Application Note

Food & Beverage Testing

Agilent

Trusted Answers

A Fast and Robust GC/MS/MS Analysis of 203 Pesticides in 10 Minutes in Spinach

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Abstract

This application note describes two approaches for achieving robust, multiresidue pesticide analysis in 10 minutes by GC/MS/MS, while maintaining sufficient chromatographic resolution for the analysis of over 200 pesticides in spinach; a challenging high chlorophyll, fresh matrix. First, the conventional 15 × 15 m (0.25 mm × 0.25 µm) midcolumn backflush configuration was used with an accelerated oven ramp, yielding an analysis time of 10 minutes. Second, a minibore 10 × 10 m (0.18 mm × 0.18 µm) midcolumn backflush configuration was used, enabling a fast 10-minute analysis time. The latter method was precisely scaled using the Agilent GC method translation technique. It was shown that midcolumn backflushing enabled method robustness and extended maintenance-free operation of the system by minimizing column trimming and source cleaning. Results demonstrate that the Agilent 7000E and 7010C triple quadrupole GC/MS systems delivered excellent linearity over a concentration range of 0.1 to 1,000 parts per billion (ppb). Method robustness was shown with 700 consecutive injections of a spinach extract, spiked with pesticides at 20 ppb, that spanned over 175 hours of continuous running of the GC/TQ.

Introduction

There is a growing demand for more rapid methods for the identification and quantitation of chemical residues in food analysis without sacrificing method robustness and chromatographic performance. Conventional methods for multiresidue pesticide analysis typically take at least 20 minutes, resulting in longer sample cycle times. As a result, the GC/MS analysis time for a batch of samples could easily span over several days. This causes a sample analysis bottleneck and limits lab productivity. Therefore, shortening the GC/MS analysis time will undoubtedly improve sample analysis throughput and eventually laboratory productivity. However, shortened GC methods usually involve trade-offs in method robustness or performance. This application note focuses on demonstrating two fast GC/MS/MS methods using (a) the Agilent 8890 GC and 7000E triple quadrupole GC/MS system and (b) the Agilent 8890 GC and 7010C triple quadrupole GC/MS system. The presented methods provide a shortened run time of 10 minutes, while maintaining robust system performance in the challenging spinach extract, without loss in sensitivity or method performance.

Two GC/TQ system midcolumn backflush configurations described in this application note provide analysis times of 10 minutes, while maintaining sufficient chromatographic resolution and MS selectivity for the analysis of 203 compounds. The conventional 20-minute GC/MS/MS method, retention time locked to the Agilent MassHunter pesticides and environmental pollutants MRM database (P&EP MRM database), was used as a benchmark for the optimized, fast analyses.

First, the conventional 15 × 15 m (0.25 mm × 0.25 µm) midcolumn backflush configuration was used with an accelerated oven ramp, yielding an analysis time of 10 minutes. This configuration did not require any hardware changes. Second, a minibore 10 × 10 m (0.18 mm × 0.18 µm) midcolumn backflush configuration was used enabling a 10-minute analysis time. This configuration required a new set of columns when compared to the conventional 15 × 15 m setup and a GC oven insert (a pillow). However, the second configuration allowed for more accurate prediction of the retention times and preserved the elution order for all tested compounds.

With both fast methods, the retention times were accurately predicted using the retention times available in the P&EP MRM database.[1] Using the GC method translation technique and maintaining the same column phase ratio allowed for accurately predicting the retention times and maintaining elution order for the 203 analyzed pesticides with the 10 × 10 m configuration. To update the retention times for the 10-minute method with the conventional 15 × 15 m configuration, a combination of pesticides and n-alkanes were used.

Midcolumn backflushing with both column configurations improved method robustness by reducing the regular maintenance frequency, such as column head trimming and source cleaning. Also, when used with a temperature-programmable multimode inlet (MMI), the liner change and other inlet maintenance procedures can be conducted much more rapidly without cooling down and venting the MS source, compared to a conventional configuration with a column connecting the inlet directly to the mass spectrometer.

The developed methods were applicable for analyzing pesticides to cover the broad range of maximum residue limits (MRLs) for different pesticides in spinach and to deliver excellent calibration performance over a dynamic range of 0.1 to 1,000 ppb.

To evaluate method robustness, a test of 700 continuous injections of the spinach extract spiked with low-level pesticides was performed. Relative standard deviation (RSD) for the response of many challenging analytes was under 15% over 700 injections. There was no need to trim the column, clean the source, or tune the MS over the test. The maintenance was limited to liner and septum replacement every 100 injections.

Experimental

GC/TQ analysis

Two column configurations used with the 8890/7000E and 8890/7010C GC/TQ combinations are shown in Figure 1. The GC was configured with the Agilent 7693A automatic liquid sampler (ALS) and 150-position tray; an MMI, operated in temperature-programmed splitless injection mode (cold splitless); a midcolumn backflush capability provided by the Agilent purged Ultimate union (PUU), installed between two identical 15 or 10 m columns; and the 8890 GC pneumatic switching device (PSD) module. The instrument operating parameters are listed in Table 1. Data were acquired in dynamic MRM (dMRM) mode, which enables the capability for large multi-analyte assays and to accurately quantitate narrow peaks by an automated and most-efficient dwell time distribution.

The dMRM capability enabled a successful analysis for a large panel of 203 pesticides with 614 total MRM transitions. The maximum number of concurrent MRM transitions with the conventional 15 × 15 m configuration and a traditional 20-minute analysis was 52. For the 10-minute analysis, the maximum number of concurrent MRM transitions with the conventional 15 × 15 m and the minibore 10 × 10 m configurations were 127 and 83, respectively (Figure 2). Furthermore, dMRM enables the analyst to add and

remove additional analytes with ease. The use of the P&EP MRM database increased the ease and speed of setting up a targeted dMRM method.

Agilent MassHunter Workstation revisions 10.1 and 10.2 including MassHunter Acquisition software for GC/MS systems 10.2, MassHunter Quantitative Analysis software 10.1, and MassHunter Qualitative Analysis software 10 packages were used in this work.

Calibration performance was evaluated using a series of matrix-matched calibration standards ranging from 0.1 to 1,000 ppb, including 0.1, 0.5, 1, 5, 10, 50, 100, 250, 500, and 1,000 ppb (w/v). The GC multiresidue pesticide kit containing 203 compounds (Restek, Bellefonte, PA, USA), regulated by the FDA, USDA, and other global governmental agencies, was used for preparing matrix-matched calibration standards. α-BHC-d6, at a final concentration of 20 ppb in vial, was used as the internal standard for quantitation of the target pesticides (Agilent Bond Elut QuEChERS IS standard number 6; part number PPS-610-1). A weighting factor of 1/x was applied to all calibration curves.

Retention time locking the 10-minute methods

Retention time locking allows a new column or instrument to have retention times that match the MRM database or an existing method exactly, allowing methods to be easily ported from one instrument to another and across instruments globally. This simplifies method maintenance and system setup. The retention times for the conventional 20-minute pesticide analysis are provided in the P&EP MRM database. The same GC column flow at which the 20-minute analysis was locked to the P&EP MRM database was used with the 10-minute method with the conventional 15 × 15 m configuration. This resulted in the new locking retention time for chlorpyrifos-methyl at 5.520 minutes. To update the retention times for the rest of the analytes, a combination of pesticides and n-alkanes were used to predict retention times for the new method based on the retention times for a 20-minute method from the P&EP MRM database.

