Qualification and Quantification of Residual Pesticides in Ginger by GCMS using Simultaneous Ultra-fast Scan and SIM

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1. Introduction

Interest in food safety has grown, resulting in a trend of tightening global regulations for residual pesticides in produce. Selected Ion monitoring (SIM) in GC/MS is a common technique used for pesticide analysis while Scan mode is often used in non-targeted screening.

This study demonstrates a SIM analysis of pesticides, including calibration and repeatability in ginger root matrix. In addition to targeted analysis, in this work we performed simultaneous untargeted analysis with Ultra-fast Scan analysis, maximizing the use of the GCMS-QP2050. NIST library search results are shown for untargeted components.

Fig. 1 GCMS-QP2050

2. Methods

GPL2005 GC/MS pesticide mixtures I to VII were diluted to 5ppb and were used as standard pesticide mixtures. The ginger sample was prepared using the QuEChERs method, and pesticide standards were added to the extract. In addition to standard pesticide mixtures, we spiked dichlorodiphenyltrichloroethane (DDT) into the sample to achieve final concentrations of 5 ppb and 500 ppb, respectively.

For GC/MS analysis, the default method available in the *Smart Pesticide Database* was used as the analytical method. The MS method was augmented and exported as a simultaneous Scan/SIM using Smart SIM+ to create an optimized MS acquisition method.

Table. 1 Analytical conditions

GC-MS:	GCMS-QP2050 (TMP exhaust: 255 L/sec)
GC	
Column:	SH-I-5Sil MS (30m×0.25 mm, 0.25µm)
Insert:	Topaz liner splitless single taper
Inlet Temp.:	250 °C
Injection Volume:	1 µL
Injection:	Splitless (high pressure 250 kPa)
Carrier Gas:	Helium
Control Mode:	Constant linear velocity
MS	
IF Temp.:	290 °C
Ion Source:	230 °C
Ionization Mode:	EI
Mode:	FASST (Scan/SIM)
Scan Range:	*m/z* 35 – 500
Scan Speed:	30,000 u/sec

3. Results

For quantitation, SIM shows great reproducibility using 5ppb spiked ginger matrix. Fig. 2 and Table. 2 show almost all compounds meet a %RSD lower than 10% (orange). Easily degradable components such as dioxathion degradation products were excluded.

For identification purposes, scan results support peak identification of target compounds. The SIM mass chromatogram and scan results for Methyl Demeton are shown in Figure 3. By comparing these results with library spectra, accurate identification is achievable.

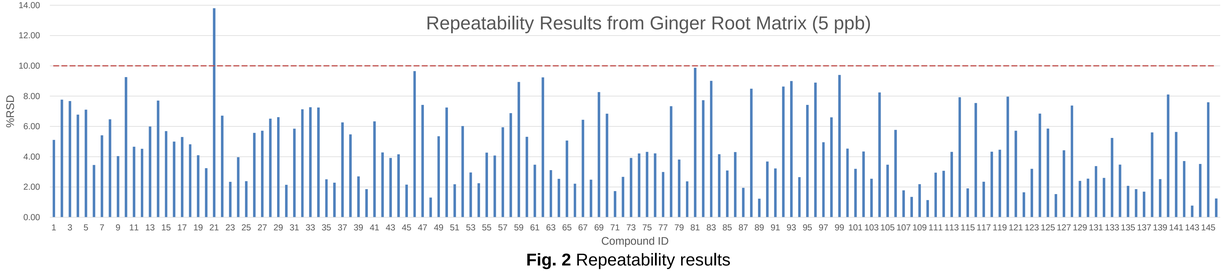

Fig. 2 Repeatability results

Table. 2 Individual repeatability result (5ppb, n=5)

