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Application News

Gas Chromatograph Mass Spectrometer GCMS-TQ™8040 NX

Multiresidue Pesticides Analysis in Cumin Seeds Using GCMS-TQ8040 NX

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User Benefits

- A QuEChERS extraction procedure has been employed for quantifying pesticides at trace levels in a complex spice matrix like cumin seeds using Ultra-fast GCMS-TQ8040 NX technique.
- GCMS Smart Pesticides Database™ Ver.2 from Shimadzu Corporation enables ease of creating and optimizing MRM method and its execution.

1. Introduction

Cumin (Cuminum cyminum) has been used as a natural medicine for over 2000 years. It shows anti-inflammatory property and supports the immune system. Cumin seeds (Figure 1) have been widely used as a spice in many food preparations. To fulfil the high-demand of such spices, pesticides are widely used. But their overuse may cause acute and permanent health problems in humans. Therefore, to protect human health, the European Union has set maximum residue limits (MRLs) for the presence of pesticides in cumin seeds[1]. Thus, increasing the importance of having analytical method for determination of residual pesticides present in it.

Fig. 1 Cumin seeds

This study reports a validated method for the determination of 169 pesticides in cumin seeds of Indian origin using Shimadzu GCMS-TQ8040 NX (Figure 2). The multi-residue extraction was performed using QuEChERS extraction method[2].

2. Materials and Methods

The reference standards for this study were procured from Restek with below catalogue number:
GC multi-residue pesticides kit – 32562
Cumin seeds procured from local market, were used to prepare matrix-matched calibration standards and spiked samples. This method is validated for criteria as mentioned in SANTE Guidelines[3].
GCMS-TQ8040 NX, manufactured by Shimadzu Corporation Japan, was used to quantify residual pesticides in cumin seeds sample.

2-1. Method development

Instrumental method was developed based on chromatographic and mass spectrometric parameters. Smart Pesticides Database for GC-MS/MS enabled quick instrumental method optimization for higher throughput. For most of the pesticides, 1 target and 2 reference MRM transitions were included in the method. Shimadzu's 'LabSolutions Insight™' software was used for data processing, which helped in evaluating validation parameters with ease. Sample Pretreatment method was optimized based on QuEChERS to give better and consistent recoveries.

2-2. Sample and standard preparation

Cumin seeds were ground and used as sample for extraction. Acetonitrile [1% acetic acid (v/v)] was used as extraction solvent along with salts as per EN 15662 method to obtain good recoveries of pesticides. After extraction, the aliquot of acetonitrile was taken for further clean up.
Optimum combination of bulk sorbents like C18, GCB (Graphitized Carbon Back), PSA (Primary secondary amine) and anhydrous $MgSO_4$ was used to minimize matrix interference. The extract was evaporated & reconstituted in ethyl acetate such that recovery samples' concentration is diluted by four times during sample preparation. Reconstituted samples were filtered through 0.22 µm PTFE filter.
Blank matrix extract prepared above was used to prepare matrix matched linearity standards, against which spiked samples were quantified. All samples were analyzed as per conditions shown in table 1.

Fig. 2 Shimadzu GCMS-TQ™8040 NX

Table 1 Instrument configuration and Analytical Conditions: GC-MS/MS

System Configuration	
GC-MS/MS	: GCMS-TQ8040 NX
Auto-injector	: AOC™-20i + s
Column	: SH-I-Rxi-5Sil MS (P/N: 221-75954-30) (30 m × 0.25 mm I.D., df = 0.25 µm)
Liner	: Topaz Liner, Splitless Single Taper w/Wool
GC	
Injector temp.	: 250 °C
Column oven temp.	: 80 °C (2 min), 20 °C/min to 180 °C (0 min), 5 °C/min to 300 °C (3 min)
Run time	: 34 min
Injection mode	: Splitless for 2 min (High pressure at 250 kPa for 2 min)
Injection volume	: 2 µL
Carrier gas	: He
Linear Velocity	: 40.4 cm/sec (Constant mode)
MS	
Ionization mode	: EI
Ion source temp.	: 230 °C
Interface temp.	: 280 °C
Solvent cut time	: 4.0 min
Loop Time	: 0.3 sec

3. Results and Discussion

Validation parameters like linearity, recovery and precision were studied to determine LOQs (Limit of quantitation) and results obtained are shown in Table 2.

