

Poster Reprint

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Analysis of Pesticides in Spices with Hydrogen Carrier Gas

Soma Dasgupta¹, Lorna De Leoz²

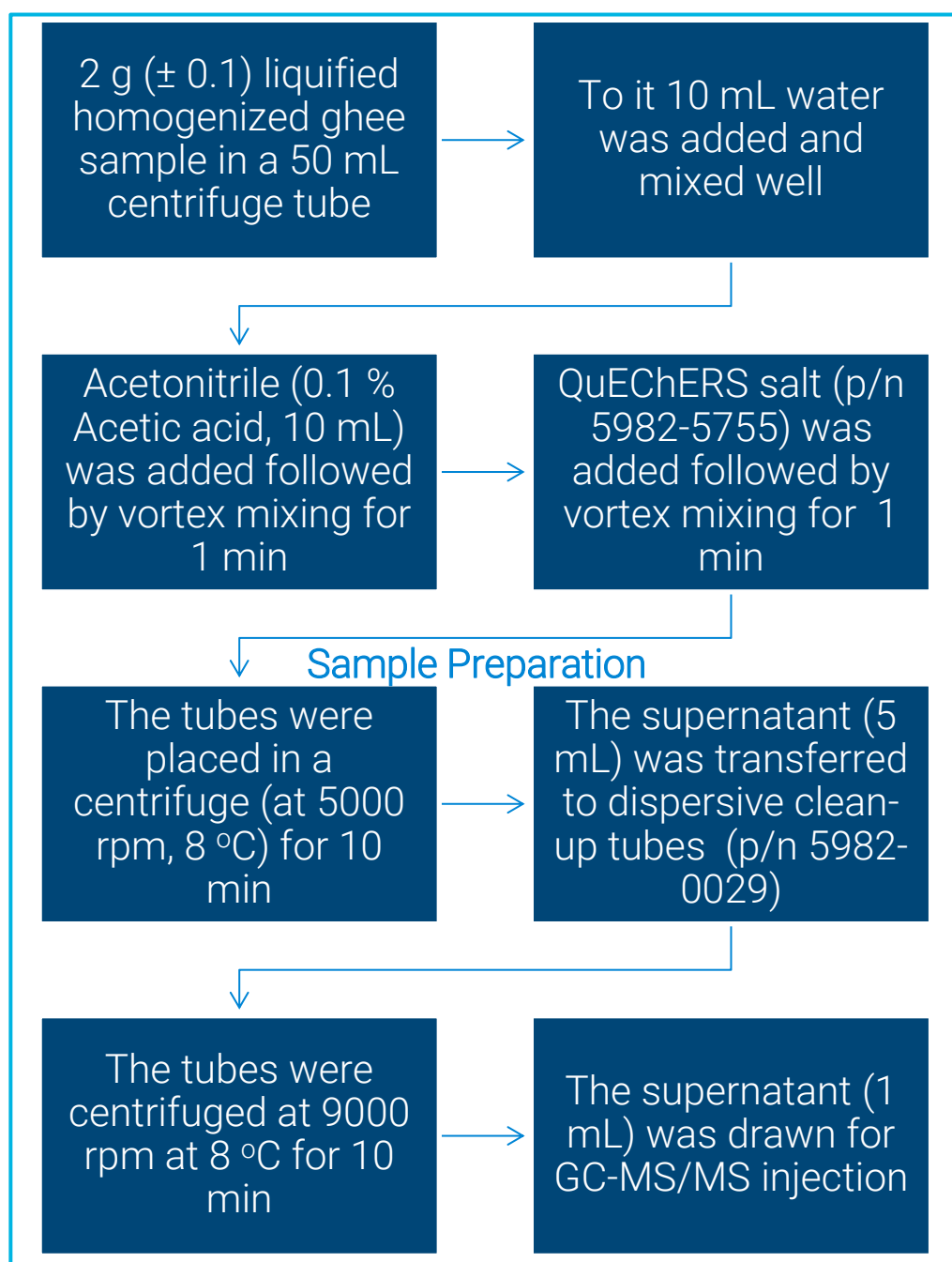
¹ Agilent Technologies, India,

² Agilent Technologies, Wilmington, Delaware, United States

Introduction

Spices are considered as one of the most challenging matrices for analysis of pesticides. Usually, the sample analysis involves 5-10 fold dilution to reduce the matrix load. Thus, lower instrument detection limits need to be achieved. Use of hydrogen as carrier gas can further pose changes in terms of sensitivity. This work describes the suitability of the hydroinert and HES ion source to achieve the required sensitivity. Samples were extracted using QuEChERS based approach followed by dispersive cleanup. Linearity was evaluated against matrix matched standards within the range of 0.5-50 ng/mL. Correlation coefficient (r^2) for calibration ranging between 0.5 -50 ng/mL was found above 0.95. Precision study was carried out by injecting 6 replicates of pre-spiked samples at 10 ng/g. The average relative standard deviation (%RSD) was <20% with recoveries ranging from 72-125%. The advantages of the method include utilization of same retention time MRMs and collision energies. This allowed for easy transition from helium to hydrogen carrier gas.

Experimental



Experimental

Matrix: Cardamom, Cumin, Coriander, Black Pepper, Chilli, Turmeric

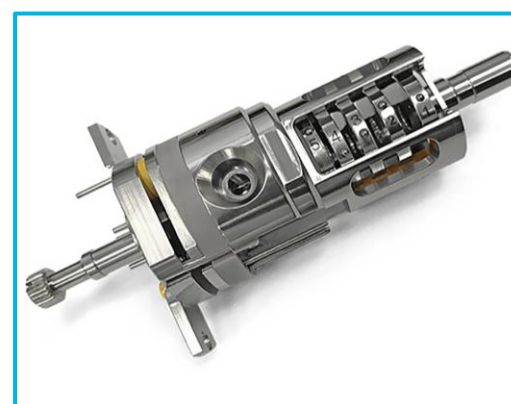


Figure 1. High Efficiency Source for GC/MS Instruments



Figure 2. HydroInert Source for 7000 TQ

