Organochlorine pesticide analysis using an Agilent Intuvo 9000 Dual ECD GC system

Technology advantage: GC Flow Chip modularity for dual column analysis

Introduction

Organochlorine pesticides are often measured following protocols from the Contract Laboratory Program (CLP) of the United States Environmental Protection Agency[1]. CLP is designed to support laboratories identifying and quantifying environmental contamination. This is important when determining what remediation steps must be taken to clean up a contaminated site. Organochlorine pesticides are common targets because they can linger in soil or sediment, and affect water sources. Laboratories following CLP protocols (EPA SW-846 Test Method 8081B[2]) for organochlorine pesticides analysis require robust, high-throughput methods of analysis, while minimizing the analytical costs. The method, written with general and nonexclusive parameters, allows the analyst to choose consumables and calibration protocols, but specifies dual column confirmation with dual electron capture detection (ECD). It also specifies inertness, measured by degradation of endrin and DDT.

This configuration is easily supported on the Agilent Intuvo 9000 GC system with additional benefits. To allow for identification and confirmation in a single run while meeting degradation limits, the modular Inert Flow Chips allow for easy configuration of two columns. Given the nature of potential samples (water to soil and sediment), the Guard Chip can simplify maintenance. With regular replacement, the Guard Chip protects downstream components from matrix, eliminating the need to trim the column. This leaves retention times unchanged, and results in less downtime.

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Experimental

An Intuvo GC system was equipped with dual ECD detectors and configured with an inlet splitter. Agilent CLP1 and CLP2 columns (123-8336-INT and 123-8232-INT) were used. A custom CLP pesticide standard diluted to 2 ppm was used to demonstrate chromatographic performance. A standard containing endrin and DDT was used to verify the inertness of the system. Table 1 gives the instrument conditions.

To meet various needs, two methods were developed. The first method provides excess resolution in a 30-minute run time. The second method is targeted for speed, with an 11-minute run time, but may not meet the resolution requirements, depending on the analytes chosen.

Results and discussion

A standard containing 20 chlorinated pesticides (Table 2) was evaluated with both the 30-minute and 11-minute methods. The 30-minute method provided excellent resolution for the 20 pesticides evaluated. This method yielded optimal resolution, and allowed for a greater number of analytes to be included (Figure 1).

GC Conditions

Table 1. Agilent Intuvo 9000 GC system parameters.

Parameter	30-Minute method	11-Minute method
Inlet	240 °C	
Pulsed splitless	60 psi for 0.3 minutes, 75 mL/min at 0.5 minutes	
Column 1	Agilent DB-CLP 1 (30 m × 320 µm, 0.25 µm)	
Column 2	Agilent DB-CLP2 (30 m × 320 µm, 0.50 µm)	
Column flow rate	3 mL/min	3 mL/min
Oven	100 °C (1 minute), 10 °C/min to 225 °C (8 minutes), 30 °C/min to 300 °C (5.25 minutes)	120 °C (0.2 minutes), 45 °C/min to 250 °C, 18 °C/min to 300 °C (5 minutes)
Guard Chip	Track oven	
Bus	260 °C	
ECDs	300 °C 30 mL/min makeup flow	

Table 2. The 20 analytes of interest, containing many chlorinated pesticides.

	Analyte		Analyte
1	α-BHC	11	4,4'-DDE
2	γ-BHC	12	Dieldrin
3	β-BHC	13	Endrin
4	Heptachlor	14	4,4-DDD
5	δ-BHC	15	Endosulfan II
6	Aldrin	16	4,4'-DDT
7	Helptachlor epoxide	17	Endrin aldehyde
8	γ-Chlordane	18	Endosulfan sulfate
9	α-Chlordane	19	Methoxychlor
10	Endosulfan I	20	Endrin ketone

