Application Note

Food Testing and Agriculture

Quantitation of Pesticides in Strawberries at Tolerance Levels Established by the US EPA

Using Agilent 8890/7000D and 8890/7010B triple quadrupole GC/MS systems

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Abstract

To test the suitability for the determination of pesticides in strawberries at the tolerances established by the US EPA, which vary between 10 and 25,000 ppb, the Agilent 8890 gas chromatograph was coupled to Agilent 7000D and 7010B triple quadrupole mass spectrometers. The instrument configuration was selected to facilitate an optimized pulsed splitless injection and midcolumn backflush, while using retention time locking (RTL) to a database of pesticides and environmental pollutants. Samples of strawberries were purchased from local grocery stores, and were used to demonstrate the capabilities of the workflow. A target list of pesticides quantitated in 12 different sources of strawberries was based on the screening results of these samples with the Agilent 5977 Series GC/MSD and the Agilent 7250 GC/Q-TOF systems.

Introduction

Concern about trace-level food and environmental pollutants is driving the demand for more rapid and reliable methods for the identification and quantitation of chemical residues. Strawberries are known to be challenging to grow commercially because of their sensitivity to poor quality water and soils, diseases, and pests. This Application Note focuses on the quantitation of GC-amenable incurred pesticides from randomly sourced commercial strawberry samples. The target list reported was based on initial screening with the 8890 coupled to the 5977 GC/MSD or 8890 coupled to the 7250 GC/Q-TOF system.

The tolerances for the maximum concentration of a pesticide residue in strawberries established by the US EPA vary in a broad concentration range.[1,2] Thus, pesticide quantitation requires a broad calibration range, while maintaining high accuracy at tolerance levels.

The analysis of pesticides in foods faces the challenge of matrix-related problems, which may cause interferences. Additionally, the high-boiling matrix that elutes after the analytes can require extended bakeout times to prevent carryover and ghost peaks in subsequent runs. The highest boiling contaminants can deposit in the head of the column, requiring more frequent column trimming and adjustment of MRM and data analysis time windows from the resulting retention time shift. Following a QuEChERS sample preparation procedure, and using midcolumn backflush and RTL, quantitation of pesticides over a broad concentration range can easily be achieved.

Experimental

The systems used were configured to increase the ease and speed of setting up a targeted MRM method, minimize the potential problems with pesticide analysis following QuEChERS extraction, and improve sample throughput while maintaining adequate sensitivity. The important techniques used are:

- **Midcolumn backflushing** is a technique in which the carrier gas flow is reversed after the last analyte has exited the column. After the MS data are collected, the oven is held at the final temperature in post run mode, and the carrier gas flow through the first column is reversed. This reversed flow carries any high boilers that were in the column at the end of data collection out of the head of the column and into the split vent trap. The ability to reverse the flow is provided by the Agilent Purged Ultimate Union (PUU). The PUU is a tee that is inserted, in this case, between two identical 15 m columns. During the analysis, a small makeup flow of carrier gas from the 8890 pressure switching device (PSD) module is used to sweep the connection. During backflushing, the makeup flow from the PSD is raised to a much higher value, sweeping high boilers backwards out of the first column and forwards from the second. For the configuration in this application, the backflushing time was 1.5 minutes.
- **Pulsed splitless injection** maximizes the transfer of analytes from the inlet onto the GC column, minimizing the residence time and breakdown in the inlet.
- **The PSD** is an 8890 pneumatics module optimized for backflushing applications. At high pressures during backflushing, the fixed restrictor can have hundreds of mL/min of wasted flow. The PSD will stay at the user-defined setpoint (default 3 mL/min) even at high pressures, which significantly reduces the required gas flow. Also, when the PSD is present in a midcolumn backflush configuration, the setup for pulsed splitless mode is simplified as the column flow for both column 1 and column 2 will be increased respectively during the pulse.
- **The Extractor EI Source** delivers high sensitivity, inertness, and wide calibration range.
- **The High Efficiency Source (HES)** creates up to 20x more ions than the Extractor, and delivers confident analysis at ultra-trace levels.
- **The Agilent MassHunter Pesticide & Environmental Pollutant MRM Database (P&EP 4)** provides up to eight MRM transitions per analyte, allowing users to build acquisition methods without having to develop MRMs. The database includes retention times for constant flow 20 and 40-minute GC oven programs.
- **Dynamic MRM mode** allows the creation of large multi-analyte assays by automatically determining the most efficient distribution of dwell times.
- **RTL** allows a new column or instrument to have retention times that precisely match the MRM database, greatly simplifying method maintenance.

