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Application News

Gas Chromatograph Mass Spectrometer

Simultaneous Analysis of Pesticide Residues in Food Using Triple Quadrupole GC-MS/MS with PTV Mode of a Multimode Injection Unit (MMI)

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User Benefits

- The GCMS-TQ™8040 RX and MMI-PTV enable analysis of more than 300 pesticide compounds in complex matrices.
- The Smart Pesticides Database™ automatically creates a multiresidue MRM acquisition method with optimized transitions.
- Peakintelligence™ for GCMS uses AI-based waveform processing for accurate analysis without parameter settings.

■ Introduction

Pesticide residue analysis is critical for ensuring food safety. Because the number of target analytes can exceed several hundred, rapid and efficient multiresidue analytical methods are needed. In this study, a simultaneous analytical method for more than 300 pesticide compounds was investigated using the GCMS-TQ™8040 RX triple quadrupole gas chromatograph-tandem mass spectrometer (Fig. 1). Samples were introduced using a Multi-Mode Injection Unit (MMI) operated in programmed temperature vaporization (PTV) mode. Method development was facilitated by Smart Pesticides Database Ver. 2.2, which includes pre-registered optimized multiple reaction monitoring (MRM) conditions, enabling straightforward transition selection and reducing matrix-derived interferences. In addition, Peakintelligence for GCMS, an AI-based data analysis tool, was used to improve the accuracy and efficiency of peak detection and integration in complex chromatograms. The proposed method was evaluated quantitatively using spinach extracts. The results demonstrated its applicability to comprehensive pesticide residue analysis in food matrices.

Fig. 1 GCMS-TQ™8040 RX

■ The Multi-Mode Injection Unit (MMI)

The Multi-Mode Injection Unit (MMI) is a GC inlet that supports various injection modes, including programmed temperature vaporization (PTV) injection and large volume injection (LVI), in addition to conventional split/splitless injection (SPL). In the PTV mode used in this study, the inlet temperature at sample injection is set low, near or below the solvent boiling point, allowing the sample to be temporarily retained in the liner. The inlet is then rapidly heated according to a predefined temperature program, vaporizing the target analytes and transferring them onto the column. Because the sample is introduced at a low temperature in PTV mode, thermal decomposition of thermally labile pesticides can be effectively suppressed (Fig. 2).

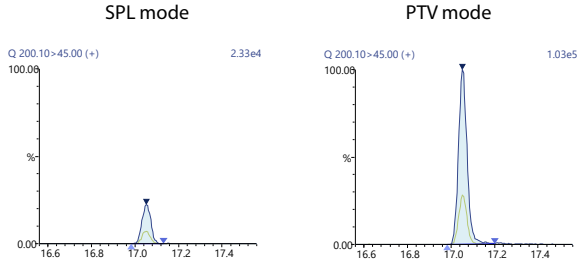

Fig. 2 Comparison of SPL and PTV Modes for Dichlofluanid Metabolite

■ Automated Development of a Multiresidue Analysis Method Using the Smart Pesticides Database

The simultaneous MRM acquisition method for pesticide residue analysis was developed using the Smart Pesticides Database. The database contains retention indices and MRM transitions for 530 pesticide compounds regulated in various countries (Fig. 3). By simply measuring *n*-alkanes, a multiresidue analytical method can be generated automatically, without using pesticide standard solutions, based on retention indices and the AART function. Because up to six transitions are registered for each pesticide, transitions that avoid matrix interference can be selectively used (Fig. 4).

Compound Name (E)	Ret. Index 1	Ret. Index 2	Ret. Index 3	Cas#	Ion1				Ion2			
	Method1	Method2	Method3		Type	m/z	CE	Ratio	Type	m/z	CE	Ratio
Clothianidin	1502	1496	1488	210880 - 92 - 5	T	132.0>71.0	14	100.00	Ref.1	132.0>45.0	22	78.58
Thiocyclam	1509	1505	1490	31895 - 21 - 3	T	135.0>71.0	8	100.00	Ref.1	135.0>58.0	24	33.28
Chloroneb	1510	1506	1502	2675 - 77 - 6	T	206.0>191.0	12	100.00	Ref.1	206.0>141.0	20	26.60
Orlaidine	1514	1509	1501	535 - 89 - 7	T	171.1>142.1	8	100.00	Ref.1	156.1>120.1	8	30.27
2-Phenylphenol	1525	1521	1511	90 - 43 - 7	T	170.1>141.1	24	100.00	Ref.1	141.1>115.1	18	73.17
Pentachlorobenzene	1528	1525	1511	608 - 93 - 5	T	248.8>214.8	18	100.00	Ref.1	248.8>178.9	28	85.94
Tebuthiuron	1530	1520	1514	34014 - 18 - 1	T	171.1>156.1	10	100.00	Ref.1	156.1>74.0	18	83.85
Isoprocarb	1539	1535	1531	2631 - 40 - 5	T	136.0>121.0	10	100.00	Ref.1	121.0>77.0	22	95.79
Molinate	1548	1543	1532	2212 - 67 - 1	T	126.1>55.0	14	100.00	Ref.1	187.1>126.1	8	38.39
XMC	1565	1559	1555	2655 - 14 - 3	T	122.1>107.1	14	100.00	Ref.1	107.1>77.0	18	44.38

Fig. 3 Registered Information in the Smart Pesticides Database

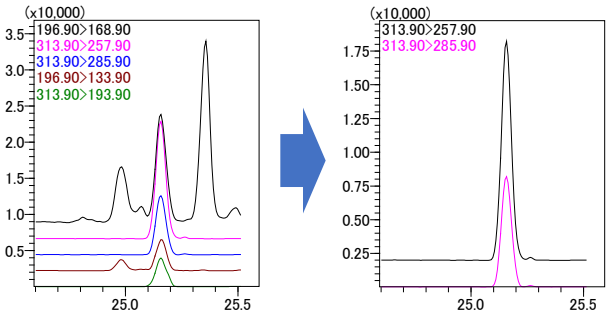

Fig. 4 Example of MRM Transition Selection for Chlorpyrifos in Spinach Extract at an Equivalent Concentration of 10 ppb

