

Analysis of PolyChlorinated Biphenyls (PCBs) by GC/MS

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Key Words

- US EPA Method 608
- PolyChlorinated Biphenyls
- Organic pollutants TG-5SiIMS
- 5% Phenyl

Introduction

US EPA 608 is an analytical method used for determination of certain PolyChlorinated Biphenyls (PCBs) and OrganoChlorine Pesticides (OCPs) using appropriate extraction techniques from municipal and industrial wastewater. The method was developed by the US Environmental Protection Agency to monitor industrial and municipal discharges under 40 CFR 136.1.

US EPA method 608 is usually performed using ECD detection with confirmational analysis by GCMS as specified in US EPA method 625. This analysis will fulfil the identification parameters for the PCBs listed and was performed using a Thermo Scientific TRACE GC in splitless injection mode coupled to a Thermo Scientific ion trap mass spectrometer.

The Thermo Scientific TraceGOLD TG-5SiIMS column fulfils the requirement of the US EPA method 625 providing excellent performance for the analysis of the listed PCBs.

Goal

To demonstrate the suitability and performance of TraceGOLD™ TG-5SiIMS for confirmational identification when using US EPA method 608.

Experimental details

A standard mix of PCBs, representative of those listed in the US EPA method 608, were analysed on a TRACE™ GC fitted with a TriPlus autosampler. An ion trap mass spectrometer was used in a segmented mode to allow precise control of ion groups for improved ion statistics and ratios. The column used for analysis of the PCB standard mixture, was a low polarity silarylene phase with selectivity similar to a 5% diphenyl/95% dimethyl polysiloxane phase. The data was acquired and processed using the Thermo Scientific Xcalibur data handling software.

Sample preparation

A pre-mixed 1ng/µL PCB standard solution was used for the analysis.

Column	Part Number
TraceGOLD TG-5SiIMS, 30 m × 0.25 mm × 0.25 µm,	26096-1420
Guard Column 2 m × 0.32 mm	260RG497
Press-Fit Union	64000-001



Thermo Scientific TriPlus Autosampler

Sample volume	1 µL
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TRACE GC Ultra

Oven Program	60 °C (5 min), 8 °C/min, 300 °C (10 min)
Equilibration Time	0.5 min
Injector	275 °C, Splitless (1 min)
Split Flow	30 mL/min
Column Flow	Helium, 1.5 mL/min (constant flow)
Transfer Line Temperature	300 °C

Thermo Scientific Ion Trap MS

MS Type	ITD 230 LT (250 L turbo pump)
MS Source Temperature	225 °C
MS Source Current	250 µA
Electron Energy	70 eV
Filament Delay	5 min
MS Acquisition Mode	El+, 45-450 amu Segmented Scan

Consumables

Consumables	Part Number
BTO 17 mm septa	31303211
3 mm ID Focus Liner, 105 mm long	45350032
Liner graphite seal	29033406
10 µL, 80 mm Syringe	36502019
Graphite ferrules to fit 0.32 mm id columns	29053487
Graphite/vespel 0.25 mm ID ferrules for GC/MS interface	29033496
2 mL clear vial and Si/PTFE seal	60180-599

Results

The US EPA method 608 covers the determination of certain organochlorine pesticides and PCBs and requires a confirmation step using GC with MS detection. The separation and identification of the PCB analytes using the TraceGOLD TG-5SilMS column on a GC/MS system fulfils this requirement. The stationary phase of the TraceGOLD TG-5SilMS column provides excellent performance due to minimal interaction of active compounds with active sites on the column, minimising peak tailing and optimising resolution. Figure 1 shows the TIC chromatogram for 1 ng/ μ L of PCBs obtained using a TraceGOLD TG-5SilMS column. Table 1 shows the peak identification of PCBs according to the retention times on TraceGOLD TG-5SilMS column.

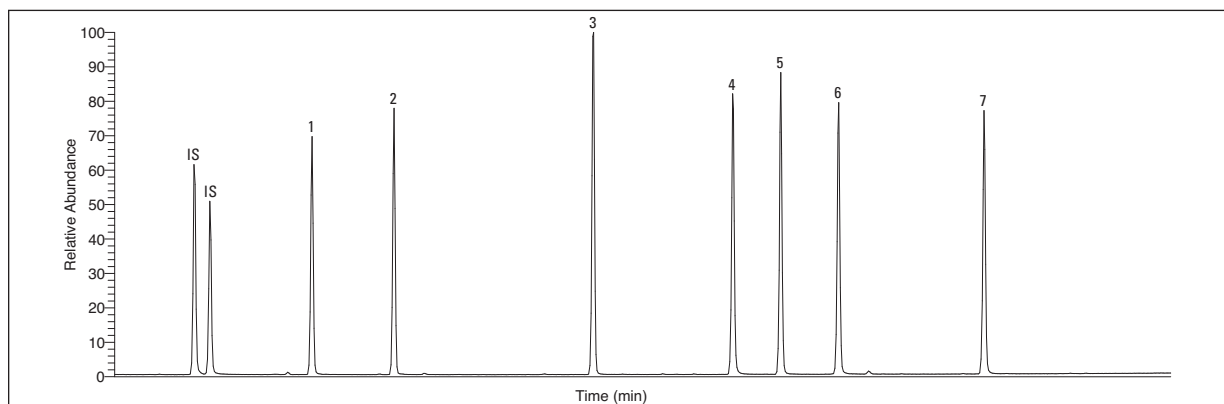


Figure 1: TIC chromatogram of 1 ng/ μ L of PCB standard mix separated on a TG-5SilMS column

PCB	Peak No.
Internal standard	IS
Internal standard	IS
CB-28 (2,4,4'-Trichlorobiphenyl)	1
CB-53 (2,2',5,5'-Tetrachlorobiphenyl)	2
CB-101 (2,2',4,5,5'-Pentachlorobiphenyl)	3
CB-118 (2,4,5,3',4'-Pentachlorobiphenyl)	4
CB-153 (2,2',4,4',5,5'-Hexachlorobiphenyl)	5
CB-138 (2,2',3,4,4',5'-Hexachlorobiphenyl)	6
CB-180 (2,2',3,4,4',5,5'-Heptachlorobiphenyl)	7

Table 1: List of PCBs according to retention

Conclusions

The TraceGOLD TG-5SilMS column demonstrated excellent performance for the analysis of PCBs with excellent peak shape and resolution. The chromatogram illustrates the superior performance of the TraceGOLD TG-5SilMS column for this analysis.

References

1. US EPA 608

Acknowledgement

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