Figure 1 shows the system configuration used.

Table 1 lists the instrument operating parameters. Isothermal, pulsed splitless injections were used to increase sample throughput for the determined target list, and to maximize transfer of the pesticides, especially the active ones, into the column. Initially, problems with analyte peak shapes were encountered due to the use of acetonitrile as the injection solvent. Acetonitrile is known to be troublesome with splitless injections into seminonpolar columns (often, a solvent vent mode of injection is used to avoid peak tailing).[3]

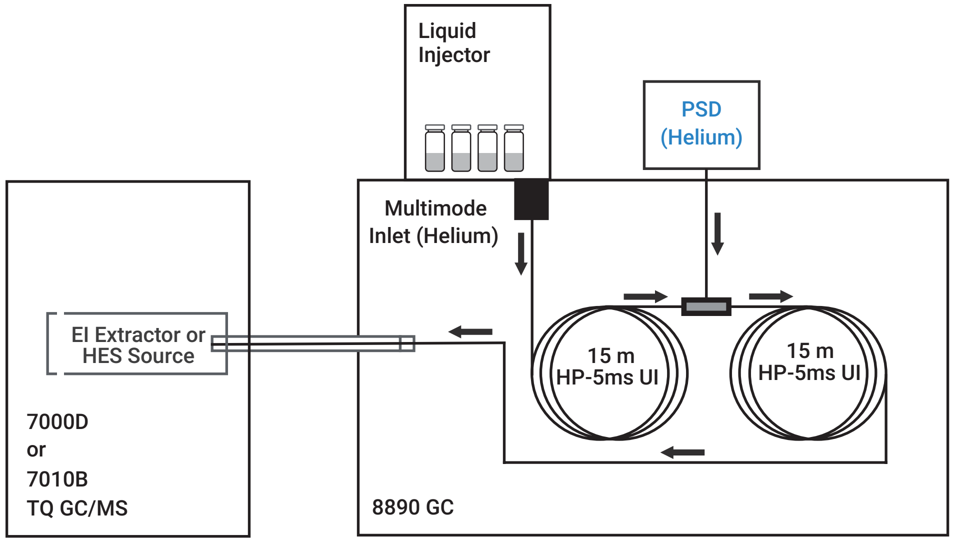

Figure 1. System configuration.

Table 1. GG/MS conditions for pesticide quantitation.

8890 GC With Fast Oven, Autoinjector, and Tray	
Multimode Inlet (MMI)	
Mode	Pulsed Splitless
Injection Pulse Pressure	50 psi until 0.75 minutes
Purge Flow to Split Vent	50 mL/min at 0.7 minutes
Septum Purge Flow Mode	Switched
Injection Volume	1.0 µL
Injection Type	Standard
L1 Airgap	0.2 µL
Inlet Temperature	280 °C
Carrier Gas	Helium
Inlet Liner	Agilent universal low pressure drop liner, with glass wool (p/n 5190-2295)
Oven	
Initial Oven Temperature	60 °C
Initial Oven Hold	1 minute
Ramp Rate 1	40 °C/min
Final Temperature 1	120 °C
Final Hold 1	0 minutes
Ramp Rate 2	5 °C/min
Final Temperature 2	310 °C
Final Hold 2	0 minutes
Total Run Time	40.5 minutes
Post Run Time	1.5 minutes
Equilibration Time	0.5 minutes

Columns	
Column 1	HP-5MS UI, 15 m × 0.25 mm, 0.25 µm (p/n 19091S-431UI)
Control Mode	Constant Flow
Flow	1.042 mL/min
Inlet Connection	MMI
Outlet Connection	PSD (PUU)
Post Run Flow (Backflushing)	–12.906 mL/min
Column 2	HP-5MS UI, 15 m × 0.25 mm, 0.25 µm (p/n 19091S-431UI)
Control Mode	Constant Flow
Flow	1.242 mL/min
Inlet Connection	PSD (PUU)
Outlet Connection	MSD
Post Run Flow (Backflushing)	13.429 mL/min
MSD	
Model	7000D or 7010B
Source	Inert Extractor Source with 3 mm drawout lens or HES
Vacuum Pump	Performance Turbo
Tune File	Atunes.eiex.tune.xml or Atunes.eihs.tune.xml
Mode	dMRM
Solvent Delay	3 minutes
EM voltage Gain Mode	10
Quad Temperature (MS1 and MS2)	150 °C
Source Temperature	280 °C
Transfer line Temperature	280 °C
He Quench Gas	2.25 mL/min
N_2 Collision Gas	1.5 mL/min

