MULTIRESIDUE PESTICIDE ANALYSIS
IMPROVING PEAK SHAPE CONSISTENCY

Technology Advantage: Agilent Intuvo 9000 GC with MS/MS

Introduction

Multiresidue pesticide analysis has become the prevalent method for analysis of food products because it allows the simultaneous determination of multiple pesticides, the number of which is continuously increasing. QuEChERS is the preferred method for sample preparation. It reduces the matrix load, but does not produce a clean sample. Despite sample cleanup, pesticide analysis can suffer from reduced response and increasingly asymmetric peaks over time. Reducing batch size or increasing the frequency of inlet or column/retention gap maintenance can be a reasonable solution for conventional gas chromatograph systems.

The Agilent Intuvo 9000 Gas Chromatograph addresses these issues, and offers the additional advantage of an innovative inert flow path[2]

A redesigned modular flow path featuring a reimagined retention gap protects the analytical column from matrix, and eliminates the need to trim the column. The innovative flow path aids in maintaining chromatographic integrity (response and peak shape) for even the most challenging analytes. Lastly, the Intuvo 9000 GC is only 27 cm wide affording additional flexibility, especially in laboratories with limited bench space.

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Experimental

A Intuvo 9000 GC was coupled to an Agilent 7000 Series triple quadrupole mass spectrometer in a simplified fashion to highlight the instrument's advantages. A split/splitless inlet was used with a standard 15 m Intuvo HP-5ms Ultra Inert Column. A custom pesticide standard was obtained from Ultra Scientific, and calibration standards ranging from 1 ppm to 5 ppb were diluted in acetone. Multiple reaction monitoring (MRM) was used by obtaining transition from the P&EP MRM database (p/n G9250AA rev A.1.01). A black tea extract was prepared using Agilent Bond Elut QuEChERS EN extraction tubes (p/n 5982-5650) followed with Agilent Bond Elut QuEChERS EN dispersive SPE tubes (p/n 5982-5256). A 3-layer sandwich injection was made using the standard, matrix, and an analyte protectant solution. Complete method details are available in a separate Application Note[3].

Results and Discussion

After calibrating with the tea extract (consisting of 24 injections), the 50 ppb standard was evaluated to determine whether it was still within a 70–120 % recovery window. Upon passing that specification, 60 injections of the 50 ppb standard (with matrix) were made. After the 60 matrix injections were completed, the liner and Intuvo Guard Chip were replaced to mimic the maintenance often performed after batch analyses on conventional GC systems. Figure 1 shows the 50 ppb standard at the recovery check after calibration, after 60 matrix injections, and after the liner and Intuvo Guard Chip replacement. Over the course of analysis, there was very little change in the chromatography. Peak shape was well maintained, as was recovery. Figure 2 highlights methacrifos, its neighboring internal standard of acenapthene-d10, and pyraclostrobin. While there is a slight difference in peak height, this is accounted for when normalized to the internal standards. Otherwise, there was no noticeable change in peak shape, and recoveries were within 70 to 120 %.

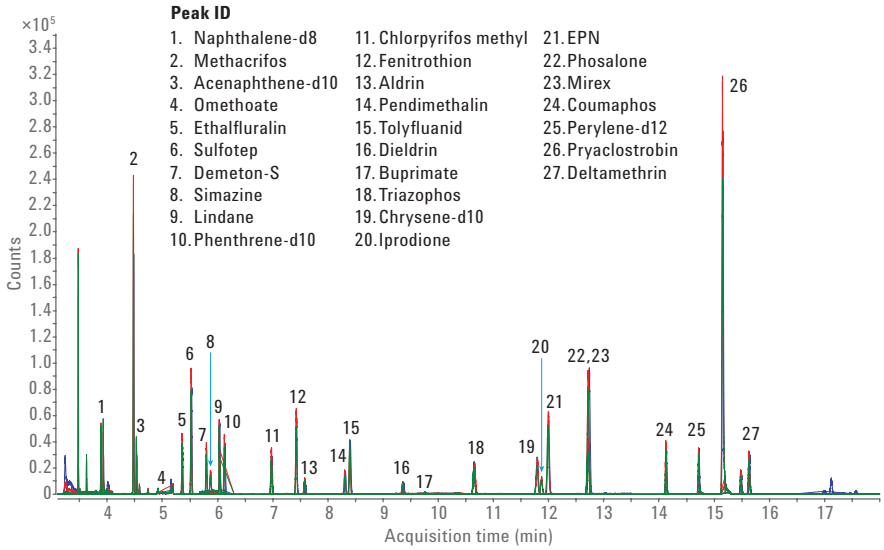

Figure 1. Overlaid chromatograms for a 50 ppb pesticide standard measured after calibration (blue), after 60 matrix injections (red), and after liner and Intuvo Guard Chip replacement (green).