An Agilent 8890 GC coupled to a 7000 (with HydroInert source) / 7010 Triple Quadrupole GC/MS system equipped with High Efficiency Source (HES) was used. The GC system was equipped with a Multi-Mode Inlet (MMI) with air cooling and a back flushing system based on a Purged Ultimate Union controlled by a PSD module.

Agilent 8890 GC parameters	
Injection volume	2 μ L
Liner	Agilent inlet liner, UI, splitless, dimpled (p/n 5190-2297)
Carrier gas	Hydrogen
Injection mode	Splitless
Injector program	70 °C (hold time 0.2 min) 280°C (ramp at 900 °C/min)
Oven program	60 °C (1 min) 170 °C (40 °C/min, 0 min) 310 °C (10 °C/min, 3 min) Total run time: 20.75 min Backflush at 325 °C for 7 mins
Column 1 and 2	HP-5ms Ultra Inert (20 m x 180 μ m x 0.18 μ m)
Column flow 1 & 2	1 and 1.2 mL/min
Agilent 7000 or 7010 MS/MS	
Source temp.	280 °C
Quadrupole temp.	Q1=Q2=150 °C
Collision cell gas flows	N ₂ : 1.5 mL/min He: Switched off when using hydrogen carrier gas
MRM parameters	As per MRM database

Results and Discussion

The oven temperature program used with H₂ as the carrier gas was maintained identical to that used with He. The analytical method originally optimized for a 20-minute runtime using He was successfully adapted for H₂. This ensured preservation of chromatographic resolution and retention time fidelity across carrier gas substitution, as evident from the elution order shown in Figure 3. The chromatograms indicate presence of high matrix load at initial 7 mins for all the spices. At RT 10-13 min, matrix load of and chilli was high. The matrix load from black pepper was very high throughout the runtime. Backflush of 7 min was used to remove the matrix.