The Agilent 5190-2293 single taper UI splitless liner (top of Figure 2) is widely used for splitless injection, and works well with most common GC solvents. However, with acetonitrile, pulsed splitless injections produced multiple peaks for each analyte. As an alternative, the Agilent 5190-2295 UI Universal Low Pressure Drop Liner (bottom of Figure 2) was found to eliminate the problem and was used for all subsequent analyses.

Table 2 provides the compound list with the retention times and suggested quantifying and qualifying MRM transitions. The transitions were sourced from the Agilent MassHunter Pesticide & Environmental Pollutant MRM Database (P&EP 4).

Strawberry samples and calibration standard preparation

Twelve different packages of organic and nonorganic strawberries were purchased at local retail stores as well as at farmers' markets in the Cupertino, CA area. Strawberries were cut into small pieces, frozen, and blended under liquid nitrogen (organic samples were blended first). A QuEChERS sample preparation was used as follows: Ten grams of each sample were weighed into a 50 mL centrifuge tube. Two ceramic homogenizers were added to each centrifuge tube, followed by the addition of 10 mL of acetonitrile (HPLC grade) to each tube. Samples were mechanically shaken for three minutes at 1,500 strokes/min. An EN Method 15662 QuEChERS extraction salt packet (p/n 5982-6650) was added to each centrifuge tube. Samples were mechanically shaken for three minutes at 1,500 strokes/min, then centrifuged for five minutes at 5,000 rpm. A 6 mL aliquot of the extract was transferred to a QuEChERS Dispersive SPE 15 mL tube (general fruits and vegetables, p/n 5982-5056). Samples were vortexed for three minutes at 1,500 strokes/min, then centrifuged for five minutes at 5,000 rpm. The sample extracts were transferred to labeled autosampler vials for analysis.

Matrix-matched calibration standards were prepared by spiking pesticides in blank matrix extract. The blank matrix extract was from one of the organic strawberry samples that had none of the pesticides identified in the preliminary screening. Calibration solutions were prepared to cover a range of 1 to 10,000 ppb, where required, to encompass the tolerances for the maximum concentration of a pesticide residue in strawberries.

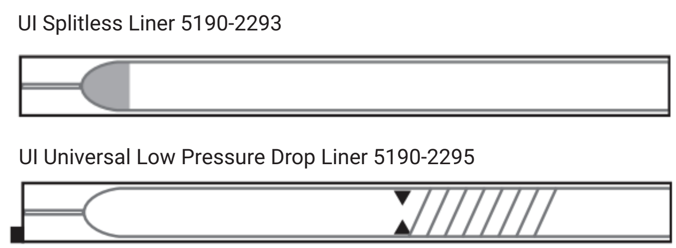

Figure 2. Liners evaluated for pulsed splitless injection.

Table 2. MRM transitions used for quantifier and qualifiers.

