Pyrethroid analysis

Introduction

Pyrethroids are potent insecticides widely used in agriculture, disease control and household products. Nearly all domestic fly aerosols use pyrethroids as the active ingredient whether these are surface or airborne sprays. Pyrethroids act by interfering with the insect's nervous system and, in high concentrations, affect humans in a similar way.

Synthetic pyrethroids are also known as endocrine disrupters. Other toxicological properties include: liver damage when subjected to long term exposure, allergic reactions and asthmatic attacks. The USEPA has found that many pyrethroids may also be carcinogenic. For this reason, the levels of synthetic pyrethroids in foods and the environment are always under close scrutiny.

Table 1. Pyrethroid structures

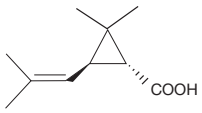
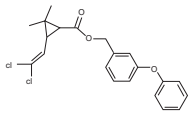
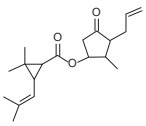
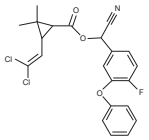
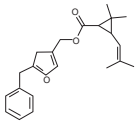
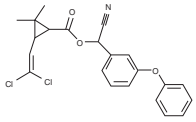
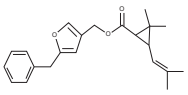
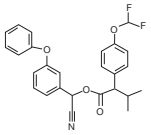
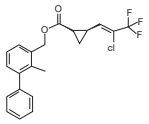
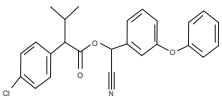
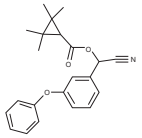
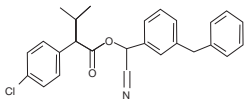
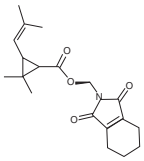
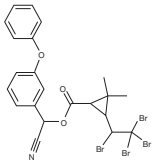
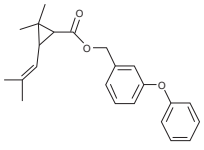
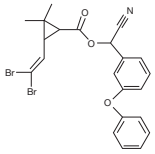
Pyrethroid	Compound Structure	Pyrethroid	Compound Structure
1. Natural Pyrethrums		9. Permethrin	
2. Allenthrin		10. Cyfluthrin	
3. Bioresmethrin		11. Cypermethrin	
4. Resmethrin		12. Flucythrinate	
5. Bifenthrin		13. Fenvalerate	
6. Fenprorathrin		14. Esfenvalerate	
7. Tetramethrin		15. Tralomethrin	
8. Sumithrin		16. Deltamethrin	

Table 1 shows the structures of the various Pyrethroids analyzed.

Analysis by GC can be complex because many of the pyrethroids can have up to 8 optical isomers thus more than one peak can represent the same pyrethroid. Therefore, overlapping of signals can be a problem (e.g. Cypermethrin with 3 chiral centers can have up to 8 optical isomers, which include 4 diastereoisomers) (Figure 3).

These identification problems have been made a thing of the past with the 5% phenyl BPX5, 35% phenyl BPX35, and 50% phenyl BPX50 columns. Each column gives excellent separation of the various isomers of the 16 components (Table 1) in less than 33 minutes, allowing a fast turnaround time of samples. With an upper temperature limit of 360°C on all three columns, high boiling point contaminants can be "baked out" without damaging the stationary phase. This ensures that contaminants won't interfere with subsequent analyses. The bleed profiles at 300°C are excellent and the exceptional peak shape of each pyrethroid indicates a high degree of inertness on all three columns.

BPX5

5% phenyl (equiv) polysilphenylene-siloxane

BPX5 replaces		
DB-5	Ultra-2	MDN-5S
DB-5MS	HP-5	CP-Sil 8CB
DB-5.625	HP-5MS	Rtx-5Sil MS
XTI-5	HP5-TA	AT-5
Rtx-5ms	SPB-5	CP-Sil 8CB M

Figure 1 shows the separation of 16 pyrethroids on BPX5. Note the superb bleed profile at 300°C. BPX5 gives separation of the synthetic pyrethroids. Breakdown in the injection port of the thermally labile pyrethroids to their parent compounds such as tralomethrin (16) to deltamethrin (17) and esfenvalerate (14) to fenvalerate (15) are the only coelutions seen.