■ Sample Preparation and Pretreatment

Spinach was selected as the food matrix because it contains pigments and other complex coexisting components. The sample was homogenized by a pre-cooled dry ice grinding method, followed by extraction and cleanup using the QuEChERS method in accordance with AOAC 2007.01. The workflow is shown in Fig. 5. For extraction, a Q-sep QuEChERS extraction salt kit (Restek; AOAC 2007.01 compliant; P/N 25852) was used. For cleanup, Q-sep QuEChERS dSPE for extract purification (Restek; AOAC 2007.01 compliant; P/N 26219) was used. Calibration curves were prepared using matrix-matched standards. A mixed pesticide standard solution was added to the pretreated blank extract to obtain final concentrations of 0.1, 0.5, 1, 5, 10, 20, 50, 100, 200, and 400 ppb.

1. Homogenize the sample to ensure uniformity
2. Weigh 15 g of the homogenized sample into a 50 mL tube for extraction
3. Add 15 mL of acetonitrile containing 1% acetic acid (v/v)
4. Add buffer salts, shake vigorously by hand for 1 min., then centrifuge for 5 min. (3,000 rpm)
5. Transfer the supernatant to a dSPE tube, shake vigorously by hand for 2 min.
6. Centrifuge for 5 min. (3,000 rpm) to obtain the test solution

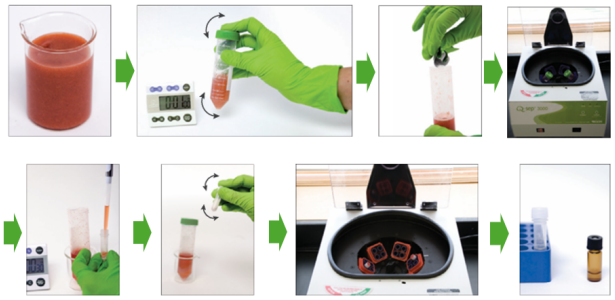

Fig. 5 Example of Sample Preparation Using QuEChERS

■ Analytical Conditions

The instrument configuration and analytical conditions are summarized in Table 1. Method 3 in the Smart Pesticides Database was used as the basis for the analytical method. To improve the peak shapes of low-boiling pesticides in acetonitrile, the initial GC oven temperature was changed to 70 ˚C.

■ Data Analysis

LabSolutions Insight, a quantitative data processing software package designed for high-throughput sample analysis, was used for data analysis, with Peakintelligence for GCMS applied as the peak integration algorithm (Fig. 6). Peakintelligence is an AI-based waveform processing algorithm that uses machine learning to emulate the peak processing performed by experienced analysts. It requires no operator-defined parameter settings and provides peak integration performance comparable to that of skilled users.

For more information on improving the efficiency of pesticide data analysis using Peakintelligence for GCMS, please refer to Application News 01-00585-EN, “Time-Saving Data Processing for Pesticide Residues with Peakintelligence for GCMS.”

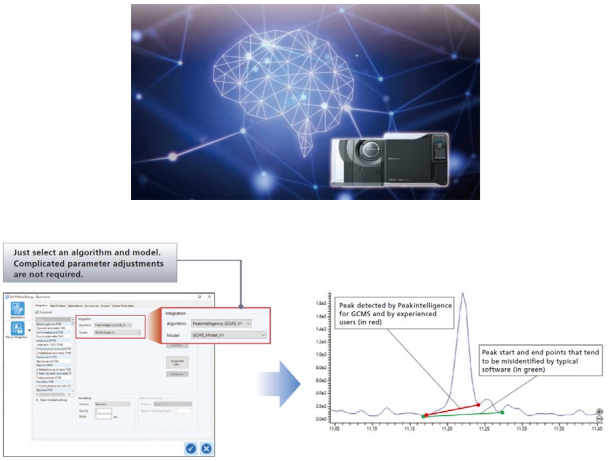

Fig. 6 Peakintelligence for GCMS

Table 1 Instrument Configuration and Analytical Conditions

Configuration	
GC-MS	: GCMS-TQ8040 RX
Injection Unit	: MMI-U
Column	: SH-I-5Sil MS (30 m × 0.25 mm I.D., 0.25 µm) (P/N: 221-75954-30)
Guard Column	: SH-I Guard / Retention Gap Column (5 m × 0.25 mm I.D.) (P/N: 227-36303-01)
Liner	: Xtra Inert Splitless Liner (P/N: 227-36701-01)

GC	
MMI Mode	: PTV injection
Injection Mode	: Pulsed Splitless
MMI Program	: 60 ˚C (0.1 min) - (900 ˚C/min) - 300 ˚C
Sampling Time	: 1 min
Carrier Gas Control	: Linear Velocity (44.1 cm/s) (He)
Pulsed Injection	: 250 kPa (1.5 min)
Injection Volume	: 2 µL
Oven Program	: 70 ˚C (3 min) - (10 ˚C/min) - 130 ˚C - (4 ˚C/min) - 200 ˚C - (8 ˚C/min) - 290 ˚C (12.25 min)

MS	
Ion Source Temp.	: 230 ˚C
IF Temp.	: 280 ˚C
Acquisition Mode	: MRM
Loop Time	: 0.4 sec

■ Quantitative Analysis

Calibration curves were prepared over a range of 0.1 to 400 ppb (up to 200 ppb for some compounds) using matrix-matched standard solutions prepared in spinach extract. Of the 386 pesticides evaluated, 357 compounds (approximately 92%) showed good linearity, with coefficients of determination (R^2) of 0.995 or higher (Fig. 7). Among these, 213 compounds (approximately 60%) were detectable at 0.1 ppb (Fig. 8).

