SHIMADZU
Excellence in Science

LCMS-8060NX

Application News

SGLC-LC/MS-081EN

Analysis of 331 Pesticides and Their Metabolites in Dried chilli

Chenyuan Zhang
Shimadzu (Shanghai) Global Laboratory Consumables Co., Ltd. (referred to as SGLC)

User Benefits

- Established an effective, fast and simple sample preparation method for analysis of pesticides in dried chilli.
- Realized simultaneous quantitative analysis of multi pesticides and metabolites, such as organophosphates, organochlorines, pyrethroids, triazole, amides, triazines and carbamate esters.
- SHIMSEN QuEChERS provides precise amounts salts in sachet packs and SPE sorbents in centrifuge tube for enhanced laboratory efficiency and throughput.

■ Introduction

Pesticides are essential tools in agriculture for protecting crops from pests and diseases, but their presence in food products must be carefully monitored to ensure consumer safety. Dried chilies, a common spice, are no exception. Pesticide residues in dried chilli can pose potential health risks and impact the quality of the final products. Therefore, rigorous analysis methods are crucial to determine the levels of pesticide residues in dried chilli and ensure compliance with regulatory standards. In this application, we present a complete workflow according to GB23200.121-2021, from sample preparation using SHIMSEN QuEChERS, to sample analysis using Shim-pack GIST C18-AQ column on Shimadzu LCMS-8060NX.

Table 1. LCMS conditions

UHPLC condition:	
LC system:	Shimadzu Nexera™ LC-40B X3
Column:	Shim-pack GIST C18-AQ, 1.9 µm, 100 × 2.1 mm *1
Column Temp.:	40 °C
Flow rate:	0.3 mL/min
Mobile phase A:	2mM ammonium formate in water containing 0.01% formic acid
Mobile phase B:	2mM ammonium formate in methanol containing 0.01% formic acid
Gradient program:	3% B (0 min) → 3%B (1 min) → 15% B (1.5 min) → 50% B (2.5 min) → 70% B (18 min) → 98% B (23 min) →98% B (27 min) →3% B (27.1 min) →3% B (30 min)
Injection volume:	2 µL (co-injection, 20 µL water)
MS conditions:	
Interface:	Heated ESI (Positive or Negative)
Interface temp:	300 °C
Collision gas:	Ar
Nebulizing gas:	N_2, 3 L/min
Heating gas flow:	Zero air, 10 L/min
DL temperature:	150 °C
Drying gas flow:	N_2, 10 L/min
Heat block temp:	400 °C
MS mode:	MRM

*1 P/N: 227-30807-02

■ Experimental

Materials:

SHIMSEN QuEChERS Extract Salt (P/N: 380-00151)
SHIMSEN QuEChERS II (P/N: 380-05300-16)
Filter and vial:
SHIMSEN Disc HPTFE syringe filter (P/N: 380-00341)
LabTotal Vial (P/N: 227-34001-01)

Sample Preparation:

2.0 g of homogenized sample in 50 mL centrifuge tube was added with 10 mL ultrapure water, and leave for 30 min. Add 15 mL 1% (v/v) acetic acid-acetonitrile solution followed by SHIMSEN QuEChERS extraction salt (6 g $MgSO_4$, 1.5 g NaOAc, P/N: 380-00151) and a single piece of ceramic homogenizer (P/N: 380-00171). The tube was shake vigorously for 1 min after which centrifuged for 5 min at 4200 rpm. 4 mL of the supernatant was transferred into SHIMSEN QuEChERS II (200 mg C18, 80 mg PSA, 20 mg GCB, 600 mg MgSO4, P/N: 380-05300-16), vortex and mix for 1min, centrifuged at 4200 rpm for 5 min, and filtered the supernatant through a 0.22 µm syringe filter for LC-MS/MS analysis. Figure 1 shows the simplified sample preparation workflow.

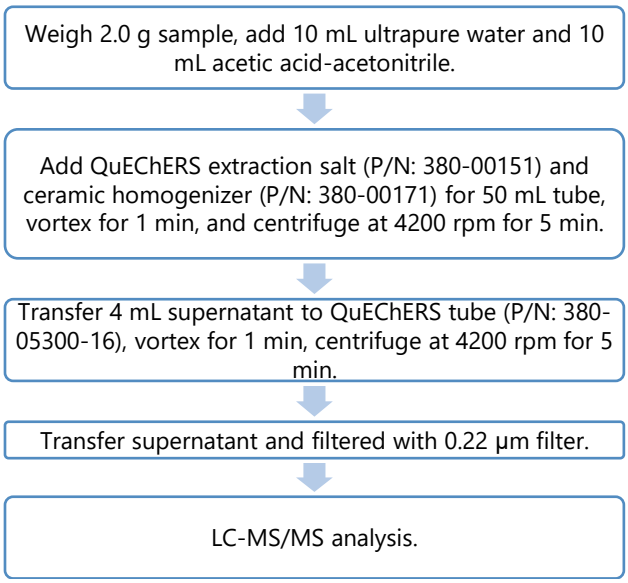

Figure 1. QuEChERS sample preparation workflow for dried chilli.

Table 2. List of MRM used for each pesticides.

No.	Compounds	Mode	Precursor ion	Quantifying ion	Q1 Pre Bias	CE	Q3 Pre Bias	Qualifying ion	Q1 Pre Bias	CE	Q3 Pre Bias
1	methamidophos	+	142.1	94	-16	-15	-17	125.1	-16	-16	-23
2	acephate	+	184.2	143	-20	-8	-15	95	-20	-23	-16
3	omethoate	+	214.1	183	-23	-10	-19	155	-23	-14	-28
4	oxamyl oxime	+	163	72.1	-11	-12	-16	90	-11	-18	-20
5	dinotefuran	+	203.1	129.1	-22	-12	-22	113.1	-22	-10	-12
6	propamocarb	+	189.2	102.1	-30	-20	-23	144.1	-30	-12	-15
7	aldicarb sulfoxide	+	207	89	-13	-15	-19	132	-19	-10	-10
8	aldicarb sulfone	+	240.1	148	-15	-12	-29	166	-13	-11	-20
9	oxamyl	+	237.1	72	-12	-10	-15	90.1	-12	-8	-20
10	nitenpyram	+	271.1	126	-14	-26	-20	189.1	-14	-13	-19
11	oxydemeton-methyl	+	247	169	-30	-24	-30	105.1	-30	-20	-30
12	demeton-S-methyl-sulfone	+	263	169	-30	-24	-30	125	-30	-30	-26
13	methomyl	+	163.1	88	-18	-8	-16	106.1	-18	-10	-19
14	monocrotophos	+	224.1	193	-15	-9	-22	127.1	-18	-20	-15
15	thiamethoxam	+	292	211.1	-30	-20	-22	181.1	-30	-30	-19
16	flonicamid	+	230.1	203.1	-25	-10	-25	174.2	-11	-25	-19
17	chlordimeform	+	197.1	46.2	-21	-35	-19	117.3	-22	-40	-24
18	phosfolan-methyl	+	228	168	-11	-25	-13	109	-11	-15	-22
19	spirotetramat-enol-glucoside	+	464	302	-22	-16	-23	216	-22	-42	-16
20	dicrotophos	+	238	112.1	-12	-12	-11	193	-12	-9	-20
21	imidacloprid	+	256.1	209.1	-29	-14	-22	175.1	-29	-17	-18
22	flumetsulam	+	326.1	129.1	-12	-15	-25	109	-12	-51	-23
23	clothianidin	+	250	169.1	-29	-12	-17	132	-29	-14	-24
24	methiocarb sulfoxide	+	242.1	185.1	-30	-24	-19	122.1	-11	-40	-26
25	imidaclothiz	+	262.1	181.1	-13	-25	-14	122	-10	-40	-15
26	vamidothion	+	287.8	118.1	-14	-35	-22	146.1	-14	-26	-16
27	3-hydroxy carbofuran	+	238.1	163.1	-27	-14	-17	181.2	-27	-10	-19
28	acetamiprid	+	223.1	126.1	-30	-22	-30	56.1	-30	-15	-23
29	mevinphos	+	225	127.1	-25	-17	-23	193	-25	-8	-20
30	methiocarb sulfone	+	258.1	122.1	-13	-23	-24	201.1	-29	-8	-14
31	carbendazim	+	192.1	160.1	-30	-39	-30	132.1	-30	-40	-24
32	dimethoate	+	230	199	-26	-15	-21	125	-26	-30	-22
33	trichlorfon	+	257	109	-10	-34	-22	220.8	-29	-11	-24
34	demeton-S-sulfoxide	+	275.1	197	-10	-17	-15	141	-10	-30	-28
35	metamitron	+	203.1	175	-10	-18	-20	104	-10	-23	-22
36	diethyl aminoethyl hexanoate	+	216.2	143.3	-24	-25	-10	100.3	-24	-25	-23
37	sulfoxaflor	+	278.1	174.2	-21	-11	-19	154.1	-20	-26	-25
38	chloridazon	+	222	92.1	-25	-26	-16	77.1	-25	-36	-30
39	demeton-S-sulfone	+	291	234.8	-14	-15	-18	263	-11	-11	-20
40	thiacloprid	+	253	126.1	-28	-30	-22	99	-28	-43	-17
41	cymoxanil	+	199.1	128.1	-21	-8	-25	111.1	-21	-18	-21
42	florasulam	+	360.1	129.1	-24	-23	-23	109.1	-24	-54	-18
43	fensulfothion oxon	+	293.1	237	-11	-29	-18	265	-11	-23	-20
44	pirimicarb-desmethyl	+	225	72.1	-30	-42	-30	180.1	-30	-15	-30
45	thiabendazole	+	202	175.1	-30	-35	-30	131.1	-30	-25	-24