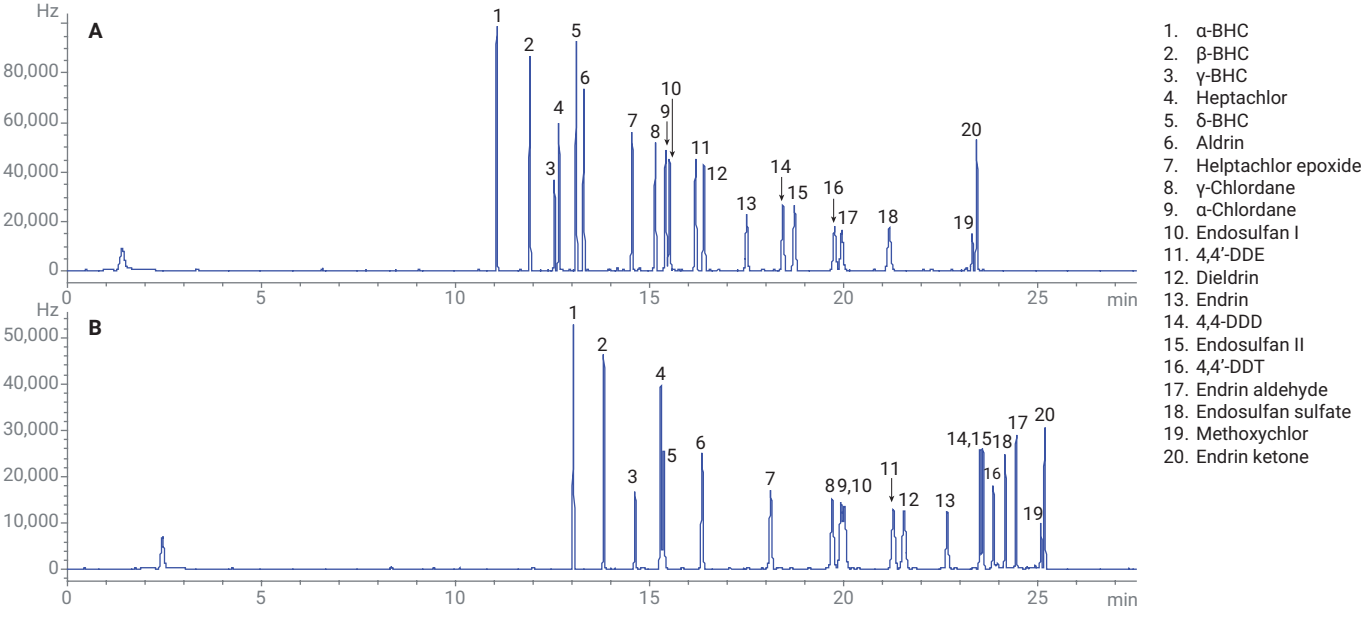

Figure 1. The Agilent DB-CLP1 (A) and DB-CLP2 (B) columns show excellent separation for the 20 analytes of interest.

The 30-minute method yielded a 7 % endrin breakdown and a 2 % DDT breakdown. EPA 8081B specifies that degradation should not exceed 15 %, if it does, maintenance or corrective action must occur.

The 11-minute method also yielded excellent resolution for the 20 pesticides evaluated, but had a lower peak capacity as a result of the increased temperature programming rate. However, it did yield separation of the 20 pesticides in under nine minutes (Figure 2). The run time could be further shortened by increasing the column flow rate to 6 mL/min.

This method yielded 8 % endrin breakdown and 3 % DDT breakdown. A robustness test was also completed with this method by monitoring endrin and DDT breakdown over multiple injections. The endrin breakdown reached 15 % (method limit) after 100 injections. This was remedied by replacing the inlet liner; the endrin breakdown then returned to <5 %.

Conclusion

The Agilent Intuvo 9000 GC system provides a simple and straightforward way to evaluate chlorinated pesticides according to CLP guidelines. Adequate resolution for the 20 target analytes was achieved with both a 30-minute and 11-minute method. Endrin and DDT yielded acceptable degradation levels, demonstrating system inertness. Endrin levels only reached method limits after 100 injections, when inlet maintenance was performed (inlet liner and septum replaced), bringing the degradation back within method limits.