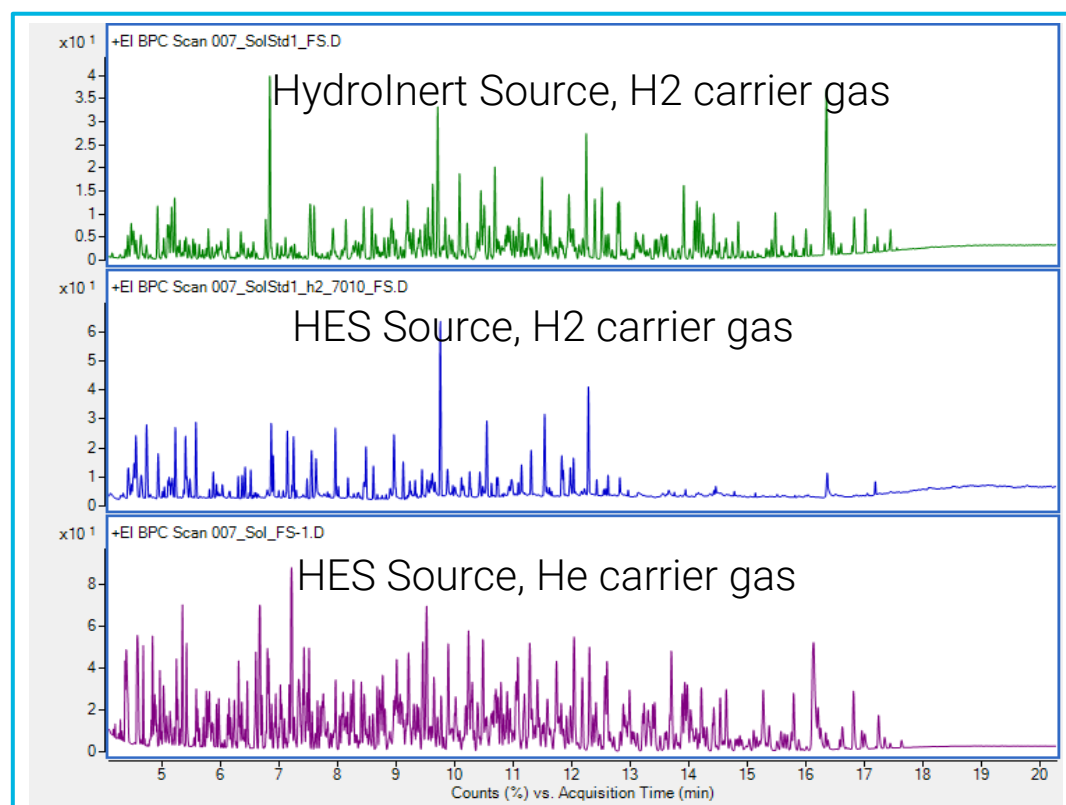


Figure 3. Full scan chromatogram of pesticide mix

Transitioning from He to H₂ can often lead to reactions at the ion source resulting in spectral changes when compared with He. Therefore, EI sources with reduced or eliminated source reactivity such as HydroInert and HES is essential to minimize or prevent the undesirable in-source reactions when using hydrogen. The tested mixture of pesticides were compared against the NIST library to ensure spectral fidelity. As evident from Figure 5, the library match percentages were acceptable and this the existing MRM database could be used for acquisition in MRM mode, without the requirement of any additional MRM development

