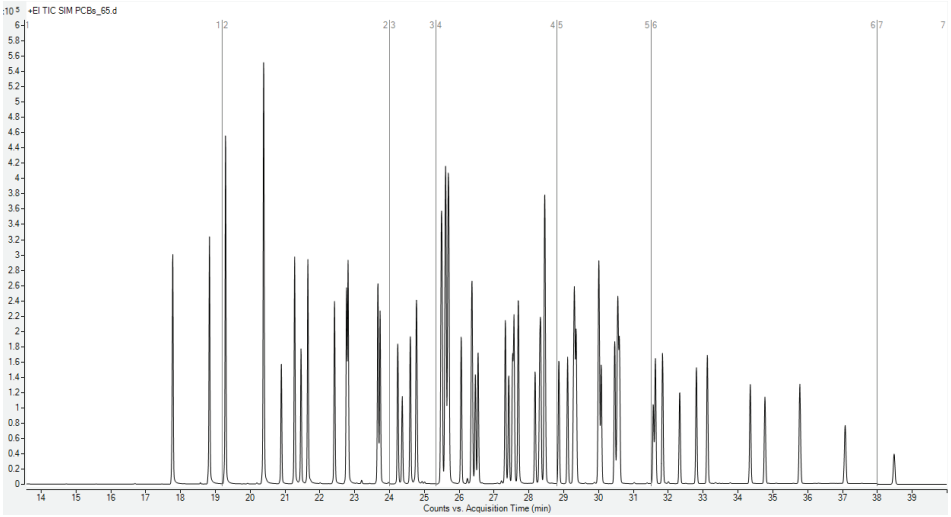

Figure 1. Separation of the 65 calibrated PCB congeners and the 32 labeled PCB congeners according to EPA method 1628.

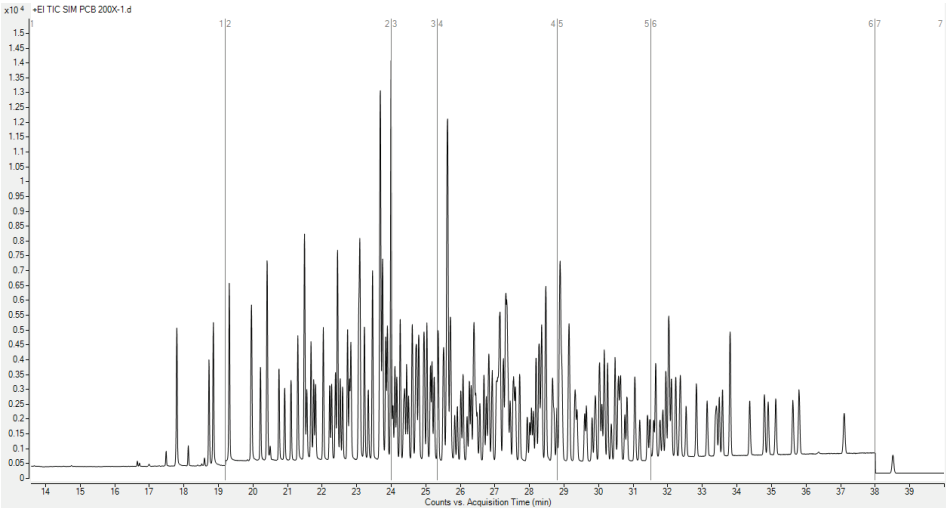

Figure 2. Separation of all 209 PCB congeners and 32 labeled PCB congeners according to EPA method 1628.

Per the method requirements for EPA method 1628, PCB congeners 28 and 31 must be chromatographically separated. The valley of the two peaks must be less than 80% of the height of the smaller peak, which is demonstrated in Figure 3. The method also requires that PCB congener 118 at 10 ppb has a signal-to-noise ratio (S/N) greater than or equal to 3:1. Figure 4 shows successfully meeting this method criteria with a S/N of 6.1 for PCB congener 118 at 10 ppb.