Table 2. List of MRM used for each pesticides.

No.	Compounds	Mode	Precursor ion	Quantifying ion	Q1 Pre Bias	CE	Q3 Pre Bias	Qualifying ion	Q1 Pre Bias	CE	Q3 Pre Bias
46	tricyclazole	+	190	163	-21	-21	-30	136	-21	-26	-24
47	fensulfothion oxon sulfone	+	309.1	175	-18	-20	-20	253	-15	-25	-20
48	phosfolan	+	256	228	-13	-12	-25	140	-13	-15	-26
49	aldicarb	+	207.9	116	-14	-6	-12	89	-15	-15	-18
50	phosmet oxon	+	302	160	-11	-40	-19	133	-11	-25	-16
51	oxadixyl	+	279.1	219.1	-30	-18	-23	133.1	-30	-30	-24
52	formothion	+	258	199	-23	-8	-16	125	-10	-23	-25
53	cinosulfuron	+	414.1	183.1	-20	-30	-19	157.1	-20	-15	-16
54	metolcarb	+	166.1	109.1	-18	-20	-20	107.1	-18	-15	-19
55	thifensulfuron-methyl	+	388.1	167.1	-19	-16	-18	141.1	-19	-22	-27
56	probenazole	+	224	41	-16	-42	-18	39	-11	-55	-17
57	phosphamidon	+	300	174.1	-15	-22	-17	127	-15	-40	-22
58	cyanazine	+	241.1	104	-30	-31	-19	68.1	-30	-39	-27
59	triasulfuron	+	402.1	167.1	-20	-18	-30	141.1	-20	-20	-26
60	phenamacril	+	217.1	104	-11	-40	-20	189.1	-11	-17	-22
61	metsulfuron-methyl	+	382.1	167.1	-14	-12	-13	199	-13	-20	-15
62	dichlorvos	+	238	109.1	-12	-21	-20	220.9	-12	-11	-15
63	thiophanate-methyl	+	343	151	-12	-19	-29	311	-12	-10	-17
64	thidiazuron	+	221.2	102	-15	-16	-18	128	-15	-17	-23
65	bendiocarb	+	224.1	167.1	-25	-15	-18	109.1	-25	-30	-20
66	spirotetramat-mono-hydroxy	+	304.1	254.1	-11	-18	-29	211	-15	-19	-16
67	propoxur	+	210.1	111.1	-23	-13	-20	168.1	-23	-7	-18
68	chlorsulfuron	+	358.1	141.1	-18	-17	-15	167	-17	-18	-30
69	carbofuran	+	222.1	123.1	-25	-30	-22	165.1	-25	-20	-17
70	fenamiphos sulfoxide	+	319.8	233	-30	-23	-26	292.1	-30	-16	-21
71	metribuzin	+	215.1	187.1	-25	-18	-18	84.1	-25	-21	-30
72	simazine	+	202.1	132	-30	-19	-25	124.1	-30	-17	-23
73	hexazinone	+	253.2	171.1	-30	-20	-18	85.1	-30	-31	-15
74	malaoxon	+	314.9	127	-15	-20	-23	99	-15	-45	-19
75	amidosulfuron	+	370.2	261.1	-13	-14	-27	218.1	-13	-23	-22
76	demeton-S-methyl	+	231	89	-21	-24	-19	61	-10	-21	-23
77	fenthion sulfoxide	+	295	280	-11	-25	-10	109	-11	-25	-20
78	fenamiphos sulfone	+	335.9	266	-16	-14	-29	188.1	-16	-35	-21
79	tebuthiuron	+	229.1	172.1	-30	-20	-30	116.1	-30	-25	-23
80	sulfentrazone	-	385	307.1	18	23	21	199	18	35	20
81	carbaryl	+	202.1	145.1	-22	-9	-26	127.1	-22	-27	-22
82	carboxin	+	236.1	143	-10	-22	-16	87	-10	-23	-19
83	ethirimol	+	210.2	140.1	-13	-22	-25	98.1	-13	-26	-16
84	fenthion sulfone	+	311	125	-11	-18	-15	233.1	-14	-23	-24

Table 2. List of MRM used for each pesticides.

No.	Compounds	Mode	Precursor ion	Quantifying ion	Q1 Pre Bias	CE	Q3 Pre Bias	Qualifying ion	Q1 Pre Bias	CE	Q3 Pre Bias
86	cyantraniliprole	+	475	286	-11	-19	-22	444	-17	-19	-24
87	pirimicarb	+	239.2	72.1	-30	-40	-30	182.2	-30	-19	-30
88	fosthiazate	+	284.1	104.1	-30	-21	-19	228	-30	-15	-24
89	spirotetramat-enol	+	302.1	216	-15	-27	-17	270	-11	-20	-15
90	disulfoton sulfoxide	+	291	185	-30	-20	-19	213	-30	-16	-23
91	chlortoluron	+	213.1	72	-10	-40	-16	46.1	-13	-25	-19
92	phorate sulfoxide	+	277	199	-10	-10	-15	96.9	-10	-34	-19
93	simetryn	+	214.2	96.2	-25	-24	-21	68.2	-24	-30	-27
94	mesosulfuron-methyl	+	504.1	182.1	-34	-25	-18	139	-34	-52	-26
95	methacrifos	+	240.8	209	-12	-10	-23	125	-12	-20	-24
96	disulfoton sulfone	+	306.8	96.9	-15	-20	-18	125	-15	-25	-23
97	tritosulfuron	+	446	195	-16	-20	-23	221	-21	-19	-12
98	phorate sulfone	+	293	171.1	-22	-9	-19	115	-22	-24	-20
99	isoprocarb	+	194.1	95	-21	-30	-17	137.1	-22	-15	-14
100	flutriafol	+	302.1	123	-15	-28	-22	109	-15	-31	-19
101	tribenuron-methyl	+	396.1	155	-19	-30	-30	181	-19	-30	-28
102	atrazine	+	216.1	174.1	-30	-17	-18	96.1	-30	-25	-17
103	imazalil	+	297	159	-15	-24	-15	201	-15	-18	-21
104	isoproturon	+	207.1	72	-23	-40	-28	165.1	-23	-20	-17
105	metazachlor	+	278.1	210.1	-30	-14	-22	134.1	-30	-35	-24
106	fensulfothion	+	309	281	-11	-15	-30	253	-11	-18	-26
107	propachlor	+	212.1	170	-30	-22	-18	94.1	-30	-20	-18
108	chlorpropham	+	214	172	-10	-10	-18	154	-18	-16	-14
109	orthosulfamuron	+	425	199.1	-30	-13	-21	227	-30	-15	-24
110	iodosulfuron-methyl-sodium	+	507.9	167	-20	-19	-19	83	-26	-53	-15
111	diuron	+	233	72	-14	-21	-15	46	-12	-16	-19
112	forchlorfenuron	+	248.1	129.1	-30	-17	-23	93.1	-30	-34	-17
113	isoxaflutole	+	360.1	251	-20	-19	-27	144	-23	-50	-30
114	metalaxyl	+	280.1	220.2	-30	-10	-24	192.2	-30	-25	-20
115	heptenophos	+	251	127	-28	-11	-25	109	-28	-29	-20
116	fensulfothion sulfone	+	325	191	-12	-23	-11	173	-21	-24	-19
117	clethodim sulfone	+	392.1	300	-11	-14	-22	164	-11	-26	-19
118	metazosulfuron	+	476.1	182	-17	-21	-21	295	-17	-16	-23
119	spirotetramat-keto-hydroxy	+	318	300	-12	-13	-23	214	-12	-25	-16
120	methidathion	+	303	145	-21	-8	-15	85.1	-21	-22	-30
121	flumorph	+	372.1	285.1	-17	-15	-29	165.2	-17	-23	-28
122	fenpropidin	+	274.1	147.1	-30	-35	-30	117.2	-14	-53	-21
123	clethodim sulfoxide	+	376.1	206	-18	-14	-16	164.1	-18	-23	-13