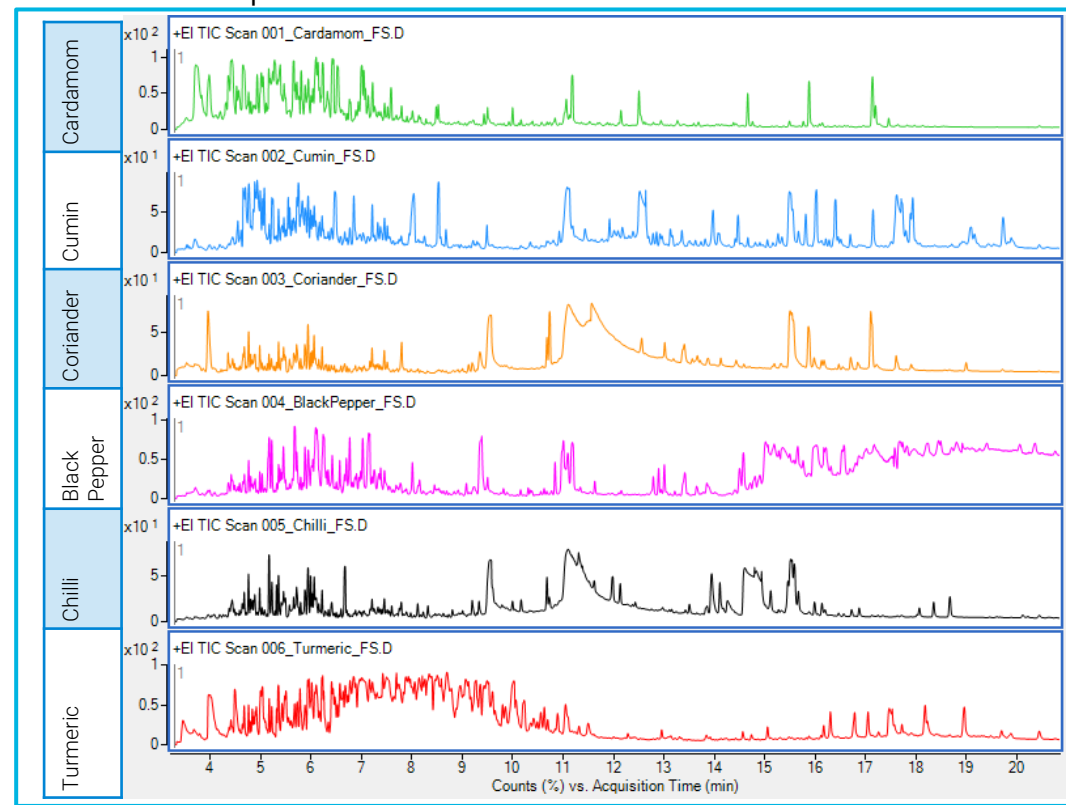


Figure 4. Full scan chromatogram of blank matrix extracts

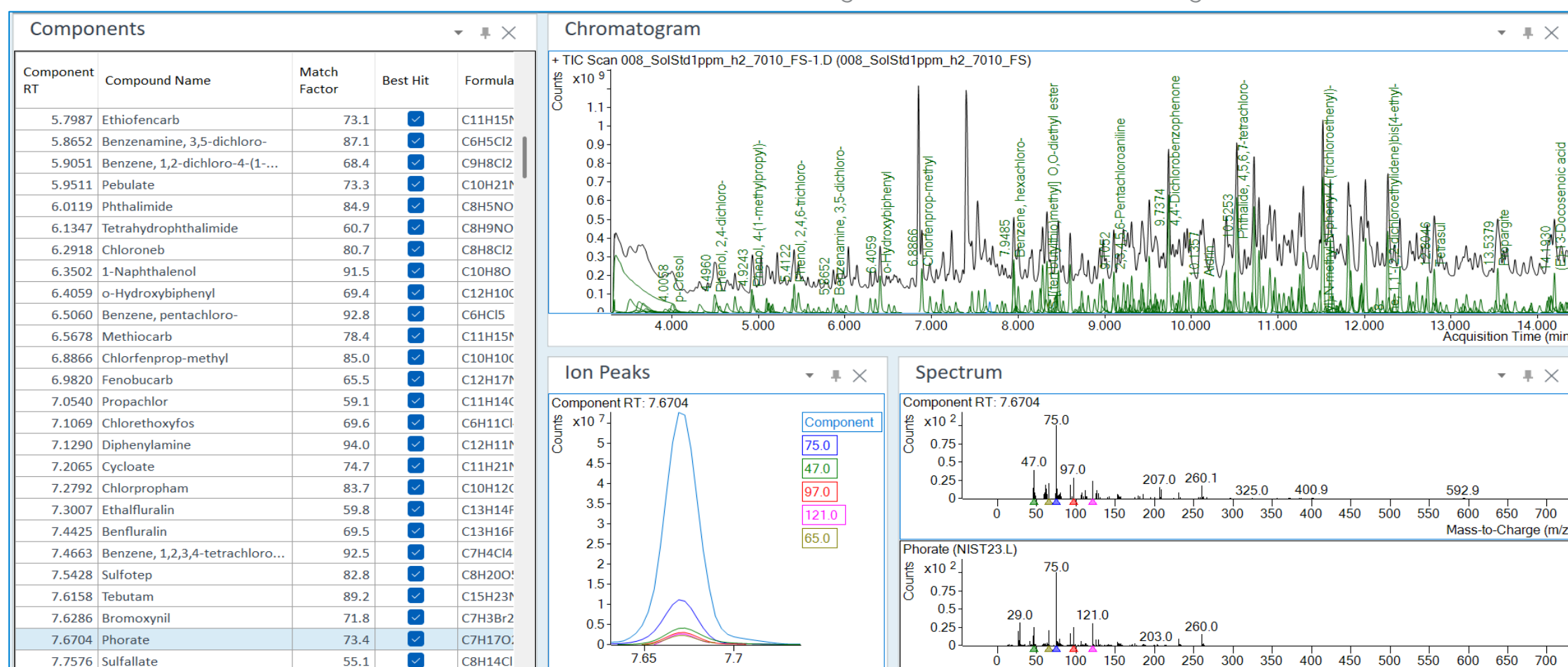


Figure 5. Extracted ion chromatograms of 150 pesticides in ghee matrix

Results and Discussion

HydroInert Source Gain factor 15 HES H2 Carrier Gas Gain factor 5 HES He Carrier Gas Gain factor 5 HydroInert Source Gain factor 15 HES H2 Carrier Gas Gain factor 5 HES He Carrier Gas Gain factor 5

