Application Note: 52022

Rapid Analysis of 303 Pesticide Residues in Green Bean Using Triple Quadrupole GC-MS/MS

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Key Words

- Pesticide Analyzer Reference
- Pesticide Residues
- SPE
- Timed-SRM
- Triple Quadrupole GC-MS/MS

Introduction

Gas chromatography (GC) with element-selective detectors or single quadrupole gas chromatography/mass spectrometry (GC/MS) are common techniques used for GC-amenable pesticide residue analysis in labs. However, as the number of pesticides increases and maximum residue levels (MRLs) decrease in regulations, these techniques require that one sample be analyzed using several injections with different methods to screen for hundreds of pesticides divided into the different groups.[1, 2] Alternatively, different types of GC columns or detection methods for confirming the multiple pesticide residues in the sample can be used.[3] To increase sample throughput and accuracy in matrix, a Thermo Scientific TSQ Quantum GC triple quadrupole mass spectrometer was used in this experiment. The TSQ Quantum GC™ has the ability to acquire 3000 timed-selective reaction monitoring (SRM) transitions per run. Using this method, over 1000 pesticides can be screened and confirmed in only one injection, with at least two SRM transitions for each compound, according to the European Council Directive 96/23/EC.[4]

The combination of triple quadrupole GC-MS/MS and ion trap GC-MS^n with QuEChERS sample preparation method has been investigated for the pesticide analysis in different food matrices.[5, 6, 7] However, solid phase extraction (SPE) as a clean-up procedure is still the official analytical method for pesticide residues analysis in some countries. This application note demonstrates the method for analysis of 303 pesticides in green bean by using an acetonitrile extract, followed by SPE clean-up steps, and analysis by the TSQ Quantum GC-MS/MS using timed-SRM mode. The linearity, average recoveries and precision of all 303 pesticides at 0.010 mg/kg in green bean have been evaluated.

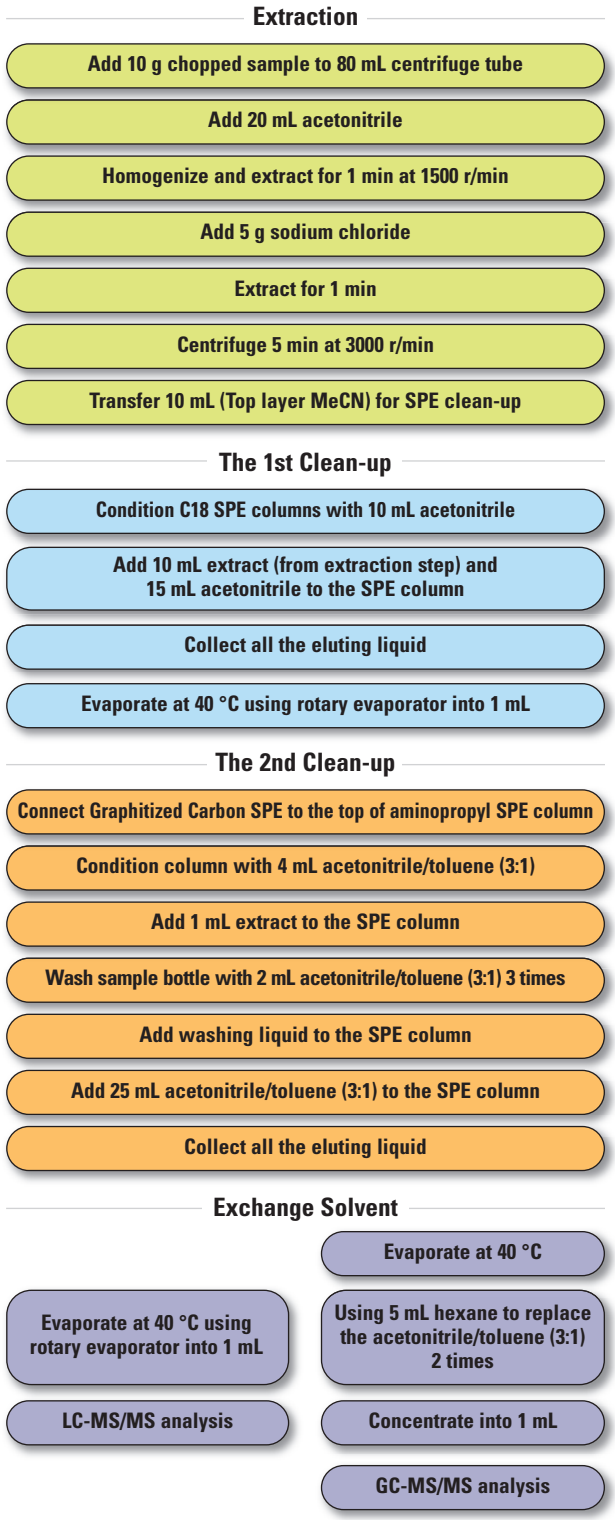


Figure 1: The sample preparation procedures including Extraction, Clean-up and Solvent Exchange