Repeatability was also evaluated (n = 6) using the calibration standard at 10 ppb. The results are summarized in Table 3. Despite the use of a challenging matrix extract, trueness values for many of the evaluated pesticides fell within the acceptance range specified in the SANTE guideline (70–120%). Repeatability (%RSD) also showed excellent performance, with most compounds well below the acceptance criterion of 20% (Fig. 9).

These results indicate that reliable measurements can be achieved even in complex matrices through the combined effects of the high sensitivity of the GCMS-TQ™8040 RX for multiresidue analysis, stable sample introduction by PTV injection, appropriate transition selection using the Smart Pesticides Database, and accurate AI-based peak processing. Representative calibration curves and chromatograms near the LOQ are shown in Fig. 11.

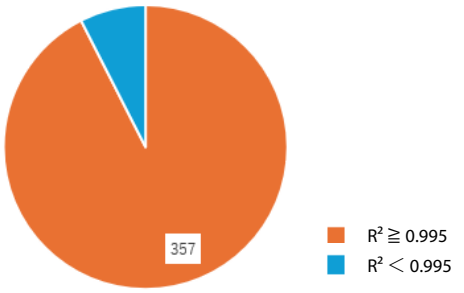

Fig. 7 Distribution of Calibration Curve Linearity ($R^2 \geq 0.995$), with numbers indicating the number of compounds

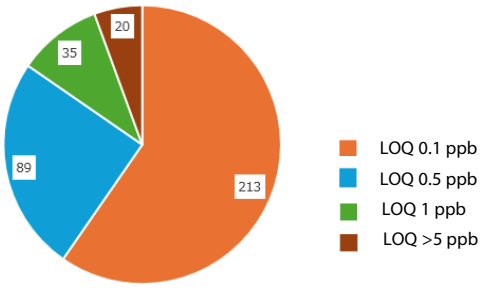

Fig. 8 Distribution of LOQs for 357 Compounds, with numbers indicating the number of compounds

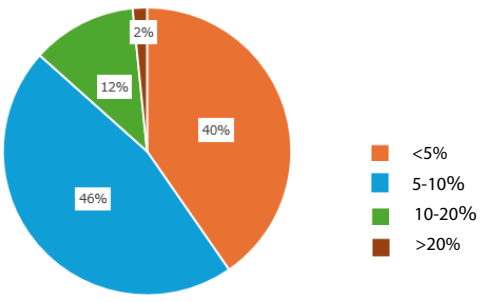

Fig. 9 Distribution of Quantitative Repeatability (%RSD)

■ Data Analysis with Peakintelligence for GCMS

Results obtained with Peakintelligence for GCMS were compared with those obtained by conventional peak processing (Chromatopac) (Fig. 10). Conventional processing occasionally resulted in incorrect peak integration, particularly at low concentrations in chromatograms with matrix-derived baseline elevation or adjacent peaks. In contrast, Peakintelligence provided appropriate peak processing for these chromatograms. Its use can reduce the time required for manual correction while enabling reliable quantitative results without operator-dependent variation.

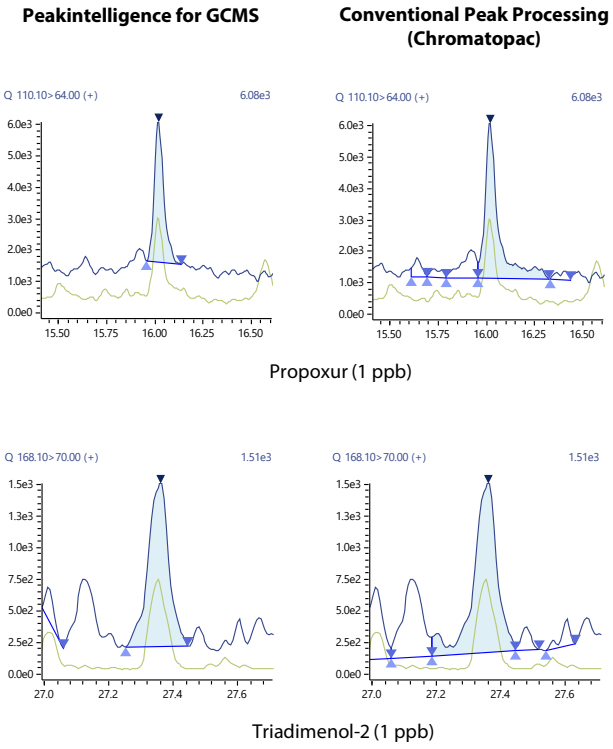

Fig. 10 Comparison of Peak Processing between Peakintelligence for GCMS and the Conventional Processing (Chromatopac)

■ Conclusion

In this Application News, pesticide residues in spinach extract were analyzed using the GCMS-TQ8040 RX in combination with a Multi-Mode Injection Unit (MMI) and advanced software tools. The GCMS-TQ8040 RX enables reliable detection of trace-level compounds in multiresidue analysis, while the PTV mode of the MMI and appropriate transition selection using the Smart Pesticides Database provide highly sensitive and selective measurements even in complex matrices. In addition, Peakintelligence for GCMS, an AI-based data analysis tool, improves integration accuracy for difficult peaks, streamlines data analysis, and reduces operator-dependent variability. Quantitative evaluation demonstrated excellent trueness and precision even for challenging matrix extracts. These results indicate that this integrated solution is well suited to routine pesticide residue analysis of complex multicomponent samples, helping laboratories improve both confidence in analytical results and operational efficiency.