Name	Classification	RT (min)	Quantifier	Qualifier 1	Qualifier 2	Qualifier 3	Qualifier 4
Acetamiprid	I	27.890	152.0 → 116.1	126.0 → 73.0	126.0 → 99.0	126.0 → 90.0	152.0 → 62.0
Allethrin	I	21.638	123.0 → 81.0	91.0 → 65.0	107.0 → 91.0	136.0 → 93.0	107.0 → 78.9
Azoxystrobin	F	37.103	344.1 → 329.0	344.1 → 171.9	344.1 → 182.9	344.1 → 155.8	387.9 → 360.0
Bifenazate	I	28.337	184.1 → 77.0	152.1 → 127.1	211.1 → 183.1	211.1 → 155.1	211.1 → 141.0
Bifenthrin	I	28.311	181.2 → 165.2	166.2 → 165.2	165.2 → 115.1	182.2 → 167.2	
Boscalid	F	33.394	140.0 → 76.0	140.0 → 112.0	111.9 → 76.0	341.9 → 139.9	341.9 → 111.8
Captan	F	21.414	151.0 → 79.0	149.0 → 77.1	148.1 → 70.0	263.9 → 79.0	149.0 → 79.1
Carbaryl	I	18.243	144.1 → 116.1	115.1 → 89.0	144.1 → 89.0	115.1 → 65.0	144.1 → 65.0
Carbofuran	I	15.170	164.2 → 149.1	149.1 → 77.1	164.2 → 103.1	149.1 → 121.1	149.1 → 103.1
Chlorantraniliprole	I	28.347	278.0 → 249.0	278.0 → 215.0			
cis-1,2,3,6-Tetrahydrophthalimide (THPI)	F*	9.913	151.1 → 80.0	79.0 → 51.0	79.0 → 77.0	151.1 → 122.1	151.1 → 106.1
Cyprodinil	F	20.897	225.2 → 224.3	224.2 → 208.2	226.2 → 225.3	225.2 → 210.3	224.2 → 131.1
p,p'-DDE	I*	23.414	246.1 → 176.2	315.8 → 246.0	317.8 → 248.0	317.8 → 246.0	176.0 → 150.1
Ethiofencarb	I	17.325	167.9 → 107.1	107.0 → 77.1	107.0 → 79.1	167.9 → 77.0	108.0 → 78.1
Etoxazole	I	28.619	141.0 → 63.1	141.0 → 113.0	204.0 → 176.1	299.9 → 269.9	299.9 → 284.9
Fenhexamid	F	26.187	97.1 → 55.1	177.1 → 78.0	177.1 → 113.0	179.0 → 78.0	179.0 → 115.0
Fenobucarb	I	12.472	121.0 → 103.1	121.0 → 77.0	149.9 → 121.1	121.0 → 93.1	102.9 → 77.0
Flonicamid	I	12.386	174.0 → 146.0	174.0 → 126.0			
Fludioxonil	F	23.383	248.0 → 127.1	248.0 → 182.1	248.0 → 154.1	154.0 → 127.1	182.0 → 154.1
Fluridone	H	34.560	328.9 → 328.1	328.0 → 258.9	328.0 → 312.8	328.9 → 258.7	328.9 → 312.7
Flutriafol	F	22.730	123.1 → 95.0	123.1 → 75.1	219.1 → 123.1	219.1 → 95.0	164.1 → 95.0
Isoprocarb I	I	11.093	121.0 → 77.1	136.0 → 121.1	121.0 → 103.1	136.0 → 77.1	121.0 → 91.1
Malathion	I	19.634	126.9 → 99.0	172.9 → 99.0	157.8 → 125.0	172.9 → 117.0	157.8 → 47.0
Metalaxyl	F	18.620	234.0 → 146.1	206.1 → 132.1	234.0 → 174.1	220.0 → 192.1	248.8 → 190.1
Metaldehyde	M	4.104	89.0 → 45.0	117.0 → 45.0			
Myclobutanil	F	23.724	179.0 → 125.1	179.0 → 90.0	150.0 → 123.0	206.0 → 179.1	244.9 → 125.0
Novaluron	I	6.479	168.0 → 139.9	168.0 → 75.9	335.0 → 167.9	168.0 → 112.0	139.9 → 75.9
Piperonyl butoxide	PS	27.225	176.1 → 103.1	176.1 → 131.1	176.1 → 117.1	149.1 → 65.1	177.0 → 119.1
Propargite	A	27.048	135.0 → 77.1	149.9 → 135.1	135.0 → 107.1	230.9 → 135.1	149.9 → 107.1
Pyrimethanil	F	16.132	198.0 → 118.1	198.0 → 183.1	198.0 → 158.1	198.9 → 184.0	117.9 → 91.0
Quinoxyfen	F	26.039	237.0 → 208.1	271.9 → 237.1	306.8 → 237.0	306.8 → 271.9	308.8 → 237.0
Tetraconazole	F	20.351	336.0 → 217.9	170.9 → 136.0	336.0 → 203.8	170.9 → 99.0	158.9 → 89.0
Thiabendazole	F	21.220	201.0 → 174.0	201.9 → 175.0	173.9 → 65.0	128.9 → 102.0	201.0 → 130.0
Thiamethoxam	I	20.583	212.0 → 139.0	212.0 → 125.0			
Trifloxystrobin	F	26.492	116.0 → 89.0	172.0 → 145.1	116.0 → 63.0	131.0 → 89.0	186.0 → 145.1
3,4,5-Trimethacarb	I	14.950	121.0 → 77.1	136.0 → 77.1	136.0 → 121.1	121.0 → 91.0	135.0 → 91.0
Tris(1-Chloro-2-propyl) phosphate	FR	16.276	277.4 → 124.9	279.4 → 125.0			