3-1. Linearity

For linearity study and quantifying spiked samples, matrix matched calibration standards were used. Multilevel calibration curve included 1, 2.5, 5, 25, 50 and 100 µg/L concentration levels. All calibration standards were found within 80 to 120 % accuracy range as per SANTE guidelines.

3-2. Recovery

Recovery was evaluated by analyzing spiked samples at 10 and 20 µg/kg (six spiked samples at each level) against matrix matched linearity plotted between 1 to 100 µg/L. Mean recoveries were found to be within 70-120 %. Representative chromatograms of recovery samples for a few compounds at their LOQ levels are shown in Figure 3.

3-3. Reproducibility (% RSD_R)

Reproducibility experiment for recoveries was performed on six different spiked samples at 10 and 20 µg/kg concentration levels. The % RSD for recovery of six spiked samples at their respective LOQ levels was found to be less than 20 %.

3-4. LOQ

The LOQ of analyte was determined based on the concentration level at which it showed accuracy within 80 to 120 %, recovery within 70 to 120 % and reproducibility within 20 %. Although few pesticides showed high recoveries up to 130 %; their reproducibility was good i.e. less than 20 %, hence their LOQs were considered at that respective concentration level. These analytes are marked with asterisk (*) in Table 2.

In all, the method successfully achieved 10 and 20 µg/kg LOQ for 157 and 12 pesticides, respectively. The LOQs were determined based on criteria mentioned in SANTE guidelines.