Table 2 Summary of Quantitative Performance Evaluation Results (Excerpt)

Compound Name	Mean conc. (ppb)	Recovery (%)	Repeatability (%RSD)	Trueness (%)
Chlofentezine deg.	9.9	99.3	2.5	-0.7
Dichlorvos	8.4	84.1	4.9	-15.9
Nereistoxin	10.3	103.0	3.0	3.0
Allidochlor	9.5	94.7	6.9	-5.3
Dichlobenil	10.2	102.0	4.1	2.0
EPTC	9.9	98.9	4.6	-1.1
Biphenyl	10.1	100.9	5.9	0.9
Mevinphos-1	9.2	92.0	10.6	-8.0
Mevinphos-2	8.8	87.8	8.4	-12.2
Butylate	10.3	103.1	3.5	3.1
Chlormephos	10.0	99.9	4.0	-0.1
Etridiazole	10.5	105.2	4.2	5.2
Thiocyclam	9.9	99.5	4.5	-0.5
Methacrifos	9.5	94.8	3.4	-5.2
Crimidine	9.3	92.6	6.1	-7.4
Chloroneb	10.3	102.8	5.9	2.8
2-Phenylphenol	9.6	96.4	2.5	-3.6
Isoprocarb	8.8	87.9	6.0	-12.1
Molinate	10.2	101.6	4.0	1.6
XMC	8.5	85.0	10.0	-15.0
Omethoate	9.3	92.7	9.6	-7.3
Tecnazene	10.9	109.1	4.9	9.1
Xylylcarb	9.6	96.4	13.3	-3.6
Propachlor	9.5	95.4	4.0	-4.6
Propoxur	9.7	96.8	6.8	-3.2
Chlorethoxyfos	11.0	110.2	3.7	10.2
Demeton-S-methyl (Methyl demeton)	8.5	85.5	5.7	-14.5
Diphenylamine	10.2	101.5	3.5	1.5
Ethoprophos	9.5	94.5	5.7	-5.5
Phenmedipham deg.	23.0	229.7	4.6	129.7
Dichlofluanid metabolite	8.2	81.8	9.9	-18.2
Ethalfluralin	10.8	108.4	6.6	8.4
Chlorpropham	9.2	92.0	3.1	-8.0
2,6-Dichlorobenzamide	10.0	100.4	6.4	0.4
Dioxabenzofos (Salithion)	9.3	93.2	4.8	-6.8
Dicrotophos	9.9	99.5	7.9	-0.5
Flusilazole metabolite	18.4	183.6	2.9	83.6
Trifluralin	10.9	109.2	4.4	9.2
Sulfotep	10.3	102.6	3.7	2.6
Benfluralin	10.8	108.2	4.7	8.2
Monocrotophos	8.4	84.0	14.7	-16.0
Cadusafos	9.7	97.4	3.9	-2.6
Di-allate-1	10.0	100.3	5.7	0.3
Phorate	10.1	100.6	5.8	0.6
alpha-BHC	9.6	95.7	4.3	-4.3
Di-allate-2	10.7	107.0	5.9	7.0
Thiometon	9.8	98.4	5.8	-1.6
Dicloran	11.4	113.8	5.0	13.8
Dimethoate	8.3	83.1	11.1	-16.9
Desmedipham deg.	11.2	112.1	5.1	12.1
Furilazole	10.3	102.7	3.7	2.7
Carbofuran	9.7	97.1	12.0	-2.9
beta-BHC	9.6	96.3	5.3	-3.7
Simazine	9.9	98.6	10.0	-1.4
Dimethipin	8.7	86.5	11.8	-13.5
Quintozene	11.7	117.4	4.9	17.4
Swep	10.5	105.0	8.9	5.0
Atrazine	9.0	90.3	6.9	-9.7
Clomazone	9.0	90.3	2.9	-9.7
Chlorbufam	11.3	112.9	4.5	12.9
gamma-BHC (Lindane)	9.8	98.5	3.9	-1.5
Propazine	9.9	99.5	3.4	-0.5
Tolylfluanid metabolite	9.2	92.5	8.7	-7.5
Dioxathion deg.	12.0	120.1	5.5	20.1
Cyanophos	9.1	91.5	5.3	-8.5
Terbufos	9.9	99.0	3.6	-1.0
Pyroquilon	9.2	91.5	6.1	-8.5
Fonofos	9.7	97.2	3.1	-2.8
Propyzamide	10.0	100.1	4.2	0.1
Chlorothalonil	10.7	106.6	15.9	6.6
Phosphamidon-1	10.5	105.0	10.3	5.0
Pyrimethanil	9.2	92.4	5.5	-7.6
Diazinon	9.5	95.3	4.7	-4.7
Disulfoton	9.9	99.1	4.8	-0.9
delta-BHC	9.1	90.7	5.4	-9.3