Experimental Conditions

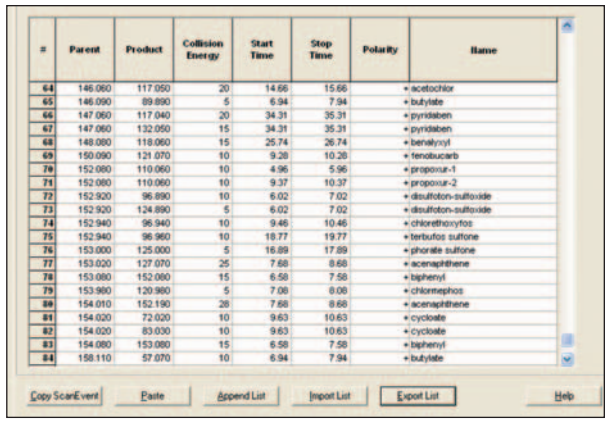
Sample Preparation

Sample preparation includes extraction, two steps of clean-up and solvent exchange according to the Chinese standard method GB/T 19648-2005. Details are given in Figure 1.

Matrix-matched calibration standards were prepared by spiking a mix of 303 pesticide standards into blank green bean and then followed by extraction for GC-MS/MS analysis given in Figure 1. The final concentrations of standards in the matrix are 0.0040, 0.0100, 0.0200, and 0.0400 mg/kg. Five replicate QC samples at 0.010 mg/kg were also prepared under the same sample preparation procedure to evaluate average recovery and precision.

Instrument Conditions

A Thermo Scientific TriPlus liquid autosampler was used to inject a 1 µL aliquot of the final extract, using a hot needle injection. The sample was then separated using a 5% diphenyl/95% dimethyl polysiloxane, 30 m × 0.25 mm i.d., 0.25 µm film thickness column. The determination of target 303 pesticides was carried out by the TSQ Quantum GC MS operated in timed-SRM mode. At least two SRM transitions for each pesticide and their collision energies were selected from the Thermo Scientific Pesticide Analyzer Reference. After a simple modification in an Excel® file, the transitions can be imported directly to the instrument method (Figure 2). Detailed GC-MS/MS conditions are given in Table 1.



#	Parent	Product	Collision Energy	Start Time	Stop Time	Polarity	Name
64	146.060	117.050	20	14.66	15.66	+	acetochlor
65	146.090	89.890	5	6.94	7.94	+	butylate
66	147.060	117.040	20	34.31	35.31	+	pyridaben
67	147.060	132.050	15	34.31	35.31	+	pyridaben
68	148.080	118.060	15	25.74	26.74	+	benalyxyl
69	150.090	121.070	10	9.28	10.28	+	fenobucarb
70	152.080	110.060	10	4.96	5.96	+	propoxur-1
71	152.080	110.060	10	9.37	10.37	+	propoxur-2
72	152.920	96.890	10	6.02	7.02	+	disulfoton-sulfoxide
73	152.920	124.890	5	6.02	7.02	+	disulfoton-sulfoxide
74	152.940	96.940	10	9.46	10.46	+	chlorethoxyfos
75	152.940	96.960	10	18.77	19.77	+	terbufos sulfone
76	153.000	125.000	5	16.89	17.89	+	phorate sulfone
77	153.020	127.070	25	7.68	8.68	+	acenaphthene
78	153.080	152.060	15	6.58	7.58	+	biphenyl
79	153.980	120.980	5	7.08	8.08	+	chlormephos
80	154.010	152.190	20	7.68	8.68	+	acenaphthene
81	154.020	72.020	10	9.63	10.63	+	cycloate
82	154.020	83.030	10	9.63	10.63	+	cycloate
83	154.080	153.080	15	6.58	7.58	+	biphenyl
84	158.110	57.070	10	6.94	7.94	+	butylate

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Figure 2: A portion of the timed-SRM instrument method automatically generated from importing transition information from a spreadsheet

Results and Discussion

The 303 pesticides, including various classes such as organochlorine, organophosphorus, carbamates, and pyrethroids, were analyzed in green bean by GC-MS/MS in 40 minutes, as shown in Figure 3. Because at least two transitions per each pesticide are required for the confirmation according to the European Council Directive 96/23/EC[3], a total of 652 SRM transitions were acquired in one analytical run by the TSQ Quantum GC using timed-SRM mode. The timed-SRM function allows all 652 transitions to be set in many small overlapping windows based on the retention times of each pesticide. The dwell time of each transition was automatically maximized for each compound to give the best sensitivity for all pesticides in one run.