Table 2. List of MRM used for each pesticides.

No.	Compounds	Mode	Precursor ion	Quantifying ion	Q1 Pre Bias	CE	Q3 Pre Bias	Qualifying ion	Q1 Pre Bias	CE	Q3 Pre Bias
124	azinphos-methyl	+	318.1	132.1	-15	-14	-23	261	-15	-7	-28
125	phenmedipham	+	318.1	168	-11	-20	-19	136	-11	-35	-16
126	clomazone	+	240.1	125	-27	-10	-22	89.1	-27	-35	-16
127	phosmet	+	318	160	-16	-30	-17	133.2	-16	-35	-25
128	chlorantraniliprole	+	484	452.9	-24	-19	-30	285.9	-24	-16	-30
129	bensulfuron-methyl	+	411.1	149.2	-20	-14	-28	182.2	-20	-35	-19
130	demeton	+	259	89	-13	-9	-18	61	-10	-32	-13
131	pyriftalid	+	319	139	-22	-27	-25	179	-22	-31	-29
132	ametryn	+	228.1	186.1	-30	-25	-19	68.1	-30	-30	-27
133	flucetosulfuron	+	488	156	-25	-21	-28	273	-25	-26	-28
134	fenobucarb	+	208.1	95	-10	-30	-20	152	-18	-15	-12
135	linuron	+	249	160.1	-27	-17	-17	182.1	-28	-14	-19
136	saflufenacil	+	501.1	349	-20	-28	-23	459	-40	-15	-21
137	pyrimethanil	+	200.1	107	-30	-25	-19	168.1	-30	-29	-30
138	propanil	+	218	162	-24	-15	-17	127	-24	-26	-23
139	albendazole	+	266	234	-12	-30	-23	191	-12	-25	-30
140	terbufos sulfone	+	321	171	-22	-12	-17	115	-22	-26	-24
141	terbufos sulfoxide	+	305	186.9	-30	-20	-30	97	-30	-52	-10
142	ethofumesate	+	304.1	241.1	-20	-13	-13	259	-22	-16	-29
143	methiocarb	+	226.1	169.1	-25	-19	-18	121.1	-25	-25	-23
144	diethofencarb	+	268.1	226.1	-30	-15	-24	180.1	-30	-25	-19
145	flurtamone	+	334.1	247.1	-12	-35	-19	303	-12	-20	-23
146	azoxystrobin	+	404.1	372.1	-30	-25	-26	329	-30	-28	-23
147	fludioxonil	+	266.1	229	-10	-14	-18	158	-10	-46	-19
148	fenamidone	+	312.1	236.1	-11	-15	-24	92.1	-11	-24	-16
149	halosulfuron-methyl	+	435	182	-16	-21	-21	139	-10	-40	-16
150	pyrisoxazole	+	289.1	151.1	-11	-14	-18	120	-11	-20	-14
151	terbuthylazine	+	230.1	174.1	-10	-25	-20	104.1	-15	-25	-22
152	dimethenamid	+	276.1	244.1	-14	-23	-25	168.1	-14	-30	-17
153	promecarb	+	208.2	109.1	-22	-10	-19	151.1	-22	-15	-16
154	ethiprole	+	397	255	-20	-45	-26	351	-20	-15	-24
155	boscalid	+	343	307.1	-12	-18	-30	271.1	-12	-30	-26
156	chlorimuron-ethyl	+	415.1	186	-20	-20	-19	83.1	-20	-43	-15
157	propyzamide	+	256.1	190	-28	-13	-20	173	-28	-20	-18
158	paclobutrazol	+	294.1	70.1	-15	-21	-28	125.1	-15	-40	-22
159	dimethomorph	+	388.1	301	-19	-30	-21	165.1	-19	-25	-30
160	mandipropamid	+	412.1	328.1	-11	-10	-22	125	-11	-25	-20
161	isoprothiolane	+	291.1	231.1	-14	-20	-25	189.1	-14	-30	-20
162	flutolanil	+	324.1	262.1	-16	-25	-27	242	-16	-20	-25
163	molinate	+	188.1	126.1	-21	-13	-13	98.1	-20	-20	-18
164	fluxapyroxad	+	382	362.1	-11	-14	-25	342.1	-11	-21	-22
165	ethoxysulfuron	+	399.1	261	-20	-15	-29	218	-20	-26	-23
166	triflusulfuron-methyl	+	493.1	264.1	-18	-15	-29	96.1	-18	-54	-18

Table 2. List of MRM used for each pesticides.

