

GC-MS/MS Analysis of Pesticides in Extra Virgin Olive Oil

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Overview

Multi-pesticide analysis in food is very challenging due to the wide variety of analytes measured, matrix complexity, and detection levels. To ensure safe levels of these pesticides in food, there is necessity for a sample preparation technique that includes matrix removal, enhanced chromatographic separation with GC column, and spectral resolution using high-end MS technique. Presented in this application note is a multi-pesticide separation from a challenging food matrix. For improving extraction and to remove matrix interference, the QuEChERS sample preparation technique was employed. This study provides a method development starting point for a multi-pesticide analysis in food with enhanced chromatographic resolution and minimal breakdown of sensitive pesticide compounds on the column, using a Zebron ZB-5MSPLUS GC-MS column.

GC-MS/MS Conditions

Column: Zebron™ ZB-5MSPLUS™

Dimension: 15 meter x 0.25 mm x 0.25 µm

Part No.: [7EG-G030-11](#)

Injection: Pulse Splitless for 1.5 min @ 250 °C, 1 µL

Recommended Liner: Zebron PLUS Z-Liner™ (Compatible with Agilent® and Thermo Scientific® GC instrument)

Liner Part No.: [AG2-0A13-05](#)

Carrier Gas: Helium @ 1.5 mL for 5 min to 1.8 mL/min (Ramp Flow)

Oven Program:	Ramp(°C/min)	Temp (°C)	Time(min)
	-	60	1.0
	40	170	0.0
	10	310	3.0

Detector: MS/MS

Detector Temperature: 300 °C

Sample Preparation

The extraction procedure is according to the European standard method EN15662. The calibration from 5 ppb to 200 ppb was prepared by the QuEChERS method with correlation coefficients greater than 0.99 for all pesticides considered in this study. **Figure 1** shows the olive oil spike at a 200 ppb concentration.

Figure 1. Multi-Pesticide Analysis of Olive Oil by GC-MS/MS on a Zebron ZB-5MSPLUS GC Column

