

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

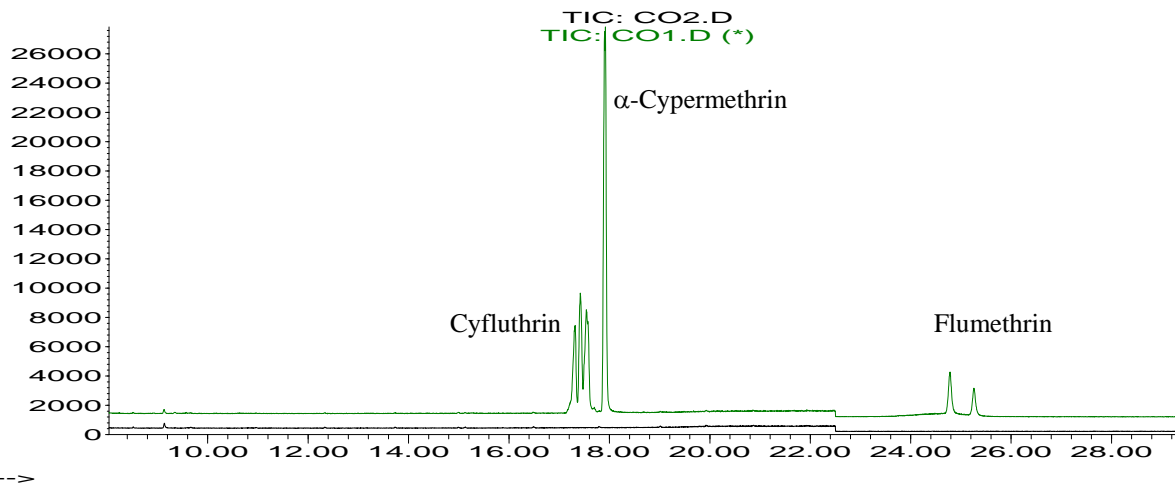


Figure: Upper trace: first injection of 100 μ L of 200 μ g/L pyrethroids in DCM; lower trace: second injection of 100 μ L DCM to check for carryover

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

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Optic Conditions:

- Liner: AT-Column
- Mode: Large Volume
- Injection volume: 100-130 μ L
- Gas Flows: Split: 50 ml/min
Vent: 120 ml/min
- Initial temperature: 43 $^{\circ}$ C
- Vent time: Auto
- Ramp rate: 1 $^{\circ}$ C/s
- Final temperature: 300 $^{\circ}$ 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 μ m film
- Initial Temperature: 56 $^{\circ}$ C hold 1 min
- Ramp 1: 25 $^{\circ}$ C/min to 195 $^{\circ}$ C hold 0 mins
- Ramp 2: 7 $^{\circ}$ C/min to 280 $^{\circ}$ C hold 0 mins
- Ramp 3: 25 $^{\circ}$ C/min to 300 $^{\circ}$ C hold 10 mins
- MSD transfer line: 280 $^{\circ}$ C
- MSD SIM mode