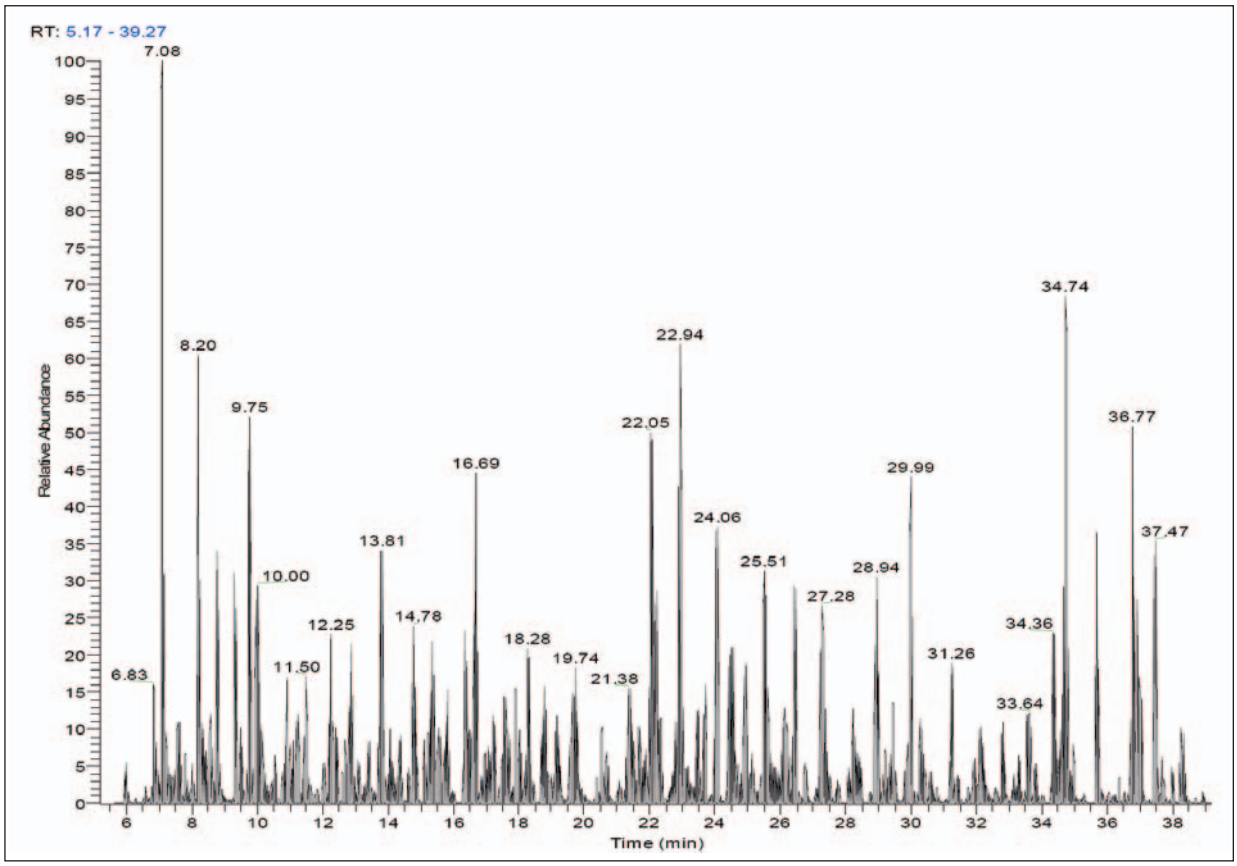

Figure 3: Total ion chromatogram demonstrating elution of 303 pesticides in 40 minutes

TriPlus™ Liquid Autosampler	
Injection Volume:	1 µL
Injection Mode:	hot needle
Pre & Post Injection Dwell Mode:	3 s and 2 s

TSQ Quantum GC Mass Spectrometer	
Ion Source Temp:	280
Emission Current:	25 µA
Ionization Mode:	EI
Ion Volume:	Closed EI
Analytical Mode:	timed-SRM (Selected Reaction Monitoring)
Scan Width:	0.002 *m/z*
Cycle Time:	0.4 s
Peak Width:	Q1 0.7 Da; Q3 0.7 Da
Collision Gas Pressure:	1.5 mTorr (Ar)
Chrom Filter Peak Width:	10 s

TRACE GC Ultra™ Gas Chromatograph	
Injection Mode:	splitless with surge
Surge Pressure:	250 kPa (1 min)
Injection Temp:	250 °C
Oven Program Temp:	50 °C for 1 min, 20 °C min to 150 °C 3 °C/min to 230 °C 10 °C/min to 300 °C, hold for 10 mins
Flow Rate:	1.5 mL/min
Transferline Temp:	280 °C

Table 1: Selected instrumental conditions for the autosampler, GC, and mass spectrometer

The responses of SRM transitions were used for quantification analysis and the ratio of two SRM transitions for each compound were used for confirmation at the same time. Two SRM transitions for Clodinafop-propargyl, Diallete-1, and Molinate show the stability of ion ratio crossing the concentration range from 0.0040 mg/kg – 0.0400mg/kg with the relative standard deviation less than 5% (Table 2).