No.	Compounds	Mode	Precursor ion	Quantifying ion	Q1 Pre Bias	CE	Q3 Pre Bias	Qualifying ion	Q1 Pre Bias	CE	Q3 Pre Bias
167	fluopicolide	+	382.9	173	-17	-22	-30	145	-17	-47	-24
168	malathion	+	331	127.1	-17	-12	-13	125	-12	-26	-25
169	mepronil	+	270.2	119.1	-30	-25	-30	228.1	-30	-18	-30
170	myclobutanil	+	289.1	70.1	-30	-21	-28	125.1	-30	-30	-22
171	triadimefon	+	294.1	69.2	-21	-22	-26	197.1	-21	-15	-21
172	propyrisulfuron	+	456	261	-17	-16	-28	196	-17	-15	-20
173	fenpropimorph	+	304.2	147.2	-30	-24	-27	119.1	-30	-30	-22
174	pyrazosulfuron-ethyl	+	415.1	182.1	-21	-18	-19	139.1	-21	-42	-24
175	bromuconazole	+	377.9	158.9	-19	-28	-30	70	-19	-23	-30
176	mefenacet	+	299.1	148.1	-15	-21	-15	120.1	-15	-40	-21
177	sedaxane	+	332	159	-13	-38	-28	292	-12	-15	-20
178	pyridaphenthion	+	341.1	189.1	-17	-15	-20	205.1	-23	-15	-22
179	methoxyfenozide	+	369.2	149.1	-18	-16	-16	313.1	-18	-8	-22
180	prometryn	+	242.2	158.1	-30	-15	-29	200.2	-30	-12	-22
181	triadimenol	+	296.1	70.1	-10	-21	-15	99.2	-14	-16	-20
182	cyproconazole	+	292.1	70.1	-30	-20	-27	125.1	-30	-30	-22
183	triazophos	+	314.1	162.2	-23	-35	-17	119.2	-23	-25	-21
184	fenpyrazamine	+	332	304	-16	-13	-17	272	-12	-13	-21
185	isazofos	+	316	164	-11	-16	-19	122	-11	-26	-10
186	procymidone	+	284	256	-23	-18	-27	67	-19	-44	-28
187	cyclosulfamuron	+	422.2	261	-30	-17	-27	218.1	-30	-28	-22
188	fenarimol	+	331	268.1	-16	-22	-28	259.1	-17	-26	-26
189	fluopyram	+	397	173	-28	-20	-18	207.9	-28	-15	-22
190	fenhexamid	+	301.9	97.1	-15	-23	-19	55.1	-15	-40	-22
191	iprovalicarb	+	321.2	119.1	-30	-19	-22	203.1	-30	-8	-22
192	triticonazole	+	318.1	70.1	-11	-21	-15	125.1	-11	-26	-25
193	tetraconazole	+	372	159.1	-27	-31	-29	70.2	-27	-24	-27
194	ethoprophos	+	243.1	131	-26	-20	-23	97	-27	-32	-17
195	spirotetramat	+	374	302	-14	-17	-23	330	-14	-15	-25
196	flufenacet	+	364	152.1	-19	-30	-15	194.1	-19	-16	-20
197	napropamide	+	272.2	129.2	-30	-16	-23	171.1	-30	-17	-18
198	acetochlor	+	270.1	224.1	-10	-8	-17	148.2	-16	-19	-18
199	chromafenozide	+	395.3	175.1	-14	-40	-20	339.2	-15	-7	-19
200	alachlor	+	270.1	238.1	-30	-10	-26	162.2	-30	-19	-30
201	epoxiconazole	+	330.1	121.2	-12	-20	-26	101	-12	-43	-21
202	cyazofamid	+	325	108.1	-11	-12	-21	261.1	-24	-11	-30
203	metolachlor	+	284.1	252.1	-30	-25	-27	176.2	-30	-20	-19
204	uniconazole	+	292.1	70.1	-21	-24	-27	125	-21	-28	-23
205	fenbuconazole	+	336.9	125.1	-26	-27	-25	70	-26	-20	-28
206	diflubenzuron	+	311	158	-11	-15	-30	141.2	-11	-24	-11
207	iprodione	+	330.1	245	-13	-16	-25	288	-25	-14	-20
208	fipronil desulfinyl	-	387	351	14	17	23	282	14	32	17
209	thifluzamide	+	528.8	148	-26	-38	-26	488.9	-34	-24	-21

Table 2. List of MRM used for each pesticides.

No.	Compounds	Mode	Precursor ion	Quantifying ion	Q1 Pre Bias	CE	Q3 Pre Bias	Qualifying ion	Q1 Pre Bias	CE	Q3 Pre Bias
210	fenothiocarb	+	254.1	72.1	-13	-10	-28	160.1	-13	-14	-16
211	picoxystrobin	+	368.1	205.1	-10	-13	-16	145	-18	-35	-29
212	rotenone	+	395.1	213.1	-14	-22	-23	192.1	-14	-22	-18
213	bupirimate	+	317.1	108	-30	-26	-19	210.2	-30	-23	-22
214	flusilazole	+	316.1	247.1	-30	-18	-27	165.1	-30	-29	-30
215	fenoxycarb	+	302.1	88.1	-15	-21	-16	116.1	-15	-10	-12
216	fenamiphos	+	304.1	217.1	-15	-15	-23	202	-15	-45	-21
217	parathion	+	292	236.2	-11	-15	-23	264	-11	-10	-26
218	cyprodinil	+	226.1	93.1	-30	-34	-16	108.1	-30	-27	-19
219	fenoxanil	+	329.1	302.1	-17	-12	-30	86.1	-17	-22	-15
220	quinalphos	+	299	163.1	-15	-20	-30	147.1	-15	-21	-27
221	dimoxystrobin	+	327	205.1	-30	-15	-30	116	-30	-35	-30
222	fipronil	-	435	330	10	16	21	250	10	28	24
223	tebufenozide	+	353.2	133.1	-18	-20	-24	297.1	-18	-8	-15
224	silthiofam	+	268.1	252	-10	-8	-26	73.1	-10	-27	-30
225	penconazole	+	284.1	70	-14	-17	-27	159	-14	-27	-30
226	penthiopyrad	+	360	276	-18	-11	-28	177	-27	-34	-17
227	chlorbenzuron	+	309	156	-21	-17	-18	111	-10	-45	-23
228	phenthoate	+	321	247	-23	-11	-17	79.1	-23	-41	-30
229	kresoxim-methyl	+	314.1	222.2	-16	-13	-24	235.1	-16	-15	-25
230	fluthiacet-methyl	+	404	274.1	-29	-30	-20	344.1	-29	-23	-26
231	diclobutrazol	+	328	70	-12	-21	-15	70	-12	-22	-15
232	pyrametostrobin	+	382.1	194.1	-14	-18	-15	163	-14	-15	-19
233	penflufen	+	318	141	-12	-20	-16	234	-12	-28	-18
234	iprobenfos	+	289.1	91.1	-30	-21	-16	205	-30	-10	-22
235	fenthion	+	279.2	169.1	-30	-17	-18	247.1	-30	-12	-18
236	etrimfos	+	293	265	-15	-16	-28	125	-15	-24	-23
237	carfentrazone-ethyl	+	429.1	412	-11	-11	-17	346	-15	-26	-29
238	tebuconazole	+	308.1	70.1	-11	-23	-15	125	-11	-31	-25
239	fipronil sulfide	-	419	262	16	29	16	383	12	13	17
240	fonofos	+	247.1	109	-27	-19	-19	137.1	-26	-10	-14
241	sulfotep	+	323	115	-16	-31	-20	171.1	-16	-15	-18
242	isofenphos-methyl	+	332.1	231	-23	-14	-25	121.1	-23	-33	-22
243	edifenphos	+	311	283	-24	-20	-30	111.1	-24	-35	-21
244	propisochlor	+	284.1	224.1	-20	-9	-24	73.1	-20	-12	-29
245	benzovindiflupyr	+	398	342	-15	-18	-23	378	-15	-14	-26
246	zoxamide	+	335.8	187	-16	-24	-20	159	-16	-41	-30
247	anilofos	+	368	199	-18	-20	-21	125	-18	-22	-22
248	propiconazole	+	342.1	159.1	-12	-25	-19	161	-12	-31	-19
249	hexaconazole	+	314.1	70.2	-15	-21	-28	159.2	-15	-29	-30
250	flubendiamide	-	681	254.1	32	26	27	273.8	32	15	28
251	diazinon	+	305	169.1	-30	-15	-18	153.1	-30	-20	-16
252	pyraflufen-ethyl	+	413	339	-20	-19	-19	253	-15	-34	-30

Table 2. List of MRM used for each pesticides.