Column part number	054101		
Phase	BPX5	Carrier gas	He, 9.5 psi
Column	30m x 0.25 mm x 0.25 µm	Carrier gas flow	0.9 mL/min
Sample	100 ppm in dichloromethane	Constant flow	On
Initial temperature	110°C, 1 min	Average linear velocity	35 cm/sec at 110°C
Rate 1	25°C/min to 150°C	Injection mode	Split (50:1)
Rate 2	12°C/min to 260°C	Injection volume	1 mL
Rate 3	15°C/min to 310°C	Injection temperature	250°C
Final temperature	310°C, 6 min	Autosampler	No
Detector	MS	Liner type	4 mm ID Single Taper

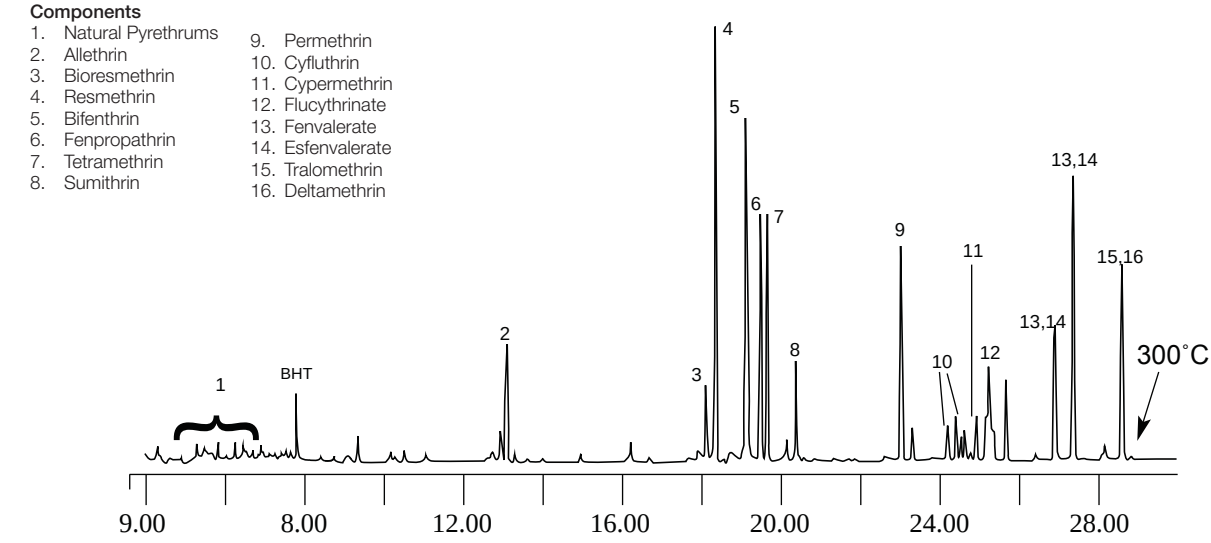


Figure 1

BPX35

35% phenyl (equiv) polysilphenylene-siloxane

BPX35 replaces		
DB-35	Rtx-35ms	SPB-35
DB-35MS	HP-35	MDN-35
Rtx-35	HP-35MS	AT-35

Figure 2 shows excellent separation of the 16 synthetic pyrethroids on BPX35. The bleed at 300°C is excellent and the signal to noise in full scan mode is 60:1 for 10 ng on column.

Figure 3 shows a chromatogram of the synthetic pyrethroid cypermethrin. Note the 4 peaks seen here representing the 4 diastereoisomers of cypermethrin. The structure of cypermethrin shown here shows the 3 chiral centers (*) of cypermethrin.

Column part number	054701		
Phase	BPX35	Constant flow	On
Column	30m x 0.25mm x 0.25 µm	Average linear velocity	35 cm/sec at 50°C
Sample	10 ppm in methanol	Injection mode	Splitless
Initial temperature	50°C, 1 min	Purge on time	0.5 min
Rate 1	30°C/min to 200°C	Purge on (split) vent	60 mL/min
Rate 2	4°C/min to 300°C	Injection volume	1 µL
Final temperature	300°C, 5 min	Injection temperature	250°C
Detector	MS	Autosampler	No
Carrier gas	He, 6.5 psi	Liner type	4 mm ID Double Taper
Carrier gas flow	0.9 mL/min		