The 10-minute analysis using the minibore 10 × 10 m configuration was precisely scaled using the method translation tool, providing a speed gain of 2. The fine tuning of the method enabled the best match between predicted and observed retention times across the elution range of 203 pesticides, which resulted in the 0.09 minutes offset. New retention times (RT) were calculated using the following equation:

$RT_{new} = RT_{old}/2 + 0.09$ minutes.

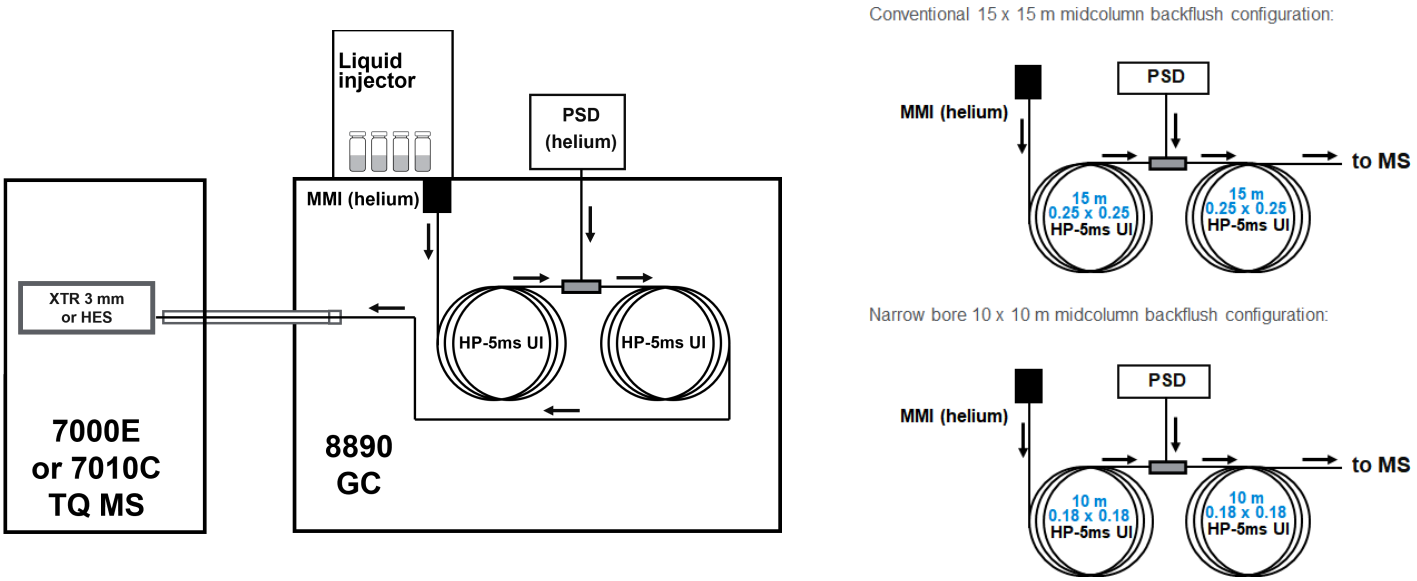


Figure 1. The Agilent GC/TQ system featuring two utilized midcolumn backflush configurations (right).

Table 1. Agilent 8890 GC and 7000 Series GC/TQ and the Agilent 8890 GC and 7010C GC/TQ system conditions enabling 10-minute pesticide analysis.

GC		
Agilent 8890 GC (220 V oven) with fast oven, auto injector, and tray		
Inlet	Multimode inlet (MMI)	
Mode	Cold splitless	
Purge Flow to Split Vent	60 mL/min at 0.75 min	
Septum Purge Flow	3 mL/min	
Septum Purge Flow Mode	Switched	
Injection Volume	1.0 µL	
Injection Type	Standard	
L1 Airgap	0.2 µL	
Gas Saver	On at 30 mL/min after 3 min	
Inlet Temperature	60 °C for 0.1 min, then to 280 °C at 600 °C/min	
Post Run Inlet Temperature	310 °C	
Post Run Total Flow	25 mL/min	
Carrier Gas	Helium	
Inlet Liner	Agilent Ultra Inert 2 mm dimpled liner	
Inlet Liner Part Number	5190-2297	
Oven		
	With 15 × 15 m	**With 10 × 10 m**
Initial Oven Temperature	60 °C	60 °C
Initial Oven Hold	1 min	0.5 min
Ramp Rate 1	80 °C/min	80 °C/min
Final Temp 1	170 °C	170 °C
Final Hold 1	0 min	0 min
Ramp Rate 2	35 °C /min	20 °C /min
Final Temp 2	310 °C	310 °C
Final Hold 2	3.625 min	1.125 min
Total Run Time	10 min	10 min
Post Run Time	1.5 min	1.5 min
Equilibration Time	0.25 min	0.25 min
		High-speed oven insert (pillow)

Column 1		
	With 15 × 15 m	**With 10 × 10 m**
Type	Agilent J&W HP-5ms Ultra Insert	Agilent J&W HP-5ms Ultra Inert
Agilent Part Number	19091S-431UI-KEY	19091S-571UI
Length	15 m	10 m
Diameter	0.25 mm	0.18 mm
Film Thickness	0.25 µm	0.18 µm
Control Mode	Constant flow	Constant flow
Flow	1.016 mL/min	1.3 mL/min
Inlet Connection	Multimode inlet (MMI)	Multimode inlet (MMI)
Outlet Connection	PSD (PUU)	PSD (PUU)
PSD Purge Flow	5 mL/min	5 mL/min
Post Run Flow (Backflushing)	–7.873	–3.174
Column 2		
	With 15 × 15 m	**With 10 × 10 m**
Type	Agilent J&W HP-5ms Ultra Inert	Agilent J&W HP-5ms Ultra Inert
Agilent Part Number	19091S-431UI-KEY	19091S-571UI
Length	15 m	10 m
Diameter	0.25 mm	0.18 mm
Film Thickness	0.25 µm	0.18 µm
Control Mode	Constant flow	Constant flow
Flow	1.216 mL/min	1.5 mL/min
Inlet Connection	PSD (PUU)	PSD (PUU)
Outlet Connection	MSD	MSD
Post Run Flow (Backflushing)	8.202	3.290

MSD		
Model	Agilent 7000 series (7000D and 7000E) or 7010C triple quadrupole GC/MS	
Source	Inert Extractor Source with a 3 mm lens or HES	
Vacuum Pump	Performance turbo	
Tune File	Atunes.eiex.jtune.xml or Atunes.eihs.jtune.xml	
Solvent Delay	3 min	
Quad Temperature (MS1 and MS2)	150 °C	
Source Temperature	280 °C	
Mode	dMRM	
He Quench Gas	2.25 mL/min	
N_2 Collision Gas	1.5 mL/min	
MRM Statistics		
	With 15 × 15 m	**With 10 × 10 m**
Total MRMs (dMRM Mode)	614	614
Minimum Dwell Time	2.33 ms	3.99 ms
Minimum Cycle Time	167.86 ms	110.38 ms
Maximum Concurrent MRMs	127	83
EM Voltage Gain Mode	10	10

Sample preparation

A sample preparation workflow chart is shown in Figure 3. The sample preparation included two major steps: sample extraction by traditional QuEChERS extraction, followed by Captiva enhanced matrix removal (EMR) pass-through cleanup. The Agilent Captiva EMR–High Chlorophyll Fresh with NH_2 (Captiva EMR–HCF1) cartridge was used for high chlorophyll fresh matrix (spinach). The new sample preparation workflow demonstrates as a simplified procedure with improvement on both sample matrix removal and targets quantitation data quality.