Concentration (mg/kg)	Clodinafop-propargyl 349-238/349-266	Diallete-1 234-192/234-150	Molinate 187-158/187-126
0.0040	78.74	84.58	33.54
0.0100	73.00	86.60	26.44
0.0200	75.71	84.47	29.68
0.0400	70.75	85.33	33.26
average	74.55	85.25	30.73
RSD%	3.45	0.98	3.36

Table 2: The ion ratios of two SRM transitions for four selected pesticides

Calibration curves from 0.004 to 0.040 mg/kg were created using matrix-matched standard calibration solutions. A summary of the linearity of calibration standards, average recovery and precision data from five replicated QC samples at 0.010 mg/kg are given in Table 3 for all 303 pesticides in green bean. The correlation coefficient (R^2) for most pesticides was greater than 0.99. The signal-to-noise ratios for all 303 pesticides at lowest calibrated level were easily more than 10:1. The average recoveries for most pesticides at 0.010 mg/kg in matrix were within the range of 73% to 110%, with average precision of 10.3% CV.

Conclusions

The analysis of 303 pesticides in green bean was evaluated in one analytical run by using the TSQ Quantum GC, with 652 SRM transitions under timed-SRM mode. This analysis was used in combination with standard sample preparation including SPE as a clean-up procedure. Sensitivity can easily reach to 0.004 mg/kg for all pesticides in green bean. Linearity and recoveries at 0.010 mg/kg were within the requirements. Confirmations were demonstrated by using two SRM transitions for each pesticide with good stability of their ion ratios at different concentrations. Timed-SRM mode is the most effective method for screening and determining the large amounts of pesticides at low levels in the sample matrix. Furthermore, the instrument setup for hundreds of pesticide SRM transitions was simplified by using conditions given in the Pesticide Analyzer Reference, which minimized complex SRM method development work.

No.	Compounds	RT (min)	R^2	0.010 mg/kg Mean	0.010 mg/kg CV (%)
1	dichlorvos	5.99	0.990	92.7	14.7
2	allidochlor	6.29	0.997	82.3	13.5
3	disulfoton-sulfoxide	6.58	0.995	109.9	5.2
4	dichlorbenil	6.82	0.993	85.3	9.2
5	EPTC	6.82	0.995	79.0	14.6
6	dichlormid	6.84	0.993	85.7	9.4
7	biphenyl	7.09	0.986	85.6	12.2
8	butylate	7.50	0.991	78.5	16.1
9	methacrifos	7.55	0.996	99.2	5.8
10	mevinphos	7.55	0.997	98.7	8.3
11	chlormephos	7.62	0.996	84.7	9.3
12	vernolate	7.67	0.994	84.2	12.3
13	propham	7.80	0.996	95.4	9.6
14	pebulate	7.81	0.994	79.1	11.1
15	acenaphthene	8.21	0.994	84.1	8.8
16	chloroneb	8.42	0.992	91.7	12.5
17	2-phenylphenol	8.56	0.997	76.4	17.4
18	crimidine	8.58	0.999	87.5	14.2
19	molinate	8.75	0.998	92.3	16.1
20	isoprocarb	8.79	0.999	98.5	8.0
21	heptanophos	9.32	0.999	97.6	7.7
22	chlorfenprop-methyl	9.51	0.998	88.0	9.5
23	thiomazin	9.74	0.993	90.4	9.9
24	tecnazene	9.74	0.995	105.2	18.6
25	fenobucarb	9.77	0.998	99.5	10.6

