

Application Note No. 065

The Analysis of Pyrethroids using the AT-Column Concentrating Technique

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- *Copes well with the difficult mixture of labile and sticky compounds*
- *No or very little optimisation is required*

Instrumentation

- ATAS Optic 2-200 programmable injector
- ATAS AT-Column kit
- HP5890 with HP5971

Sample analysed

Cyfluthrin, α -Cypermethrin and Flumethrin in dichloromethane

Principles

- Sample is injected under AT-Column conditions
- An equilibrium is formed between the solvent vapour pressure and carrier gas pressure keeping the solvent in the liner
- Solvent is vented, analytes are concentrated and transferred onto the head of the capillary column
- GC oven temperature program starts

Chromatogram

Abundance

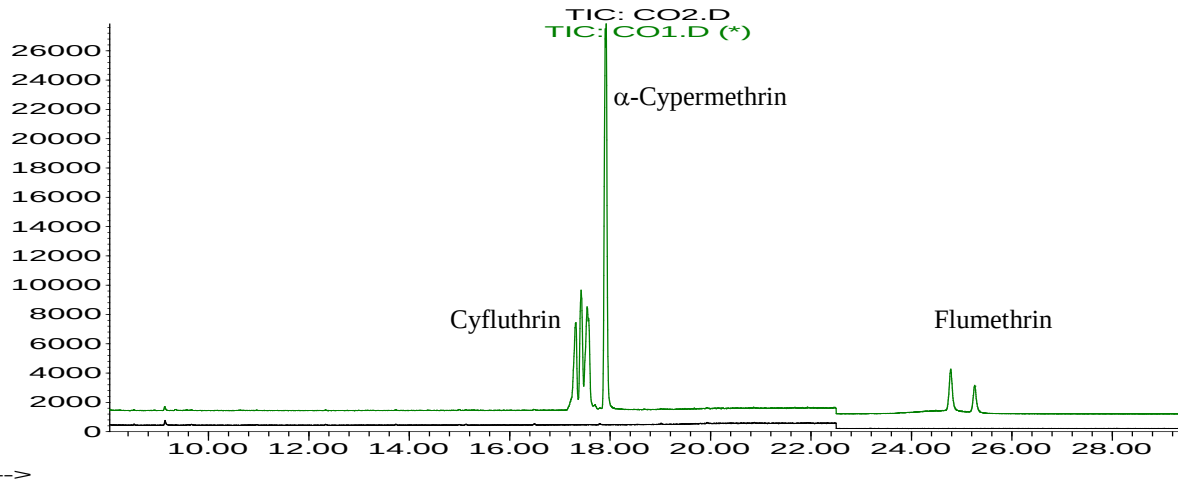


Figure: Upper trace: first injection of 100 μ L of 200 μ g/L pyrethroids in DCM; lower trace: second injection of 100 μ L DCM to check for carryover

Results

- RSDs: α -Cypermethrin 2.95%; Flumethrin 5.35% (standardised to Cyfluthrin)
- Carryover: None detected for any compound (see Figure)
- LOD: The required detection limit of 4 μ g/L was easily reached for all compounds

We would like to thank Anthony Gravell from the Environment Agency NLS, Llanelli, Wales, for his kind permission to publish this information and Alan Tuckley from Agilent Technologies for providing the column.

Appendix

Optic Conditions:

- Liner: AT-Column
- Mode: Large Volume
- Injection volume: 100-130 µL
- Gas Flows: Split: 50 ml/min
Vent: 120 ml/min
- Initial temperature: 43 °C
- Vent time: Auto
- Ramp rate: 1 °C/s
- Final temperature: 300 °C
- Splitopen time: 0:00 m:s
- Purge pressure: 3.63 psi
- Transfer pressure: 8.00 psi
- Transfer time: 1:30 m:s
- Initial pressure: 8.00 psi
- Final pressure: 22 psi
- Solvent threshold: 15

GC-MS conditions:

- Column: HP5-MS 30m x 0.25mm i.d. x 0.25 µm film
- Initial Temperature: 56 °C hold 1 min
- Ramp 1: 25 °C/min to 195 °C hold 0 mins
- Ramp 2: 7 °C/min to 280 °C hold 0 mins
- Ramp 3: 25 °C/min to 300 °C hold 10 mins
- MSD transfer line: 280 °C
- MSD SIM mode