No.	Compounds	Mode	Precursor ion	Quantifying ion	Q1 Pre Bias	CE	Q3 Pre Bias	Qualifying ion	Q1 Pre Bias	CE	Q3 Pre Bias
253	coumaphos	+	363	227	-18	-26	-23	307.1	-18	-18	-21
254	pyrimorph	+	385.2	242.1	-14	-27	-18	272.1	-14	-33	-21
255	benalaxyl	+	326.2	148.2	-16	-13	-15	294.1	-16	-15	-20
256	chlorfenvinphos	+	358.9	155.1	-18	-12	-30	99	-18	-33	-18
257	metconazole	+	320	70.1	-23	-22	-15	125.1	-11	-40	-25
258	phorate	+	261	75	-29	-10	-30	47	-17	-55	-11
259	fipronil sulfone	-	451	415	17	17	19	282	17	27	18
260	famoxadone	+	392	331	-11	-12	-25	238	-13	-20	-10
261	tolclofos-methyl	+	301.1	125.2	-14	-16	-16	175.1	-20	-23	-20
262	clofentezine	+	303	138.1	-21	-14	-26	102.1	-21	-34	-19
263	prochloraz	+	376	308	-19	-11	-21	266	-19	-17	-29
264	phoxim	+	299	77.1	-30	-20	-30	129.1	-30	-25	-13
265	oxadiargyl	+	340.9	150.9	-13	-27	-27	223	-13	-15	-24
266	pyraclostrobin	+	388.1	194.1	-19	-20	-21	163.1	-19	-35	-30
267	bitertanol	+	338.2	269.2	-17	-9	-29	99.1	-17	-15	-18
268	pirimiphos-methyl	+	306.1	108.1	-30	-31	-19	95	-30	-29	-17
269	triflumuron	+	359.1	156	-17	-16	-30	139	-17	-33	-26
270	phosalone	+	368	182.1	-30	-14	-19	111	-30	-39	-20
271	diniconazole	+	326.1	70	-12	-24	-15	159	-12	-27	-18
272	benzoximate	+	364.1	199	-13	-12	-23	105	-27	-26	-21
273	pyraoxystrobin	+	413.1	205.1	-15	-18	-16	145	-15	-10	-17
274	disulfoton	+	275.2	89.1	-12	-15	-20	60.8	-10	-32	-12
275	chlorpyrifos-methyl	+	321.9	125.1	-22	-23	-23	125.1	-28	-15	-24
276	bifenox	+	359	310	-12	-15	-17	342	-10	-7	-27
277	metrafenone	+	409	209.1	-15	-17	-16	227.1	-20	-22	-18
278	pencycuron	+	329.1	125.1	-17	-15	-22	218.1	-17	-15	-23
279	cyflufenamid	+	413.2	295.1	-20	-10	-30	203	-20	-30	-20
280	ametoctradin	+	276.2	176.1	-10	-35	-20	149	-10	-35	-17
281	difenoconazole	+	406.1	251	-30	-25	-27	337.1	-30	-17	-24
282	EPN	+	324	156.9	-12	-20	-18	296.1	-11	-13	-16
283	cadusafos	+	271.1	159	-30	-20	-29	97	-30	-25	-18
284	isopyrazam	+	360.1	244	-11	-24	-25	320.1	-11	-21	-22
285	dimepiperate	+	264.1	146.1	-29	-7	-15	91.1	-29	-36	-16
286	spinosad A	+	732.4	142	-20	-27	-17	98.1	-20	-55	-21
287	diflufenican	+	395.1	266	-14	-35	-21	246	-14	-34	-19
288	ipconazole	+	334.2	70.1	-22	-26	-21	125.1	-13	-43	-13
289	triflumizole	+	346.1	73.1	-17	-25	-30	278	-17	-22	-30
290	indoxacarb	+	528.1	293	-26	-15	-21	249.1	-26	-17	-27
291	trifloxystrobin	+	409.1	186.1	-20	-40	-20	145	-20	-20	-26
292	prosulfocarb	+	252	91	-12	-45	-19	128	-12	-15	-15
293	cycloxydim	+	326.2	280.2	-12	-12	-21	180.2	-12	-20	-14
294	amisulbrom	+	466	227	-10	-19	-13	148	-10	-47	-29
295	pretilachlor	+	312.2	252.2	-15	-29	-28	176.2	-15	-35	-18
296	clethodim	+	360.2	164.1	-18	-19	-17	268.1	-18	-11	-30
297	hexaflumuron	-	459	438.9	16	12	29	175.1	16	36	29
298	fenaminstrobin	+	434.1	171	-21	-40	-20	212	-12	-14	-25

Table 2. List of MRM used for each pesticides.

No.	Compounds	Mode	Precursor ion	Quantifying ion	Q1 Pre Bias	CE	Q3 Pre Bias	Qualifying ion	Q1 Pre Bias	CE	Q3 Pre Bias
299	fluoroglycofen-ethyl	+	465.1	344	-17	-15	-26	223	-17	-33	-17
300	profenofos	+	372.9	302.8	-18	-25	-30	345	-18	-20	-24
301	quizalofop-ethyl	+	373	299	-13	-28	-20	270.9	-11	-26	-28
302	fenoxaprop-ethyl	+	362.1	288	-28	-26	-20	121.1	-28	-30	-23
303	oxyfluorfen	+	362	316	-18	-19	-25	140	-27	-52	-27
304	spinosad D	+	746.4	142	-28	-30	-17	98	-22	-55	-12
305	oxaziclomefone	+	376.1	190.1	-19	-34	-20	161.1	-19	-40	-28
306	diclofop-methyl	+	358	281	-25	-15	-22	120.2	-18	-27	-26
307	cyflumetofen	+	465.2	173	-14	-24	-18	145	-14	-55	-28
308	metamifop	+	441.1	288	-16	-24	-16	180	-21	-19	-21
309	terbufos	+	289	103.2	-14	-9	-18	57.1	-14	-24	-24
310	enestroburin	+	400.1	178	-19	-25	-14	137	-15	-15	-16
311	teflubenzuron	-	379	339	13	11	22	359	13	6	24
312	sethoxydim	+	328.1	178.1	-12	-19	-21	282.2	-12	-12	-22
313	fluazifop-butyl	+	384	328	-14	-11	-16	282	-14	-10	-30
314	furathiocarb	+	383.2	252.1	-27	-13	-27	195	-27	-10	-21
315	picolinafen	+	377.1	238.1	-19	-40	-24	359.1	-19	-25	-17
316	imibenconazole	+	411	125.1	-20	-31	-22	171	-20	-20	-18
317	propaquizafop	+	444.1	100.1	-23	-19	-19	371	-23	-16	-18
318	buprofezin	+	306.1	116.1	-30	-23	-12	201.1	-30	-20	-22
319	lactofen	+	479.2	344	-18	-25	-25	223.1	-18	-25	-25
320	tolfenpyrad	+	384.1	197.1	-10	-35	-12	154.1	-10	-35	-29
321	metaflumizone	-	505.1	302	24	21	30	285	24	48	28
322	oxadiazon	+	345	303	-16	-13	-13	220	-24	-18	-23
323	fluazinam	-	463	416	22	20	13	398	13	17	17
324	butachlor	+	312.2	238.1	-23	-11	-28	162	-16	-22	-13
325	pyriproxyfen	+	322.1	96.1	-30	-10	-10	185.1	-30	-20	-20
326	piperonyl butoxide	+	356.3	177.1	-24	-31	-19	119	-24	-22	-22
327	coumoxystrobin	+	437.1	205.1	-12	-10	-16	145.1	-12	-35	-17
328	ethion	+	385	199	-19	-15	-22	143	-19	-20	-25
329	pyribenzoxim	+	610.1	413.1	-22	-25	-30	180.1	-22	-20	-19
330	emamectin benzoate	+	886.5	158.1	-40	-25	-17	82.1	-40	-55	-15
331	spinetoram L	+	760.7	142.2	-22	-32	-25	98.2	-28	-55	-18
332	chlorpyrifos	+	351.9	199.9	-27	-18	-21	97	-27	-25	-18
333	spinetoram J	+	748.5	142.1	-40	-33	-14	98.2	-30	-55	-21
334	lufenuron	-	509	326	36	17	21	339	36	11	22
335	pendimethalin	+	282.2	212.1	-30	-10	-23	194	-30	-18	-20
336	hexythiazox	+	353.1	228	-18	-20	-24	168.1	-18	-30	-30
337	triallate	+	304	143	-15	-27	-25	86	-15	-17	-16
338	tralkoxydim	+	330.2	284.2	-16	-10	-30	138.1	-16	-25	-25
339	flucythrinate	+	469	412	-23	-14	-22	181	-30	-36	-10
340	flufenoxuron	+	489	158.1	-11	-20	-12	141.2	-11	-39	-17
341	propargite	+	368.2	231.2	-26	-17	-25	175.2	-26	-10	-19
342	dinocap	-	295.1	209	11	32	22	134.1	21	51	25
343	etoxazole	+	360.1	141.1	-30	-13	-26	113.1	-30	-35	-21
344	butralin	+	296.2	240.1	-14	-12	-25	222.1	-14	-21	-24
345	fenpropathrin	+	350.3	97.2	-12	-45	-19	125.2	-10	-23	-28

Table 2. List of MRM used for each pesticides.