Table 2 Summary results of GC-MS/MS analysis

#	Compound Name	Ret. Time (min)	Target MRM (m/z)	CE (V)	Determination Coefficient (R^2)	LOQ mg/kg	% Accuracy at LOQ	% Recovery at LOQ	% RSD_R (n=6)
1	Dichlobenil	6.9	170.90>136.00	14	0.9927	0.01	93.38	94.64	3.45
2	Biphenyl	7.219	154.10>128.10	22	0.9926	0.01	86.58	119.50	1.47
3	Mevinphos-1	7.468	192.00>127.00	12	0.9923	0.01	92.11	83.42	10.81
4	Etridiazole	7.685	210.90>182.90	10	0.9907	0.01	92.77	88.68	9.13
5	Pebulate	7.748	161.10>128.10	6	0.9902	0.01	103.05	87.00	11.45
6	Methacrifos	8.004	208.00>180.00	8	0.9890	0.01	91.85	98.72	7.16
7	Chloroneb	8.111	193.00>113.00	18	0.9922	0.01	95.9	105.86	7.77
8	2-Phenylphenol	8.292	170.10>141.10	24	0.9907	0.01	88.23	105.25	4.75
9	Pentachlorobenzene	8.319	249.90>214.90	18	0.9917	0.01	101.4	70.59	9.03
10	Tecnazene	8.93	260.90>202.90	14	0.9911	0.01	98.36	96.60	10.94
11	Propachlor	9.05	176.10>57.00	8	0.9899	0.01	90.96	94.75	10
12	2,3,5,6-Tetrachloroaniline	9.282	228.90>158.00	18	0.9899	0.01	99.89	86.37	9.54
13	Diphenylamine	9.236	169.10>66.00	24	0.9883	0.01	107.61	99.21	9.15
14	Cycloate	9.327	154.20>72.00	6	0.9879	0.01	88.23	110.78	16.61
15	Ethalfluralin	9.357	276.00>202.00	18	0.9853	0.01	103.37	107.62	13.8
16	Chlorpropham	9.493	127.10>65.00	22	0.9872	0.01	97.43	102.36	13.91
17	Trifluralin	9.508	306.10>264.10	8	0.9923	0.01	89.53	98.95	6.93
18	Benfluralin	9.566	292.10>264.00	8	0.9883	0.01	93.32	95.40	10.51
19	Sulfotep	9.611	322.00>202.00	10	0.9891	0.01	92.39	101.18	9.62
20	Di-allate-1	9.876	234.10>150.00	20	0.9891	0.01	81.41	89.79	9.23
21	Phorate	9.883	260.00>75.00	8	0.9931	0.01	107.03	119.57	5.47
22	alpha-BHC	10.042	180.90>144.90	16	0.9872	0.01	99.55	81.83	10.57
23	Hexachlorobenzene	10.148	283.80>248.80	24	0.9902	0.01	89.6	71.07	6.55
24	Pentachloroanisole	10.249	264.80>236.80	16	0.9899	0.01	86.09	72.11	4.72
25	Dicloran	10.306	206.00>176.00	10	0.9929	0.01	95.89	105.47	11
26	Atrazine	10.532	215.10>58.00	14	0.9856	0.02	90.85	109.27	12.97
27	beta-BHC	10.59	180.90>144.90	16	0.9900	0.01	94.8	92.72	10.37
28	Clomazone	10.587	204.10>107.00	20	0.9927	0.01	101.44	106.16	10.21
29	Quintozene	10.678	294.80>236.80	16	0.9894	0.01	100.52	99.46	17.89
30	Pentachlorobenzonitrile	10.76	274.80>239.80	18	0.9894	0.01	91.33	94.15	14.32
31	gamma-BHC (Lindane)	10.758	180.90>144.90	16	0.9879	0.01	99.08	82.78	7.76
32	Profluralin	10.667	318.10>199.10	16	0.9900	0.01	97.3	108.00	16.32
33	Terbuthylazine	10.836	229.10>173.10	6	0.9921	0.01	99.58	86.60	13.61
34	Terbufos*	10.788	231.00>128.90	26	0.9911	0.01	96.22	128.92	4.41

Table 2 Summary results of GC-MS/MS analysis (Contd.)