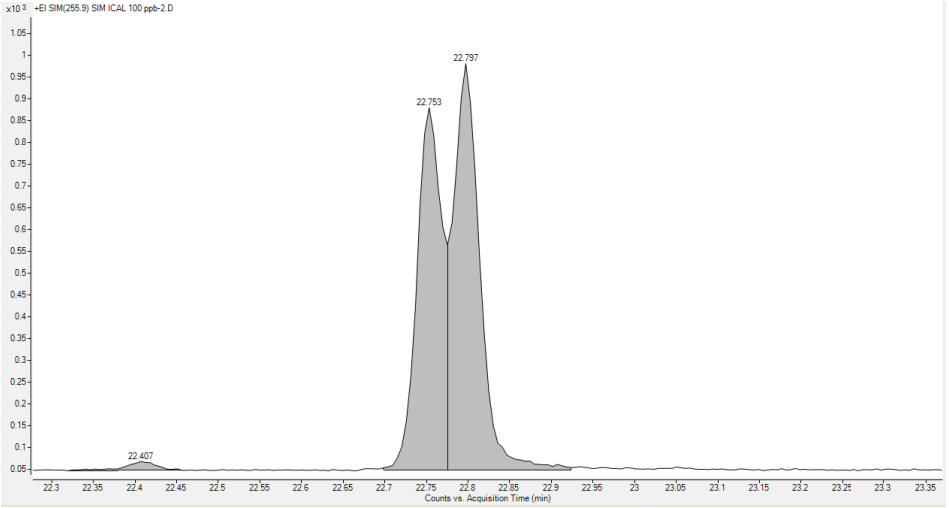

Figure 3. PCB congeners 28 and 31 mixed at approximately 1:1.

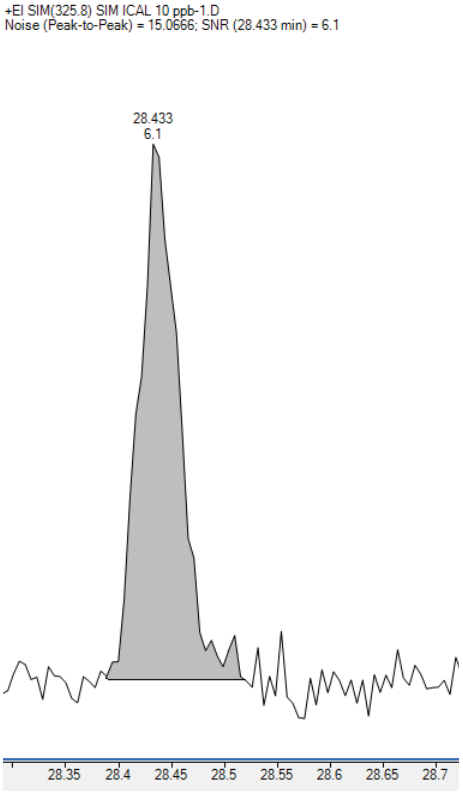

Figure 4. PCB congener 118 at 10 ppb.

As specified in EPA method 1628, 65 PCB congeners were calibrated for. For each of these compounds, nine-point (n = 3) calibration curves were generated ranging from 5 to 1,000 ppb. Five example calibration curves are shown in Figures 5 to 9. These calibration curves present a representative sampling of the total 65 PCB congener calibration curves. Congeners 28 and 31 were calibrated for with excellent recovery and calibration for the heaviest congeners despite lower chromatographic resolution. PCB congener 101 was chosen to represent the calibration of a midpoint congener. For all 65 compounds, a strong linear correlation was observed with $R^2 > 0.991$ for each. The average response factor RSDs ranged from 0.906 to 11.34%, meeting the requirements for EPA method 1628 where RSD must be less than 20%.

