Rapid Contract Laboratory Program (CLP) Pesticides Analysis with 0.32 mm ID Capillary GC Columns Utilizing Hydrogen Carrier Gas

Application Brief

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Environmental

Analysis time is often a limiting factor in sample turnaround for a laboratory. In order to be competitive, a contract laboratory must analyze a high volume of samples quickly and efficiently, while providing high quality results. Many laboratories prefer the higher sample loading capacity of the 0.32 mm id medium bore capillary columns, but do not want to contend with the longer analysis times usually associated with these columns. A typical CLP pesticides analysis run time using a 30-meter 0.32 mm id column can be over 20 minutes to resolve all 20 pesticides. This application brief is for laboratories that prefer using 30-meter 0.32 mm id medium bore capillary columns for pesticide determination and are looking for high sample throughput. Other application notes have demonstrated fast CLP pesticides analyses using Agilent J&W DB-17ms and Agilent J&W DB-XLB 0.18 mm id High Efficiency columns when sample capacity is not a major concern. [1,2]

Highlights

- **Rapid pesticides analysis** while maintaining sample loading capacity with 0.32 mm id columns
- **Increased speed of analysis** using hydrogen carrier and flow programming
- **Resolution of traditional CLP pesticides** plus seven additional target pesticides in under eight minutes using Agilent J&W DB-17ms and DB-XLB columns
- **Capillary Flow Technology (CFT):** Agilent's reusable CFT 2-way splitter without makeup gas simplifies maintenance of dual column system

Agilent Technologies

Agilent J&W DB-17ms and DB-XLB capillary columns are the ideal choice for low level pesticide determination. The DB-17ms and DB-XLB columns offer excellent separation of the CLP pesticides for primary and confirmational analysis. [3] Both columns feature low bleed which improves the signal-to-noise ratio and detection limits, along with higher upper temperature limits which allow quicker run times. In this example, a gas chromatographic analysis of an expanded list of 27 target CLP pesticides and two surrogate standards was accomplished in under eight minutes using hydrogen carrier and flow programming.

A common technique in decreasing analysis time is the use of hydrogen as the carrier gas. This allows shorter retention times with minimal or no loss in resolution. Agilent's GC Method Translation software [4] can be used to convert the chromatographic conditions for helium carrier to hydrogen carrier gas. This software is available for free download from the internet. The speed of analysis can be further increased by employing flow programming to help elute highly retained peaks more rapidly. Flow programming is controlled by electronic pneumatic control (EPC) to eliminate pressure reproducibility problems.

This analysis is typically done in dual column mode for simultaneous primary and confirmation analysis using a quartz y-splitter to connect the columns and retention gap. In this application, an Agilent capillary flow technology (CFT) 2-way splitter without makeup gas (Agilent p/n G3181B) was employed [5]. A diagram of the splitter and column setup is shown in Figure 1. Installation of the retention gap and columns into the CFT splitter module uses ferrules and internal nuts similar to a typical column installation. Because the column connections are individually connected into the splitter, inlet and column maintenance can be done independent of the other connections in the flow path, minimizing instrument downtime.

Experimental

An Agilent 7890A GC system with µECD equipped with an Agilent 7683B automatic liquid sampler was used for this experiment. Table 1 lists the chromatographic conditions used for this analysis. Table 2 lists flow path consumable supplies used in this experiment.

Table 1. Chromatographic Conditions for CLP Pesticide Standard Analysis

GC:	Agilent 7890A GC system with µECD
Sampler:	Agilent 7683B automatic liquid sampler, 5.0 µL syringe (Agilent p/n 5181-1273) 0.5 µL splitless injection
Carrier:	Hydrogen 83.8 cm/s, Ramped flow 4.25 mL/min hold 4.6 min; 100 mL/min^2 to 8.5 mL/min hold 1.6 min; 100 mL/min^2 to 9.5 mL/min
Inlet:	Pulsed splitless; 250 °C, Pulse pressure 40 psi until 0.2 min Purge flow 30 mL/min at 1 min, gas saver 50 mL/min after 2 min
Inlet Liner:	Deactivated dual taper direct connect (Agilent p/n G1544-80700)
Retention Gap:	1 m 0.32 mm id Hi-Temp Deactivated fused silica tubing (Agilent p/n 160-2855-5)
Column 1:	Agilent J&W DB-17ms 30 m × 0.32 mm, 0.25 µm (Agilent p/n 123-4732)
Column 2:	Agilent J&W DB-XLB 30 m × 0.32 mm, 0.5 µm (Agilent p/n 123-1236)
Oven:	150 °C (0.1 min) to 230 °C (75 °C/min); 20 °C/min to 235 °C; 1 °C/min to 237 °C (1.25 min); 10 °C/min to 250 °C (0.4 min); 120 °C/min to 330 °C hold 1 min.
Detection:	µECD 330 °C, N_2 makeup; constant column + makeup = 62.01 mL/min