No.	Compounds	Mode	Precursor ion	Quantifying ion	Q1 Pre Bias	CE	Q3 Pre Bias	Qualifying ion	Q1 Pre Bias	CE	Q3 Pre Bias
346	fenpyroximate	+	422.2	366.1	-30	-30	-26	138.1	-30	-25	-26
347	proquinazid	+	373	331	-18	-23	-18	289	-14	-40	-22
348	flumetralin	+	422.1	107.1	-22	-54	-22	143	-15	-47	-27
349	chlorfluazuron	+	540	382.9	-26	-21	-27	158	-26	-20	-30
350	spirodiclofen	+	411.1	71.2	-21	-16	-28	313.1	-21	-11	-22
351	deltamethrin	+	523	281	-36	-16	-22	506	-38	-11	-28
352	fenazaquin	+	307	161.1	-15	-10	-30	131	-15	-46	-24
353	fenvalerate	+	437	167.1	-17	-15	-26	125	-16	-40	-24
354	pyridaben	+	365.1	147.1	-18	-42	-27	309	-18	-23	-22
355	bioresmethrin	+	339.2	171.1	-24	-25	-18	128.1	-24	-25	-23
356	tau-fluvalinate	+	503.1	208	-40	-13	-22	181	-40	-30	-18
357	methoprene	+	279.2	191.2	-10	-9	-15	237.2	-10	-9	-28
358	abamectin	+	890.5	305.2	-34	-25	-22	567.3	-34	-14	-30
359	permethrin	+	408.2	183.1	-14	-14	-20	355.2	-21	-8	-27
360	etofenprox	+	394	177.1	-19	-26	-20	107	-19	-33	-19
361	bifenthrin	+	440.3	181.1	-16	-21	-18	166.2	-16	-43	-18
362	pyridalyl	+	491.9	110.9	-18	-27	-19	108.9	-18	-28	-20
363	ivermectin	+	892.5	569.2	-26	-16	-40	307.1	-26	-28	-20
364	benazolin-ethyl	+	272	198	-13	-15	-25	170	-18	-25	-19
365	pirimicarb-desmethyl-formamido	+	253.1	72	-20	-25	-20	225	-17	-10	-18
366	prochloraz metabolite BTS44595	+	325	282.1	-11	-15	-21	284.1	-11	-15	-21
367	prochloraz metabolite BTS44596	+	353	308	-12	-14	-17	310	-12	-14	-17
368	pyrethrin I	+	329.2	161.1	-11	-10	-19	133	-11	-19	-25
369	pyrethrin II	+	373.2	161.1	-11	-11	-19	133.1	-13	-20	-28
370	triflumizole metabolite FM-6-1	+	295	43.1	-10	-23	-18	73	-10	-18	-15
371	isocarbophos	+	231	121	-16	-19	-23	109	-11	-24	-13
372	cyazofamid metabolite CCIM	-	216	179.1	10	31	16	180.2	15	25	17
373	isoxaflutole-diketonitrile	-	358.1	79	12	23	26	64	12	50	11
374	spiromesifen	+	388	273.1	-14	-15	-21	255.2	-14	-27	-19
375	novaluron	+	493	158	-15	-18	-28	141.1	-15	-40	-27

[a] Pesticide contains two chromatograpeseedhic peaks, which are either its cis-trans isomers or non-enantiomeric isomers. The sum of the peak areas need to be used for quantification.

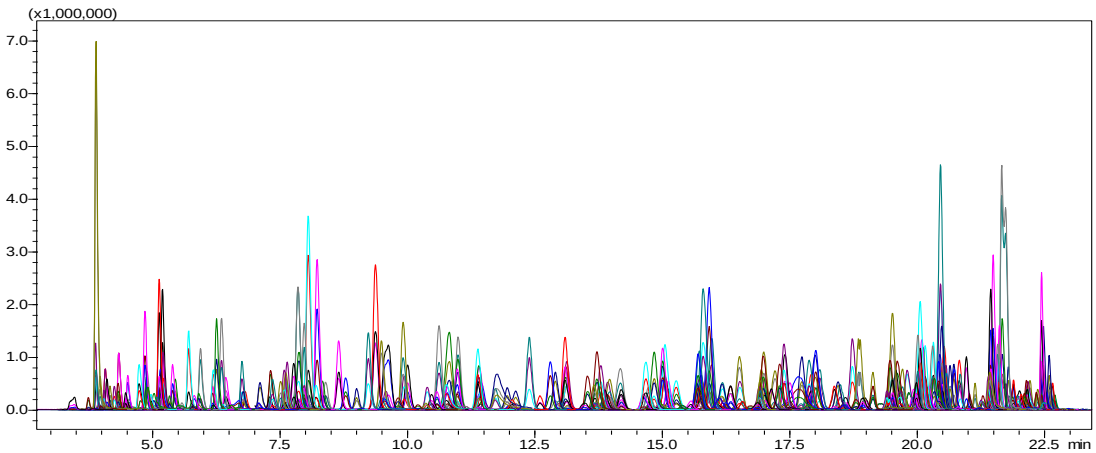

Figure 2. MRM chromatogram of 331 pesticides and their metabolites standards in dried chilli matrix (concentration: 50 ng/mL).

Table 3. List of recovery and %RSD for each pesticides.

No.	Compounds	Spiked amount (0.05 mg/kg, n=3)	
		Avg recovery/%	%RSD
1	methamidophos	81.41	1.37
2	acephate	85.54	0.33
3	omethoate	85.11	1.50
4	oxamyl oxime	85.97	1.46
5	dinotefuran	90.60	4.50
6	propamocarb	85.78	3.23
7	aldicarb sulfoxide	89.15	3.29
8	aldicarb sulfone	89.29	1.91
9	oxamyl	88.40	4.17
10	nitenpyram	79.15	3.26
11	oxydemeton-methyl	83.43	1.86
12	demeton-S-methyl-sulfone	84.58	4.78
13	methomyl	85.05	1.57
14	monocrotophos	83.59	1.34
15	thiamethoxam	86.81	3.18
16	flonicamid	99.38	8.99
17	chlordimeform	77.32	4.34
18	phosfolan-methyl	87.39	3.30
19	spirotetramat-enol-glucoside	73.54	4.23
20	dicrotophos	84.98	7.11
21	imidacloprid	88.94	2.47
22	flumetsulam	82.93	1.73
23	clothianidin	97.53	1.50
24	methiocarb sulfoxide	89.88	4.51
25	imidaclothiz	82.22	4.22
26	vamidothion	88.10	4.06
27	3-hydroxy carbofuran	89.45	2.86
28	acetamiprid	87.05	3.91
29	mevinphos*	85.84	1.53
30	methiocarb sulfone	80.63	2.92
31	carbendazim	73.94	1.36
32	dimethoate	86.90	3.71
33	trichlorfon	83.07	6.24
34	demeton-S-sulfoxide	90.39	2.70
35	metamitron	81.16	9.06
36	diethyl aminoethyl hexanoate	80.77	1.59
37	sulfoxaflor*	79.11	5.37
38	chloridazon	81.03	0.93
39	demeton-S-sulfone	91.54	2.67
40	thiacloprid	82.40	0.30
41	cymoxanil	89.37	4.22
42	florasulam	88.30	3.40
43	fensulfothion oxon	83.30	0.73
44	pirimicarb-desmethyl	88.13	2.41
45	thiabendazole	56.65	0.90
46	tricyclazole	74.04	2.18
47	fensulfothion oxon sulfone	85.34	3.23
48	phosfolan	88.27	1.32
49	aldicarb	82.66	2.22
50	phosmet oxon	87.02	3.06
51	oxadixyl	86.12	5.80
52	formothion	88.86	2.35
53	cinosulfuron	88.43	0.97
54	metolcarb	81.12	1.81
55	thifensulfuron-methyl	82.37	0.80
56	probenazole	88.57	1.60
57	phosphamidon*	87.55	2.35
58	cyanazine	87.89	3.10
59	triasulfuron	86.60	0.60
60	phenamacril	83.01	1.02
61	metsulfuron-methyl	82.45	3.43
62	dichlorvos	84.42	2.16
63	thiophanate-methyl	83.65	2.34
64	thidiazuron	57.79	5.60
65	bendiocarb	85.27	2.74
66	spirotetramat-mono-hydroxy	92.67	1.59
67	propoxur	87.76	2.74
68	chlorsulfuron	74.64	1.43
69	carbofuran	85.67	0.34
70	fenamiphos sulfoxide	87.54	1.29
71	metribuzin	72.71	2.00
72	simazine	80.01	2.45
73	hexazinone	86.85	0.87
74	malaoxon	93.74	2.26
75	Amidosulfuron	72.90	1.52
76	demeton-S-methyl	99.28	4.65
77	fenthion sulfoxide	81.70	6.42