#	Compound Name	Ret. Time (min)	Target MRM (m/z)	CE (V)	Determination Coefficient (R^2)	LOQ mg/kg	% Accuracy at LOQ	% Recovery at LOQ	% RSD_R (n=6)
35	Fonofos	10.911	246.00>137.10	6	0.9904	0.01	102.67	100.22	7.34
36	Propyzamide	10.911	172.90>109.00	26	0.9859	0.01	87.02	103.47	4.23
37	Diazinon	10.906	304.10>179.10	10	0.9839	0.01	88.3	99.81	19.14
38	Fluchloralin	10.929	306.00>264.00	8	0.9947	0.01	107.11	111.89	10.06
39	Pyrimethanil	11.088	198.10>118.10	28	0.9907	0.01	103.41	98.18	5.79
40	Isazofos	11.202	257.00>162.00	8	0.9874	0.01	93.09	93.50	19.47
41	Tefluthrin	11.205	177.00>127.10	16	0.9895	0.01	89.67	102.42	4.62
42	delta-BHC	11.409	180.90>144.90	16	0.9904	0.01	90.92	91.27	8.51
43	Tri-allate	11.399	268.10>184.00	20	0.9881	0.01	97.14	92.91	14.55
44	Pentachloroaniline	11.838	262.90>191.90	22	0.9887	0.01	87.4	79.73	12.61
45	Endosulfan ether	11.859	240.90>205.90	16	0.9965	0.01	92.27	96.59	15.84
46	Dimethachlor	11.973	197.10>148.10	10	0.9912	0.01	94.36	106.99	4.76
47	Propanil*	12.056	160.90>99.00	24	0.9895	0.01	101.84	129.01	6.71
48	Acetochlor	12.081	223.10>132.10	22	0.9907	0.01	90.77	98.83	18.33
49	Chlorpyrifos-methyl	12.115	285.90>93.00	22	0.9905	0.01	89.77	90.45	11.76
50	Vinclozolin	12.199	285.00>212.00	12	0.9890	0.01	104.45	99.96	12.51
51	Parathion-methyl	12.263	263.00>109.00	14	0.9870	0.01	89.65	104.65	6.97
52	Tolclofos-methyl	12.296	264.90>249.90	14	0.9869	0.01	87.93	96.74	10.06
53	Alachlor	12.309	188.10>160.10	10	0.9892	0.01	88.26	97.60	10.81
54	Transfluthrin	12.324	163.10>127.10	6	0.9885	0.01	85.91	86.69	9.31
55	Heptachlor	12.56	271.80>236.90	20	0.9927	0.01	95.98	80.80	11.83
56	Metalaxyl (Mefenoxam)	12.51	249.20>146.10	22	0.9864	0.02	99.08	98.09	4.34
57	Fenchlorphos	12.568	284.90>269.90	16	0.9888	0.01	98.53	97.67	13.05
58	Pirimiphos-methyl	12.858	290.10>125.00	22	0.9891	0.01	88.63	103.80	10.39
59	Prodiamine	12.914	321.10>279.10	6	0.9977	0.01	105.16	107.90	11.33
60	Fenitrothion	12.942	277.00>260.00	6	0.9919	0.01	83.97	106.47	17.43
61	Pentachlorothioanisole	13.127	295.80>262.90	14	0.9897	0.01	88.29	76.69	19.9
62	Malathion	13.16	173.10>99.00	14	0.9876	0.01	94.3	108.70	10.28
63	Metolachlor (S-Metolachlor)	13.361	162.10>133.10	16	0.9888	0.01	91.71	101.90	6.16
64	Fenthion*	13.486	278.00>169.00	14	0.9970	0.01	88.46	121.12	11.6
65	Chlorthal-dimethyl	13.544	298.90>220.90	24	0.9905	0.01	99.9	103.01	10.46
66	Anthraquinone	13.569	180.10>152.10	14	0.9914	0.01	87.43	97.16	8.85
67	Parathion	13.603	291.10>109.00	14	0.9950	0.01	89.61	109.23	11.53
68	Triadimefon	13.726	208.10>127.00	14	0.9860	0.01	102.05	96.65	15.82
69	4,4'-Dichlorobenzophenone	13.817	139.00>111.00	14	0.9911	0.01	97.73	93.25	8.1
70	Fenson	13.974	141.00>77.00	16	0.9870	0.01	95.42	103.98	6.57
71	Pirimiphos ethyl	13.983	304.10>168.10	12	0.9877	0.01	100.64	95.09	10.07
72	Bromophos	14.013	330.90>315.90	14	0.9904	0.01	90.01	91.29	14.06
73	MGK 264-1	14.074	164.10>93.00	10	0.9903	0.01	111.68	100.91	18.8
74	Isopropalin	14.147	280.10>238.10	8	0.9914	0.01	96.05	98.75	9.2
75	Isodrin	14.363	192.90>157.00	20	0.9903	0.01	98.24	117.57	7.62
76	Pendimethalin	14.354	252.10>162.10	10	0.9910	0.01	86.78	100.67	13.81
77	Cyprodinil	14.422	224.10>208.10	16	0.9861	0.01	90.07	88.76	18.71
78	Metazachlor	14.426	209.10>132.10	18	0.9917	0.01	101.33	107.48	16.11
79	MGK 264-2	14.443	164.10>98.00	12	0.9909	0.01	108.43	92.09	12.51

Table 2 Summary results of GC-MS/MS analysis (Contd.)