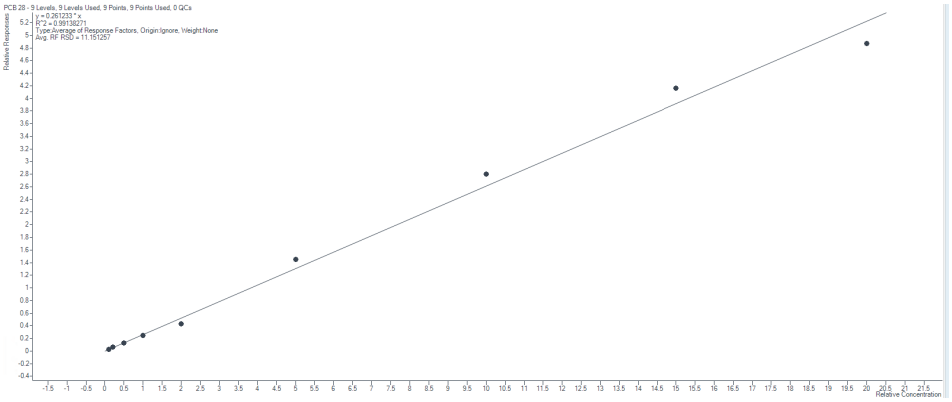

Figure 5. Calibration curve for PCB congener 28.

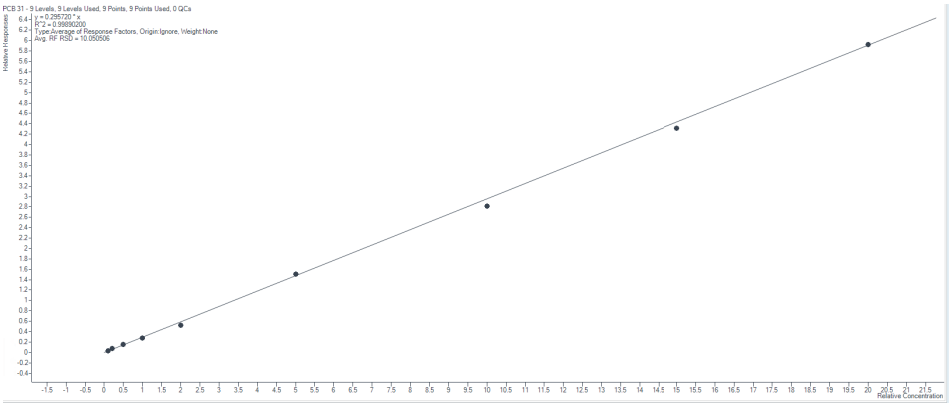

Figure 6. Calibration curve for PCB congener 31.

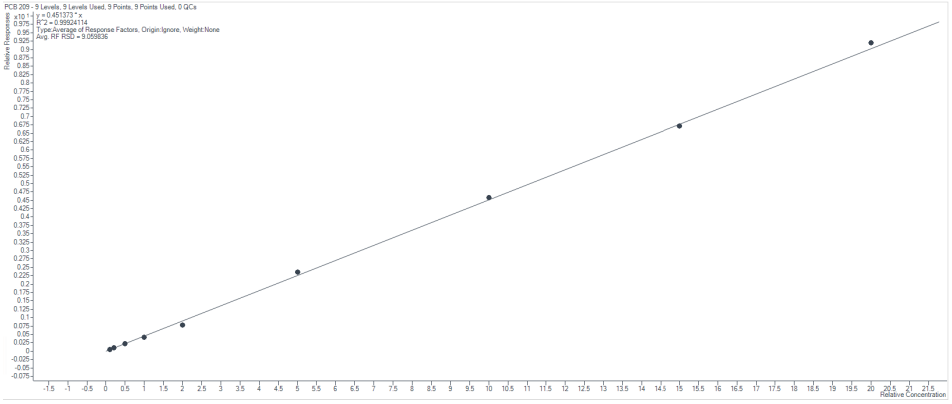

Figure 7. Calibration curve for PCB congener 209.

To establish the MDL for each of the congeners, 10 g of dry sand was extracted as described in the method. The MDL ranges specified in the method range from < 0.01 to 0.4 ng/g. The MDL established in the sand matrix meets the criteria outlined in the method.