No.	Comppounds	Spiked amount (0.05 mg/kg, n=3)	
		Avg recovery	%RSD
78	fenamiphos sulfone	91.14	2.34
79	tebuthiuron	85.04	1.16
80	Sulfentrazone	90.51	6.09
81	carbaryl	83.74	2.24
82	carboxin	79.25	0.51
83	ethirimol	61.54	3.10
84	fenthion sulfone	102.11	1.50
85	penoxsulam	99.18	3.76
86	cyantraniliprole	82.86	1.48
87	pirimicarb	85.82	2.59
88	fosthiazate	86.89	0.40
89	spirotetramat-enol	105.71	3.50
90	disulfoton sulfoxide	92.51	3.05
91	chlortoluron	82.34	1.50
92	phorate sulfoxide	90.47	3.01
93	simetryn	79.18	1.48
94	mesosulfuron-methyl	99.37	5.77
95	methacrifos*	86.41	0.43
96	disulfoton sulfone	87.65	1.62
97	tritosulfuron	73.39	4.73
98	phorate sulfone	86.64	1.68
99	isoprocarb	83.13	1.64
100	flutriafol	91.39	3.67
101	tribenuron-methyl	78.34	2.38
102	atrazine	85.04	1.42
103	imazalil	77.33	0.42
104	isoproturon	81.64	1.49
105	metazachlor	88.21	4.32
106	fensulfothion	86.63	1.20
107	propachlor	87.22	3.08
108	chlorpropham	86.83	0.87
109	orthosulfamuron	80.40	3.68
110	iodosulfuron-methyl-sodium	70.84	2.68
111	diuron	80.89	1.37
112	forchlorfenuron	61.84	5.80
113	isoxaflutole	94.90	0.94
114	metalaxyl	87.11	1.02
115	heptenophos	83.59	0.23
116	fensulfothion sulfone	81.86	12.49
117	clethodim sulfone	91.98	3.00
118	metazosulfuron	62.95	4.09
119	spirotetramat-keto-hydroxy	93.03	2.64
120	methidathion	86.48	3.27
121	flumorph	90.14	1.58
122	fenpropidin	77.88	0.28
123	clethodim sulfoxide*	83.14	2.87
124	azinphos-methyl	84.78	6.59
125	phenmedipham	84.61	2.83
126	clomazone	83.97	1.71
127	phosmet	90.66	2.03
128	chlorantraniliprole	90.74	3.30
129	bensulfuron-methyl	88.32	1.45
130	demeton	83.07	0.10
131	pyriftalid	85.71	1.54
132	ametryn	83.70	0.71
133	flucetosulfuron*	83.97	1.87
134	fenobucarb	86.38	1.49
135	linuron	87.13	0.84
136	saflufenacil	92.80	2.92
137	pyrimethanil	72.35	3.56
138	propanil	82.71	3.49
139	albendazole	34.43	5.81
140	terbufos sulfone	92.23	3.64
141	terbufos sulfoxide	86.42	0.79
142	ethofumesate	86.25	2.40
143	methiocarb	86.48	4.19
144	diethofencarb	86.07	2.83
145	flurtamone	88.71	2.28
146	azoxystrobin	91.68	0.55
147	fludioxonil	91.57	1.12
148	fenamidone	92.20	1.60
149	halosulfuron-methyl	73.19	4.26
150	pyrisoxazole*	85.22	2.88
151	terbuthylazine	87.17	2.41
152	dimethenamid	85.59	3.12
153	promecarb	85.96	2.34
154	ethiprole	77.04	6.45
155	boscalid	91.09	1.63
156	chlorimuron-ethyl	76.32	2.90