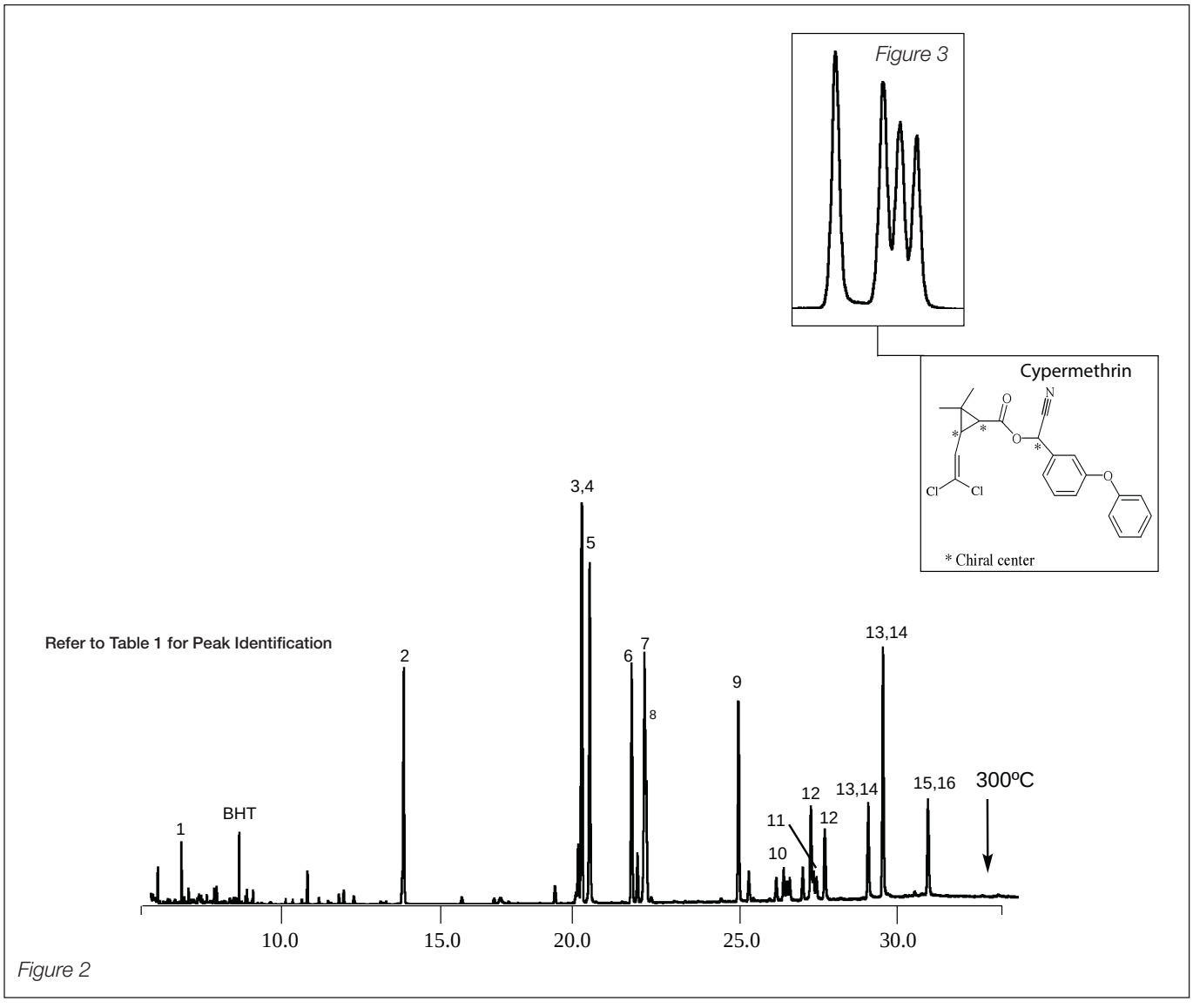


Figure 2

BPX50

50% phenyl (equiv) polysilphenylene-siloxane

BPX50 replaces		
OV-17	DB-17ht	HP-17
SP-2250	Rtx-50	AT-50
DB-17	SPB-50	007-17
DB-17MS	HP-50+	

Figure 4 shows the separation of the synthetic pyrethroids as run on BPX50. Note the run time on this chromatogram is 16 minutes for complete elution of the 16 pyrethroids. This allows analytical laboratories to have higher sample throughput.