As shown in Figure 3, samples were first extracted using the traditional Agilent Bond Elut QuEChERS EN extraction kit (part number 5982-5650CH). Homogenized fresh spinach (10 g) was used for extraction. The 10 mL of ACN with 1% acetic acid was then added, followed by extraction. After extraction, 3 mL of crude extract was transferred to a Captiva EMR–HCF1 cartridge (part number 5610-2088) for pass-through cleanup. The sample eluent was collected and further dried by anhydrous $MgSO_4$ (part number 5982-0102). Samples were then ready for GC/TQ analysis. The Agilent positive pressure manifold 48 processor (PPM-48; part number 5191-4101) was used for Captiva EMR pass-through cleanup processing.

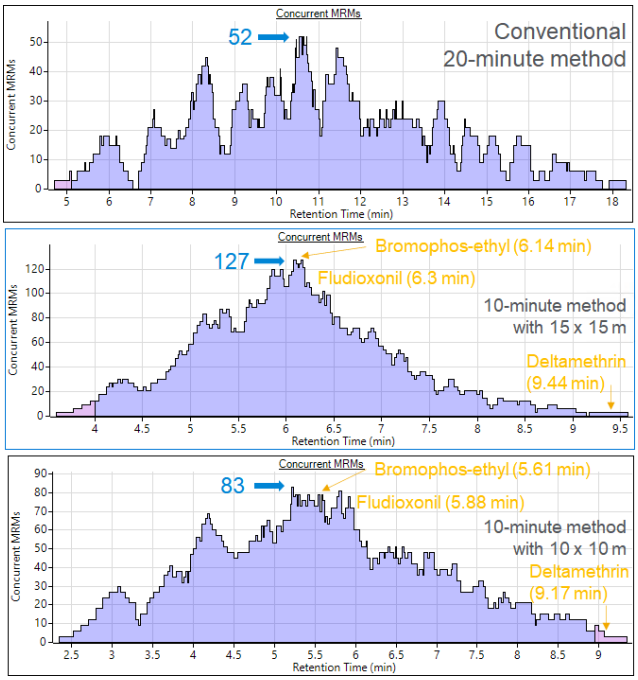


Figure 2. The distribution of 614 dMRM transitions with the 20-minute conventional pesticide analysis, the 10-minute analysis employing the conventional 15 × 15 m configuration, and the 10-minute method employing the minibore 10 × 10 m column configuration.

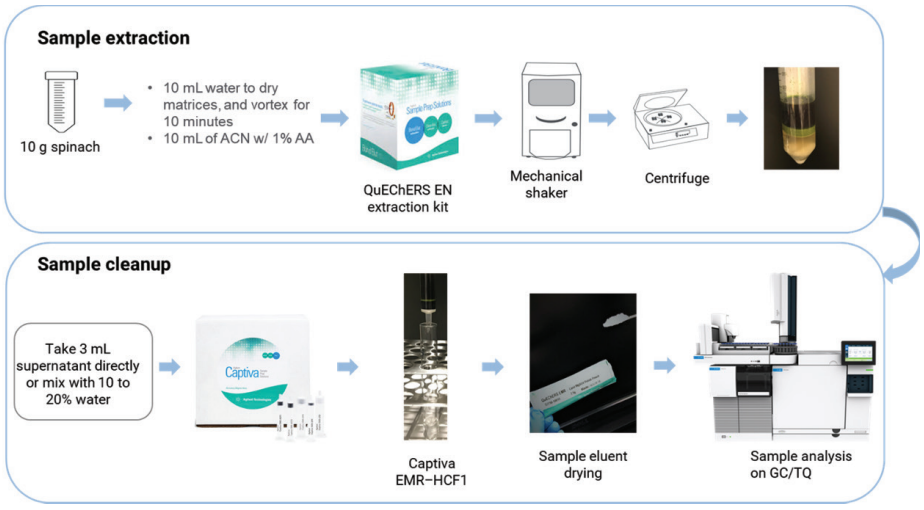


Figure 3. Sample preparation flowchart including traditional Agilent QuEChERS extraction, followed by Agilent Captiva EMR pass-through cleanup.

Results and discussion

Maintaining chromatographic resolution with the 10-minute analysis of over 200 pesticides

The presented GC midcolumn backflush configurations, including the conventional 15 × 15 m and the minibore 10 × 10 m configurations, enabled the 10-minute analysis of 203 pesticides with three MRM transitions acquired per each compound. Figure 4 demonstrates that the chromatographic resolution with the fast, 10-minute method was largely maintained with the conventional 15 × 15 m setup (Figure 4A) and completely preserved with the minibore 10 × 10 m setup (Figure 4B). The GC method translation technique used for transferring the method to the 10 × 10 m configuration allowed for preserving the relative elution order of the compounds.

Sensitivity and calibration performance over a wide dynamic range with the 10-minute separations

The method sensitivity achieved with the different column configurations and 10-minute separations was comparable to that observed with the conventional 20-minute method. Both 10-minute methods with the 15 × 15 m and the 10 × 10 m column configurations allowed for detecting all the targeted pesticides below their regulated MRLs, even for the most challenging ones. For example, deltamethrin, a challenging compound for GC/MS, was shown to be accurately quantitated in spinach down to 0.1 ppb with the 7010C GC/TQ and 1 to 5 ppb with the 7000 series GC/TQ (Figure 5A). While deltamethrin does not have an established MRL in spinach, it is regulated in many other food commodities including vegetable groups 8 and 9, and subgroups IB and IC, with the MRLs at 40 to 300 ppb.[2] The observed calibration ranges with the 7010 GC/TQ and the 7000 series GC/TQ would allow analysts to meet their analytical needs for the analysis of deltamethrin in various food matrices.

While deltamethrin is known to be challenging for GC/MS analysis, its elution at the end of the 10-minute analysis results in few concurrent MRM transitions. With only a few concurrent MRM transitions, the MRMs monitored for deltamethrin have relatively long dwell times (above 50 ms) even with the fast 10-minute methods (Figure 2). On the contrary, fludioxonil, a fungicide with an established MRL of 10 ppb in spinach[3], elutes during the crowded segment of the MRM methods with 120 and 80 concurrent MRM transitions in the 15 × 15 m method and the 10 × 10 m method configurations, respectively. Despite relatively short dwell times of 3 and 4.9 ms with the two configurations, fludioxonil was accurately quantitated down to 0.1 ppb with both the 7010C and the 7000 series GC/TQ systems with at least ten data points across the peak (Figure 5B). The 7010C GC/TQ equipped with the high efficiency source (HES) demonstrated superior sensitivity compared to the 7000 series GC/TQ. It allows for accurate quantitation below 0.1 ppb, even though this was not required in this work, as the MRLs for pesticides regulated in most food commodities by US EPA do not require sub-0.1 ppb quantitation. Similarly, bromophos-ethyl eluted in a crowded retention time window with a high number of concurrently monitored MRM transitions, leading to a short dwell time of 2.7 and 4.7 ms with the 15 × 15 m and the 10 × 10 m configurations, respectively. Bromophos-ethyl has recommended tolerances ranging from 20 to 2,000 ppb in various commodities.[4] Figures 5B and 5C demonstrate that fludioxonil and bromophos-ethyl were accurately quantitated over the wide concentration range of 0.1 to 1,000 ppb with excellent sensitivity and linearity in the challenging spinach matrix and at least nine data points across the peak.