Table 3 continue. List of recovery and %RSD for each pesticides

No.	Compounds	Spiked amount (0.05 mg/kg, n=3)		No.	Comppounds	Spiked amount (0.05 mg/kg, n=3)	
		Avg recovery/%	%RSD			Avg recovery/%	%RSD
157	propyzamide	88.16	6.49	237	carfentrazone-ethyl	91.64	1.21
158	paclobutrazol	88.96	3.49	238	tebuconazole	91.38	2.54
159	dimethomorph	92.44	2.56	239	fipronil sulfide	89.97	1.67
160	mandipropamid	91.17	4.07	240	fonofos	92.36	7.74
161	isoprothiolane	92.14	1.41	241	sulfotep	88.72	3.18
162	flutolanil	95.85	1.66	242	isofenphos-methyl	91.88	5.25
163	molinate	82.95	3.14	243	edifenphos	87.82	2.92
164	fluxapyroxad	89.19	2.83	244	propisochlor	83.93	5.54
165	ethoxysulfuron	73.18	2.65	245	benzovindiflupyr	91.18	3.12
166	triflusulfuron-methyl	84.70	0.15	246	zoxamide	86.35	2.32
167	fluopicolide	84.17	1.66	247	anilofos	91.46	2.61
168	malathion	89.60	1.05	248	propiconazole	93.90	2.08
169	mepronil	88.57	4.59	249	hexaconazole	90.80	2.34
170	myclobutanil	88.60	3.07	250	flubendiamide	107.44	4.14
171	triadimefon	86.42	2.44	251	diazinon	91.78	3.07
172	propyrisulfuron	82.63	3.44	252	pyraflufen-ethyl	96.56	1.87
173	fenpropimorph	81.05	5.33	253	coumaphos	88.55	6.56
174	pyrazosulfuron-ethyl	76.77	2.15	254	pyrimorph	91.90	4.73
175	bromuconazole	89.33	3.01	255	benalaxyl	89.78	2.26
176	mefenacet	91.25	3.26	256	chlorfenvinphos*	94.03	3.51
177	sedaxane*	83.70	5.38	257	metconazole	87.88	3.24
178	pyridaphenthion	93.31	1.77	258	phorate	86.75	10.40
179	methoxyfenozide	95.41	2.22	259	fipronil sulfone	89.93	3.88
180	prometryn	84.18	0.93	260	famoxadone	101.25	19.11
181	triadimenol	101.41	1.81	261	tolclofos-methyl	99.87	6.07
182	cyproconazole	92.31	4.38	262	clofentezine	89.80	6.97
183	triazophos	92.04	3.41	263	prochloraz	93.05	2.83
184	fenpyrazamine	89.21	3.12	264	phoxim	89.49	2.81
185	isazofos	91.22	1.60	265	oxadiargyl	86.01	19.61
186	procymidone	86.27	14.48	266	pyraclostrobin	90.99	2.12
187	cyclosulfamuron	94.50	5.70	267	bitertanol	76.09	18.11
188	fenarimol	91.65	2.31	268	pirimiphos-methyl	89.83	3.11
189	fluopyram	84.16	2.23	269	triflumuron	83.73	5.10
190	fenhexamid	89.17	1.43	270	phosalone	87.96	1.94
191	iprovalicarb*	91.47	0.73	271	diniconazole	87.24	2.54
192	triticonazole	89.62	7.61	272	benzoximate	92.98	4.09
193	tetraconazole	96.32	3.01	273	pyraoxystrobin	89.57	2.46
194	ethoprophos	87.53	3.21	274	disulfoton	72.93	16.13
195	spirotetramat	87.38	0.59	275	chlorpyrifos-methyl	92.53	0.77
196	flufenacet	89.61	1.06	276	bifenox	68.57	24.53
197	napropamide	86.74	0.83	277	metrafenone	88.09	0.23
198	acetochlor	86.68	3.14	278	pencycuron	84.07	3.13
199	chromafenozide	93.88	1.76	279	cyflufenamid	94.35	1.14
200	alachlor	88.44	1.76	280	ametoctradin	71.31	2.90
201	epoxiconazole	93.71	2.82	281	difenoconazole*	94.43	7.50
202	cyazofamid	90.69	0.47	282	EPN	16.85	29.95
203	metolachlor	90.21	1.93	283	cadusafos	87.24	2.64
204	uniconazole	89.13	3.39	284	isopyrazam*	85.89	1.06
205	fenbuconazole	94.81	4.61	285	dimepiperate	85.80	1.24
206	diflubenzuron	86.46	6.78	286	spinosad A	76.51	2.22
207	iprodione	149.05	42.70	287	diflufenican	101.29	2.90
208	fipronil desulfinyl	93.38	2.77	288	ipconazole*	93.93	1.82
209	thifluzamide	93.85	1.05	289	triflumizole	100.59	7.96
210	fenothiocarb	84.59	7.09	290	indoxacarb	107.37	3.91
211	picoxystrobin	94.75	0.87	291	trifloxystrobin	98.79	3.98
212	rotenone	97.73	1.34	292	prosulfocarb	83.44	2.95
213	bupirimate	78.56	5.34	293	cycloxydim	81.63	5.07
214	flusilazole	97.05	5.89	294	amisulbrom	97.06	14.61
215	fenoxycarb	87.82	1.61	295	pretilachlor	90.53	1.44
216	fenamiphos	84.49	1.41	296	clethodim	84.49	2.89
217	parathion	47.33	54.65	297	hexaflumuron	95.80	3.33
218	cyprodinil	58.38	3.41	298	fenaminstrobin	87.02	0.99
219	fenoxanil*	96.55	5.28	299	fluoroglycofen-ethyl	95.66	2.63
220	quinalphos	90.95	1.11	300	profenofos	89.48	2.56
221	dimoxystrobin	90.87	4.15	301	quizalofop-ethyl	86.33	3.85
222	fipronil	98.59	3.09	302	fenoxaprop-ethyl	88.28	0.97
223	tebufenozide	91.54	1.30	303	oxyfluorfen	87.35	11.84
224	silthiofam	114.71	2.85	304	spinosad D	71.94	2.32
225	penconazole	87.44	3.85	305	oxaziclomefone	89.07	2.93
226	penthiopyrad	90.98	3.41	306	diclofop-methyl	66.62	8.17
227	chlorbenzuron	75.63	8.30	307	cyflumetofen	89.91	1.52
228	phenthoate	87.48	5.13	308	metamifop	90.55	4.98
229	kresoxim-methyl	93.99	1.23	309	terbufos	84.18	10.09
230	fluthiacet-methyl	86.58	5.29	310	enestroburin	89.42	2.57
231	diclobutrazol	94.76	1.36	311	teflubenzuron	84.79	8.50
232	pyrametostrobin	87.55	1.96	312	sethoxydim	77.69	4.03
233	penflufen	85.11	3.59	313	fluazifop-butyl	85.11	3.71
234	iprobenfos	91.96	4.64	314	furathiocarb	86.04	0.68
235	fenthion	91.61	3.09	315	picolinafen	82.88	2.25
236	etrimfos	83.59	1.99	316	imibenconazole	83.08	6.22

Table 3 continue. List of recovery and %RSD for each pesticides.

No.	Comppounds	Spiked amount (0.05 mg/kg, n=3)	
		Avg recovery/%	%RSD
317	propaquizafop	89.67	2.45
318	buprofezin	84.75	3.69
319	lactofen	88.07	1.77
320	tolfenpyrad	86.45	2.99
321	metaflumizone	88.83	5.53
322	oxadiazon	77.29	12.43
323	fluazinam	93.81	7.04
324	butachlor	85.69	3.19
325	pyriproxyfen	84.31	3.69
326	piperonyl butoxide	88.96	2.10
327	coumoxystrobin	79.42	4.05
328	ethion	85.02	1.18
329	pyribenzoxim	91.67	5.19
330	emamectin benzoate	70.26	3.30
331	spinetoram L	68.80	13.10
332	chlorpyrifos	87.93	6.35
333	spinetoram J	72.81	5.94
334	lufenuron	89.76	5.50
335	pendimethalin	76.69	5.95
336	hexythiazox	84.51	4.07
337	triallate	77.59	7.91
338	tralkoxydim	80.77	0.45
339	flucythrinate	106.79	28.47
340	flufenoxuron	88.14	4.58
341	propargite	87.92	5.60
342	dinocap*	81.48	2.40
343	etoxazole	81.59	4.57
344	butralin	84.43	1.88
345	fenpropathrin	86.00	8.25
346	fenpyroximate	83.18	5.00
347	proquinazid	66.29	3.00
348	flumetralin	79.83	6.83
349	chlorfluazuron	78.70	2.35
350	spirodiclofen	80.30	1.42
351	deltamethrin	99.45	8.01
352	fenazaquin	72.23	3.92
353	fenvalerate	70.30	10.72
354	pyridaben	85.37	3.88
355	bioresmethrin	78.92	8.87
356	tau-fluvalinate	77.65	11.41
357	methoprene	84.23	0.70
358	abamectin	101.64	17.90
359	permethrin*	95.17	14.82
360	etofenprox	74.85	1.16
361	bifenthrin	88.57	2.59
362	pyridalyl	59.47	0.78
363	ivermectin	70.91	9.95
364	benazolin-ethyl	84.92	3.44
365	pirimicarb-desmethyl-formamido	87.88	1.50
366	prochloraz metabolite BTS44595	102.73	2.15
367	prochloraz metabolite BTS44596	103.26	0.31
368	pyrethrin I	82.11	8.03
369	pyrethrin II	96.41	9.36
370	triflumizole metabolite FM-6-1	84.39	0.74
371	isocarbophos	86.87	2.68
372	cyazofamid metabolite CCIM	83.24	1.42
373	isoxaflutole-diketonitrile	65.39	1.67
374	spiromesifen	72.53	2.22
375	novaluron	73.71	9.76

■ Results and Discussion

Dried chilli blank matrix was spiked with a standard solution of pesticides to a final concentration of 0.05 mg/kg. QuEChERS method sample preparation was performed according to Figure 1. Figure 2 shows the MRM chromatogram of the pesticide standards in dried chilli matrix. Three independent experiment was performed to determine average recovery and %RSD. Results show 359 compounds having a good recovery rate between 70.0%-120.0%. %RSD show 370 compounds were below 20%. Recovery and %RSD for all the compounds are shown in Table 3.

■ Conclusion

This study presents a method for the determination of residues of 331 pesticides and their metabolites in dried chilli. Shimadzu SHIMSEN QuEChERS products were used for clean-up of the dried chilli, followed by analysis using Shim-pack GIST C18-AQ column on Shimadzu LCMS-8060NX. The recovery and reproducibility was determined using 0.05 mg/kg spiked dried chilli and dried chilli blank sample. The method has high recovery and good reproducibility, providing a reference for the determination of residues of 331 pesticides and their metabolites in dried chilli.

SGLC-LC/MS-081EN First Edition: April 2024

SHIMADZU

Shimadzu (Shanghai) Global Laboratory Consumables Co.,Ltd.
www.sglc.shimadzu.com.cn
www.shimadzumall.com
Contact:contact@sglc.shimadzu.com.cn