TECHNICAL ARTICLE

MARKET FOCUSED - Environmental
PYRETHROID ANALYSIS

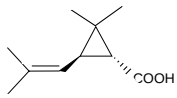
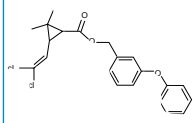
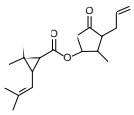
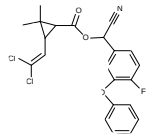
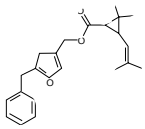
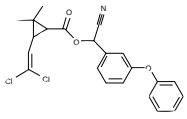
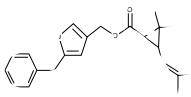
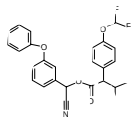
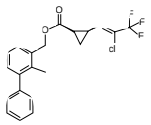
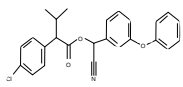
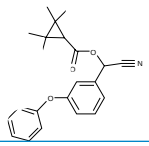
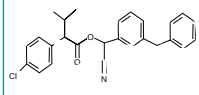
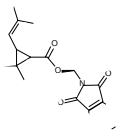
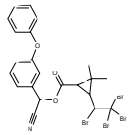
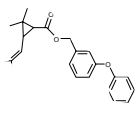
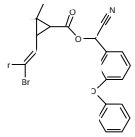
Pyrethroids are potent insecticides widely used in agriculture, disease control and household products. Nearly all domestic fly aerosols use pyrethroids as the active ingredient whether these are surface or airborne sprays. Pyrethroids act by interfering with the insect's nervous system and, in high 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 of the pyrethroids may also be carcinogenic. For this reason, the levels of synthetic pyrethroids in foods and the environment are always under close scrutiny.

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 SGE's 5% phenyl BPX5, 35% phenyl BPX35, and 50% phenyl BPX50 columns. Each column gives superb 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.

Table 1 shows the structures of the various Pyrethroids analyzed

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	

BPX5
5% phenyl (equiv) polysilphenylene-siloxane

BPX5 Replaces

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

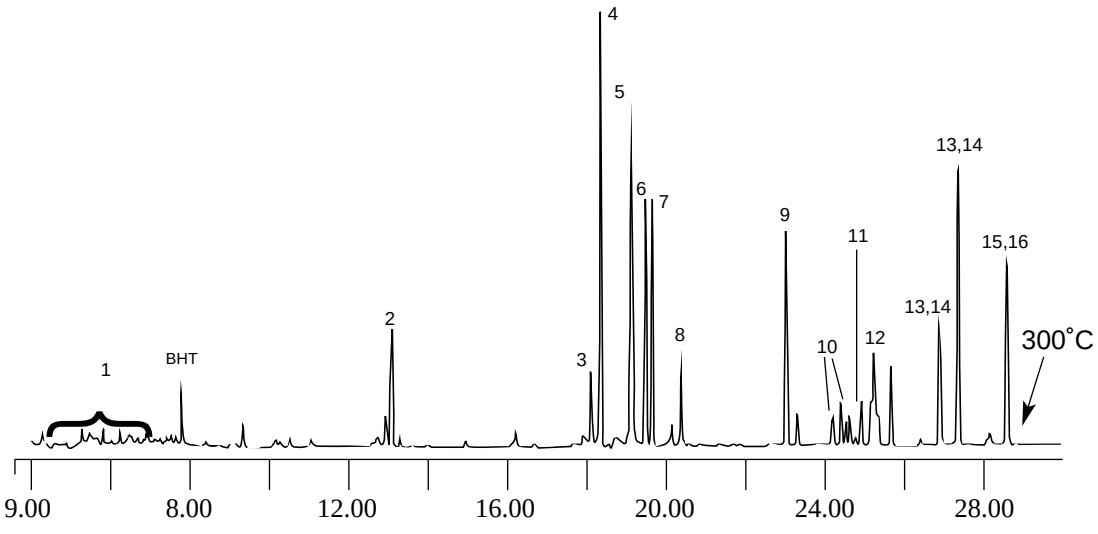
Figure 1 shows the separation of 16 pyrethroids on BPX5. Note the superb bleed profile at 300°C. BPX5 gives excellent separation of the synthetic pyrethroids. Breakdown in the injection port of the thermally labile pyrethroids to there parent compounds such tralomethrin (16) to deltamethrin (17) and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