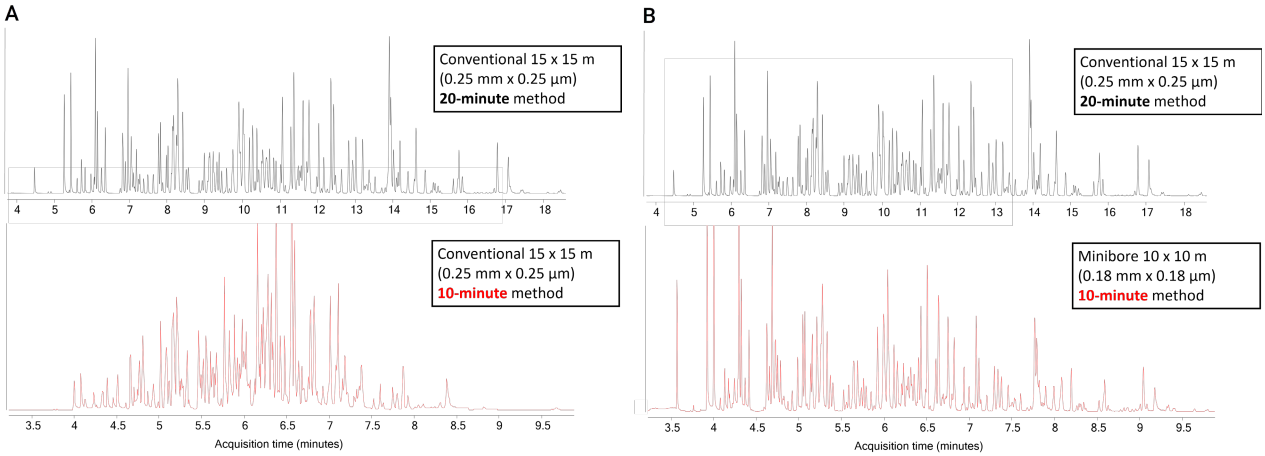
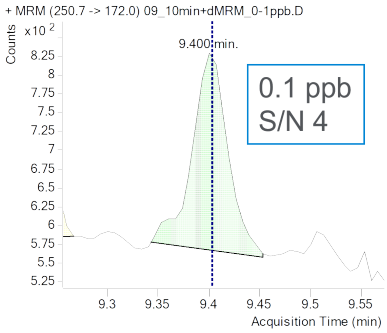
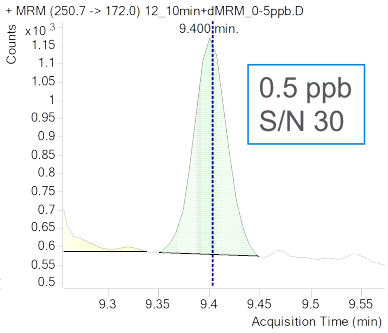


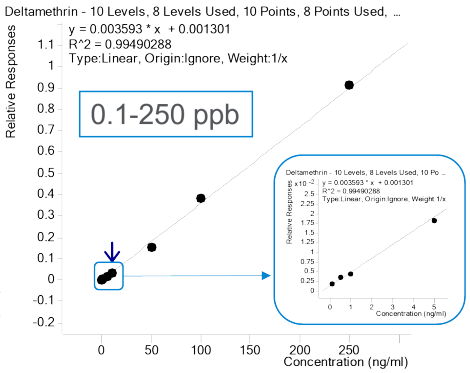
Figure 4. MRM total ion current chromatograms (TIC) or a mixture of 203 pesticides acquired with (A) the conventional 15 × 15 m configuration and (B) with the minibore 10 × 10 m configuration.