Compound Name	Mean conc. (ppb)	Recovery (%)	Repeatability (%RSD)	Trueness (%)
Terbacil	9.8	98.3	7.5	-1.7
Isazofos	9.9	99.3	2.7	-0.7
Prohydrojasmon-1	9.6	95.6	3.6	-4.4
Tri-allate	9.8	98.0	6.5	-2.0
MCPA-thioethyl	9.7	97.0	4.1	-3.0
Etrimfos	9.7	97.1	5.1	-2.9
Tefluthrin	10.1	101.4	4.7	1.4
Iprobenfos	9.7	96.6	4.7	-3.4
Oxabetrinil	9.4	94.4	18.2	-5.6
Tebupirimfos	10.0	100.3	4.4	0.3
Benoxacor	10.3	102.8	3.8	2.8
Prohydrojasmon-2	10.8	108.2	9.8	8.2
Formothion	11.2	112.3	9.1	12.3
MCPB-ethyl	10.1	101.4	3.9	1.4
Benfuresate	9.7	96.7	3.6	-3.3
Phosphamidon-2	10.0	100.4	11.3	0.4
Dimethenamid (Dimethenamid-P)	9.7	97.5	4.4	-2.5
Dichlofenthion	9.8	98.3	3.3	-1.7
Propanil	10.8	107.9	3.2	7.9
Bromobutide	10.9	108.8	3.9	8.8
Terbucarb	10.1	101.4	4.5	1.4
Chlorpyrifos-methyl	9.6	95.7	7.0	-4.3
Metribuzin	10.7	107.2	3.9	7.2
Acetochlor	9.7	97.4	2.4	-2.6
Oxpoconazole-formyl deg.	11.3	112.9	2.7	12.9
Vinclozolin	9.5	94.6	7.3	-5.4
Parathion-methyl	11.5	115.2	4.1	15.2
Tolclofos-methyl	9.6	96.0	5.4	-4.0
Simeconazole	9.3	92.6	6.0	-7.4
Alachlor	9.8	98.3	2.8	-1.7
Spiroxamine-1	10.0	100.3	4.3	0.3
Simetryn	10.2	102.2	5.4	2.2
Metalaxyl (Mefenoxam)	9.9	98.7	2.9	-1.3
Fenchlorphos	9.2	92.5	5.2	-7.5
Ametryn	9.8	98.3	2.4	-1.7
Cinmethylin	9.5	95.1	5.5	-4.9
Prometryn	10.5	105.0	4.2	5.0
2-(1-Naphthyl)acetamide	8.5	84.9	3.3	-15.1
Dithiopyr	10.0	100.5	4.4	0.5
Fenitrothion	10.8	107.9	5.1	7.9
Pirimiphos-methyl	9.7	97.4	4.4	-2.6
Terbutryn	9.9	99.3	3.9	-0.7
Spiroxamine-2	9.5	94.7	5.8	-5.3
Ethofumesate	9.3	92.9	6.4	-7.1
Dichlofluanid	11.8	118.4	10.8	18.4
Quinoclamine	11.8	118.2	7.5	18.2
(E)-Dimethylvinphos	9.7	96.6	8.6	-3.4
Bromacil	9.9	98.9	10.0	-1.1
Esprocarb	9.6	95.5	5.3	-4.5
Malathion	9.9	98.8	6.0	-1.2
Metolachlor (S-Metolachlor)	9.6	95.6	4.0	-4.4
Chlorpyrifos	9.7	96.9	5.2	-3.1
Thiobencarb	9.9	98.7	5.5	-1.3
(Z)-Dimethylvinphos	9.4	94.1	7.2	-5.9
Fenthion	9.4	93.7	4.0	-6.3
Chlorthal-dimethyl	9.2	91.6	6.4	-8.4
Cyanazine	10.6	105.7	7.7	5.7
Diethofencarb	9.4	93.8	4.2	-6.2
Parathion	10.6	105.7	5.2	5.7
Fenpropimorph	9.9	99.0	4.1	-1.0
Phthalide	8.9	89.1	8.7	-10.9
Triadimefon	9.2	92.0	5.8	-8.0
Isocarbophos	10.2	102.4	8.4	2.4
Isofenphos oxon	10.0	99.7	7.8	-0.3
Tetraconazole	9.8	98.4	7.3	-1.6
Bromophos	9.8	98.2	4.4	-1.8
Nitrothal-isopropyl	12.6	125.8	4.0	25.8
Fosthiazate-1	9.9	98.8	14.0	-1.2
Diphenamid	8.6	85.6	4.7	-14.4
Fosthiazate-2	9.7	96.6	12.3	-3.4
Thiamethoxam deg.	10.2	102.4	5.0	2.4
Cyprodinil	9.3	92.6	4.8	-7.4
Pendimethalin	11.4	114.4	5.6	14.4
(E)-Chlorfenvinphos	9.3	92.8	6.3	-7.2
Penconazole	8.9	89.0	6.4	-11.0

Table 2 Summary of Quantitative Performance Evaluation Results
(Continued)

