Rapid Analysis of CLP Pesticides Using High-Temperature DB-35ms and DB-XLB Columns

Application

Environmental

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Abstract

Arylene-phase column pairs (primary and confirmation) permit high oven temperature for rapid analysis of CLP chlorinated pesticides. The columns are also suitable for phenoxy acids, haloacetic acids, polychlorinated biphenyls and Environmental Protection Agency 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 (GC) 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 Technologies' J&W Brand DB-35ms (primary) and DB-XLB (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-35ms uses arylene-phase technology to provide improved thermal stability through a "stiffening" of the polymer backbone. 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-XLB uses a proprietary second-generation arylene technology giving it the same 360°C upper temperature limit and the lowest bleed of any phase available.

Experimental

The columns and related inlet parts are described in Table 1.

Table 1. Columns and Related Parts

Phase/Description	id (mm)	Film (µm)	Length (m)	Part Number
DB-35ms	0.32	0.25	30	123-3832
DB-XLB	0.32	0.50	30	123-1236
Quartz deactivated splitter	-	-	-	5181-3398
Deactivated fused silica guard column	0.53	-	5	160-2535-5

This is a small sampling of the many DB columns and dimensions available.

Results and Discussion

Simple window diagramming identified the exact film thickness necessary to allow DB-XLB to give optimum confirmation power when run using the primary column temperature program. Figure 1 shows the optimized primary and confirmation chromatograms for the DB-35ms/DB-XLB column pair.

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-35ms/DB-XLB. In short, you are ready sooner after column installation.

Environmental laboratories are also interested in other gas chromatograpy/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. This column pair provides baseline resolution of all 8151A analytes on both columns in just over 16 minutes. In addition, DB-35ms and DB-XLB provide the best confirmation and the fewest coelutions of any dual column pair commercially available for 508.1 pesticides.

Conclusions

The DB-35ms and DB-XLB column pair perform 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.

For More Information

For more information on our products and services, visit our Web site at www.agilent.com/chem.

Column: **DB-35ms**
30 m × 0.32 mm id, 0.25 µm
P/N: 123-3832

Column: **DB-XLB**
30 m × 0.32 mm id, 0.50 µm
P/N: 123-1236

Carrier: Helium at 45 cm/sec
(EPC in constant flow mode)

Oven: 110 °C for 0.5 min
110-320 °C at 15 °/min
320 °C for 2 min

Injector: Splitless, 250 °C
30 sec purge activation time
50 pg per component

Detector: Micro ECD, 350 °C
Nitrogen makeup gas
(column + makeup flow =
30 mL/min constant flow)

1. Tetrachloro-m-xylene (SS)
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 (SS)

SS-Surrogate Standard

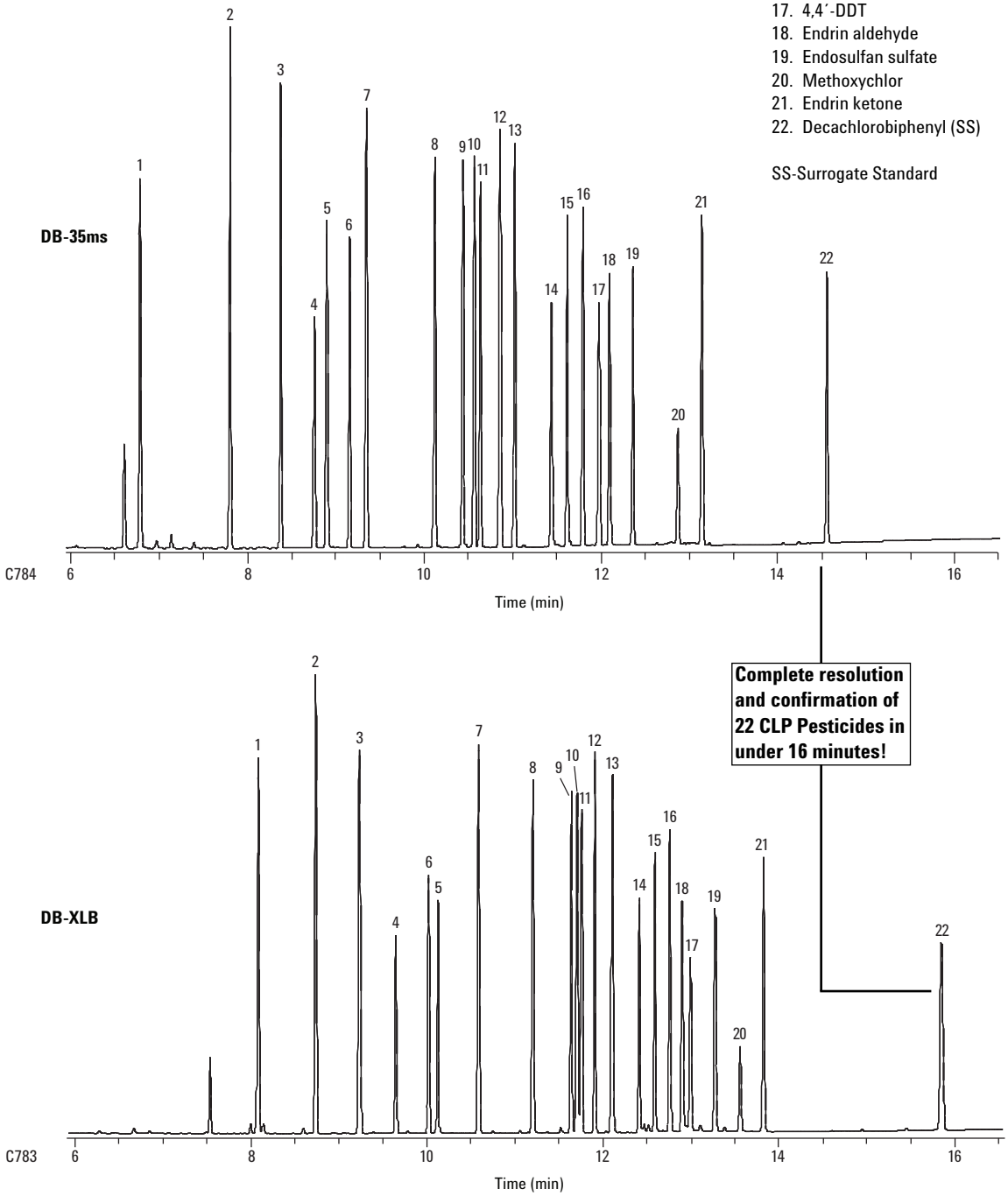

Figure 1. CLP Pesticides

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