I - Insecticide
F - Fungicide
F* - Fungicide metabolite
I* - Insecticide break-down product
H - Herbicide
M - Molluscicide
PS - Pesticide synergist
A - Acaricide
FR - Flame retardant

Results and discussion

Pesticides incurred in strawberries

Thirty-seven pesticides and pollutants were detected at some level through preliminary screening using the 8890/5977 Series GC/MSD with an RTL pesticide library and an 8890/7250 GC/Q-TOF with the Pesticide Personal Compound Database and Library (PCDL).[4] Using the new accurate mass screening approach, GC/Q-TOF was generally able to identify a higher number of pesticides in each sample compared to the GC/MSD. Twenty-eight of the detected pesticides were quantified in 12 strawberry samples, while nine pesticides were determined to be at or below the lowest calibration level prepared, and therefore not determined.

Two pesticides, acetamiprid and myclobutanil, were found in strawberry samples at concentrations exceeding the the tolerances for the maximum concentration of a pesticide residue in strawberries established by the US EPA[1,2] (Table 3). The insecticide thiamethoxam and the pesticide synergist piperonyl butoxide were found at a concentration above 10 ppb. These compounds do

Table 3. Quant results for 12 strawberry samples achieved with 8890/7000D triple quadrupole GC/MS system.

		Linear Calibration			Extended Calibration*			Organic Strawberries			Nonorganic Strawberries								
Compound	Tolerance (ppb)	Low calib. limit (ppb)	High linear calib. limit (ppb)	CF R2	Low calib. limit (ppb)	High calib. limit (ppb)	CF R2	O-1	O-2	O-3	N/O-1	N/O-2	N/O-3	N/O-4	N/O-5	N/O-6	N/O-7	N/O-8	N/O-9
3,4,5-Trimethacarb	NT**	5	500	0.9951	5	8,000	0.9985	7	6	6	5	6	<LOQ	5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Acetamiprid	600	500	1000	0.9961	500	10,000	0.9991					2,845							
Azoxystrobin	10,000	500	10,000	0.9930	500	10,000	0.9930												534
Bifenazate	1,500	100	4,000	0.9886	100	5,000	0.9941				<LOQ	498			177				
Bifenthrin	3,000	1	5,000	0.9902	1	10,000	0.9929	<LOQ			91	745	611	649	<LOQ	18	287	252	<LOQ
Boscalid	4,500	1	5,000	0.9961	1	10,000	0.9971	<LOQ	<LOQ	<LOQ	<LOQ	165	<LOQ	<LOQ	<LOQ	<LOQ	43	<LOQ	<LOQ
Captan	20,000	10	1,000	0.9794	10	10,000	0.9849	349			296	>10,000*	56		7,719	>10,000*	2,039	200	5,655
Carbaryl	4,000	1	1,000	0.9900	1	10,000	0.9978	85											
Chlorantraniliprole	1,000	20	1,000	0.9958	20	10,000	0.9988												<LOQ
cis-1,2,3,6-Tetrahydrophthalimide	25,000	1	1,000	0.9887	1	10,000	0.9987	465	15	<LOQ	1,275	1,850	87	15	837	856	461	105	1,098
Cyprodinil	5,000	1	500	0.9915	1	10,000	0.9987				29	232	<LOQ	279	289	102	5	<LOQ	<LOQ
DDE-*p,p'*	NT	1	1,000	0.9911	1	1,000***	0.9911	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Etoxazole	500	5	2,000	0.9891	5	2,000	0.9891							79					
Fenhexamid	3,000	50	5,000	0.9940	50	10,000	0.9977					298	<LOQ	<LOQ	561	<LOQ	<LOQ		
Flonicamid	1,500	1	1,000	0.9766	1	1,000***	0.9766	8	2	1	1	904	381	77	92	195	146	111	84
Fludioxonil	2,000	1	1,000	0.9910	1	8,000	0.9987				30	459	<LOQ	548	617	195	14	<LOQ	
Flutriafol	1,500	5	2,000	0.9893	5	2,000	0.9893				31	<LOQ	7					7	<LOQ
Malathion	8,000	1	1,000	0.9905	1	8,000	0.9994	48	<LOQ	<LOQ	5	<LOQ	61	<LOQ	<LOQ	<LOQ	<LOQ	48	<LOQ
Metalaxyl	10,000	1	500	0.9974	1	10,000	0.9959				48	5		3			<LOQ		90
Myclobutanil	500	1	1,000	0.9958	1	10,000	0.9987	1			<LOQ	4	9	706	20	<LOQ	<LOQ	14	1
Novaluron	500	1	250	0.9910	1	8,000	0.9901	462			29	284	136	<LOQ	110	<LOQ	<LOQ	376	428
Piperonyl butoxide	NT	5	1,000	0.9961	5	5,000	0.9964	1,618	<LOQ										
Pyrimethanil	3000	1	500	0.9968	1	10,000	0.9963	<LOQ	<LOQ	<LOQ	243	117	<LOQ	<LOQ	273	5	<LOQ	<LOQ	<LOQ
Quinoxyfen	900	1	1,000	0.9754	1	1,000***	0.9754				2	<LOQ	4			74	1	4	37
Tetraconazole	2,500	1	1,000	0.9867	1	8,000	0.9991					75	<LOQ		22	<LOQ			153
Thiametoxam	NT	20	1,000	0.9920	20	1,000***	0.9920	<LOQ	<LOQ		<LOQ	28			50	<LOQ	<LOQ		<LOQ
Trifloxystrobin	1,100	1	1,000	0.9978	1	10,000	0.9978				14			135	5	152			
Tris(1-Chloro-2-propyl) phosphate	****	1	250	0.9773	1	1,000***	0.9895	<LOQ	20	10	2	3	8	11	<LOQ	<LOQ	8	<LOQ	1