Table 3: Summary of linearity, average recovery and precision for 303 pesticides

No.	Compounds	RT (min)	R^2	0.010 mg/kg Mean	0.010 mg/kg CV (%)	No.	Compounds	RT (min)	R^2	0.010 mg/kg Mean	0.010 mg/kg CV (%)
26	propachlor	9.82	0.996	86.6	10.3	78	iprobenfos	14.08	0.996	94.4	11.5
27	propoxur-2	9.84	0.997	94.7	9.0	79	benoxacor	14.19	0.998	100.5	5.6
28	chlorethoxyfos	9.97	0.999	88.0	7.5	80	monalide	14.19	0.998	86.9	16.0
29	demeton-s-methyl	9.98	0.999	76.5	12.0	81	pentachloroaniline	14.29	0.996	91.8	8.4
30	cycloate	10.13	0.997	90.0	5.9	82	pirimicarb	14.40	0.995	92.2	12.5
31	ethoprophos	10.14	0.999	101.3	5.9	83	benfuresate	14.64	0.995	98.0	18.6
32	chlorpropham	10.44	0.999	93.5	8.3	84	desmetryn	14.68	0.994	90.2	13.9
33	chlordimeform	10.55	1.000	87.3	9.1	85	dichlorfenthion	14.79	0.998	97.5	8.4
34	ethalfluralin	10.56	0.997	92.7	16.2	86	dimethachlor	14.81	0.999	91.0	8.8
35	dioxabenzofos	10.79	0.996	95.3	5.5	87	dimethenamid	14.85	0.999	92.6	9.3
36	trifluralin	10.86	0.987	91.7	6.7	88	bromobutide	14.87	0.998	90.7	17.5
37	benfluralin	10.94	0.997	89.9	7.9	89	phosphamidon	14.91	0.997	95.3	19.8
38	tebutam	11.04	0.998	91.7	10.4	90	sporidesmin-1	15.14	0.994	97.2	14.2
39	sulfotep	11.04	0.997	97.5	13.7	91	vinclozolin	15.19	0.996	89.9	11.9
40	diallate-1	11.13	0.999	94.5	6.6	92	chlorpyrifos-methyl	15.19	0.996	89.9	12.0
41	phorate	11.15	0.991	104.2	7.4	93	methyl-parathion	15.21	0.991	95.5	11.9
42	alpha-BHC	11.26	1.000	93.9	13.6	94	plifenate	15.23	0.990	110.7	14.3
43	sulfallate	11.27	0.998	81.5	12.4	95	terbucarb	15.25	0.994	96.3	11.0
44	diallate-2	11.42	0.999	101.7	16.8	96	heptachlor	15.34	0.998	93.3	8.2
45	hexachlorobenzene	11.51	0.999	79.7	4.6	97	toloclofs-methyl	15.37	0.997	93.9	10.0
46	thiometon	11.51	0.998	76.7	8.8	98	simeconazole	15.38	0.998	102.2	9.1
47	simetone	11.56	0.999	97.1	8.2	99	simetryn	15.43	0.996	87.1	15.0
48	atratone	11.81	0.999	89.7	11.5	100	alachlor	15.56	0.998	91.9	13.5
49	simazine	12.03	0.998	78.9	15.4	101	transfluthrin	15.57	0.996	95.8	15.2
50	prometon	12.05	0.999	94.8	4.6	102	tridiphane	15.76	0.999	87.0	8.6
51	atrazine	12.21	0.999	91.7	14.9	103	ronnel	15.84	0.999	87.9	8.1
52	clomazone	12.26	0.999	94.1	3.3	104	prometryn	15.85	0.997	103.3	4.1
53	beta-BHC	12.26	0.991	96.4	7.6	105	metalaxyl	15.87	0.996	94.5	10.8
54	propazine	12.37	1.000	95.8	11.5	106	prosulfocarb	16.00	0.996	99.4	17.5
55	gamma-HCH	12.43	0.999	90.1	11.5	107	sporidesmin-2	16.39	0.994	94.3	11.9
56	terbumeton	12.44	0.993	92.2	19.9	108	terbutryn	16.41	0.997	102.8	4.9
57	quintozene	12.63	0.996	96.9	10.1	109	dithiopyr	16.50	1.000	97.1	6.5
58	terbufos	12.71	0.998	91.0	10.0	110	fenitrothion	16.52	0.999	89.0	5.0
59	cyanophos	12.75	0.993	97.9	8.4	111	esprocarb	16.63	0.999	92.6	6.8
60	terbuthylazine	12.76	0.998	93.4	11.3	112	pirimiphos-methyl	16.71	0.999	101.5	13.9
61	trietazine	12.80	1.000	91.6	11.5	113	ethofumesate	16.73	0.993	106.9	15.2
62	fonofos	12.82	0.999	97.4	6.4	114	aldrin	16.87	0.997	101.7	17.1
63	propetamphos	12.84	0.996	94.7	10.5	115	thiobencarb	16.97	0.988	104.0	17.5
64	propyzamide	12.88	0.999	97.1	12.1	116	dipropetryn	17.08	1.000	100.3	10.0
65	profluralin	12.91	0.999	93.7	10.0	117	malathion	17.17	0.995	106.9	8.7
66	pyrimethanil	13.09	0.993	104.7	8.2	118	metolachlor	17.25	0.997	95.0	8.4
67	diazinon	13.31	0.997	73.4	14.7	119	phoratesulfone	17.26	1.000	87.1	12.4
68	delta-BHC	13.44	0.999	82.4	5.3	120	fenthion	17.45	0.998	96.8	2.7
69	fluchloralin	13.44	0.999	101.3	9.2	121	dimethylvinphos	17.49	0.998	92.6	6.2
70	secbumeton	13.57	0.991	80.8	9.7	122	diethofencarb	17.49	0.997	91.4	7.5
71	dinitramine	13.61	0.997	85.3	12.1	123	aspon	17.51	0.998	101.9	10.2
72	mexacarbate	13.71	0.999	82.4	10.8	124	fenpropimorph	17.53	0.998	93.7	12.8
73	triallate	13.74	0.996	90.6	9.4	125	chlorpyrifos	17.54	0.999	99.1	4.7
74	tefluthrin	13.82	0.998	96.5	7.9	126	dicofol	17.55	0.998	89.9	14.5
75	isazofos	13.83	0.993	93.9	18.9	127	parathion	17.57	0.997	92.8	5.8
76	etrimfos	13.92	0.999	93.5	18.5	128	isomethiozin	17.64	0.989	81.6	15.1
77	sebutylazin	14.02	0.991	101.0	16.8	129	triadimefon	17.70	0.994	122.4	11.3