Compound Name	Mean conc. (ppb)	Recovery (%)	Repeatability (%RSD)	Trueness (%)
Tolylfluanid	10.9	109.4	10.4	9.4
Dimethametryn	10.4	104.1	5.9	4.1
(Z)-Pyrifenox	8.7	86.6	4.3	-13.4
Fipronil	9.9	98.9	3.3	-1.1
Chlozolinate	11.5	115.5	3.3	15.5
Ethychlozate	9.2	92.3	5.4	-7.7
Thiabendazole	8.8	87.8	10.7	-12.2
Isofenphos	10.1	100.5	3.7	0.5
(Z)-Chlorfenvinphos	9.3	92.9	6.7	-7.1
Captan	32.9	329.4	3.6	229.4
Allethrin-1,2	8.6	86.0	8.9	-14.0
Mecarbam	10.6	105.7	15.5	5.7
Phenthoate	9.9	99.5	5.1	-0.5
Diclocymet-1	9.4	94.0	3.0	-6.0
Quinalphos	9.6	95.8	6.6	-4.2
Dimepiperate	9.2	92.1	4.4	-7.9
Folpet	23.5	235.2	5.1	135.2
Procymidone	9.0	89.5	5.0	-10.5
Triadimenol-1	8.6	86.3	9.5	-13.7
Allethrin-3,4 (Bioallethrin)	9.9	99.3	4.7	-0.7
Zoxamide deg.	9.7	96.5	4.4	-3.5
Chinomethionat	10.9	108.6	6.1	8.6
Chlorbenside	10.6	105.7	3.3	5.7
Methidathion	10.2	101.7	6.9	1.7
Ferimzone	11.8	118.4	4.3	18.4
Triadimenol-2	9.4	94.3	7.4	-5.7
Bromophos-ethyl	9.5	95.5	3.4	-4.5
Diclocymet-2	9.7	96.6	3.4	-3.4
Methoprene	9.8	97.9	12.7	-2.1
(E)-Pyrifenox	8.8	87.6	7.0	-12.4
Propaphos	8.9	88.9	7.3	-11.1
Paclobutrazol	10.0	99.8	2.7	-0.2
Tetrachlorvinphos	9.7	96.8	9.5	-3.2
Trichlamide	10.3	103.0	5.5	3.0
alpha-Endosulfan	9.7	97.1	4.7	-2.9
Disulfoton sulfone	10.1	100.7	11.0	0.7
Fenothiocarb	9.9	99.3	4.8	-0.7
Butachlor	10.0	99.7	4.8	-0.3
Ditalimfos	9.8	98.5	6.3	-1.5
Imazamethabenz-methyl-1	10.5	104.9	7.2	4.9
Flutriafol	9.1	90.6	4.0	-9.4
Butamifos	10.0	100.2	3.3	0.2
Imazamethabenz-methyl-2	8.4	84.4	11.5	-15.6
Napropamide	8.4	84.5	5.6	-15.5
Tricyclazole	6.5	64.9	13.8	-35.1
Chlorfenson	9.4	93.6	6.7	-6.4
Fenamiphos	9.9	98.9	4.2	-1.1
Hexaconazole	9.9	98.9	9.8	-1.1
Flutolanil	9.2	91.9	5.2	-8.1
Prothiofos	9.5	95.3	5.3	-4.7
Fludioxonil	8.6	86.3	7.0	-13.7
(E)-Metominostrobin	9.4	93.9	3.3	-6.1
Isoprothiolane	8.7	86.6	6.5	-13.4
Pretilachlor	9.7	96.9	5.8	-3.1
Profenofos	9.1	90.9	9.6	-9.1
Uniconazole (Uniconazole-P)	9.5	95.1	4.7	-4.9
Tribufos	9.4	93.6	4.5	-6.4
Myclobutanil	8.9	89.3	5.2	-10.7
Oxadiazon	9.3	92.8	6.4	-7.2
Flamprop-methyl	9.3	92.6	5.2	-7.4
Thifluzamide	9.2	92.1	5.0	-7.9
Carboxin	10.1	100.8	3.4	0.8
Imibenconazole-debenzyl	10.1	101.3	11.0	1.3
Diclobutrazol	9.3	93.4	6.3	-6.6
Flusilazole	9.2	91.6	5.8	-8.4
Buprofezin	9.5	95.4	7.2	-4.6
Azaconazole	8.9	89.0	6.0	-11.0
Bupirimate	9.8	98.1	4.6	-1.9
Oxyfluorfen	12.0	120.4	3.0	20.4
(Z)-Metominostrobin	9.2	91.9	3.6	-8.1
Kresoxim-methyl	9.5	94.8	6.9	-5.2
Cyproconazole-1	9.8	97.7	10.1	-2.3
Cyproconazole-2	8.8	88.1	6.8	-11.9
Chlorfenapyr	9.6	95.6	8.7	-4.4
Isoxathion	10.4	104.3	10.0	4.3

Compound Name	Mean conc. (ppb)	Recovery (%)	Repeatability (%RSD)	Trueness (%)
Cyflufenamid	10.3	103.1	5.2	3.1
Nitrofen	10.9	109.0	6.0	9.0
Chlorthiophos-1	9.2	92.0	14.0	-8.0
Fenoxanil	9.5	95.5	7.0	-4.5
1,1-Dichloro-2,2-bis(4-ethylphenyl)ethane	9.6	96.2	4.2	-3.8
beta-Endosulfan	10.4	104.0	5.9	4.0
Chlorthiophos-2	9.5	95.4	5.5	-4.6
Chlorobenzilate	9.4	93.6	4.9	-6.4
Chloropropylate	9.4	94.2	3.8	-5.8
Fensulfothion	10.2	101.7	5.8	1.7
Diniconazole	9.3	92.9	4.4	-7.1
(Z)-Pyriminobac-methyl	9.6	96.5	4.7	-3.5
Flufenpyr-ethyl	9.6	96.1	7.9	-3.9
Oxadixyl	8.9	88.7	4.1	-11.3
Ethion	10.1	100.8	4.3	0.8
Chlorthiophos-3	9.4	93.8	5.5	-6.2
Mepronil	8.5	84.7	5.8	-15.3
Fluacrypyrim	9.6	95.5	7.8	-4.5
Sulprofos	9.6	95.5	4.0	-4.5
Triazophos	8.8	87.5	8.5	-12.5
Azamethiphos	17.9	179.4	12.1	79.4
Chlornitrofen	11.7	117.4	3.5	17.4
Benalaxyl	9.1	90.9	7.9	-9.1
Isoxadifen-ethyl	10.5	104.8	4.1	4.8
Carbophenothion	8.7	87.3	9.1	-12.7
Edifenphos	10.0	100.4	7.5	0.4
Carfentrazone-ethyl	9.5	95.3	5.7	-4.7
Cyanofenphos	8.9	89.5	7.6	-10.5
Endosulfan sulfate	9.6	95.7	11.8	-4.3
Norflurazon	8.8	88.3	5.9	-11.7
Quinoxyfen	9.1	91.0	5.7	-9.0
Propiconazole-1	9.6	96.3	4.4	-3.7
Lenacil	8.8	88.2	5.7	-11.8
Trifloxystrobin	10.2	101.9	5.8	1.9
Propiconazole-2	9.7	96.5	4.6	-3.5
(E)-Pyriminobac-methyl	9.4	94.1	4.2	-5.9
Pyraflufen-ethyl	9.4	94.0	4.9	-6.0
Hexazinone	9.5	94.5	4.5	-5.5
Thenylchlor	9.5	95.0	5.3	-5.0
Tebuconazole	8.9	89.2	3.0	-10.8
Diclofop-methyl	9.1	91.2	6.6	-8.8
Propargite-1	14.9	149.0	7.3	49.0
Propargite-2	14.2	142.1	7.3	42.1
Diflufenican	9.6	96.4	3.7	-3.6
Resmethrin-1	9.7	97.0	10.1	-3.0
Piperonyl butoxide	9.3	92.5	5.5	-7.5
Nitralin	37.7	376.8	0.9	276.8
Epoxiconazole	8.7	87.2	6.0	-12.8
Resmethrin-2 (Bioresmethrin)	9.4	93.5	5.7	-6.5
Zoxamide	9.8	97.7	11.8	-2.3
Mefenpyr-diethyl	9.6	95.6	4.8	-4.4
Pyributicarb	9.2	91.9	4.4	-8.1
Chlomethoxyfen (Chlormethoxynil)	11.3	113.3	5.6	13.3
Pyridaphenthion	9.9	99.5	7.4	-0.5
Iprodione	9.2	92.2	8.5	-7.8
Bromuconazole-1	9.2	91.8	5.2	-8.2
Phosmet	9.4	94.3	6.2	-5.7
Tetramethrin-1	9.2	91.9	6.7	-8.1
EPN	11.5	115.0	4.5	15.0
Bromopropylate	9.6	96.3	3.5	-3.7
Piperophos	9.5	94.8	5.0	-5.2
Bifenthrin	10.0	99.5	3.7	-0.5
Picolinafen	9.2	92.3	6.2	-7.7
Tetramethrin-2	9.2	92.3	4.9	-7.7
Fenoxycarb	8.6	86.4	12.9	-13.6
Bifenazate	9.3	93.4	10.9	-6.6
Methoxychlor	10.3	102.8	6.5	2.8
Etoxazole	9.3	93.0	14.1	-7.0
Fenamidone	8.9	89.0	5.3	-11.0
Fenpropathrin	9.8	98.2	4.4	-1.8
Indanofan	10.4	104.1	5.6	4.1
Bromuconazole-2	8.5	85.0	6.7	-15.0
Anilofos	10.4	104.0	7.1	4.0
Tebufenpyrad	9.1	91.0	3.8	-9.0
Bifenox	20.4	204.3	3.5	104.3