Table 2 Flow Path Supplies

CFT device:	2-way splitter accessory without makeup gas (Agilent p/n G3181B) Alternative: Deactivated quartz y-splitter (Agilent p/n 5181-3398)
CFT fittings:	Internal nut (Agilent p/n G2855-20530) Swaging nut (Agilent p/n G2855-20555)
CFT ferrules:	SilTite ferrules, 0.32 mm id (Agilent p/n 5188-5362)
Vials:	Amber crimp cap glass vials (Agilent p/n 5183-4496)
Vial caps:	Crimp caps (Agilent p/n 5282-1210)
Vial inserts:	100 µL glass/polymer feet (Agilent p/n 5181-8872)
Syringe:	5 µL (Agilent p/n 5181-1273)
Septum:	Advanced Green (Agilent p/n 5183-4759)
Inlet seal:	Gold plated inlet seal (Agilent p/n 5188-5367)
Inlet liners:	Deactivated dual taper direct connect (Agilent p/n G1544-80700)
Ferrules:	0.5 mm id short; 85/15 Vespel/graphite (Agilent p/n 5062-3514)
20x magnifier:	20x Magnifier loop (Agilent p/n 430-1020)

Sample Preparation

Neat standards of Hexachlorobenzene, Telodrin, o,p'-DDD, o,p'-DDE, o,p'-DDT, Kepone, and Mirex were acquired from ChemService, Inc (West Chester, PA). Individual pesticides solutions were prepared in 2,2,4-trimethylpentane. Two CLP organochlorine pesticide standard mixes were purchased from Accustandard (New Haven, CT). A working standard was prepared by mixing the individual pesticide solutions with the CLP standard mixes and diluting with 2,2,4-trimethylpentane to a 5ng/mL concentration. 2,2,4-Trimethylpentane used was JT Baker Ultra Resi grade purchased thorough VWR International, West Chester, PA 19380-USA. 2,2,4-Trimethylpentane was used as a reagent blank and syringe wash solvent.

Discussion of Results

In this application brief, 27 organochlorine pesticides and two surrogate standards were resolved using an Agilent J&W DB-17ms 30 m × 0.32 mm, 0.25 µm (p/n 123-4732) for primary analysis and confirmatory analysis on the Agilent J&W DB-XLB 0.32 mm id column (p/n 123-1236). An example chromatogram of the dual column analysis for a 1.25-pg on column loading for the low level target CLP pesticides is shown in Figure 2.

Using hydrogen carrier and flow programming, separation for the 27 organochlorine analytes was achieved in under 8 minutes. This analysis yields a time savings of more than 10 minutes over typical CLP pesticides analyses while maintaining resolution and peak shape. Expanded view chromatograms of the 1.25-pg on column loading for the low level target CLP pesticides on each column is shown in Figures 3 and 4.