Table 3 Continued: Summary of linearity, average recovery and precision for 303 pesticides

No.	Compounds	RT (min)	R^2	0.010 mg/kg Mean	0.010 mg/kg CV (%)	No.	Compounds	RT (min)	R^2	0.010 mg/kg Mean	0.010 mg/kg CV (%)
130	chlorthal-dimethyl	17.73	0.997	98.9	9.7	182	diclobutrazole	22.70	0.995	98.3	9.1
131	Isocarbophos	17.92	0.994	94.3	14.4	183	oxadiazone	22.77	0.998	90.2	10.8
132	fenson	17.97	0.993	92.2	12.0	184	myclobutanil	22.81	0.996	101.6	5.5
133	isofenphosoxon	18.02	0.995	103.8	3.7	185	buprofezin	22.92	0.995	90.5	18.2
134	tetraconazole	18.14	0.991	94.0	6.2	186	azaconazole	22.94	0.998	100.4	4.6
135	crufomate	18.20	0.997	103.8	6.0	187	flamprop-methyl	22.94	0.995	96.3	8.5
136	isodrin	18.23	0.997	92.4	8.7	188	flusilazole	23.02	0.990	104.7	11.6
137	flurochloridone	18.25	0.998	88.3	11.4	189	endrin	23.09	0.995	100.9	11.4
138	bromofos	18.30	0.999	92.2	9.3	190	methoprotryne	23.17	0.999	98.6	9.1
139	butralin	18.39	0.995	100.1	5.5	191	bupirimate	23.29	0.997	102.0	19.4
140	diphenamid	18.44	0.995	97.8	17.6	192	nitrofen	23.35	0.994	97.7	14.1
141	octachlorostyrene	18.76	1.000	86.1	7.7	193	metominostrobin-Z	23.43	0.994	100.6	8.0
142	cyprodinil	18.78	0.999	92.7	7.9	194	thifluzamide	23.43	0.992	97.0	11.6
143	isopropalin	18.84	0.998	95.7	5.8	195	kresoxim-methyl	23.45	0.991	95.2	10.5
144	heptachlor epoxide	18.86	0.999	98.0	12.3	196	isoxathion	23.48	0.994	91.8	11.0
145	pendimethalin	19.12	0.999	94.2	13.7	197	perthane	23.71	0.995	105.4	9.7
146	terbufos sulfone	19.15	0.999	96.1	10.0	198	fenoxanil	23.88	0.999	111.8	8.1
147	penconazole	19.17	0.999	93.6	8.3	199	fluazifop-p-butyl	24.04	0.998	97.3	15.3
148	dimethametryn	19.23	0.998	98.3	7.9	200	chloropropylate	24.07	0.998	102.5	7.7
149	dimepiperate	19.59	0.999	97.7	8.6	201	chlorobenzilate	24.08	0.999	97.1	8.7
150	chlorfenvinphos	19.67	0.997	94.5	11.3	202	fensulfothin	24.39	0.997	93.9	14.8
151	isofenphos	19.69	0.998	98.5	9.6	203	pp-DDD	24.46	0.993	92.6	9.0
152	quinalphos	19.74	0.997	96.3	14.5	204	op-DDT	24.56	1.000	99.2	6.7
153	phenthoate	19.81	0.998	86.3	8.9	205	flamprop-isopropyl	24.71	0.998	101.3	4.0
154	mcpa-butoxyethylester	19.89	0.999	96.7	12.2	206	oxadixyl	24.84	0.998	99.6	8.0