Extended calibration* was achieved with alternate fit such as second order (quadratic) allowed by USDA regulations.
NT** - No tolerances for the maximum concentration of a pesticide residue in strawberries established by the US EPA.
1,000*** - 1,000 ppb was the highest calibration standard used in this case.
**** - No information on tolerances for the maximum concentration is available.

not have established tolerances for the maximum concentration in strawberries, and would be reported as Presumptive Tolerance Violations[2] (highlighted in red in Table 3).

Quantitation of pesticides at their tolerance level with the 8890/7000D and 8890/7010B triple quadrupole GC/MS systems

The tolerances for the maximum concentration of a pesticide residue in strawberries established by the US EPA vary over a broad range between 20 and 25,000 ppb.[1] Moreover, the concentration of pesticides that do not have the established tolerance levels for strawberries should not exceed 10 ppb.[2] To ensure accurate quantitation of regulated and prohibited pesticides in one GC/MS analysis without the need for re-analyzing the sample and further processing, quantitation of the incurred pesticides requires an extended calibration range.

The 8890/7000D triple quadrupole GC/MS system equipped with the Inert Extractor source enabled quantifying all the compounds suspected to be present at their established tolerance levels while maintaining an accuracy of ±20 %. An extended calibration range permits quantifying pesticides with tolerance levels ranging from 10 ppb (no tolerance established) to 10,000 ppb in one GC/MS run (Table 3). A linear calibration fit can be achieved over a typical working range up to 500 ppb for 26 out of 28 pesticides. No calibration was performed below 1 ppb because the lowest reporting limit for strawberries is 10 ppb.

Boscalid is a fungicide regulated in strawberries at a fairly high concentration of 4,500 ppb. A linear calibration fit over the range of 1 to 5,000 ppb encompasses the tolerance level. If accurate quantitation above the reporting limit is desired, an extended calibration range of 1 to 10,000 ppb can be used (Figure 3).