ID	Compound Name	%RSD	ID	Compound Name	%RSD	ID	Compound Name	%RSD
1	Dichlorvos	5.11	50	Metalaxyl	7.25	99	Oxyfluorfen	9.40
2	Nereistoxin	7.77	51	Fenchlorphos	2.18	100	Bupirimate	4.53
3	Biphenyl	7.67	52	Prometryn	6.02	101	Carboxin	3.20
4	Chlormephos	6.78	53	Dithiopyr	2.96	102	Kresoxim-methyl	4.34
5	Methacrifos	7.11	54	Pirimiphos-methyl	2.25	103	Diclobutrazol	2.55
6	Chloroneb	3.46	55	Terbutryn	4.27	104	(Z)-Metominostrobin	8.24
7	2-Phenylphenol	5.41	56	Fenitrothion	4.08	105	Azaconazole	3.47
8	Tecnazene	6.47	57	Ethofumesate	5.95	106	Cyflufenamid	5.77
9	Xylylcarb	4.04	58	(E)-Dimethylvinphos	6.88	107	1,1-Dichloro-2,2-bis(4-ethylphenyl)ethane	1.78
10	Chlorethoxyfos	9.26	59	Chlorpyrifos	8.93	108	Flufenpyr-ethyl	1.35
11	Diphenylamine	4.66	60	(Z)-Dimethylvinphos	5.31	109	(Z)-Pyriminobac-methyl	2.19
12	Phenmedipham deg.	4.52	61	Diethofencarb	3.47	110	Chloropropylate	1.14
13	Trifluralin	5.99	62	Fenthion	9.24	111	Chlorobenzilate	2.95
14	Dioxabenzofos	7.70	63	Cyanazine	3.11	112	Fensulfothion	3.07
15	Benfluralin	5.69	64	Chlorthal-dimethyl	2.54	113	Ethion	4.32
16	Sulfotep	5.00	65	Parathion	5.07	114	Mepronil	7.92
17	Phorate	5.30	66	Fenpropimorph	2.22	115	Carfentrazone-ethyl	1.91
18	Thiometon	4.82	67	Isofenphos oxon	6.44	116	Chlornitrofen	7.54
19	Dimethoate	4.09	68	Tetraconazole	2.49	117	Pyraflufen-ethyl	2.35
20	Clomazone	3.24	69	Isocarbophos	8.27	118	Diflufenican	4.33
21	Quintozene	13.8	70	Phthalide	6.84	119	Mefenpyr-diethyl	4.46
22	Propazine	6.71	71	Bromophos	1.73	120	Chlomethoxyfen	7.96
23	Tolylfluanid metab.	2.34	72	Diphenamid	2.67	121	Pyridaphenthion	5.71
24	Dioxathion deg.	3.96	73	(E)-Chlorfenvinphos	3.92	122	Bromopropylate	1.65
25	Propentamphos	2.39	74	Pendimethalin	4.21	123	Picolinafen	3.20
26	Terbufos	5.57	75	Cyprodinil	4.32	124	Tebufenpyrad	6.85
27	Fonofos	5.71	76	Fipronil	4.22	125	Furametpyr	5.86
28	Diazinon	6.52	77	Dimethametryn	2.99	126	Leptophos	1.53
29	Chlorothalonil	6.61	78	Chlozolinate	7.34	127	Cyhalofop-butyl	4.42
30	Pyrimethanil	2.14	79	(Z)-Chlorfenvinphos	3.82	128	Pyraclofos	7.37
31	Disulfoton	5.85	80	Phenthoate	2.37	129	Fenoxaprop-ethyl	2.41
32	Prohydrojasmon-1	7.13	81	Diclocymet-1	9.87	130	Fluquinconazole	2.55
33	Isazofos	7.26	82	Quinalphos	7.73	131	Pyridaben	3.38
34	Terbacil	7.25	83	Dimepiperate	9.01	132	Dioxathion	2.60
35	delta-BHC	2.51	84	Procymidone	4.17	133	Butafenacil	5.24
36	Etrimfos	2.29	85	Bromophos-ethyl	3.09	134	Etobenzanid	3.48
37	Tri-allate	6.26	86	Diclocymet-2	4.30	135	Cafenstrole	2.08
38	MCPA-thioethyl	5.47	87	Propaphos	1.95	136	Halfenprox	1.86
39	Tebupirimfos	2.70	88	Tetrachlorvinphos	8.49	137	Silafluofen	1.69
40	Benoxacor	1.86	89	Paclobutrazol	1.23	138	Fluridone	5.60
41	Phosphamidon-2	6.33	90	Fenamiphos	3.68	139	Pyrimidifen	2.52
42	Dichlofenthion	4.28	91	Napropamide	3.23	140	Flumioxazin	8.11
43	Dimethenamid	3.92	92	Chlorfenson	8.63	141	Fenvalerate-1	5.64
44	Metribuzin	4.16	93	(E)-Metominostrobin	9.00	142	Fenvalerate-2	3.71
45	Chlorpyrifos-methyl	2.15	94	Prothiofos	2.65	143	Flumiclorac-pentyl	0.77
46	Vinclozolin	9.65	95	Isoprothiolane	7.42	144	Azoxystrobin	3.53
47	Parathion-methyl	7.42	96	Profenofos	8.89	145	Tolfenpyrad	7.59
48	Tolclofos-methyl	1.30	97	Thifluzamide	4.96	146	Cinidon-ethyl	1.24
49	Simetryn	5.35	98	Flusilazole	6.60			

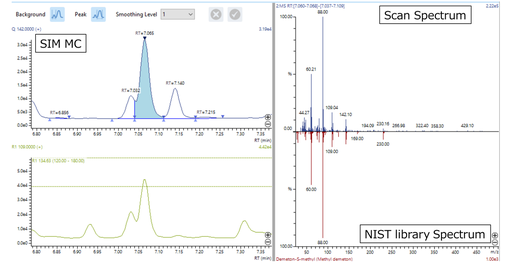

Fig. 3 SIM mass chromatograms and Spectra of Methyl Demeton

For non-targeted qualification, scan results from simultaneous SIM/scan analysis facilitate peak identification. Figure 4 illustrates scan results for DDT mass chromatography in ginger root. Figure 5 highlights that library search results can be employed for DDT identification without compromising accuracy.

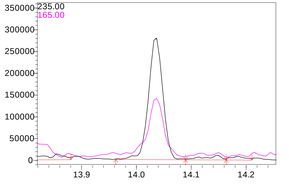

Fig. 4 Mass chromatogram of DDT from Scan result

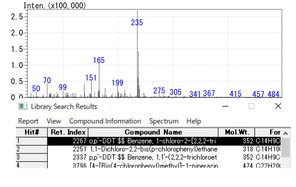

Fig. 5 DDT mass spectrum and library search result

4. Conclusion

The GCMS-QP2050 demonstrates sensitivity and reproducibility in analyzing pesticides in produce using SIM data obtained from simultaneous Scan/SIM analysis. Ultra-fast scan supports accurate identification of target compounds alongside SIM analysis without interference. Scan results provide MS spectrum patterns for identifying unknown compounds, assisting in non-target screening.