#	Compound Name	Ret. Time (min)	Target MRM (m/z)	CE (V)	Determination Coefficient (R^2)	LOQ mg/kg	% Accuracy at LOQ	% Recovery at LOQ	% RSD_R (n=6)
80	Fipronil	14.783	366.90>212.90	30	0.9862	0.01	84.62	115.44	9.3
81	Penconazole	14.562	248.10>157.10	26	0.9898	0.01	84.54	106.48	13.65
82	Chlozolinate	14.607	330.90>258.90	6	0.9890	0.02	111.66	87.21	14.36
83	Bromfenvinfos-methyl	14.7	294.90>109.00	16	0.9889	0.01	107.15	88.66	16.79
84	(Z)-Chlorfenvinphos	14.681	267.00>159.00	18	0.9927	0.01	91.06	103.08	12.97
85	Quinalphos	14.835	146.10>118.00	10	0.9902	0.01	95.29	97.51	14.93
86	Procymidone	14.978	283.00>96.00	10	0.9868	0.01	85.44	106.62	13.67
87	Triflumizole	15.081	206.10>179.10	14	0.9841	0.02	99.79	98.26	7.48
88	Folpet	15.068	259.90>130.00	14	0.9975	0.02	97.96	80.11	12.02
89	Bromophos-ethyl	15.277	358.90>302.90	16	0.9917	0.01	104.2	97.46	6.61
90	trans-Chlordane	15.338	374.80>265.90	26	0.9901	0.01	95.52	94.70	18.27
91	Chlorbenside	15.31	125.00>89.00	16	0.9920	0.01	103.83	104.26	10.56
92	o,p'-DDE	15.43	246.00>176.00	30	0.9871	0.01	92.26	93.44	6.56
93	Tetrachlorvinphos	15.506	328.90>109.00	20	0.9907	0.01	106.82	99.13	16.44
94	cis-Chlordane	15.75	374.80>265.90	26	0.9901	0.01	94.37	81.47	12.22
95	trans-Nonachlor	15.841	406.80>299.90	24	0.9879	0.01	87.93	101.78	16.14
96	Iodofenphos	16.101	376.90>361.80	22	0.9916	0.01	84.35	94.48	9.96
97	Flutolanil	16.224	173.00>95.00	26	0.9893	0.01	98.68	101.01	19.88
98	Chlorfenson	16.087	175.00>111.00	12	0.9906	0.01	91.47	104.51	3.58
99	Prothiofos	16.208	266.90>238.90	10	0.9917	0.01	88.48	95.23	10.06
100	Fludioxonil	16.471	248.00>127.00	26	0.9903	0.01	94.56	99.69	3.43
101	Pretilachlor	16.284	262.10>202.10	10	0.9880	0.01	104.29	91.33	13.51
102	p,p'-DDE	16.482	246.00>176.00	30	0.9885	0.01	104.9	83.15	9.79
103	Oxadiazon	16.513	258.00>175.00	8	0.9881	0.01	106.77	90.18	18.3
104	Myclobutanil	16.747	179.10>125.00	14	0.9865	0.01	109.61	114.48	16.01
105	o,p'-DDD	16.696	235.00>165.00	24	0.9894	0.01	97.74	94.44	8.3
106	Flusilazole	16.772	233.10>165.10	14	0.9911	0.02	90.04	101.02	10.67
107	Oxyfluorfen	16.711	361.00>300.00	14	0.9865	0.02	93.17	88.00	7.82
108	Bupirimate	16.784	273.10>193.10	8	0.9824	0.01	92.39	113.19	19.34
109	Fluazifop-P-butyl	17.267	282.10>91.00	18	0.9860	0.01	86.64	88.80	12.14
110	Nitrofen	17.225	202.00>139.00	24	0.9876	0.01	88.67	80.64	16.39
111	1,1-Dichloro-2,2-bis(4-ethylphenyl)ethane	17.272	223.20>167.10	14	0.9897	0.01	99.22	96.97	9.01
112	Chlorthiophos-2	17.494	324.90>268.90	14	0.9892	0.02	96.94	97.64	8.2
113	Chlorobenzilate	17.531	139.00>111.00	16	0.9868	0.01	91.09	103.49	5.71
114	cis-Nonachlor	17.819	406.80>299.90	24	0.9873	0.01	102.26	87.18	9.14
115	p,p'-DDD	17.817	235.00>165.00	24	0.9897	0.01	101.85	105.89	2.72
116	o,p'-DDT	17.878	235.00>165.00	24	0.9903	0.01	97.22	95.28	4.65
117	Chlorthiophos-3	17.946	324.90>268.90	14	0.9883	0.01	92.59	101.21	9.97
118	Triazophos	18.46	257.00>162.00	8	0.9913	0.01	99.13	112.41	11.12
119	Sulprofos	18.424	322.00>156.00	8	0.9930	0.01	115.18	115.28	19.96
120	Carfentrazone-ethyl	18.685	340.10>312.10	14	0.9880	0.01	95.35	106.62	11.32
121	4,4'-methoxychlor olefin	18.762	238.10>223.10	12	0.9874	0.01	87.55	98.64	9.31
122	Carbophenothion	18.73	341.90>157.00	14	0.9894	0.01	99.34	107.32	5.23
123	Edifenphos	18.885	173.00>109.00	10	0.9905	0.01	87.96	97.33	8.61
124	Norflurazon	18.885	145.00>95.00	18	0.9884	0.01	91.93	112.21	9.46

