ANALYSIS OF SYNTHETIC PYRETHROIDS USING CAPILLARY COLUMNS OF VARIOUS POLARITIES

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Introduction

Pyrethroids are extremely potent insecticides that are widely used in agriculture, disease control and in household products. For example, many domestic fly sprays use pyrethroids as the active insecticide. Pyrethroids act by interfering with the insect's nervous system, and in high concentrations, can affect humans in a similar way. Side effects can include asthma attacks, liver damage and allergic reactions and many of the pyrethroids are also carcinogenic. They can remain present in the environment (e.g. soil) between 1-16 days and as a result, can reach natural water ways for which they are especially toxic to fish and other aquatic organisms.
The levels of synthetic pyrethroids in foods and the environment are always under close scrutiny.

Analysis

Laboratory analysis of synthetic pyrethroids by gas chromatography is often a difficult and confusing task. As synthetic pyrethroids are based upon the natural pyrethrin structure extracted from chrysanthemum flowers, namely the chrysanthemic acid (Figure 1) where the R group can be any type of substituent, some pyrethroids may act as a precursor to other active pyrethroids.

The analysis may also be confusing because some synthetic pyrethroids, such as Cypermethrin (Figure 2a) can have 8 isomers due to the presence of chiral centers. This makes analysis of synthetic pyrethroids by gas chromatography confusing when multiple peaks represent a single pyrethroid (Figure 2b), with some of these peaks co–eluting.

There are several choices of columns for these types of analyses. Phases such as BPX5, BPX35 and BPX50 (where the percentage phenyl content is progressively increased and as the names suggest, BPX5: 5% phenyl content, BPX35 35% phenyl and BPX50 50% phenyl) are excellent for this type of analysis. All of these columns are suitable for analysing synthetic pyrethroids.

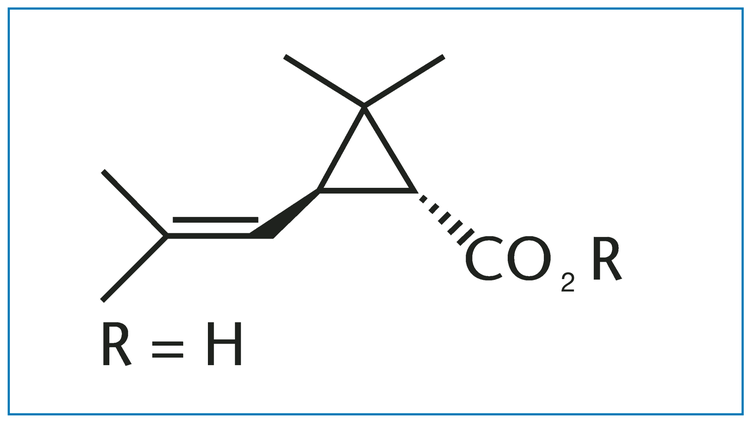
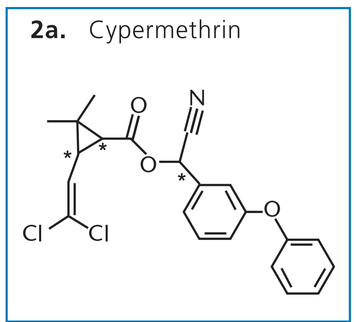


Figure 1. Chrysanthemic acid

2a. Cypermethrin



2b. Cypermethrin chromatographic peaks

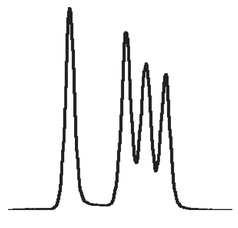


Figure 2. Chromatogram of the synthetic pyrethroid cypermethrin. Note the 4 peaks seen here representing the 4 diastereoisomers of cypermethrin. The structure of cypermethrin shown here shows the 3 chiral centers (*) of cypermethrin.

