

Analysis of Chlorinated Pesticides by GC/MS

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Key Words

- US EPA Method 608, 617, 8080
- Chlorinated Pesticides
- Organic pollutants
- TG-5SiIMS
- 5% Phenyl

Introduction

The US EPA methods listed are analytical methods used for determination of certain PolyChlorinated Biphenyls (PCBs) and OrganoChlorine Pesticides (OCPs) using appropriate extraction techniques from municipal and industrial wastewater. The methods were developed by the US Environmental Protection Agency to monitor industrial and municipal discharges under 40 CFR 136.1. These US EPA methods are usually performed using two detection systems. The first (usually ECD) for screening (quantification and identification) and a second confirmational analysis by GCMS. This analysis will fulfil the identification parameters for the organochlorine pesticides listed below and which correspond to those listed in the US EPA methods.

The analysis 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 provides excellent performance for the analysis of OCPs in accordance to the US EPA methods 608, 617 and 8080.

Goal

To demonstrate the suitability and performance of TraceGOLD™ TG-5SiIMS for confirmational identification when using US EPA methods 608, 617, 8080.

Experimental details

A standard mix of organochlorine pesticides representative of those listed in the US EPA methods 608,617 and 8080 were run 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 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 1 ng/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 requirements for US EPA methods 608, 617 and 8080 were achieved with the separation and identification of a range of organochlorine pesticides using the TraceGOLD TG-5SilMS on a GC/MS system. The stationary phase of the TraceGOLD TG-5SilMS 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/μL of organochlorine pesticides obtained using a TraceGOLD TG-5SilMS column. Table 1 shows the peak identification of the organochlorine pesticides according to the retention times on TraceGOLD TG-5SilMS column.

Conclusions

The TraceGOLD TG-5SilMS column demonstrated excellent performance for the analysis of organochlorine pesticides with excellent peak shape and resolution. The chromatogram illustrates the superior performance of the TraceGOLD TG-5SilMS for this analysis, in accordance with the EPA methods 608, 617 and 8080.

References

1. USA EPA 608

Acknowledgement

Many thanks to the dedicated team at Cromlab S.L., Barcelona, Spain for all of their help providing the data for this application.

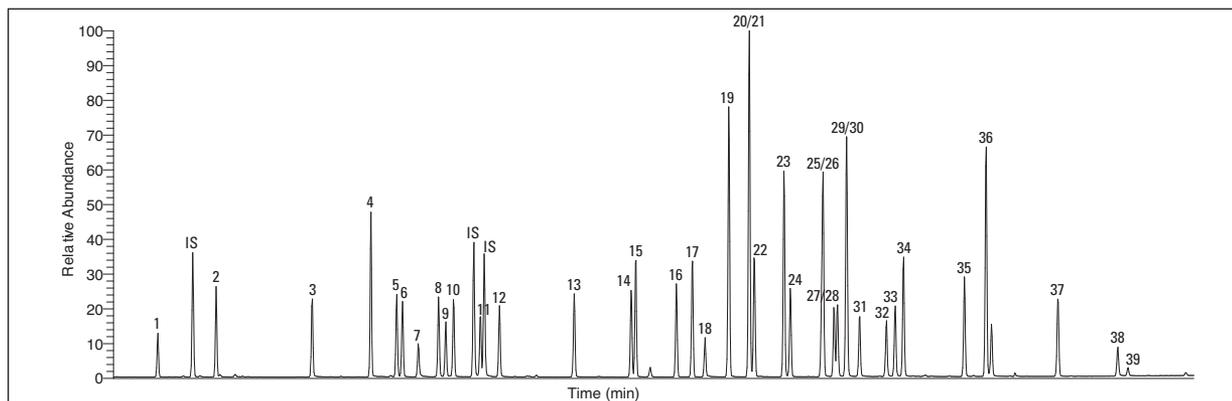


Figure 1: TIC chromatogram of 1 ng/μL of Organochlorine Pesticide standard mix separated on a TG-5SilMS column

Compound Name	Peak No.	Compound Name	Peak No.	Compound Name	Peak No.
Etridiazole	1	delta-HCH	12	Perthane	26
Internal Standard	IS	Heptachlor	13	Chlorobenzilate	27
Chloroneb	2	Aldrin	14	Endosulfan-II	28
Propachlor	3	DCPA	15	cis-Nonachlor	29
Trifluralin	4	Isodrin	16	pp-DDD	30
alfa-HCH	5	Heptaclor-epoxide	17	Endrin-aldehyd	31
HCB	6	Captan	18	Carbofenthion	32
Dicloran	7	gamma-Chlordane	19	Endosulfan-sulfat	33
beta-HCH	8	Endosulfan-I	20	pp-DDT	34
Quintozene	9	alfa-Chlordane	21	Endrin-Ketone	35
gamma-HCH	10	trans-Nonachlor	22	Methoxychlor	36
Internal Standard	IS	pp-DDE	23	Mirex	37
Chlorotalonil	11	Dieldrin	24	cis-Permethrin	38
Internal Standard	IS	Endrin	25	trans-Permethrin	39

Table 1. Peak identification list of Organochlorine Pesticide standard mix

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