Figure 1

Phase:	**BPX5, 0.25µm film**
Sample:	**100ppm in Dichloromethane**
Column:	**30m x 0.25 mm ID**
Initial Temp:	110°C, 1 min.
Rate 1:	25°C/min to 150°C,
Rate 2:	12°C/min to 260°C
Rate 3:	15°C/min to 310°C
Final Temp:	310°C, 6 min.
Detector Type:	Mass Spectrometer
Carrier Gas:	He, 9.5 psi
Carrier Gas Flow:	0.9 mL/min.
Constant Flow:	On
Average Linear Velocity:	35 cm/sec at 110°C
Injection Mode:	Split
Split Ratio:	50:1
Injection Volume:	1 mL
Injection Temperature:	250°C
Autosampler:	No
Liner Type:	4 mm ID Single Taper Liner
Liner Part Number:	**092017**
Column Part Number:	**054101**

COMPONENTS

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



BPX35
35% phenyl (equiv) polysilphenylene-siloxane

BPX35

Replaces

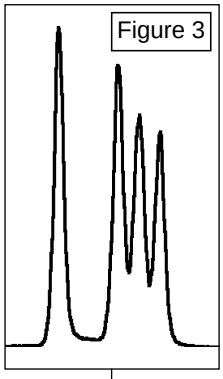
DB-35
DB-35MS
Rtx-35
Rtx-35ms
HP-35
HP-35MS
SPB-35
MDN-35
AT-35

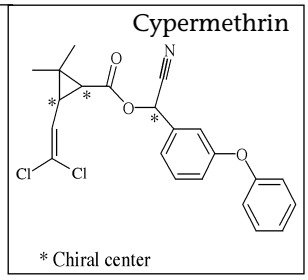
Figure 2 shows excellent separation of the 16 synthetic pyrethroids on BPX35. The bleed at 300°C is excellent and the signal to noise in full scan mode is 60:1 for 10ng on column.

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.