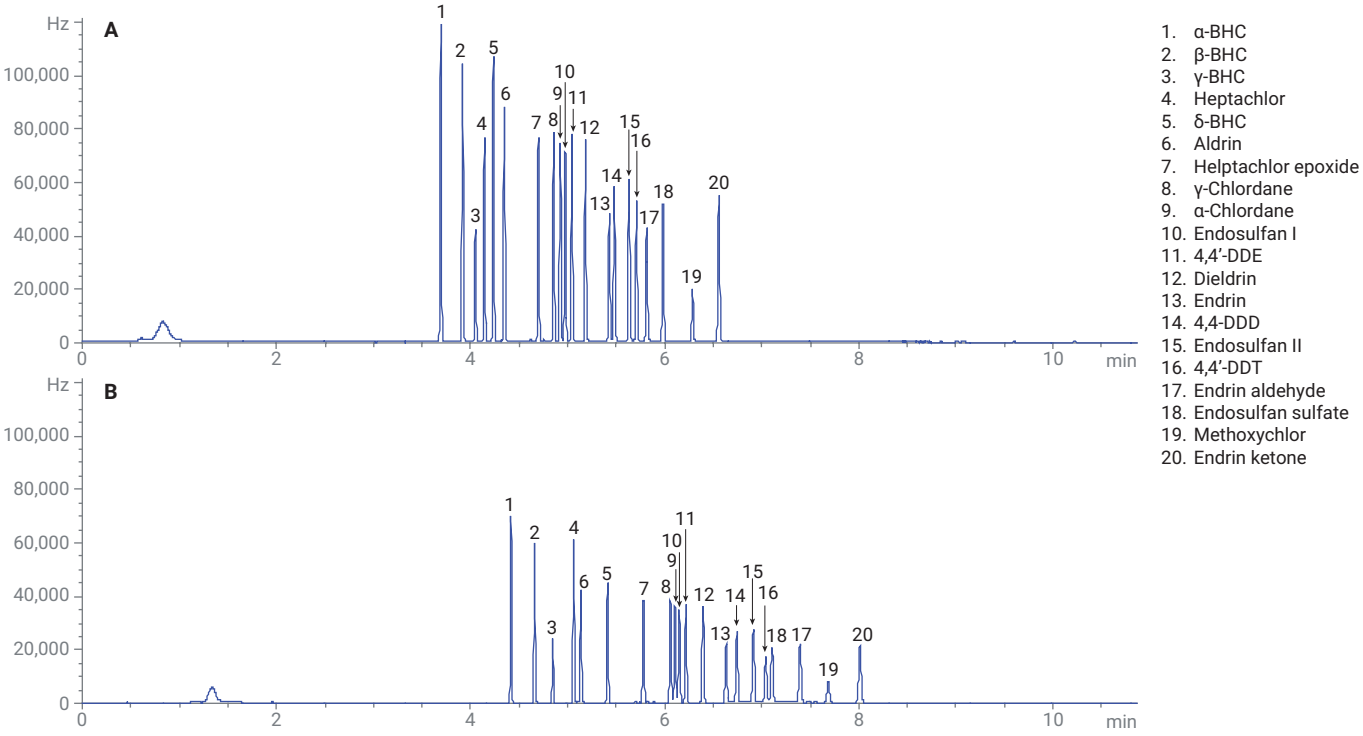

Figure 2. The Agilent DB-CLP1 (A) and DB-CLP2 (B) columns achieve excellent resolution in under nine minutes.

References

1. Introduction to the Contract Laboratory Program, *EPA 540-R-07-02*, January **2007**.
2. Organochlorine Pesticides by Gas Chromatography, *EPA Method 8081B*, February **2007**.

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