Column part number	054751		
Phase	BPX50	Constant flow	On
Column	30 m x 0.25 mm x 0.25 µm	Average linear velocity	35 cm/sec at 50°C
Sample	10 ppm in methanol	Injection mode	Splitless
Initial temperature	50°C, 1 min	Purge on time	0.5 min
Rate 1	30°C/min to 200°C	Purge on (split) vent	60 mL/min
Rate 2	4°C/min to 300°C	Injection volume	1 µL
Final temperature	300°C, 5 min	Injection temperature	250°C
Detector	MS	Autosampler	No
Carrier gas	He, 6.5 psi	Liner type	4 mm ID Double Taper
Carrier gas flow	0.9 mL/min		

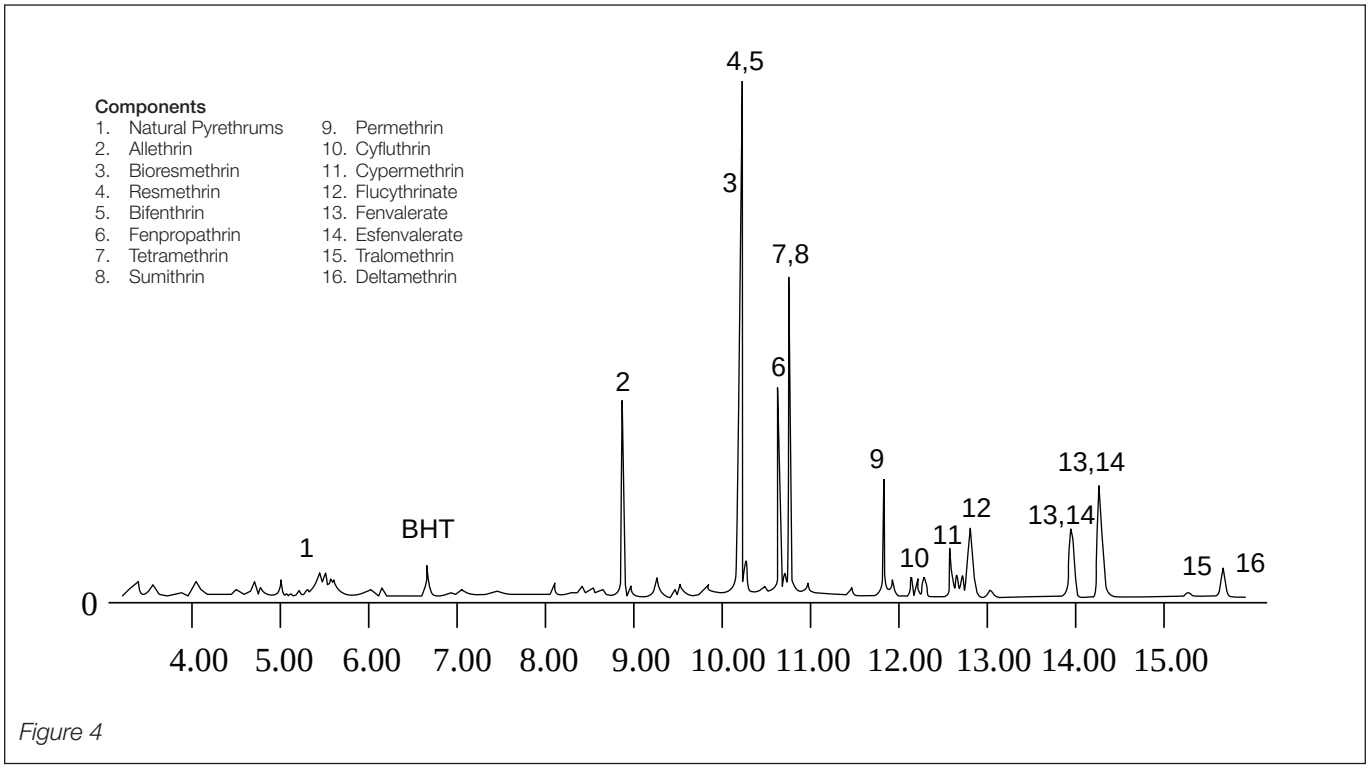


Figure 4

Summary

BPX5, BPX35 and BPX50 columns show unparalleled performance for the separation of synthetic pyrethroids. These columns can be conditioned at the end of each analysis to remove any high boiling point contaminants without any degradation to the stationary phase and give excellent signal-to-noise with very little on-column breakdown. BPX5, BPX35 and BPX50 are the columns of choice for all pyrethroid analyses.

Information and support

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Specifications are subject to change without notice.