Multiresidue Pesticide Analysis with the Agilent Intuvo 9000 GC and Agilent 7000C Triple Quadrupole GC/MS

Introduction

As pesticide use has increased so has the level of concern among environmentalists, regulators, and consumers. Regulations regarding the maximum limit of pesticide residues that can be found in or on foods (MRLs) have been established nearly worldwide, including countries in North America (United States and Canada), Europe (European Union), Asia (Japan), and Australia. In the United States, MRLs can range from 0.02 ppm to 100 ppm depending on the matrix and pesticide in question while the European Commission has a default value of 0.05 ppm [1].

The analysis of pesticides in food can quickly become complex with increasing target compound and commodity lists and decreasing detection requirements. Having a robust method on an easy-to-use platform that integrates seamlessly to a large database is desired to facilitate this analysis. Coupling Agilent’s 7000C Triple Quadrupole GC/MS to Agilent’s newest GC platform (Intuvo 9000 Gas Chromatograph) delivers a streamlined workflow that involves the implementation an inert microfluidic retention gap (guard chip) for multidrug pesticide analyses. Calibration curves for various target pesticides in varied matrices showed excellent linearity for concentrations ranging from 5 ng/mL to 500 ng/mL (97% of the compounds analyzed maintained a R2 > 0.995). Excellent response and peak shape consistency were obtained with the implementation of the Intuvo guard chip which protects downstream components and eliminates the need to trim the column after matrix evaluation. All analyzed pesticides obtained a %RSD of repeated measurements ≤30% with regular maintenance, including liner and Intuvo guard chip replacements, peak shape, response, and analyzes calibration can be preserved for up to 500 injections [2].

Sample Preparation

Sample preparation is an essential part of successful chromatography. It extends column lifetime, reduces the need for repeated samples, and minimizes interferences that can jeopardize your separation, detection, and quantification. Many laboratories focused on pesticide residue analysis in food commodities routinely use the Quick, Easy, Cheap, Effective, Rugged, and Safe (QEChem) method [3]. This straightforward sample preparation allows for the analysis of hundreds of pesticides at low concentrations with a single extraction. However, sample cleanup steps are becoming more common within the laboratory. Not only will this influence the analysis quality, but it will increase the frequency of GC/MS system maintenance. Three diverse matrices were selected to demonstrate matrix differences. These matrices included organic honey, jasmine rice, and black loose-leaf tea. Each matrix was extracted with their specified QEChemS methodology (Figure 1), in which specific dispersive SPME (dSPME) were used for matrix cleanup.

Results and Discussion

Meeting MRL Requirements

Agilent’s Intuvo 9000 GC and the 7000C Series Triple Quadrupole GC/MS system can confirm and quantitate pesticide residues at the low ppb level in organic honey, jasmine rice, and loose-leaf black tea that meet both the EU and the USDA MRL requirements. Note that the commodities analyzed are more aligned with EU commodities. Generally EU MRLs are lower than those of the USDA.

Table 1. MRL requirements and results for Organic Honey

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Table 2. MRL requirements and results for Loose-leaf Black Tea

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Table 3. MRL requirements and results for Jasmine Rice

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Experimental

Instrumentation

All analyses were run on an Agilent Intuvo 9000 GC equipped with an Agilent 7693B Autosampler and an Agilent 7000C Triple Quadrupole GC/MS (Figure 1). The Intuvo 9000 inert flow path was configured with a multimode inlet (MMI) equipped with two Agilent J&W HP-5ms Ultra Inert Intuvo GC columns (15 m x 0.25 mm, 0.25 µm film thickness). The Table below displays the GC/MS/MS method parameters. The Intuvo GC was also configured with a multimode inlet (MMI) equipped with a 4 mm ultrainert, stainless, single-taper, glass wool liner (p/n 5190-2293), and an MMI guard chip (p/n 54987-06605).

Table 4. Surrogate Recovery Data (n = 12, 25–25 ppb)

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Conclusions

Agilent’s Intuvo 9000 GC and the 7000C Series Triple Quadrupole GC/MS system can confirm and quantitate pesticide residues at the low ppb level in complex extracts that will meet both the EU and the USDA MRL requirements. Calibration curves for targeted pesticides in organic honey, jasmine rice, and loose-leaf tea showed excellent linearity (97% of the compounds analyzed maintained a R2 > 0.990) for concentrations ranging from 5 to 500 ppb. For all compounds analyzed in honey and rice the LOQs were found to be below 10 ppb. For 94% of the compounds analyzed in the tea the LOQs were found to be below 100 ppb. All analyzed pesticides obtained a %RSD of repeated measurements ≤30% with recovery errors ≤30%. Noteworthy is that the commodities analyzed are more aligned with EU commodities. Generally EU MRLs are lower than those of the USDA.

References


Figure 1. Sample Prep Procedure

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Figure 2. Agilent Intuvo 9000 GC equipped with an Agilent 7693B Autosampler and an Agilent 7000C Triple Quadrupole GC/MS and B) Intuvo Column Configuration for a Multiresidue Pesticide Workflow

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Figure 3. Trans-Chloride at Spb in Tea. A) overlay of MRM transitions, and B) view of each transition separately.

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Figure 4. DGT-qSpb at Spb in Honey. A) overlay of MRM transitions, and B) view of each transition separately.

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Figure 5. Linoleic acid in Spb at 17:0 and at 18:0. A) overlay of MRM transitions, and B) view of each transition separately.

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Figure 6. Linoleic acid in Spb at 17:0 and at 18:0. A) overlay of MRM transitions, and B) view of each transition separately.

Figure 6. Linoleic acid in Spb at 17:0 and at 18:0. A) overlay of MRM transitions, and B) view of each transition separately.

Figure 7. Linoleic acid in Spb at 17:0 and at 18:0. A) overlay of MRM transitions, and B) view of each transition separately.