Table 2 Summary of Quantitative Performance Evaluation Results
(Continued)

Compound Name	Mean conc. (ppb)	Recovery (%)	Repeatability (%RSD)	Trueness (%)
Clomeprop	8.7	87.0	6.2	-13.0
Furametpyr	8.5	85.5	4.2	-14.5
Phenothrin-1	9.1	90.6	7.3	-9.4
Tetradifon	9.2	92.0	3.9	-8.0
Iprodione metabolite	11.0	109.8	15.0	9.8
Phosalone	9.2	92.1	8.6	-7.9
Phenothrin-2	9.4	93.6	6.2	-6.4
Leptophos	8.9	89.4	6.6	-10.6
Pentoxazone	9.3	93.0	5.5	-7.0
Azinphos-methyl	10.6	106.1	9.4	6.1
Pyriproxyfen	10.0	100.2	11.9	0.2
Mefenacet	9.3	92.7	6.9	-7.3
Cyhalothrin-1	11.6	116.0	5.6	16.0
Cyhalofop-butyl	8.6	85.8	5.4	-14.2
Furametpyr metabolite	8.1	80.8	8.9	-19.2
Cyhalothrin-2	11.6	115.8	3.9	15.8
Acrinathrin-1	12.2	121.8	10.3	21.8
Fenarimol	8.8	88.1	2.8	-11.9
Pyrazophos	9.0	89.6	4.8	-10.4
Acrinathrin-2	10.9	108.7	5.9	8.7
Azinphos-ethyl	10.1	101.4	5.9	1.4
Dialifos	9.8	97.7	4.5	-2.3
Pyraclofos	9.6	96.5	10.9	-3.5
Fenoxaprop-ethyl (Fenoxaprop-P-ethyl)	9.6	96.3	6.8	-3.7
Spirodiclofen	10.4	104.0	30.5	4.0
Bitertanol-1	9.5	94.8	10.3	-5.2
Permethrin-1	8.6	86.3	3.3	-13.7
Bitertanol-2	9.0	89.6	16.1	-10.4
Fluquinconazole	8.8	88.4	3.8	-11.6
Pyridaben	8.9	89.3	5.0	-10.7
Permethrin-2	10.0	99.7	3.6	-0.3
Dioxathion	10.1	100.7	4.2	0.7
Oxpoconazole	8.5	84.9	8.6	-15.1
Butafenacil	9.7	97.2	4.9	-2.8
Etobenzanid	9.3	92.8	5.4	-7.2
Cafenstrole	11.3	113.2	9.7	13.2
Fenbuconazole	8.4	83.8	6.5	-16.2
Cyfluthrin-1	11.4	113.6	3.7	13.6
Cyfluthrin-2	12.4	124.0	5.8	24.0
Cyfluthrin-3	12.5	124.8	3.3	24.8
Cyfluthrin-4	10.4	103.8	10.0	3.8
Cypermethrin-1	14.6	145.7	54.7	45.7
Halfenprox	10.1	101.2	2.5	1.2
Cypermethrin-2	14.1	140.7	41.0	40.7
Quizalofop-ethyl (Quizalofop-P-ethyl)	8.9	88.6	7.0	-11.4
Cypermethrin-3	13.2	131.9	60.3	31.9
Flucythrinate-1	9.5	94.6	5.1	-5.4
Cypermethrin-4	15.0	150.2	62.4	50.2
Etofenprox	9.5	94.8	4.2	-5.2
Flucythrinate-2	9.9	99.2	4.9	-0.8
Silafluofen	8.9	89.2	5.6	-10.8
Fluridone	9.0	89.6	14.0	-10.4
Pyrimidifen	8.9	88.8	3.9	-11.2
Flumioxazin	9.7	97.2	9.3	-2.8
Fenvalerate-1	10.9	109.0	5.1	9.0
Pyraclostrobin	8.6	85.9	13.4	-14.1
Fluvalinate-1	10.4	103.9	7.0	3.9
Fenvalerate-2 (Esfenvalerate)	10.8	108.1	6.8	8.1
Fluvalinate-2	11.2	111.7	9.4	11.7
Difenoconazole-1	8.5	84.9	6.7	-15.1
Difenoconazole-2	8.2	82.3	4.5	-17.7
Pyrazoxyfen	5.8	57.9	23.9	-42.1
Deltamethrin-1 (Tralomethrin deg.-1)	11.3	113.0	6.2	13.0
Indoxacarb	8.2	82.2	12.7	-17.8
Deltamethrin-2 (Tralomethrin deg.-2)	11.0	109.9	5.7	9.9
Flumiclorac-pentyl	9.2	92.5	4.9	-7.5
Azoxystrobin	9.1	90.9	8.6	-9.1
Dimethomorph-1	9.5	95.0	4.6	-5.0
Famoxadone	8.8	88.4	8.0	-11.6
Tolfenpyrad	9.4	94.4	4.3	-5.6
Dimethomorph-2	8.9	89.0	5.8	-11.0
Imibenconazole	9.2	92.3	9.3	-7.7
Cinidon-ethyl	9.4	93.5	3.6	-6.5
Fluthiacet-methyl	11.4	113.5	11.6	13.5