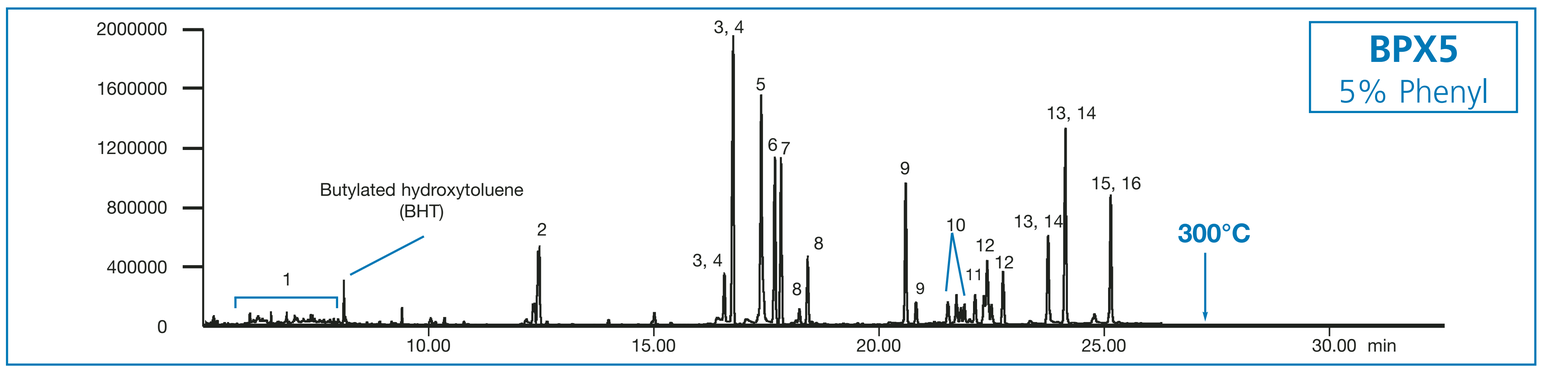


Figure 3. Separation of 16 pyrethroids on BPX5. Note the superb bleed profile at 300°C. BPX5 gives excellent separation of synthetic pyrethroids. Visit www.sge.com for all experimental conditions.

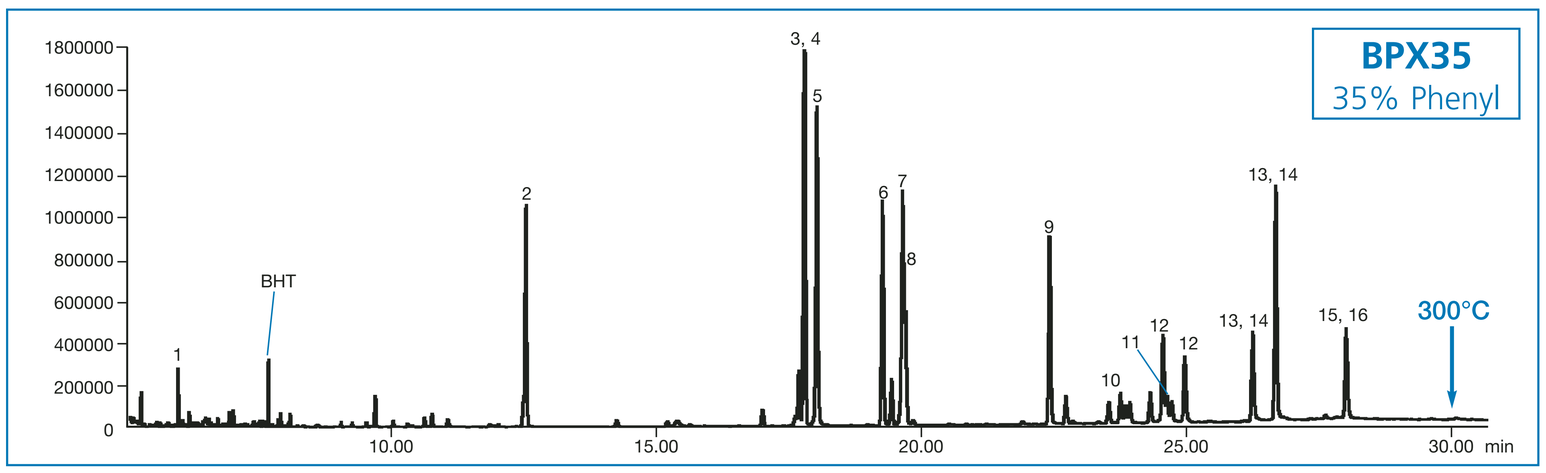


Figure 4. Unparalleled separation of the 16 synthetic pyrethroids on BPX35. The bleed at 300°C is also excellent.