A) Deltamethrin

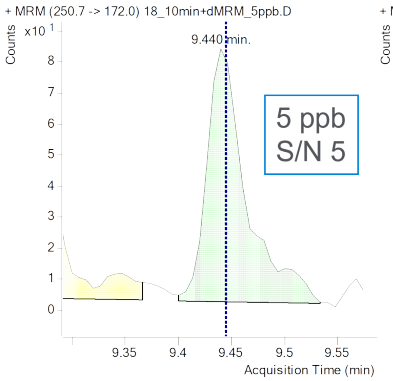
7010C
15 x 15 m

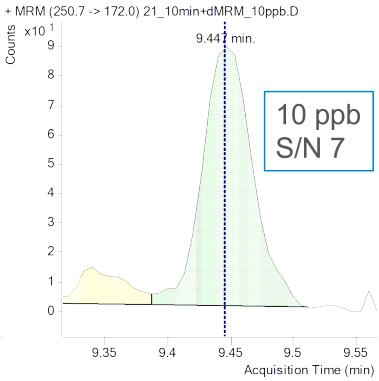


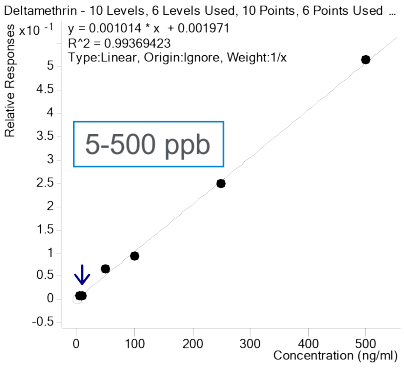




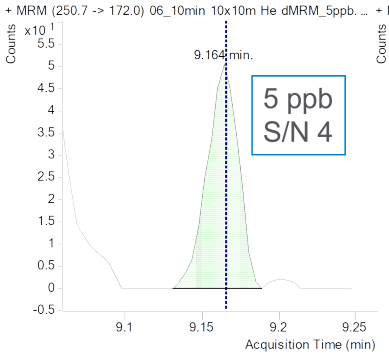
7000E
15 x 15 m

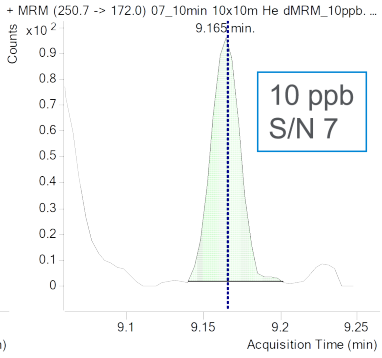


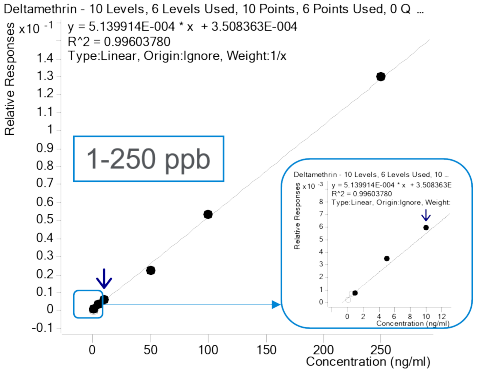




7000 series
10 x 10 m

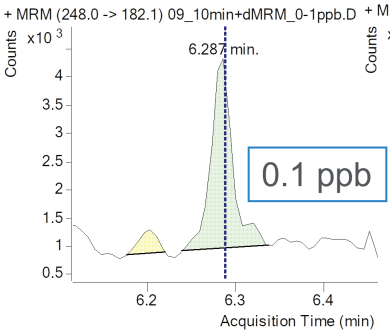


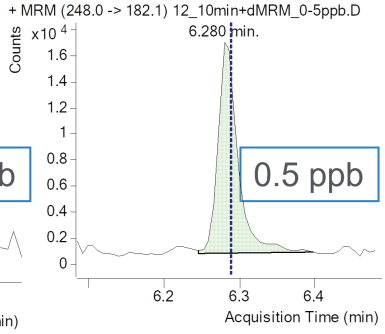


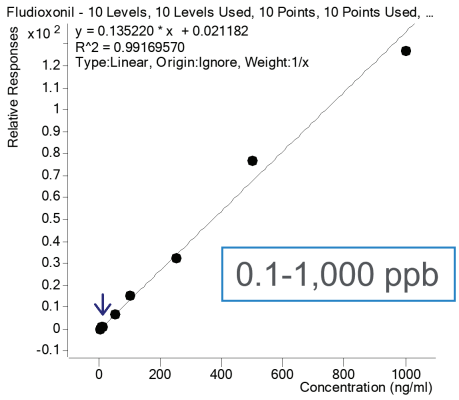


B) Fludioxonil

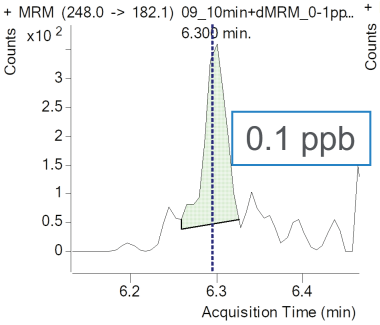
7010C
15 x 15 m

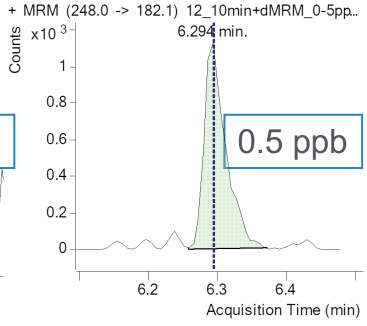


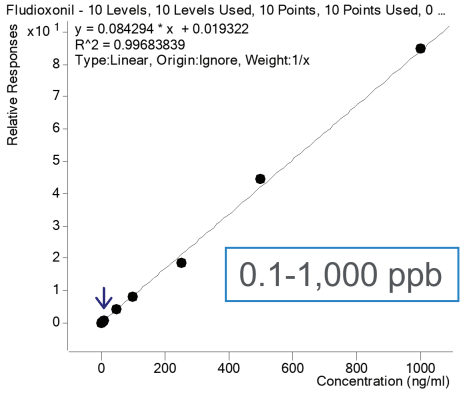




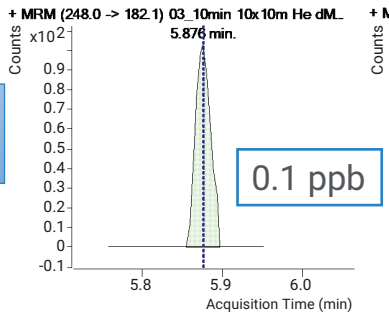
7000E
15 x 15 m

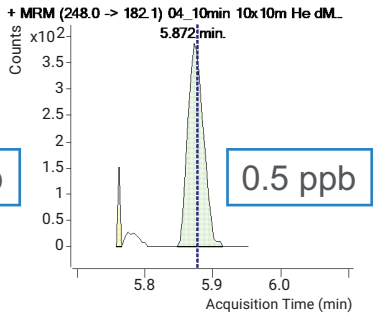


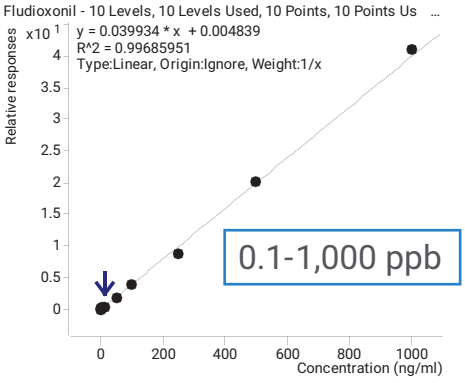




7000 series
10 x 10 m







C) Bromophos-ethyl

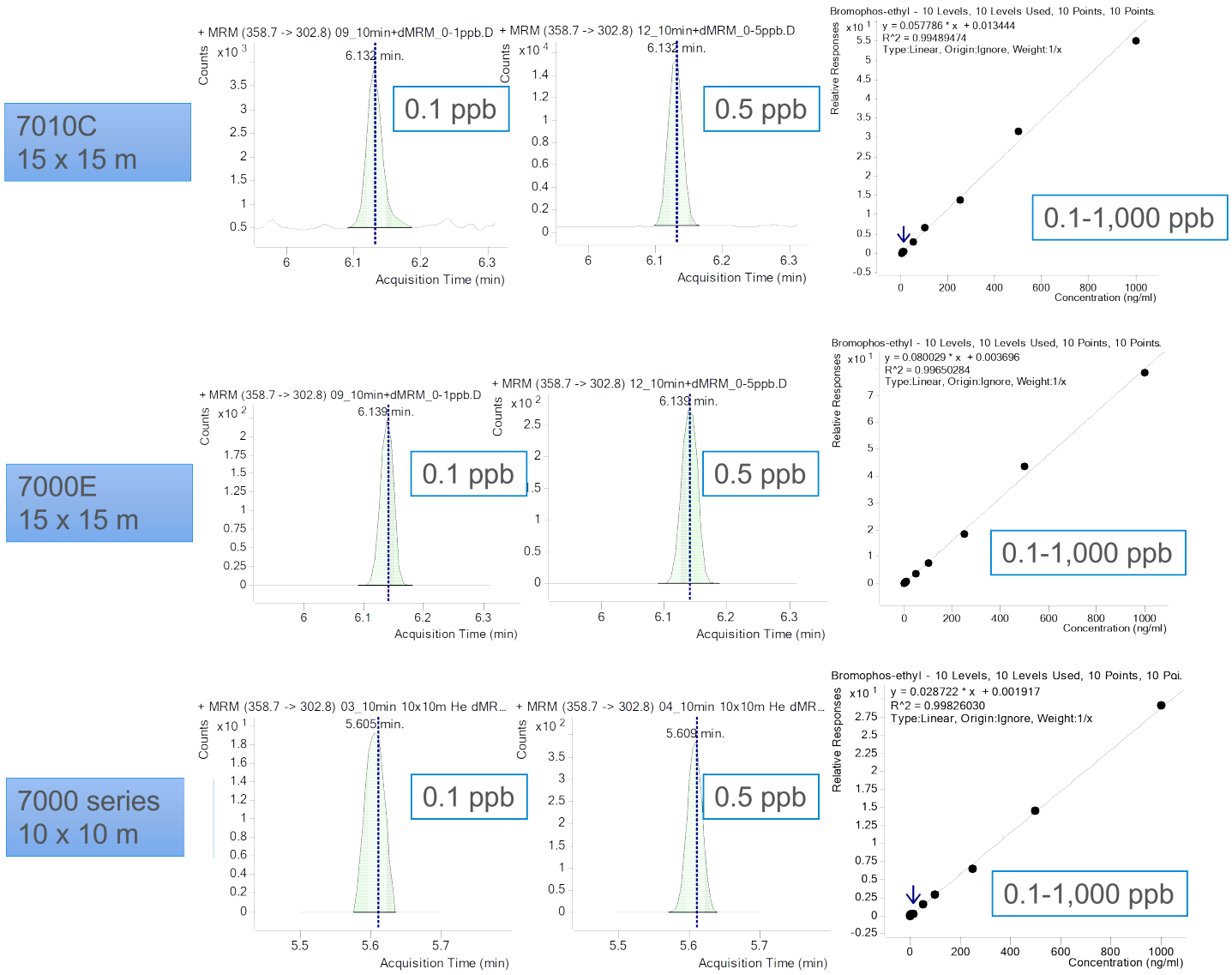


Figure 5. MRM chromatograms and matrix-matched calibration curves in spinach for (A) deltamethrin, (B) fludioxonil, and (C) bromophos-ethyl observed with different column configurations and 10-minute separations using the Agilent 7010C and 7000 series triple quadrupole GC/MS systems.