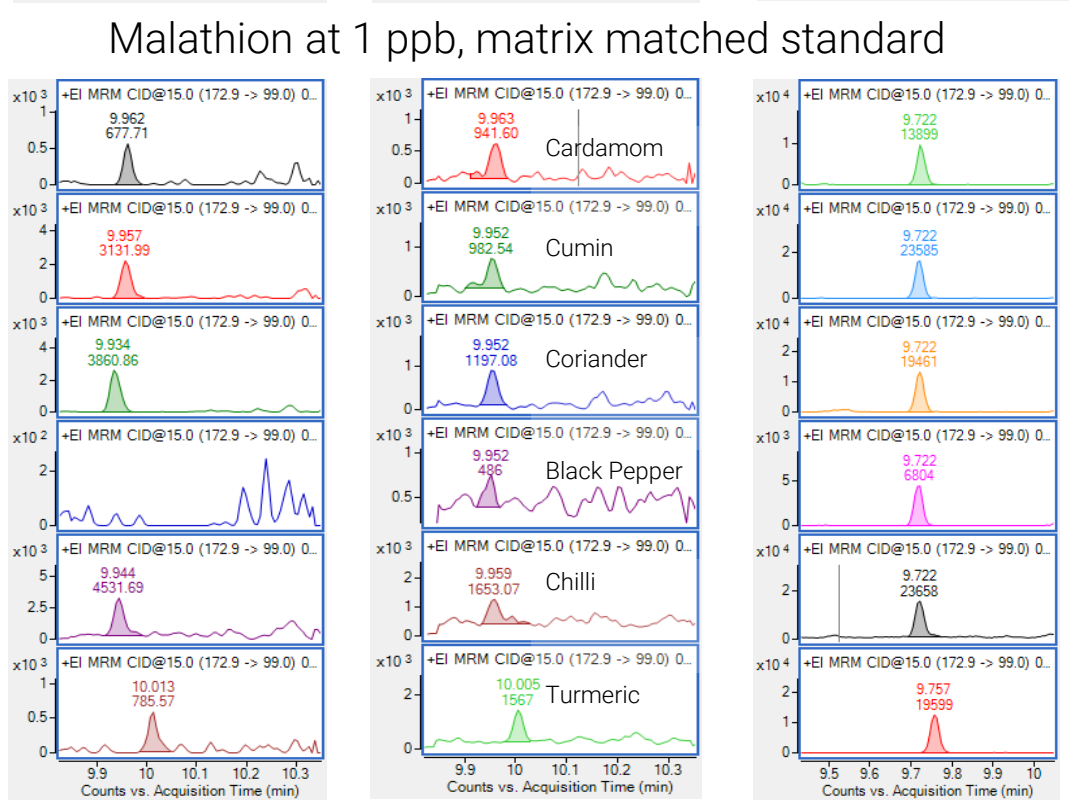
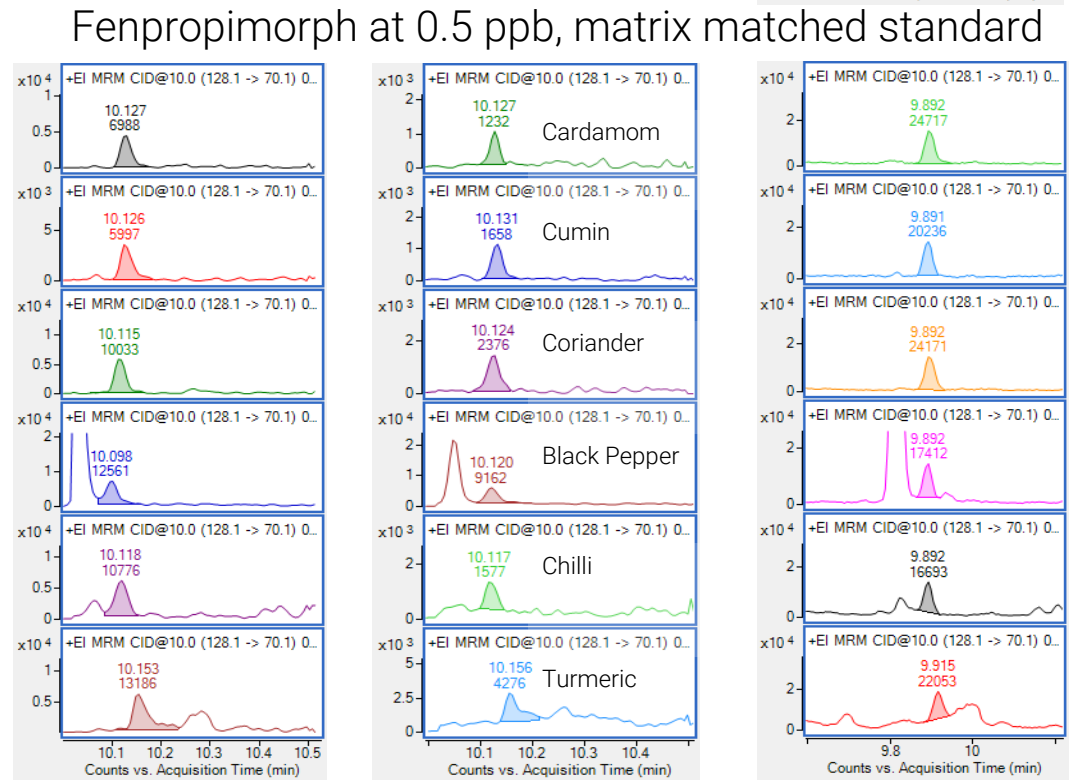
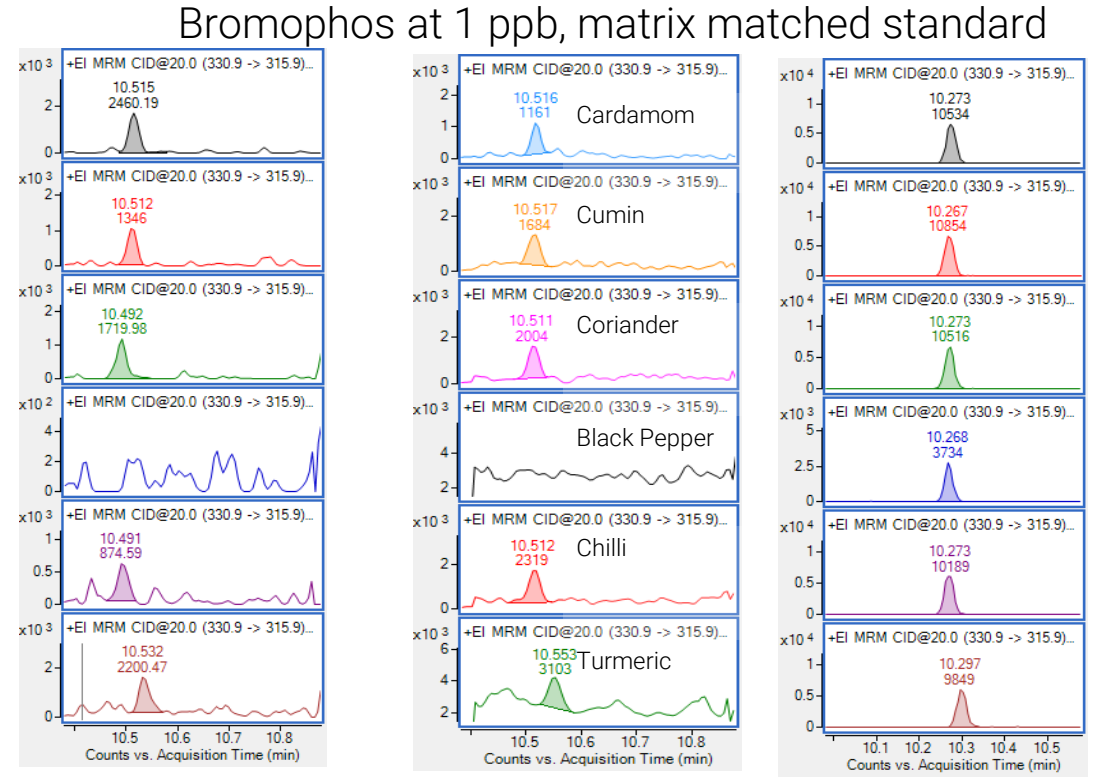
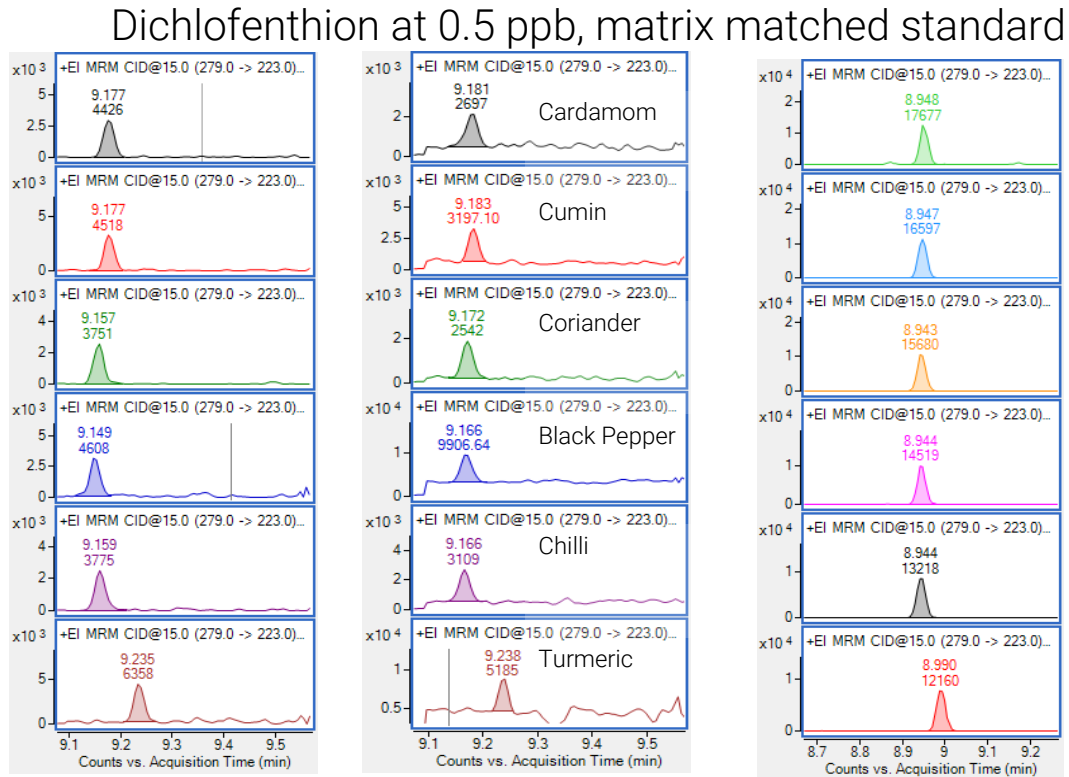


Figure 6. Chromatograms of representative pesticides

The lowest calibration levels for the target analytes ranged from 0.5 to 2 ng/g. The samples were subjected to 5-fold dilution, resulting in LOQs spanning 2.5 to 5 ng/g. Mean recovery rates were within the range of 70–130%. Matrix effects were found to significantly impact analyte response, as demonstrated in Figure 6. Although malathion was detectable at concentrations below 1 ppb across most matrices, its detection threshold increased to above 2 ppb in black pepper, indicating substantial matrix suppression. A comparable trend was observed for bromophos, suggesting matrix-dependent variability in analytical sensitivity.

Conclusions

Agilent GC/TQ systems with HydroInert and HES sources delivered consistent sensitivity and spectral fidelity using hydrogen carrier gas, without re-optimizing MRM transitions or collision energies. Retention times and elution order were preserved across complex spice matrices.

The systems achieved LOQs of 2.5 to 5 ppb after five-fold dilution, even in heavily loaded matrices like black pepper, confirming reliable quantitation in high-background spice samples.

References

GC/MS/MS Pesticide Residue Analysis. A reference guide. 5994-7435EN

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