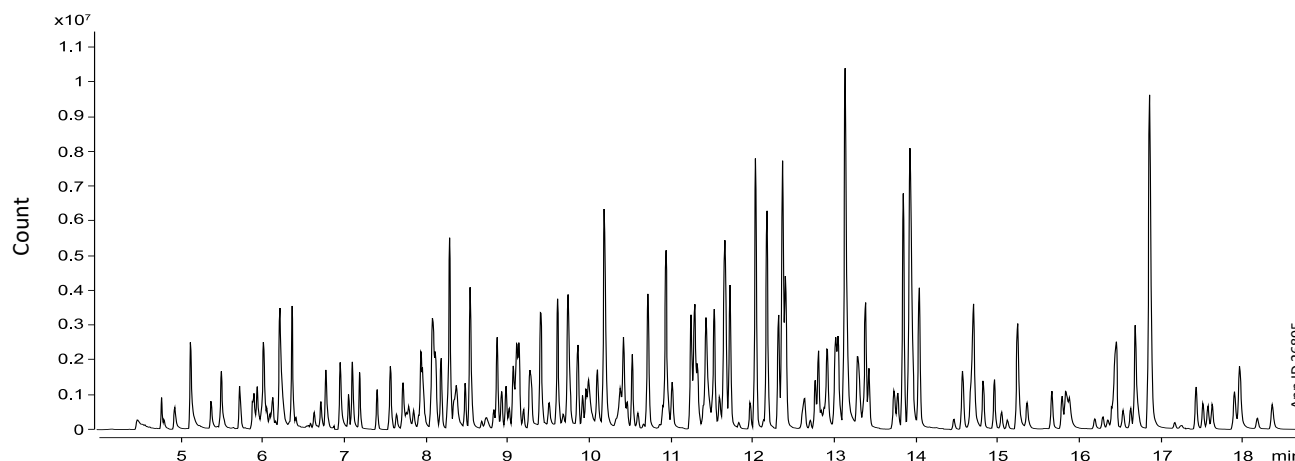


Table 1. MRM Transitions and Retention Profile for Multi-Pesticide Analysis

Compound Name	Precursor ion	Product ion	RT (min)	Dwell (ms)	CE (V)	Compound Name	Precursor ion	Product ion	RT (min)	Dwell (ms)	CE (V)
Methamidophos	141	95, 79, 64	4.58	13.2	5	Acetochlor	223	147, 132	9.10	43.4	15
Dichlorvos	184.9	83	4.68	14.6	10	Chlorpyrifos-methyl	286	271, 93	9.14	12.2	15
Dichlorvos	144.9	109	4.68	14.5	10	Vinclozolin	212	172	9.19	109.1	15
Dichlorvos	109	79	4.68	14.5	5	Vinclozolin	212	145	9.19	109.1	25
Allidochlor	134	56	4.91	25.8	5	Transfluthrin	163	143, 127	9.24	25.9	20
Allidochlor	132	56.1	4.91	25.8	5	Propisochlor	162	147, 120	9.25	21.5	15
Linuron (artifact)	187	159, 124	5.00	10.3	10	Alachlor	269	188, 160	9.26	34.4	5
Diuron artifact	187	124, 100	5.03	13.3	20	Alachlor	237	160	9.26	27.4	5
Dichlobenil	171	136, 100	5.26	13.8	15	Parathion Methyl	263	109, 79	9.27	13.2	10
Biphenyl	154	128, 115	5.44	32.4	25	Tolclofos Methyl	265	220, 93	9.29	25.9	25
Biphenyl	153	127	5.44	29.1	25	Metalaxyl	249	190, 146	9.37	11.4	5
Mevinphos	192	164, 127, 66	5.61	15.4	5	Fenchlorphos	285	240, 93	9.44	14.2	30
Pebulate	203	128, 57	5.82	12.8	0	Heptachlor	274	239, 237	9.47	10.8	15
Pebulate	161	128	5.82	12.8	5	Heptachlor	237	143	9.47	10.8	35
Etridiazole	211	183, 140	5.85	15.5	10	Pirimiphos Methyl	290	151, 125	9.58	15.2	15
Phthalimide (Folpet deg)	147	103, 76	5.86	12.2	5	Prodiamine	321	279, 203	9.58	18.3	5
Phthalimide (Folpet deg)	104	50	5.86	12.3	25	Prodiamine	275.1	255.1	9.58	18.6	10
Tetrahydrophthalimide	151	122, 80, 79	5.98	33.2	5	Prosulfocarb	251	128.2	9.59	24.7	5
Methacrifos	240	180	6.06	12.2	5	Prosulfocarb	128	86.1	9.59	24.7	0
Methacrifos	208	93	6.06	12.2	10	Prosulfocarb	91	65	9.59	39.8	15
2-Phenyl phenol	169	141, 115	6.34	40.3	15	Fenitrothion	277	109	9.68	14.1	15
Pentachlorobenzene	252	215	6.36	12.8	20	Fenitrothion	260	125	9.68	13.6	10
Pentachlorobenzene	250	215, 179	6.36	12.8	20	Malathion	173	127, 99	9.79	10.8	5
Molinate	187	126.1	6.39	13.1	5	Malathion	158	125	9.79	11.4	10
Molinate	126.2	98.1, 83, 55	6.39	13.1	5	Metolachlor	238	162, 113	9.93	15.4	10
Tetrachloronitrobenzene	260.9	203, 179	6.84	33.5	10	Chlorpyrifos	314	286, 258	9.94	13.7	5
Propachlor	211	120	6.90	19.6	18	Chlorthal-dimethyl	301	273, 223	10.00	12.1	15
Propachlor	196	120	6.90	19.6	10	Fenthion	278	169, 125	10.03	13.6	15
Propachlor	176	57	6.90	19.6	5	Fenthion	278	125	10.03	13.6	15