The biggest challenge with multiresidue pesticide analysis is that the MRLs established for pesticides in different food commodities vary significantly. This may require undesirable sample re-injection if the method calibration ranges do not encompass all the MRLs for the compounds of interest. A broad dynamic calibration range is desirable to use the more generic quantitation method for analyzing different pesticides in the commodity and for various foods and to simplify the sample pretreatment before instrument detection, such as further dilution. Figure 6 summarizes the calibration performance for the 203 pesticides that were analyzed in spinach with the 10-minute separations using the conventional 15 × 15 m configuration coupled with the 7010C and the 7000E GC/TQ, and the minibore 10 × 10 m configuration coupled with the 7000 series GC/TQ. The graph shows the number of compounds with the calibration correlation coefficient $R^2 > 0.99$, using the different regression fit (linear or quadratic), within the different calibration ranges.

Most of the target compounds demonstrated linear calibration curves over a wide range of either 0.1 to 1,000 ppb or 0.5 to 1,000 ppb, enabling their reliable quantitation at the varying MRLs established for different compounds.

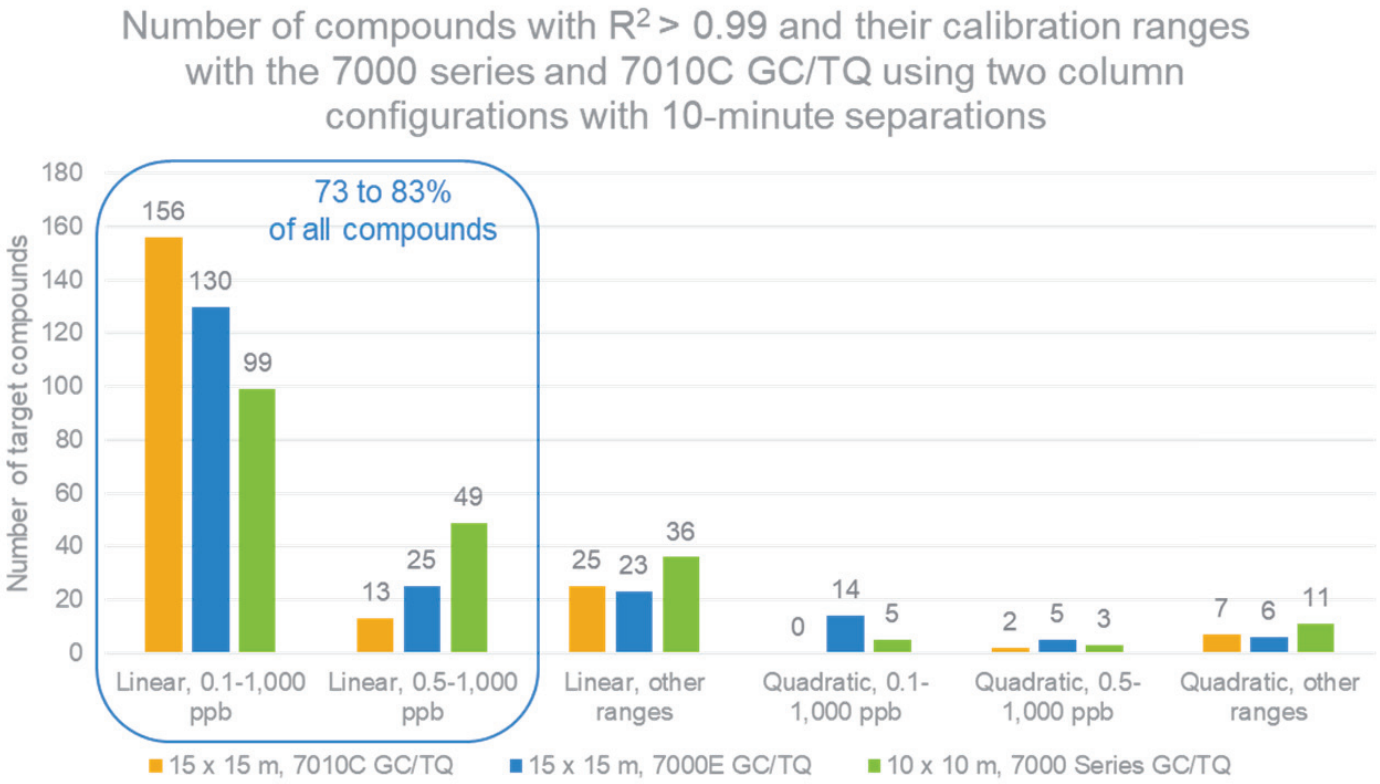


Figure 6. Calibration performance for the 203 pesticides with the 10-minute methods using the conventional 15 × 15 m configuration, coupled with the Agilent 7010C and 7000E triple quadrupole GC/MS systems, and the minibore 10 × 10 m configuration, coupled with the Agilent 7000 series triple quadrupole GC/MS in spinach. The graph shows the number of compounds and their calibration ranges.

Method robustness with 700 injections of a spinach extract

The robustness of the 10-minute analysis was demonstrated by analyzing a challenging, highly pigmented spinach extract spiked with pesticides at 20 ppb. The area of the analytes was monitored over 700 consecutive injections. Analyte response, normalized by the internal standards (ISTD), remained consistent over 700 injections that spanned over 175 hours of continuous running with the 10-minute method, using the conventional 15 × 15 m column configuration coupled with the 7000E GC/TQ. The only maintenance procedure performed during the robustness testing involved septum and liner replacement every 100 injections.

There was no need to perform inlet cleaning, GC column trimming, or MS source cleaning, or retune the MS during the entire study that involved over 1,000 injections (robustness testing over 700 runs and additional analyses performed for system evaluation and calibration).

The keys to successful and robust pesticide analysis that enables stable GC/TQ performance for over 700 injections are described in the application note 5994-4965EN.[5] The best practices used in this work included:

- Simplified and improved sample preparation achieved with the novel and improved Captiva EMR pass-through cleanup following traditional QuEChERS extraction
- Evaluation of in-source loading of the matrix in full scan data acquisition mode
- Postrun backflushing enabled with the conventional 15 × 15 m and the minibore 10 × 10 m midcolumn backflush configurations
- Leak-free GC/TQ system enabled with the self-tightening collared column nuts and CFT gold-plated flexible metal ferrules
- Use of temperature-programmed MMI with a 2 mm Ultra Inert dimpled liner (no glass wool)