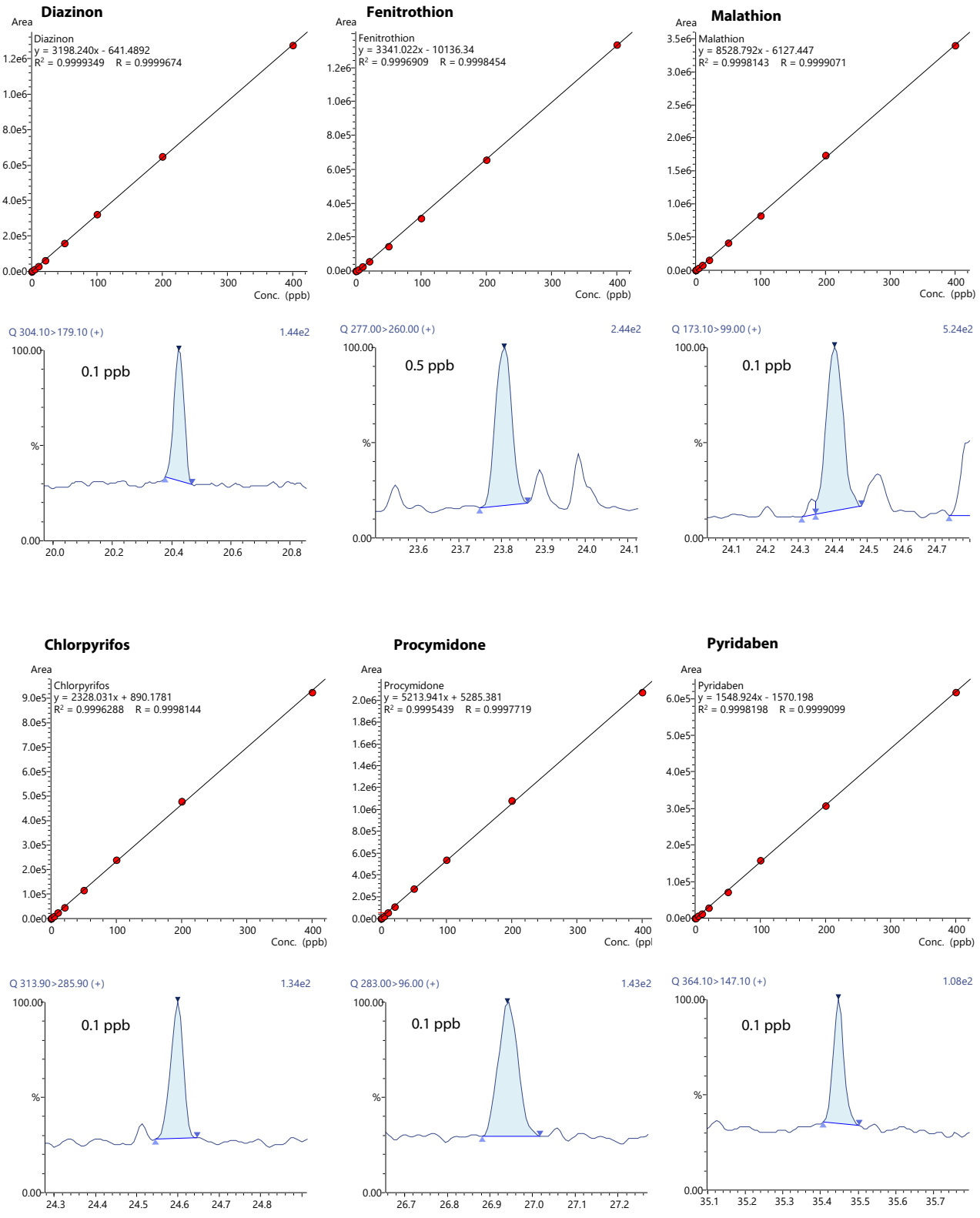

Fig. 11 Calibration Curves and Chromatograms at the Limit of Quantification (LOQ)

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