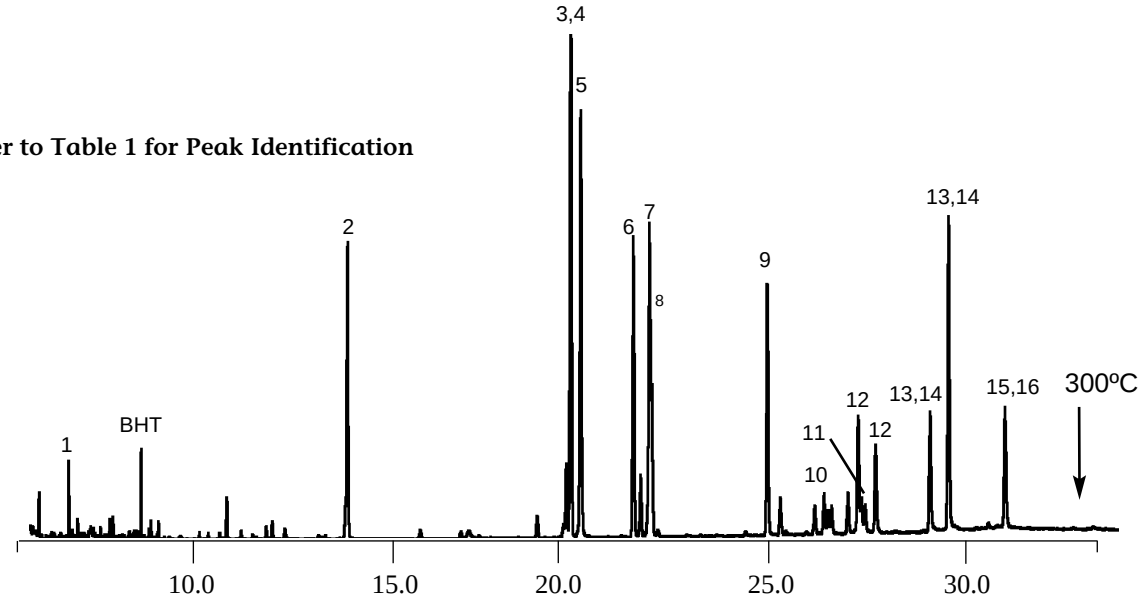
Figure 2 & Figure 3

Phase:	**BPX35 0.25µm film**
16 Pyrethroids:	**10ppm in methanol**
Column:	**30m x 0.25mm ID**
Initial Temp:	50°C, 1 min
Rate 1:	30°C/min to 200°C
Rate 2:	4°C/min to 300°C
Final Temp:	300°C for 5 min
Detector Type:	Mass Spectrometer
Carrier Gas:	He, 6.5 psi
Carrier Gas Flow:	0.9mL/min
Constant Flow:	On
Average Linear Velocity:	35cm/sec at 50°C
Injection Mode:	Spiltless
Purge on Time:	0.5min
Purge on (Split) Vent:	60mL/min
Injection Volume:	1µL
Injection temperature:	250°C
Autosample:	No
Liner Type:	4mm ID Double Taper Liner
Liner Part Number:	**092018**
Column part Number:	**054701**





Refer to Table 1 for Peak Identification



BPX50

50% phenyl (equiv) polysilphenylene-siloxane

Figure 4 show 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.

BPX50 Replaces

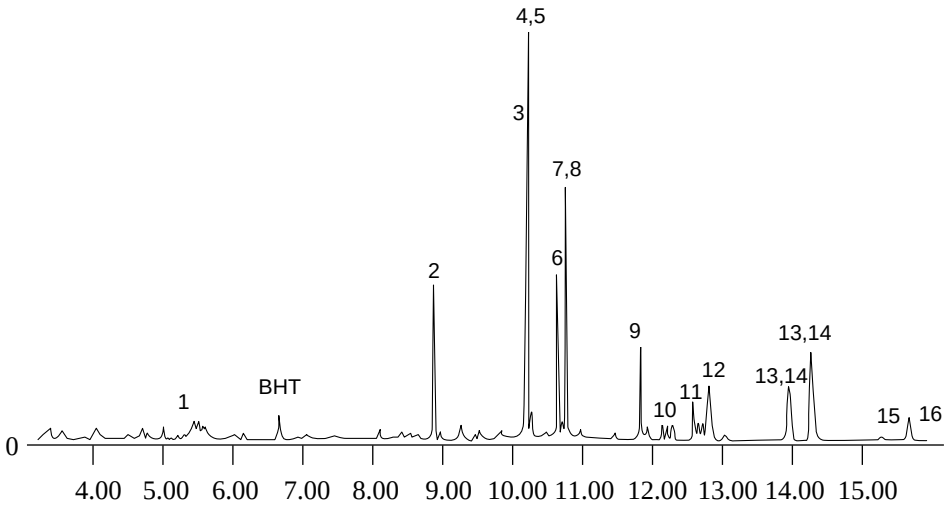
OV-17
SP-2250
DB-17
DB-17MS
DB-17ht
Rtx-50
SPB-50
HP-50+
HP-17
AT-50
007-17

Figure 4

Phase:	**BPX50 0.25µm film**
16 Pyrethroids:	**10ppm in methanol**
Column:	**30m x 0.25mm ID**
Initial Temp:	50°C, 1 min
Rate 1:	30°C/min to 200°C
Rate 2:	4°C/min to 300°C
Final Temp:	300°C for 5 min
Detector Type:	Mass Spectrometer
Carrier Gas:	He, 6.5psi
Carrier Gas Flow:	0.9mL/min
Constant Flow:	On
Average Linear Velocity:	35cm/sec at 50°C
Injection Mode:	Splitless
Purge On Time:	0.5min
Purge on (Split) Vent:	60mL/min
Injection Volume:	1µL
Injection Temperature:	250°C
Autosampler:	No
Liner Type:	4mm ID Double Taper Liner
Liner Part Number:	**092018**
Column Part Number:	**054751**