Table 2 Summary results of GC-MS/MS analysis (Contd.)

#	Compound Name	Ret. Time (min)	Target MRM (m/z)	CE (V)	Determination Coefficient (R^2)	LOQ mg/kg	% Accuracy at LOQ	% Recovery at LOQ	% RSD_R (n=6)
125	Endosulfan sulfate	18.991	271.80>236.90	18	0.9898	0.01	104.51	95.88	17.28
126	Lenacil	19.072	153.10>136.10	14	0.9870	0.02	106.62	108.93	11.19
127	p,p'-DDT	19.101	235.00>165.00	24	0.9895	0.01	89.41	86.14	3.25
128	Hexazinone	19.333	171.10>71.00	16	0.9898	0.01	98.86	104.41	5.27
129	2,4'-Methoxychlor	19.325	227.10>121.10	16	0.9908	0.01	96.59	97.69	2.15
130	Piperonyl butoxide	19.877	176.10>131.10	12	0.9912	0.01	87.74	95.14	11
131	Nitralin*	19.923	316.10>274.00	8	0.9943	0.01	100.78	121.15	10.95
132	Pyridaphenthion	20.42	340.00>199.10	8	0.9918	0.01	91.26	111.90	10.62
133	Iprodione*	20.494	314.00>245.00	12	0.9903	0.01	89.89	128.81	12.29
134	Tetramethrin-1	20.567	164.10>107.10	14	0.9878	0.01	99.6	108.98	6.54
135	Endrin ketone	20.501	316.90>244.90	20	0.9891	0.02	90.35	96.02	13.03
136	Phosmet	20.631	160.00>77.00	24	0.9910	0.01	98.03	107.11	14.13
137	Bifenthrin	20.783	181.10>166.10	12	0.9890	0.01	95.54	102.51	5.06
138	EPN	20.745	156.90>77.00	24	0.9931	0.01	87.95	75.64	12.58
139	Bromopropylate	20.824	340.90>182.90	18	0.9865	0.01	95.13	102.50	6.35
140	Tetramethrin-2	20.857	164.10>107.10	14	0.9853	0.01	103.25	99.36	10.02
141	Methoxychlor	20.989	227.10>169.10	24	0.9909	0.01	90.67	109.29	5.26
142	Fenpropathrin	21.1	265.10>210.10	12	0.9916	0.01	94.19	106.27	17.94
143	Tebufenpyrad	21.29	333.10>171.10	20	0.9888	0.01	91.69	108.22	11.28
144	Tetradifon	21.693	355.90>228.90	12	0.9929	0.01	103.11	101.71	11.31
145	Phosalone	21.908	182.00>102.00	14	0.9926	0.01	97.13	103.25	9.85