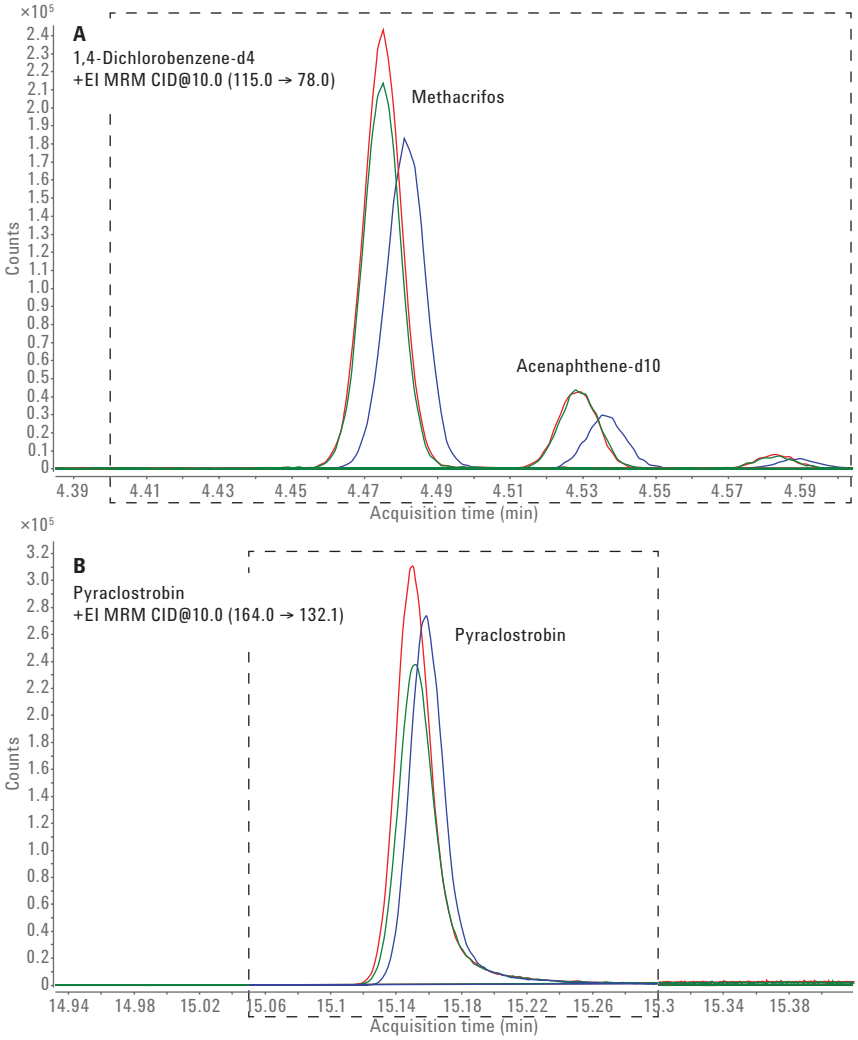

Figure 2. Methacrifos and pyraclostrobin are shown to demonstrate the peak fidelity maintained over the course of nearly 100 matrix injections using an 15 m Intuvo HP-5ms Ultra Inert Column. The dashed box shows the MRM transition window used.

Conclusion

Analyzing pesticides with the Agilent Intuvo 9000 GC using a 15 m Intuvo HP-5ms Ultra Inert Column yields consistent results throughout the batch. Peak shape is well maintained throughout calibration and matrix samples. For the application shown here, nearly 100 injections were performed before maintenance was performed. Maintenance on the 9000 Intuvo GC has been simplified as a result of the Intuvo Guard Chip, a reimagined retention gap. Retention time windows are maintained after replacing the Intuvo Guard Chip, further simplifying the analysis.

References

1. Analysis of Semivolatiles Intuvo Guard Chip Protection, *Agilent Technologies*, publication number 5991-7182EN, **2016**.
2. Veeneman, R.; Stevens, J. Multiresidue Pesticide Analysis with the Agilent Intuvo 9000 GC and Agilent 7000 Series Mass Spectrometer. *Agilent Technologies Application Note*, publication number 5991-7216EN, **2016**.

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