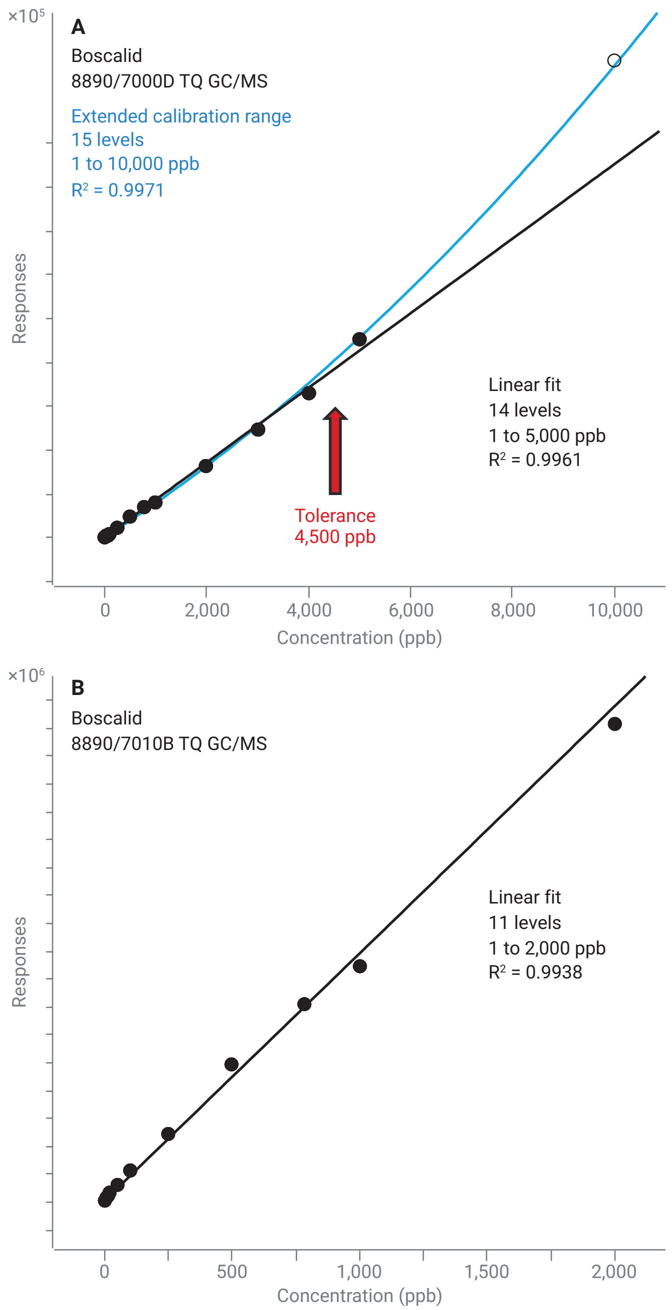

Figure 3. Matrix-matched calibration curves for boscalid with the 8890/7000D and 8890/7010B triple quadrupole GC/MS systems. Extended calibration to 10,000 ppb using the 7000D was achieved with an alternate fit such as second order (quadratic), allowed by the US EPA regulations.

Acetamiprid is an insecticide with a tolerance level of 600 ppb. Both triple quadrupole GC/MS systems, the 8890/7000D with the Inert Extractor Source and the 8890/7010B with the HES, allow for quantifying acetamiprid at its tolerance level in strawberries.

Thiamethoxam is an insecticide with no tolerance level established for strawberries, so its reporting limit is 10 ppb. This insecticide is known to present a challenge for GC/MS analysis due to its thermally labile nature, thus, it is commonly analyzed by LC/MS. The 8890/7010B triple quadrupole GC/MS system, equipped with the HES source, enabled quantifying thiamethoxam between 1 to 1,000 ppb when using a hot, isothermal, pulsed splitless injection. By comparison, the calibration range for thiamethoxam is between 20 to 1,000 ppb with the 8890/7000D triple quadrupole GC/MS system, equipped with the Inert Extractor source (1,000 ppb was the highest concentration possible with the stock solution used). The signal-to-noise ratio (S/N) at 10 ppb was 19.6 with the HES source, and 2.2 with the Inert Extractor source (Figure 4). A good qualifier/quantifier ratio for thiamethoxam was maintained at the tolerance level of 10 ppb with the HES source. The above reported calibration ranges for thiamethoxam are expected to be extended to significantly lower levels with a cold, temperature programed, splitless injection using the MMI.[5]

The 8890/7010B triple quadrupole GC/MS system with the HES source is the system of choice when quantitation needs to be performed at the trace and ultra-trace levels with the recommended analyte loading not exceeding 1 ng on-column. Lower limits of quantitation (LOQs) were achieved with the 8890/7010B triple quadrupole GC/MS system than with the 7000D. Note that the 7010B with the HES source will, in many cases, achieve lower LOQs than the lowest calibration standard of 1 ppb used in this study. It was not necessary to perform quantitation below 1 ppb, because the lowest reporting limit for residual pesticides in strawberries does not exceed 10 ppb.