155	zoxamide	19.92	0.997	88.5	15.8	207	pyriminobac-methyl(Z)	24.94	0.999	97.1	7.2
156	methidathion	20.38	0.995	97.4	18.7	208	ethion	24.96	0.999	100.9	6.8
157	triflumizole	20.39	0.957	78.2	17.2	209	tetrasul	24.99	0.997	96.3	7.9
158	bromophos-ethyl	20.56	1.000	93.8	10.1	210	chlorthiophos	25.14	0.999	96.8	7.3
159	endosulfan-1	20.62	0.981	98.8	16.0	211	mepronil	25.48	0.997	98.2	7.5
160	propaphos	20.68	0.997	87.6	3.8	212	sulprofos	25.49	0.998	84.7	4.6
161	pyrifenox-2	20.68	0.998	75.7	17.5	213	triazophos	25.75	0.995	95.3	5.9
162	tetrachlorvinphos	21.05	0.996	97.2	11.2	214	ofurace	25.81	0.996	100.0	6.9
163	trans-nonachlor	21.11	0.998	100.3	7.6	215	isoxadifen-ethyl	25.93	0.990	96.0	15.6
164	flutriafol	21.28	0.999	97.7	6.4	216	famphur	25.99	0.994	92.4	11.6
165	butachlor	21.28	0.998	79.6	17.3	217	endosulfan-sulfate	26.00	0.984	95.4	11.8
166	flumetralin	21.40	0.999	94.8	6.5	218	benalyxyl	26.08	0.985	97.4	16.5
167	chlorfenson	21.44	0.994	94.3	9.0	219	edifenphos	26.09	0.995	92.2	9.8
168	napropamide	21.57	0.996	94.1	9.2	220	quinoxyfen	26.12	0.998	95.2	4.0
169	hexaconazole	21.62	0.989	97.9	14.3	221	cyanfenphos	26.22	0.995	97.6	17.6
170	iodofenphos	21.68	0.999	90.2	6.6	222	diofenolan	26.26	0.993	89.0	14.9
171	butamifos	21.68	0.995	99.2	13.8	223	propiconazole-1	26.41	0.995	94.1	11.6
172	bromfenvinfos	21.77	0.996	95.5	13.1	224	pp-DDT	26.45	0.998	94.6	7.2
173	prothiophos	21.90	0.995	91.9	9.2	225	carfentrazone-ethyl	26.49	0.999	99.4	9.0
174	fluorodifen	21.96	0.988	97.2	19.0	226	propiconazole-2	26.75	0.996	103.1	6.9
175	dieldrin	21.99	0.990	109.8	13.8	227	Clodinafop-propargyl	27.09	0.998	99.3	8.4
176	flutolanil	22.05	0.999	102.8	4.5	228	tebuconazole	27.29	0.995	97.7	8.2
177	profenofos	22.10	0.997	94.4	18.7	229	thenylchlor	27.30	0.998	97.0	11.7
178	pp-DDE	22.22	1.000	92.9	10.3	230	pyraflufen ethyl	27.51	0.997	99.0	5.9
179	metominostrobin-E	22.34	0.999	104.8	5.4	231	diclofop methyl	27.77	0.999	98.2	6.7
180	tribufos	22.34	0.987	90.9	14.3	232	diflufenican	28.09	0.995	84.8	9.8
181	pretilachlor	22.36	0.997	95.3	17.1	233	piperonyl butoxide	28.24	0.999	97.0	3.8

Table 3 Continued: Summary of linearity, average recovery and precision for 303 pesticides