Components (for Figure 3, 4 & 5)

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

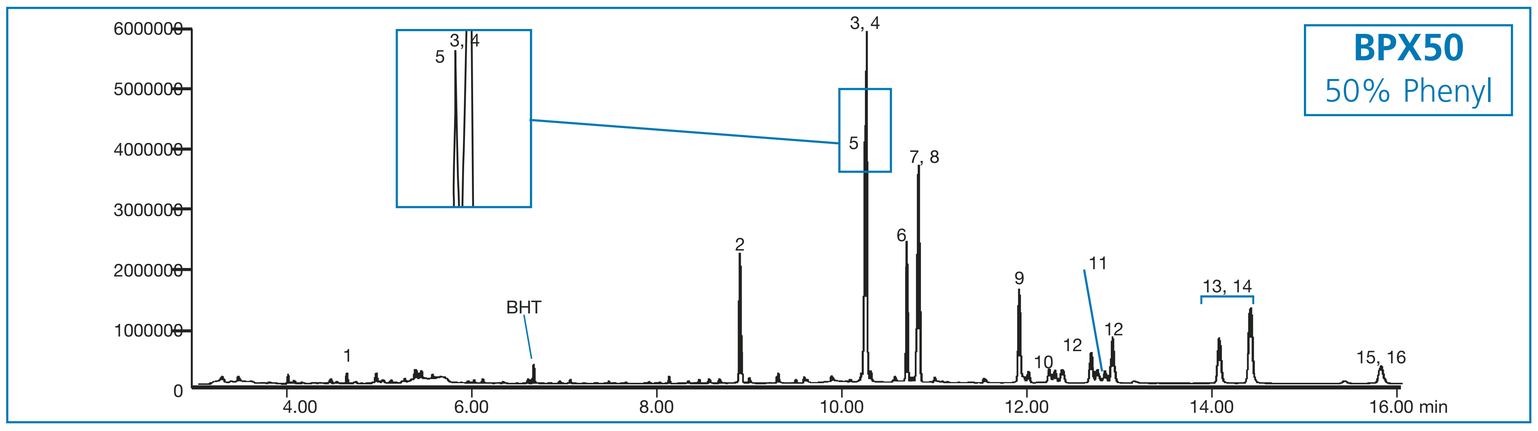


Figure 5. Separation of the synthetic pyrethroids on a BPX50. Note the run time on this chromatogram is 16 minutes for complete elution of the 16 pyrethroids. This allows analytical laboratories to have a higher sample throughput.

BPX5, 35 and 50 provide outstanding resolution of 16 target synthetic pyrethroids in less than 32 minutes.

Most laboratories will use a relatively non-polar column (i.e. 5% phenyl) as the quantitation column. BPX35 and BPX50 are excellent confirmation columns for BPX5 by providing different retention times, elution orders and degrees of overlap of the various isomers. These columns also have the added advantage of being thermally stable at 360°C allowing the baking out of any high boiling contaminants without damaging the phase. This bake out ensures that these contaminants do not interfere with the retention times and elevated baselines of target compounds for future analyses. These columns exhibit exceptional peak shape of each pyrethroid; indicating thier high degree of inertness.

Summary

BPX5, BPX35 and BPX50 columns demonstrate unparalleled performance for the separation of 16 synthetic pyrethroids. The columns can be conditioned at the end of each analysis to remove any high boiling point contaminants without any degradation to the stationary phase. The bleed levels are exceptionally low at 300°C and the columns produce excellent peak shape indicating a high degree of inertness.

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