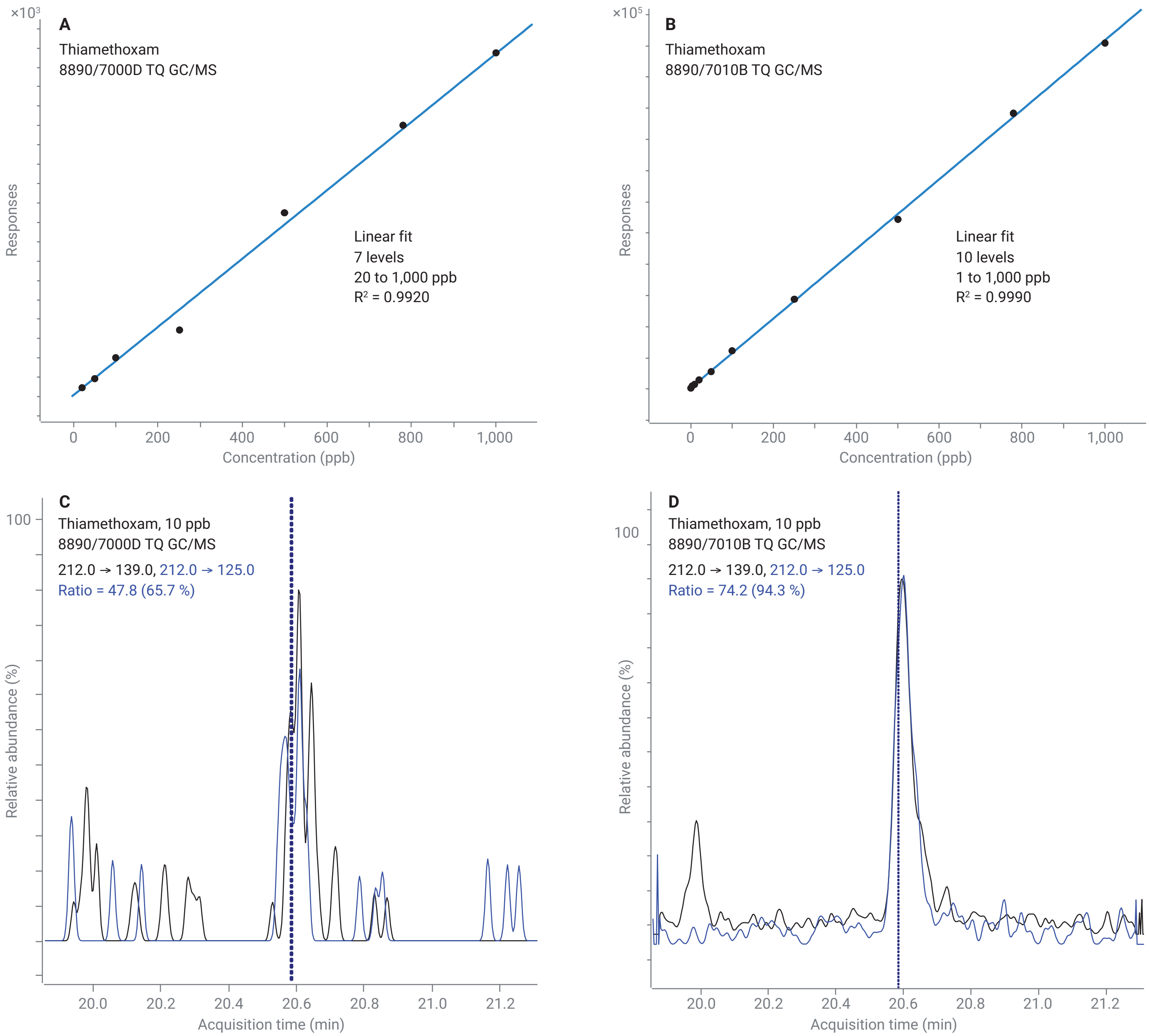

Figure 4. Matrix-matched calibration curves and MRM chromatograms for quantifier and qualifier for thiamethoxam.

Conclusions

The 8890/7000D and 8890/7010B triple quadrupole GC/MS systems were demonstrated to be suitable for quantifying incurred pesticides in strawberries at required regulatory levels ranging from 10 to 25,000 ppb. Isothermal, pulsed splitless injection produces suitably inert sample transfer at the required levels, and allows for increased sample throughput. Midcolumn backflush reduces both the run time and frequency of instrument maintenance. MRM transitions for the compounds suspected present were provided by the MassHunter Pesticide & Environmental Pollutant MRM Database (P&EP 4), which, along with the use of Dynamic MRM, greatly simplified creating an acquisition method. The compounds identified in the preliminary screening were quantitated at the tolerance level using matrix-matched calibration standards.

References

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