No.	Compounds	RT (min)	R^2	0.010 mg/kg Mean	0.010 mg/kg CV (%)	No.	Compounds	RT (min)	R^2	0.010 mg/kg Mean	0.010 mg/kg CV (%)
234	epoxiconazole	28.35	1.000	104.0	3.5	269	pyrazophos	33.56	0.999	95.0	8.0
235	fluotrimazole	28.44	0.999	102.8	3.6	270	dialifos	33.64	0.998	94.0	10.2
236	endrin ketone	28.44	0.994	111.2	12.7	271	pyraclofos	33.77	0.996	98.0	7.4
237	nitralin	28.76	0.996	93.8	14.4	272	fenoxaprop-ethyl	34.01	1.000	94.0	10.0
238	mefenpyr-diethyl	28.90	0.998	93.4	4.8	273	bitertanol	34.35	1.000	99.7	5.5
239	pyributicarb	28.96	0.999	98.0	4.6	274	permethrin-1	34.63	1.000	100.0	9.0
240	benzoylprop-ethyl	28.98	0.998	98.1	7.9	275	pyridaben	34.73	0.999	100.9	7.2
241	bromuconzole	29.03	0.997	96.0	6.3	276	fluquinconazole	34.91	0.997	100.3	8.5
242	phosmet	29.19	0.990	93.4	16.9	277	permethrin-2	34.91	1.000	103.3	9.3
243	pyridaphenthion	29.32	0.989	97.7	8.9	278	coumaphos	34.97	0.999	89.0	15.5
244	bromopropylate	29.39	0.999	97.5	4.0	279	prochloraz	35.10	0.998	82.8	16.6
245	epn	29.45	0.997	99.1	6.9	280	dioxathion	35.26	0.999	89.8	10.6
246	picolinafen	29.82	0.998	96.5	11.5	281	butafenacil	35.65	0.998	100.1	6.1
247	piperophos	29.84	0.998	95.2	10.2	282	fenbuconazole	35.65	0.998	98.5	6.9
248	methoxychlor	29.93	1.000	95.7	6.9	283	cyfluthrin-1	35.85	1.000	96.8	13.2
249	bifenthrin	30.01	0.998	97.6	7.5	284	cyfluthrin-2	36.03	1.000	106.7	16.9
250	fenpropathrin	30.24	0.997	103.0	13.6	285	cyfluthrin-3	36.15	0.982	93.7	11.7
251	fenazaquin	30.30	1.000	115.1	15.2	286	cyfluthrin-4	36.22	0.999	102.0	10.9
252	fenamidone	30.36	0.988	74.3	16.7	287	Cypermethrin-1	36.34	0.994	93.8	8.0
253	tebufenpyrad	30.38	1.000	98.1	7.6	288	halfenprox	36.37	0.977	102.9	13.9
254	etoxazole	30.38	0.991	86.4	7.8	289	Cypermethrin-2	36.52	0.998	107.0	6.4
255	anilofos	30.60	0.999	96.3	6.8	290	flucythrinate-1	36.73	0.998	101.3	3.2
256	tetradifon	30.78	0.997	102.3	9.4	291	etofenprox	36.78	1.000	100.0	7.0
257	mirex	31.28	0.999	95.6	8.5	292	pyridalyl	36.91	0.999	76.4	10.3
258	phosalone	31.39	0.999	97.6	8.0	293	silafluofen	37.03	0.996	90.3	14.1
259	phenothrin	31.43	0.995	103.9	10.6	294	flucythrinate-2	37.04	0.999	97.7	7.2
260	leptophos	31.44	0.997	100.6	10.6	295	pyrimidifen	37.46	0.999	81.5	9.5
261	flurtamone	31.68	0.982	93.0	7.2	296	fenvalerate-1	37.68	0.998	98.5	8.4
262	pyriproxyfen	31.93	1.000	96.7	13.4	297	flumioxazin	37.74	0.998	98.6	10.6
263	mefenacet	32.06	0.998	98.3	5.2	298	fenvalerate-2	37.97	0.998	94.3	9.2
264	cyhalothrin-1	32.59	0.998	100.0	11.4	299	difenconazole-1	38.23	0.998	94.4	6.9
265	fenarimol	32.80	0.999	95.6	4.6	300	difenconazole-2	38.32	0.999	98.7	4.7
266	cyhalothrin-2	33.15	0.996	95.8	4.3	301	deltamethrin-1	38.43	0.994	94.5	15.1
267	azinphos-ethyl	33.29	0.996	99.6	7.7	302	deltamethrin-2	38.69	0.999	99.1	12.4
268	pyriftalid	33.38	1.000	87.9	10.2	303	flumiclorac-pentyl	38.90	0.999	98.0	5.3

Table 3 Continued: Summary of linearity, average recovery and precision for 303 pesticides

References

1. The Japanese Positive List System for Agricultural Chemical Residues in Foods (enforcement on May 29, 2006)
2. GB/T 19648-2005 (466 Pesticides Analysis Method in Fruit and Vegetable GC-MS and LC-MS-MS)
3. GB/T 5009.218-2008 (Determination of Multi Pesticide Residues in Fruits and Vegetables)
4. European Council Directive 96/23/EC
5. AN51880: Rapid Analysis of Pesticides in Difficult Matrices Using GC/MS/MS
6. TN10239: Multi-residue Pesticide Analysis in Rice by a Modified QuEChERS Extraction and Ion Trap GC/MS^n Analysis
7. TN10295: Multi-residue Pesticide Analysis in Green Tea by a Modified QuEChERS Extraction and Ion Trap GC/MS^n Analysis

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