GCxGC Separation of the Pesticides Beta-Hexachlorocyclohexane and Trifluralin

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Key Words: GCxGC-TOFMS, Pesticides

1. Introduction

The pesticides beta-hexachlorocyclohexane (beta-HCH) and Trifluralin can coelute on 100%-dimethyl-polysiloxane and 95%-dimethyl-5%-diphenyl-polysiloxane Gas Chromatography (GC) columns. Although Mass Spectrometry (MS) can identify them based on their unique spectra, it is necessary to have them chromatographically separated when a non-specific detector like an Electron Capture Detector (ECD) is used for their analysis. ECDs are often used in routine pesticide monitoring due to lower cost and less maintenance versus MS.

Comprehensive two-dimensional GC (GCxGC) is one way to solve this type of coelution problem. GCxGC increases peak capacity by applying two independent separations to a sample in one analysis with one detector. GCxGC involves serially connected columns (differing phases), separated by a thermal modulator. A separation is performed on the first column, and then effluent from the first column is continually (and quickly) focused and "injected" onto the second column. By keeping the second column short, a series of high-speed chromatograms are generated, and the first column separation can be maintained. Separation results can be plotted as a retention plane (column 1 time x column 2 time), also known as a contour plot.

This note shows the GCxGC separation of beta-hexachlorocyclohexane and Trifluralin.

2. Experimental Conditions

Standards

Pesticide standards were obtained from Restek Corporation.

Instrumentation

LECO Pegasus® III GC-TOFMS (Time-of-Flight Mass Spectrometer) and LECO Pegasus 4D GCxGC-TOFMS systems were used for this work. The 4D contains a LECO quad jet—dual-stage thermal modulator and independently heated secondary oven. The one-dimensional GC work was done with a DB-1 (J&W Scientific) column. The column set for GCxGC used a DB-1 in the first dimension, and an Rtx-CLPesticidesII (Restek Corporation) column in the second dimension.

3. Results and Discussion

Figure 1 illustrates the one-dimensional chromatographic coelution of beta-HCH and Trifluralin on DB-1, which results in the mixed mass spectrum seen in Figure 2. Although spectral deconvolution algorithms are an integral part of the ChromaTOF® software used for system control and data processing on Pegasus units, there has to be at least some small difference in peak apexes to produce spectra that are not coeluted. In this example, the coelution of beta-HCH and Trifluralin is exact. As mentioned in the introduction, this is likely not of concern for quantitative purposes when using MS, as unique ions can be selected as quantification masses for both pesticides. But for ECD, chromatographic separation of these pesticides is necessary, and can be easily accomplished with GCxGC (Figure 3).

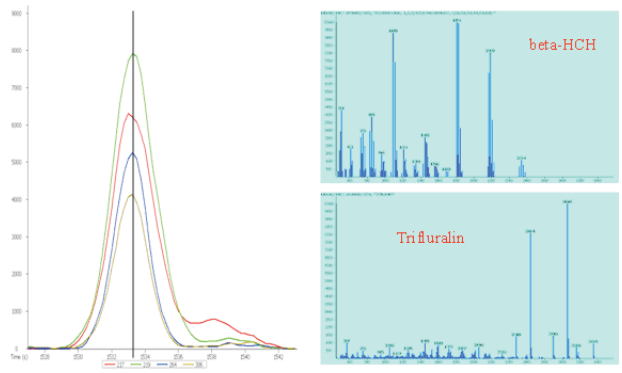

Figure 1. Coelution of beta-HCH and Trifluralin on DB-1. The red (217) and green (219) ion plots represent beta-HCH, and the blue (264) and brown (306) ion plots represent Trifluralin. Non-coeluted mass spectra for each pesticide are shown on the right.

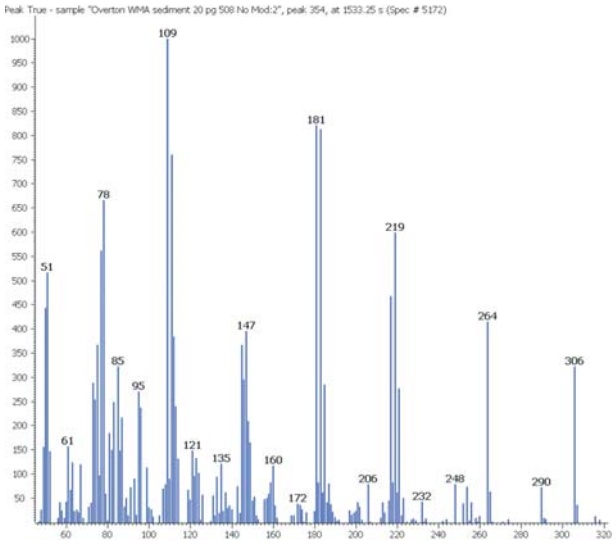

Figure 2. Mixed mass spectrum of beta-HCH and Trifluralin resulting from their exact coelution on DB-1. The 264 and 306 ions can be attributed to Trifluralin, and the main beta-HCH ions are in the 109/181/219 groups.

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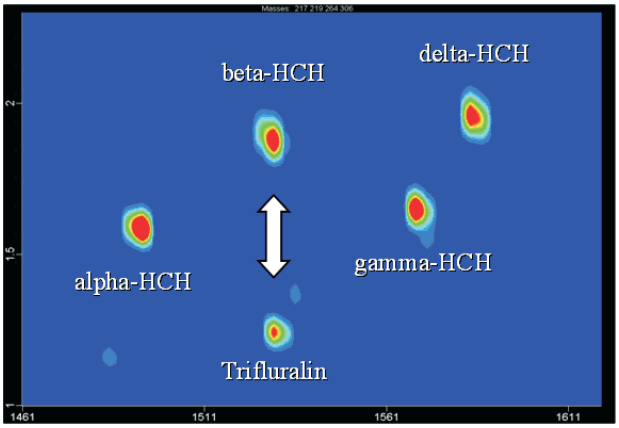

Figure 3. Contour plot showing chromatographic separation of beta-HCH and Trifluralin in the second dimension (Rtx-CLPesticidesII, Y-axis). They coelute in the first dimension (DB-1, X-axis) as noted by the white arrow.

4. Conclusions

GCxGC is a powerful way to increase peak capacity and promote separation of compounds that might coelute in a one-dimensional GC analysis. Chromatographic separations are especially important when non-specific detectors are used.

5. Acknowledgment

Frank Dorman at Restek Corporation provided the Rtx-CLPesticidesII GC column.

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