Locally sourced soil was prepared and analyzed for all 209 native PCBs. Additional soil samples were spiked with the calibrated 65 PCBs at 10 ppb. No PCBs were found in the soil, and spiked recoveries fell within the range of 50 to 150% recovery for the 65 PCBs.

Conclusion

An Agilent 8890 GC with an Agilent 5977C GC/MSD was successfully used to analyze PCB congeners according to EPA method 1628. This method uses low-resolution SIM mode to quantitate 65 congeners while being able to see all 209 native congeners. All method requirements were met, including for linearity, resolution, and sensitivity.

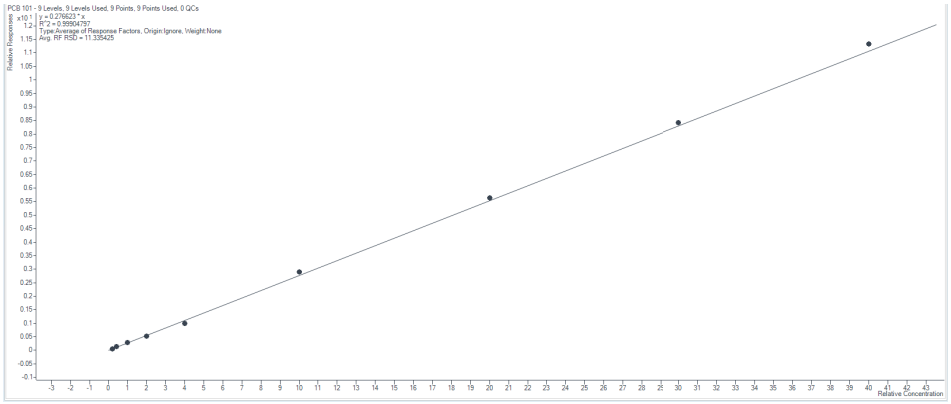

Figure 8. Calibration curve for PCB congener 101.

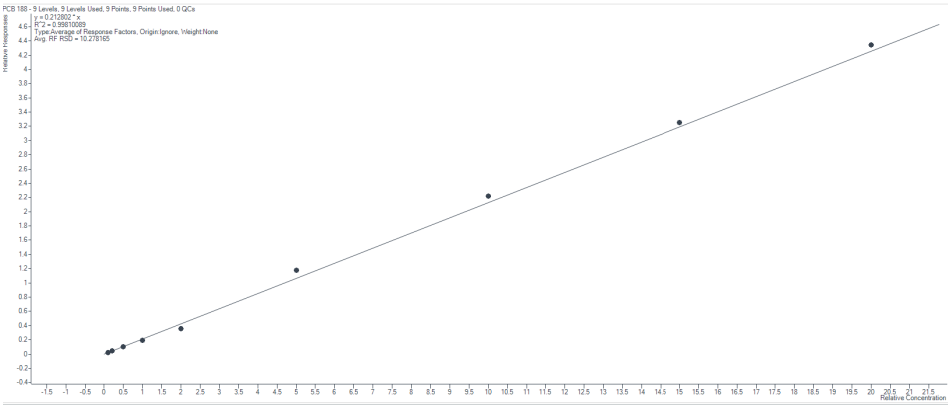

Figure 9. Calibration curve for PCB congener 188.

References

1. Learn about Polychlorinated Biphenyls. United States Environmental Protection Agency. Website: https://www.epa.gov/pcbs/learn-about-polychlorinated-biphenyls (accessed 2024-10-07).
2. Method 1628 Polychlorinated Biphenyl (PCB) Congeners in Water, Soil, Sediment, Biosolids, and Tissue by Low-resolution GC/MS using Selected Ion Monitoring. United States Environmental Protection Agency. Website: https://www.epa.gov/system/files/documents/2021-07/method-1628_pcb-congeners-by-low-resolution-gc-ms_july-2021.pdf (accessed 2024-10-07)

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