COMPONENTS

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



ORDERING INFORMATION

	ID(mm) (mm)	Length (m)	Film Thickness (µm)	Temperature Limits (°C)	Part No.
BPX5	0.10	10	0.10	-40 to 360/370	054099
	0.22	12	0.25	-40 to 360/370	054112
		25	0.25	-40 to 360/370	054113
		30	0.25	-40 to 360/370	054142
		50	0.25	-40 to 360/370	054114
	0.25	15	0.25	-40 to 360/370	054100
		15	1.00	-40 to 360/370	054121
		30	0.25	-40 to 360/370	054101
		30	0.50	-40 to 360/370	0541025
		30	1.00	-40 to 360/370	054122
		60	0.25	-40 to 360/370	054102
	0.32	15	0.25	-40 to 360/370	054144
		15	1.00	-40 to 360/370	054152
		25	0.25	-40 to 360/370	054119
		25	0.50	-40 to 360/370	054125
		30	0.25	-40 to 360/370	054145
		30	0.50	-40 to 360/370	0541205
		30	1.00	-40 to 360/370	054145
		60	0.25	-40 to 360/370	054146
		60	1.00	-40 to 360/370	054154
	0.53	12	1.00	-40 to 360/370	054130
		15	1.00	-40 to 360/370	054147
		15	1.50	-40 to 350/360	0541347
		15	3.00	-40 to 350/360	054159
		25	1.00	-40 to 360/370	054131
		30	0.50	-40 to 360/370	0541345
		30	1.00	-40 to 360/370	054148
		30	1.50	-40 to 350/360	0541348
		30	3.00	-40 to 350/360	054160
		60	1.00	-40 to 360/370	054158
		60	3.00	-40 to 350/360	054164
BPX35	0.10	10	0.1	0 to 360/370	054699
	0.22	15	0.25	0 to 360/370	054713
		30	0.25	0 to 360/370	054714
	0.25	15	0.25	0 to 360/370	054700
		15	1	0 to 360/370	054703
		30	0.25	0 to 360/370	054701
		30	0.5	0 to 360/370	0547025
		30	1	0 to 360/370	054704
		60	0.25	0 to 360/370	054702
		60	1	0 to 360/370	054705
	0.32	15	0.25	0 to 360/370	054723
		15	0.5	0 to 360/370	054718
		15	1	0 to 360/370	054716
		30	0.25	0 to 360/370	054724
		30	0.5	0 to 360/370	0547158
		30	1	0 to 360/370	054717
		60	0.25	0 to 360/370	054725
	0.53	15	0.5	0 to 360/370	054734
		15	1	0 to 360/370	054736
		30	0.5	0 to 360/370	054735
		30	1	0 to 360/370	054737
BPX50	0.10	10	0.10	0 to 360/370	054740
	0.25	15	0.25	0 to 360/370	054750
		30	0.25	0 to 360/370	054751
		60	0.25	0 to 360/370	054752
	0.32	15	0.25	0 to 360/370	054760
		30	0.25	0 to 360/370	054761
	0.53	15	0.50	0 to 360/370	054770
		30	0.50	0 to 360/370	054771
		30	1.00	0 to 360/370	054772
SolGel-1ms™	0.25	30	0.25	0 to 370/380	054795
		60	0.25	0 to 370/380	054793
	0.32	30	0.25	0 to 370/380	054798
		60	0.25	0 to 370/380	054794

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.

For information on SGE's complete range of GC accessories, please refer to our latest catalog, or contact your nearest SGE office or distributor.

www.sge.com

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