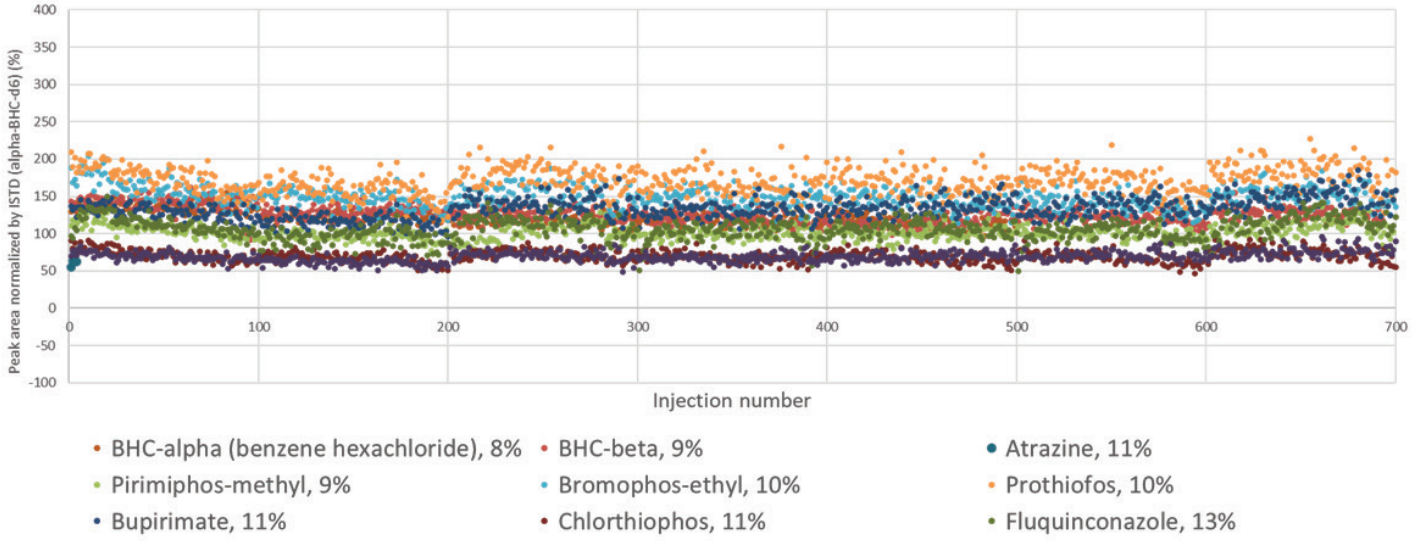


Figure 7. Stability of the peak area for pesticides spiked at 20 ppb into spinach extract, normalized by the ISTD, over 700 consecutive injections. The 10-minute analysis using the conventional 15 × 15 m column configuration coupled with the Agilent 7000E triple quadrupole GC/MS.

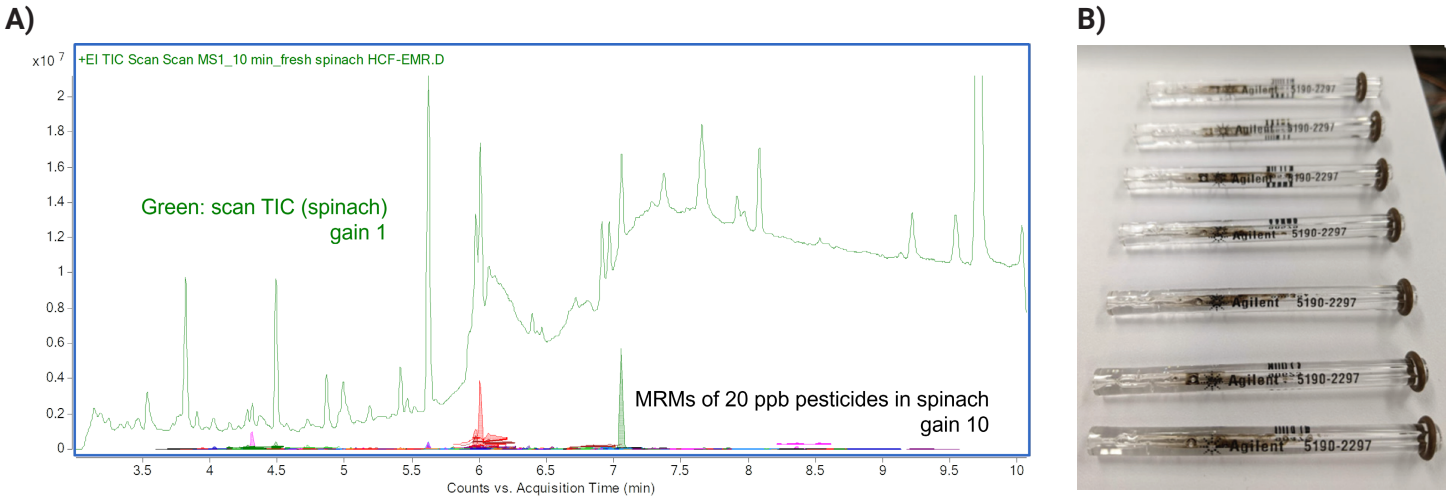


Figure 8. (A) TIC of a full scan chromatogram acquired for spinach extract and the MRM TIC for 20 ppb pesticides. (B) The GC inlet liners replaced after 100 injections when analyzing spinach extract during the robustness evaluation.

Highly pigmented spinach extract selected for the robustness testing was demonstrated to have a relatively high background in full scan data acquisition mode, as shown in Figure 8A, compared to the abundance of the MRM signal for pesticides at 20 ppb. The liners replaced after 100 injections, seven times during the robustness study, are shown in Figure 8B. This indicates that spinach extract truly presents a challenge for GC/MS analysis, hence, served as a suitable matrix for robustness performance evaluation.

Conclusion

This application note described two GC/TQ system configurations using midcolumn backflush that both enable robust pesticide analysis in 10 minutes, while maintaining sufficient chromatographic resolution for 203 compounds. The conventional 15 × 15 m (0.25 mm × 0.25 µm) and the minibore 10 × 10 m (0.18 mm × 0.18 µm) midcolumn backflush configurations were used to achieve a 10-minute analysis time. Results demonstrate that excellent linearity, over a calibration dynamic range of 0.1 to 1,000 ppb or 0.5 to 1,000 ppb, was achieved with the Agilent 7010C and 7000 series triple quadrupole GC/MS systems. Method robustness was shown with 700 consecutive injections of spinach extract spiked with pesticides at 20 ppb.

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Appendix 1

Compounds analyzed in this work and their observed retention times with two-column configurations and 10-minute separations.

