Rapid Analysis of CLP Pesticides Using High-Temperature Agilent J&W DB-CLP1 and DB-CLP2 Columns

Application Note

Environmental

Authors

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Abstract

Agilent J&W DB-CLP1 and DB-CLP2 are a pair of arylene-phase columns (primary and confirmation) that permit high oven temperature for rapid analysis of Contract Laboratory Program (CLP) chlorinated pesticides. The columns are also suitable for phenoxy acids, haloacetic acids, polychlorinated biphenyls, and United States Environmental Protection Agency (US EPA) Method 508.1 pesticides.

Introduction

Accurate identification and confirmation of trace level chlorinated pesticides are difficult tasks facing environmental laboratories. The chromatographic system, including the analytical columns, must be optimized. The gas chromatography columns must possess the selectivity, inertness, and thermal stability needed to achieve optimum resolution and sensitivity in the shortest possible time. These needs are realized with Agilent J&W DB-CLP1 (primary) and DB-CLP2 (confirmation) columns.

The excellent selectivity of high phenyl content phases for chlorinated pesticides is well documented. However, these phases typically suffer from poor thermal stability resulting in high bleed and excessively long analysis times. DB-CLP1 uses arylene-phase technology to provide improved thermal stability through a "stiffening" of the polymer backbone. Arylene MS-grade columns use special surface deactivations and siloxane chemistries, which enhance the chromatographic performance of siloxane polymers. The arylene inclusion in the siloxane polymer strengthens the polymer backbone, reducing the amount of stationary phase degradation, thus reducing column bleed.

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The result is increased sensitivity and an upper temperature limit of 360 °C. The column bleed contribution to background noise is reduced, giving a much improved signal-to-noise ratio and increased usable sensitivity compared to standard 35%-phenyl phases. The high thermal limit translates into shorter analysis times, increased column lifetime, and the ability to periodically bake the column at a high temperature to remove semivolatile contaminants.

DB-CLP2 uses a proprietary, second-generation arylene technology giving it the same 360 °C upper temperature limit and the lowest bleed of any such phase available.

Experimental

Conditions

Column 1:	Agilent DB-CLP1, 30 m × 0.32 mm, 0.25 µm (p/n 123-8232)
Column 2:	Agilent DB-CLP2, 30 m × 0.32 mm, 0.5 µm (p/n 123-8336)
Carrier:	Helium, constant flow 2.5 mL/min
Oven:	90 °C (0.5 min) 35 °C/min to 175 °C 12 °C/min to 300 °C (1.75 min)
CFT device:	Inert tee (p/n G3184-60065), split ratio 1:1
Retention gap:	5 m x 0.32 mm id deactivated fused silica tubing
Inlet:	2 µL splitless 250 °C, purge flow 60 mL/min at 0.5 min
GC/µECD:	Agilent 7890A GC
Sampler:	Agilent 7683B Automatic Liquid Sampler, 5.0 µL tapered syringe (p/n 5181-1273)
µECD:	325 °C, constant column + makeup (N_2) = 30 mL/min

Flow path supplies

Vials and caps:	MS certified amber crimp top glass vials and caps kit (p/n 5190-2283)
Vial inserts:	250 µL glass/polymer feet (p/n 5181-8872)
Syringe:	5 µL tapered (p/n 5181-1273)
Septum:	Advanced Green (p/n 5183-4759)
Inlet liner:	Agilent Ultra Inert double tapered liner (p/n 5190-3983)
Ferrules:	0.5 mm id short; 85/15 Vespel/graphite (p/n 5062-3514)
CFT fittings:	Internal nut (p/n G2855-20530)
CFT ferrules:	SilTite ferrules, 0.32 mm id (p/n 5188-5362)
Magnifier:	20× magnifier loop (p/n 430-1020)

Results and Discussion

Figure 1 shows the optimized primary and confirmation chromatograms for the DB-CLP1/DB-CLP2 column pair using helium carrier for CLP Pesticides.

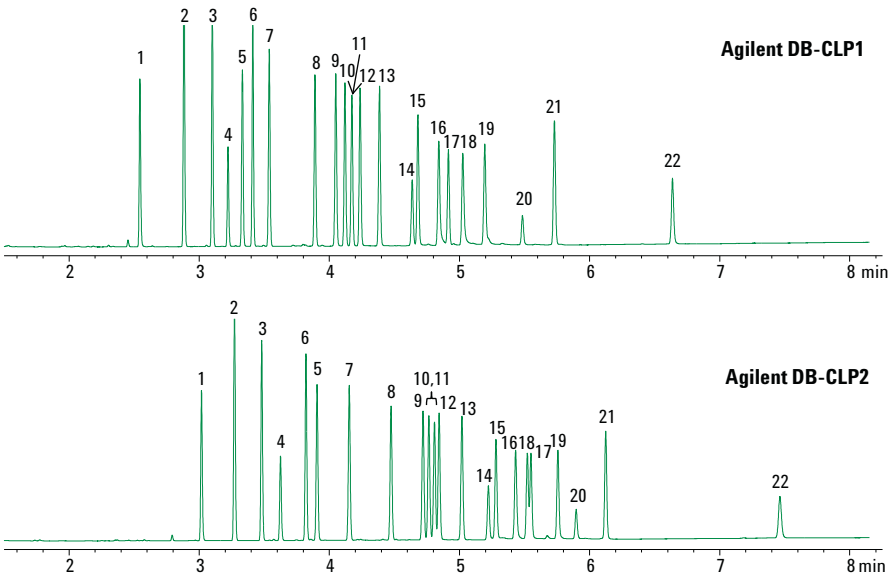

Figure 1. CLP pesticides analyzed on Agilent J&W DB-CLP1 and DB-CLP2 columns.

Peaks	
1.	Tetrachloro-m-xylene*
2.	α-BHC
3.	γ-BHC
4.	β-BHC
5.	Heptachlor
6.	δ-BHC
7.	Aldrin
8.	Heptachlor epoxide
9.	γ-Chlordane
10.	α-Chlordane
11.	Endosulfan I
12.	4,4′-DDE
13.	Dieldrin
14.	Endrin
15.	4,4′-DDD
16.	Endosulfan II
17.	4,4′-DDT
18.	Endrin aldehyde
19.	Endosulfan sulfate
20.	Methoxychlor
21.	Endrin ketone
22.	Decachlorobiphenyl*

*surrogate standard

Because these columns are designed for enhanced thermal resistance, it is not necessary to bake them excessively upon installation to reduce bleed to acceptable levels. A simple 1 to 2 hour conditioning period is typically more than adequate. Conditioning columns overnight is a common requirement with cyanopropyl- and trifluoropropyl-containing CLP pesticide columns. This practice can result in increased column activity and decreased column life time but is not required with DB-CLP1 or DB-CLP2. In short, chromatography can proceed sooner after column installation.

Environmental laboratories are also interested in other gas chromatography/electron capture detector (GC/ECD) methods with the same dual column pair used for the chlorinated pesticides. These methods include phenoxy acid herbicides (EPA Method 8151A), haloacetic acids (EPA Method 552.2), PCBs (EPA Method 8082), and EPA Method 508.1 pesticides.

Conclusions

The Agilent J&W DB-CLP1 and DB-CLP2 column pair performs rapid, high-resolution separations of CLP pesticides. The high temperature limit and low column bleed make these columns attractive for analyses of similar semivolatile sample mixtures. This column pair is the pair of choice for both CLP pesticides and US EPA methods calling for dual-µECD detection.

For More Information

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