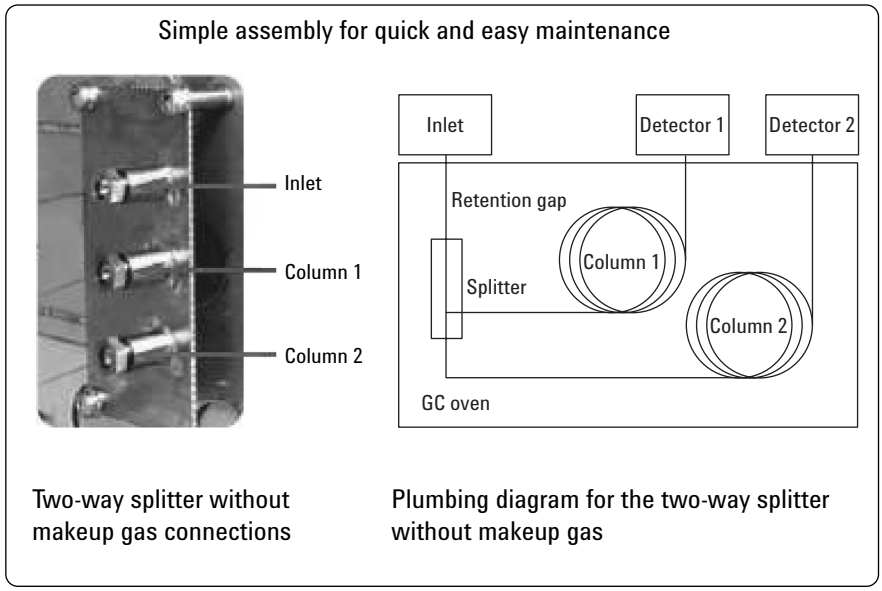

Figure 1. Agilent capillary flow technology 2-way splitter without makeup gas (p/n G3181B) and diagram of instrument setup of simultaneous confirmation from a single injection onto both the primary and confirmation columns.

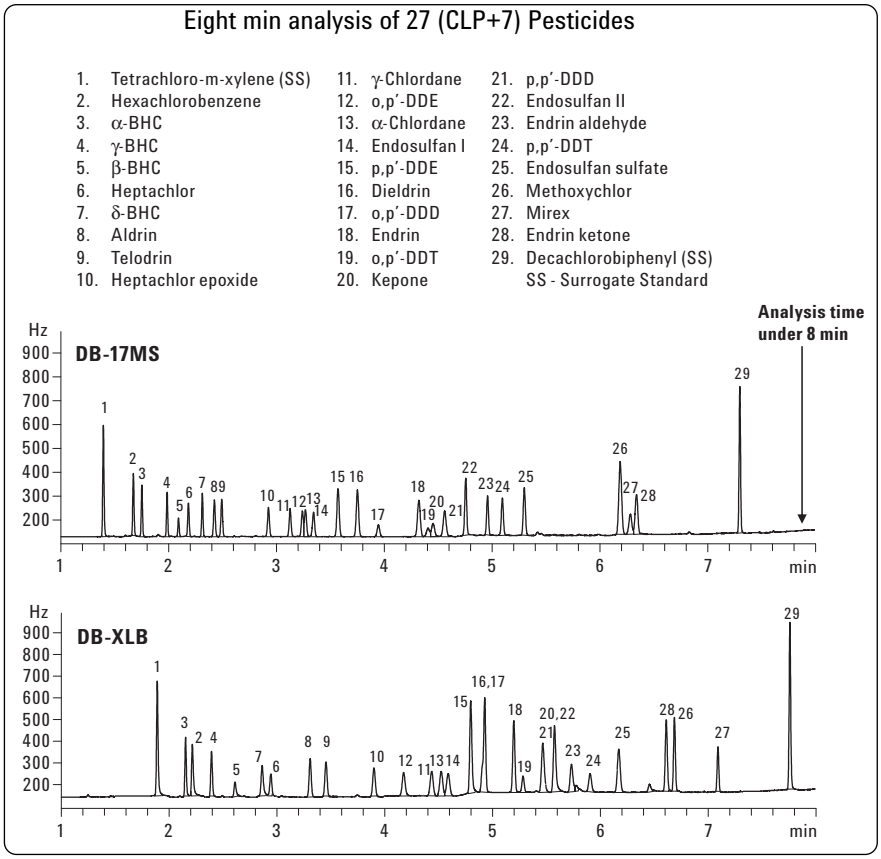

Figure 2. Chromatogram of the 1.25 pg on column loading of low level target CLP pesticide standard solution on a dual column analysis using Agilent J&W DB-17ms and DB-XLB capillary GC columns. Chromatographic conditions are listed in Table 1.