Name	Retenion Time (min)		Name	Retenion Time (min)	
	15 × 15 m	10 × 10 m		15 × 15 m	10 × 10 m
Allidochlor	3.773	2.542	BHC-gamma (Lindane, gamma HCH)	5.201	4.174
Dichlorobenzonitrile, 2,6-	3.972	2.720	Pyrimethanil	5.222	4.246
Biphenyl	4.055	2.812	Tefluthrin	5.223	4.310
Mevinphos, E-	4.110	2.901	Fonofos	5.225	4.223
3,4-Dichloroaniline	4.193	2.954	Pentachloronitrobenzene	5.227	4.210
Pebulate	4.223	3.006	Pentachlorobenzonitrile	5.247	4.228
Etridiazole	4.246	3.016	Disulfoton	5.273	4.312
N-(2,4-dimethylphenyl)formamide	4.305	3.091	Isazofos	5.285	4.361
cis-1,2,3,6-Tetrahydrophthalimide	4.312	3.090	Terbacil	5.285	4.323
Methacrifos	4.321	3.129	Triallate	5.322	4.379
Chloroneb	4.375	3.171	BHC-delta	5.330	4.351
2-Phenylphenol	4.444	3.228	Chlorothalonil	5.350	4.392
Pentachlorobenzene	4.495	3.276	Propanil	5.463	4.570
Propachlor	4.702	3.546	Endosulfan ether	5.466	4.523
Tecnazene	4.712	3.547	Transfluthrin	5.476	4.658
Diphenylamine	4.734	3.582	Dimethachlor	5.477	4.596
Cycloate	4.757	3.626	Pentachloroaniline	5.482	4.552
Chlorpropham	4.769	3.656	Acetochlor	5.502	4.641
2,3,5,6-Tetrachloroaniline	4.793	3.633	Vinclozolin	5.503	4.654
Trifluralin	4.798	3.724	Parathion-methyl	5.526	4.668
Benfluralin	4.811	3.740	Chlorpyrifos-methyl	5.526	4.668
Ethalfluralin	4.812	3.670	Tolclofos-methyl	5.559	4.710
Sulfotep	4.869	3.789	Alachlor	5.564	4.725
Diallate I	4.928	3.846	Propisochlor	5.579	4.765
Phorate	4.932	3.852	Metalaxyl	5.583	4.763
BHC-beta	5.010	4.115	Ronnel	5.614	4.791
BHC-alpha (benzene hexachloride)	5.011	3.918	Prodiamine	5.622	4.871
Hexachlorobenzene	5.069	3.987	Heptachlor	5.630	4.763
Atrazine	5.072	4.048	Pirimiphos-methyl	5.650	4.892
Dichloran	5.072	3.998	Fenitrothion	5.676	4.891
Pentachloroanisole	5.083	4.013	Malathion	5.696	4.962
Clomazone	5.122	4.092	Linuron	5.708	4.927
Profluralin	5.123	4.156	Dichlofluanid	5.745	4.980
Terbuthylazine	5.155	4.163	Pentachlorothioanisole	5.767	4.972
Terbufos	5.173	4.178	Aldrin	5.768	5.061
Propyzamide	5.175	4.188	Fenthion	5.779	5.057
Diazinon	5.191	4.244	Metolachlor	5.783	5.046
Fluchloralin	5.199	4.261	Chlorpyrifos	5.790	5.075

Name	Retenion Time (min)		Name	Retenion Time (min)	
	15 × 15 m	10 × 10 m		15 × 15 m	10 × 10 m
Parathion	5.793	5.081	Chlorfenson	6.275	5.784
Triadimefon	5.811	5.100	Nonachlor, trans-	6.279	5.787
DCPA (Dacthal, Chlorthal-dimethyl)	5.829	5.124	Dieldrin	6.279	5.955
Anthraquinone	5.831	5.053	Fludioxonil	6.294	5.876
Dichlorobenzophenone, 4,4'-	5.840	5.110	Prothiofos	6.300	5.844
Pirimiphos-ethyl	5.869	5.241	Oxadiazon	6.303	5.920
MGK-264	5.881	5.315	Pretilachlor	6.303	5.895
Isopropalin	5.898	5.267	Iodofenphos	6.304	5.828
Fenson	5.902	5.194	Profenofos	6.312	5.877
Diphenamid	5.908	5.235	Oxyfluorfen	6.314	5.960
Bromophos	5.918	5.237	DDE-p,p'	6.342	5.906
Cyprodinil	5.941	5.314	Bupirimate	6.361	6.014
Pendimethalin	5.975	5.356	Myclobutanil	6.364	5.970
Chlozolinate	5.976	5.378	Chlorfenapyr	6.365	6.122
Allethrin	5.979	5.393	Flusilazole	6.370	5.995
Triflumizole	5.979	5.473	Fluazifop-p-butyl	6.388	6.090
Fipronil	5.993	5.431	DDD-o,p'	6.404	5.990
Penconazole	5.998	5.375	Tricyclazole	6.412	5.932
Metazachlor	5.999	5.358	Endrin	6.423	6.153
Chlorfenvinphos	6.016	5.436	Ethylan	6.453	6.121
Heptachlor exo-epoxide	6.016	5.402	Nitrofen	6.477	6.101
Isodrin	6.018	5.319	Chlorobenzilate	6.506	6.189
Captan	6.020	5.472	Ethion	6.571	6.315
Tolylfluanid	6.026	5.413	DDD-p,p'	6.582	6.280
Bromfenvinfos-methyl	6.036	5.436	DDT-o,p'	6.582	6.318
Quinalphos	6.047	5.463	Chlorthiophos	6.587	6.338
Triadimenol	6.053	5.476	Endosulfan II (beta isomer)	6.603	6.235
Procymidone	6.090	5.515	Triazophos	6.644	6.428
Folpet	6.127	5.513	Sulprofos	6.659	6.420
Paclobutrazol	6.137	5.653	Nonachlor, cis-	6.667	6.341
Chlorbenside	6.137	5.549	Carfentrazone-ethyl	6.668	6.509
Bromophos-ethyl	6.139	5.609	Methoxychlor olefin	6.702	6.519
DDE-o,p'	6.176	5.631	Endrin aldehyde	6.709	6.402
Tetrachlorvinphos	6.181	5.680	Carbophenothion	6.726	6.513
Chlordane-trans	6.187	5.610	Norflurazon	6.754	6.576
Chlordane-cis	6.196	5.744	Edifenphos	6.786	6.566
Fenamiphos	6.227	5.797	Lenacil	6.787	6.588
Flutolanil	6.233	5.801	DDT-p,p'	6.805	6.615
Bromfenvinfos	6.252	5.800	Iprodione	6.826	6.947
Flutriafol	6.255	5.764	Methoxychlor, o,p'-	6.846	6.703
Endosulfan I (alpha isomer)	6.274	5.724	Endosulfan sulfate	6.852	6.610

Name	Retenion Time (min)		Name	Retenion Time (min)	
	15 × 15 m	10 × 10 m		15 × 15 m	10 × 10 m
Piperonyl butoxide	6.854	6.788	Acrinathrin	7.415	7.607
Propargite	6.856	6.760	Leptophos	7.417	7.413
Resmethrin	6.857	6.756	Pyrazophos	7.556	7.660
Hexazinone	6.861	6.708	Fenarimol	7.631	7.641
Tebuconazole	6.886	6.739	Mirex	7.636	7.533
Captafol	6.890	6.805	Pyraclofos	7.645	7.728
Nitralin	6.913	6.862	Azinphos-ethyl	7.675	7.700
Bifenthrin	7.044	7.057	Permethrin, (1R)-cis-	7.785	7.901
Pyridaphenthion	7.048	7.004	Permethrin, (1R)-trans-	7.842	7.962
Tetramethrin I	7.052	6.999	Pyridaben	7.916	7.980
Fenpropathrin	7.106	7.121	Coumaphos	7.964	8.028
Bromopropylate	7.109	7.061	Fluquinconazole	7.964	8.023
EPN	7.112	7.061	Prochloraz	7.988	8.058
Tebufenpyrad	7.130	7.152	Cyfluthrin I	8.157	8.184
Methoxychlor, p,p'-	7.131	7.111	Cypermethrin I	8.250	8.339
Phosmet	7.135	7.054	Flucythrinate I	8.359	8.444
Endrin ketone	7.189	7.033	Acequinocyl	8.409	8.534
Phenothrin I	7.230	7.243	Ethofenprox	8.431	8.485
Azinphos-methyl	7.330	7.405	Fluridone	8.708	8.662
Tetradifon	7.330	7.305	Fenvalerate I	8.881	8.799
Cyhalothrin (Lambda)	7.334	7.438	Fluvalinate-tau I	8.970	8.894
Pyriproxyfen	7.358	7.406	Deltamethrin	9.444	9.166
Phosalone	7.389	7.387			

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