146	Leptophos	21.931	376.90>361.90	24	0.9879	0.01	97.98	93.11	10.77
147	Azinphos-methyl*	22.008	160.10>77.00	20	0.9912	0.01	92.35	121.09	7.51
148	Pyriproxyfen	22.246	136.10>96.00	14	0.9865	0.01	119.9	92.22	8.94
149	Mirex	22.58	271.80>236.80	18	0.9889	0.01	90.36	72.76	13.26
150	Acrinathrin-2	23.058	181.10>152.10	26	0.9945	0.01	98.71	116.17	8.19
151	Pyrazophos	22.953	221.10>149.10	14	0.9915	0.02	93.5	99.86	8.84
152	Fenarimol	22.914	251.00>139.00	14	0.9922	0.01	86.81	96.65	12.15
153	Azinphos-ethyl	23.134	160.10>132.10	4	0.9914	0.01	87.27	97.17	7.48
154	Pyraclofos	23.478	194.00>138.00	22	0.9934	0.01	98.57	115.13	11.68
155	Coumaphos	24.335	362.00>109.00	16	0.9910	0.01	101.44	115.51	14.88
156	trans-Permethrine	24.368	183.10>153.10	14	0.9905	0.01	101.16	101.61	13.39
157	Fluquinconazole	24.358	340.00>298.00	20	0.9898	0.01	110.15	109.16	11.53
158	Pyridaben	24.375	147.10>117.10	22	0.9875	0.01	84.05	93.05	12.63
159	Acequinocyl deg.	25.173	342.20>188.10	14	0.9932	0.02	104.53	81.62	3.34
160	Cyfluthrin-1	25.191	163.10>127.10	6	0.9892	0.01	92.45	76.03	10.29
161	Cyfluthrin-2	25.407	163.10>127.10	6	0.9920	0.01	90.19	115.64	8.81
162	Cyfluthrin-3	25.515	163.10>127.10	6	0.9910	0.01	86.57	107.02	14.7
163	Cyfluthrin-4	25.607	163.10>127.10	6	0.9917	0.01	101.48	105.35	7.76
164	Flucythrinate-1	26.148	157.10>107.10	12	0.9902	0.01	93.12	109.70	6.21
165	Flucythrinate-2	26.52	157.10>107.10	12	0.9912	0.01	99.65	104.66	3.36
166	Fluridone*	26.846	328.10>259.00	24	0.9901	0.01	103.83	128.64	6.97
167	tau-Fluvalinate-1	27.779	250.10>55.00	18	0.9901	0.01	104.88	103.28	6.71
168	tau-Fluvalinate-2	27.916	250.10>55.00	18	0.9904	0.01	101.6	99.98	9
169	Deltamethrin (Tralomethrin deg.)	28.914	252.90>93.00	20	0.9940	0.01	95.89	82.47	11.12