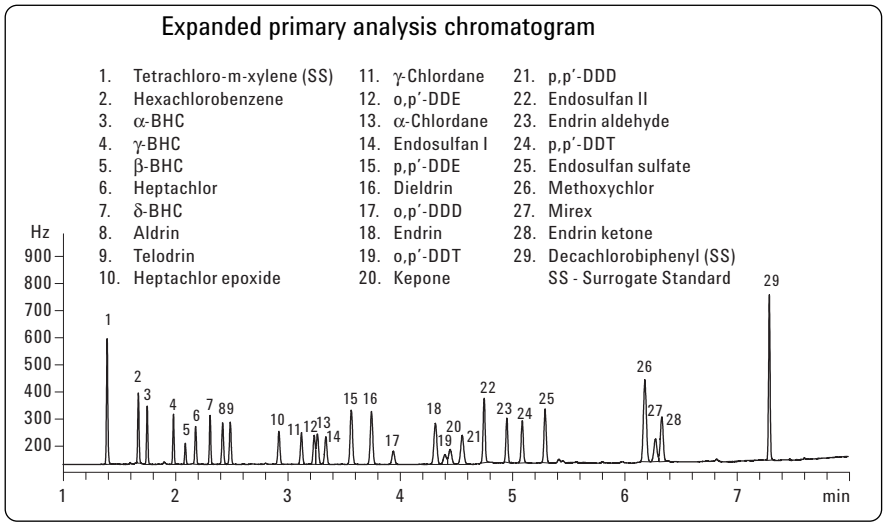

Figure 3. Chromatogram of the 1.25 pg on column loading of low level target CLP pesticide standard solution on an Agilent J&W DB-17ms 30 m × 0.32 mm × 0.25 µm capillary GC column (p/n 123-4732). Chromatographic conditions are listed in Table 1.

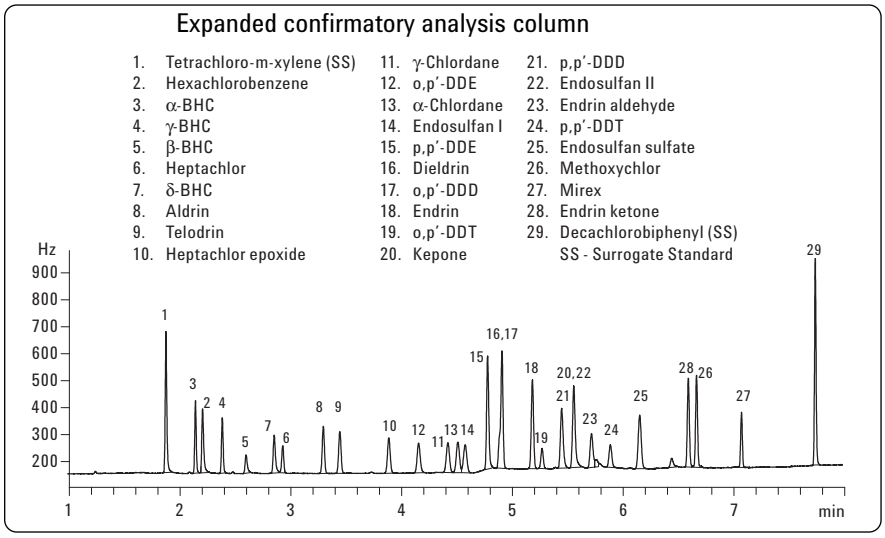

Figure 4. Chromatogram of the 1.25 pg on column loading of low level target CLP pesticide standard solution on an Agilent J&W DB-XLB 30 m × 0.32 mm × 0.5 µm capillary GC column (p/n 123-1236). Chromatographic conditions are listed in Table 1.

Conclusions

This application successfully demonstrates a rapid CLP pesticides method using Agilent J&W DB-17ms and DB-XLB 0.32 mm id medium bore capillary columns. By using hydrogen as the carrier gas and employing flow programming, complete primary and confirmatory analysis of 27 organochlorine pesticides was accomplished in less than eight minutes. This allows for greater sample throughput in less time thereby increasing laboratory productivity.

References

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2. K. Lynam and W. Long, "Contract Laboratory (CLP) Pesticide Analysis with 0.18 mm ID High Efficiency GC Columns Utilizing Helium Carrier Gas," Agilent publication 5989-7818EN, January 7, 2008
3. Doris Smith and Ken Lynam, "A 0.32 mm ID Capillary Column Approach to Contract Laboratory Program (CLP) Pesticides Analysis," Agilent Technologies publication 5990-4069EN, May 21, 2009
4. To download Agilent Method Translation software, please visit the link below: http://www.chem.agilent.com/cag/servsup/usersoft/files/GCTS.htm
5. Agilent G3181B Two-Way Splitter Kit Without Makeup Gas Installation and Operation Guide: http://www.chem.agilent.com/Library/usermanuals/Public/G3181-90120_045611.pdf

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