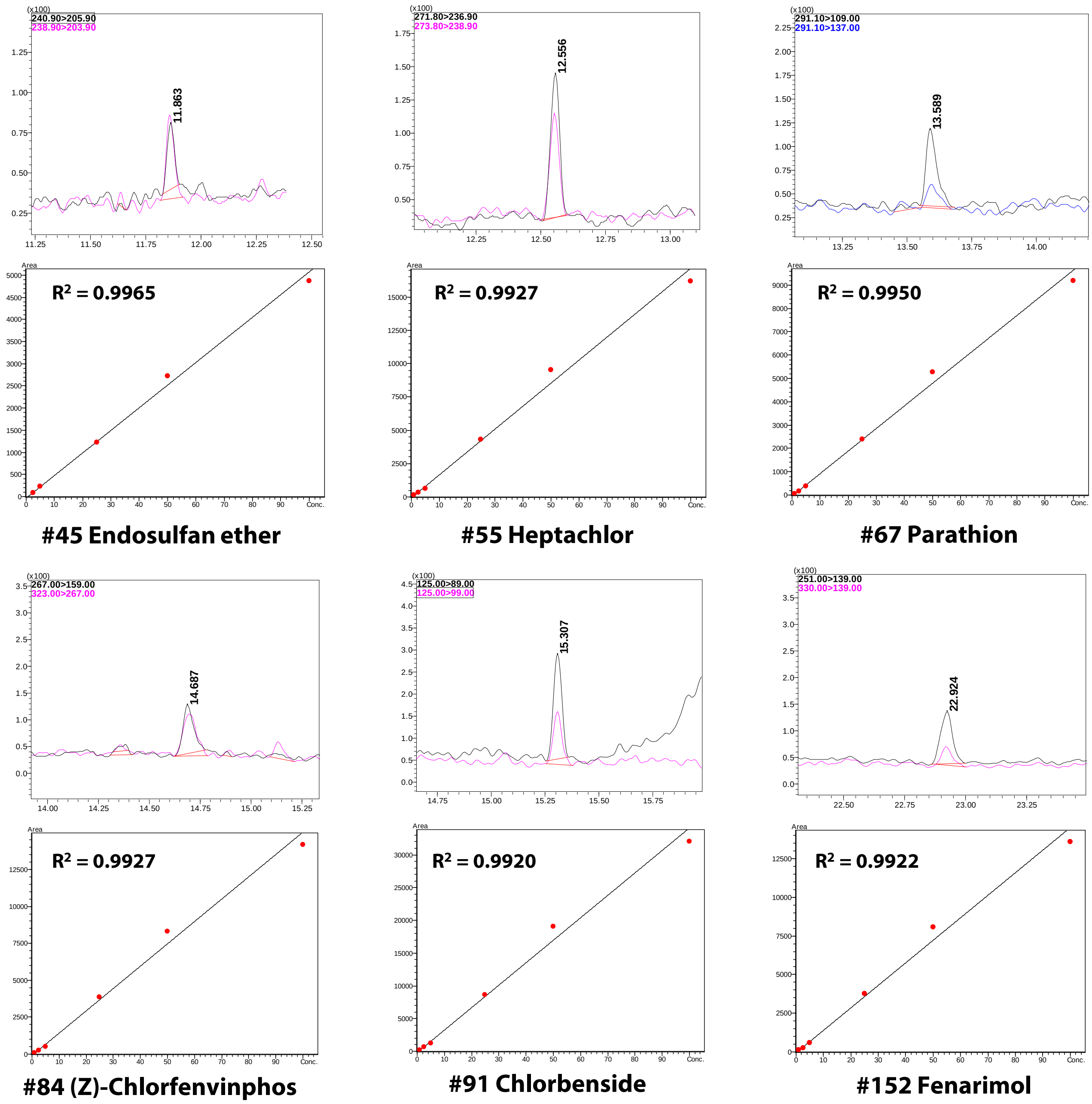

Fig. 3 Representative linearity graphs and chromatograms at LOQ level for some compounds

4. Conclusion

A validated method was developed as per SANTE guidelines for determination of 169 pesticides in cumin seeds.

QuEChERS' extraction was used for quantifying residual pesticides in this complex spice matrix containing essential oils, cuminaldehyde, pigments etc. The instrumental MRM method was effortlessly created using Smart Pesticides Database.

The combination of highly sensitive Shimadzu GC-MS/MS and reliable method showed reproducibility < 20% (as per SANTE guidelines) at LOQ levels. This enables its use in testing laboratories for multi-residue analysis in cumin seeds.

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