Tecnazene	261	203	6.90	34.5	10	Parathion Ethyl	291	109, 91, 81	10.09	13.5	10
Tecnazene	215	179	6.90	34.5	10	Aldrin	263	228	10.12	27.4	20
Diphenylamine	169	167, 66	7.10	12.8	20	Aldrin	255	220	10.12	27.4	20
Ethafluralin	316	276, 202	7.14	16.7	10	Triadimefon	208	127, 111	10.14	25.9	15
Trifluralin	306	264	7.25	44.1	5	Antraquinone	208	152	10.16	25.8	20
Trifluralin	290	248	7.25	50.7		Antraquinone	180	152	10.16	22.6	10
Chlorpropham	213	171, 127	7.25	15.6	5	4,4'-Dichlorobenzophenone	250	215, 139	10.23	38	5
Benfluralin	292	264, 160	7.28	31.5	5	Pirimiphos Ethyl	318	182, 166	10.28	15.2	10
Sulfotep	322	174, 146	7.38	29.4	15	Diphenamid	167	165, 152, 115	10.36	12.8	20
Diallate	234	192, 150	7.49	14.4	15	Bromophos methyl	331	286, 93	10.36	28.4	30
Hexachlorocyclohexane-alfa	219	183, 109	7.72	10.5	5	Fenson	268	141, 77	10.38	13.6	5
Hexachlorocyclohexane-alfa	217	181	7.72	9.9	5	Pendimethalin	252	191, 162	10.53	12.8	10
Hexachlorobenzene	284	249, 214	7.78	10.6	15	Fipronil	367	255, 213	10.54	13.1	25
Dimethoate	229	87	7.87	13.2	5	Cyprodinil	224	118, 104	10.57	10.9	40
Dimethoate	125	47	7.87	13.2	30	Metazachlor	209	132, 117	10.60	11.9	15
Dimethoate	87	46	7.87	12.8	20	MGK-264	164.2	98, 67.1	10.61	13.9	10
Dichloran	206	176, 148	7.91	14.6	10	Isodrin	262.8	227.9, 193	10.64	9.2	20
Simazine	201	138, 44	7.94	28.3	10	Chlozolinat	331	259	10.65	11.8	5
Atrazine	215	138, 58	8.00	22.6	15	Chlozolinat	259	188	10.65	13.8	10
Hexachlorocyclohexane-beta	219	183, 109	8.09	9.9	5	Chlorphenvinfos	323	267	10.71	15.5	10
Hexachlorocyclohexane-beta	217	181	8.09	9.9	5	Chlorphenvinfos	267	159	10.71	15.6	15
Clomazone	204	107, 78	8.10	11.6	20	Heptachlor-epoxide-B-exo	357	284, 265	10.79	10.8	20
Profluralin	318	199, 155	8.11	18.8	15	Heptachlor-epoxide-B-exo	353	282, 263	10.79	10.8	20
Propyzamide	254	226	8.17	24.9	15	Triadimenol	168	70, 43	10.80	32.5	10
Propyzamide	254	191	8.17	24.9	20	Quinalphos	298	190, 156	10.85	26.8	15
Terbutylazine	214	132, 104	8.22	28.5	10	Trifluzol	278	73, 43	10.90	44.1	5
Terbufos	231	129, 97	8.24	28.5	20	Procyimidone	285	96	10.90	15.7	10
Lindane-gamma	219	183, 109	8.25	12.3	5	Procyimidone	283	96	10.90	17.3	10
Diazinon	304	179, 137	8.29	14.1	15	Procyimidone	283	67	10.90	17.3	40
Diazinon	199	135	8.29	13.8	10	Bromophos ethyl	359	331, 303	11.09	28.4	5
Fonofos	246	137, 109	8.30	11.8	5	Captan	264	106, 79	11.12	28	10
Fluchloralin	326	63	8.32	13.1	10	Captan	151	80	11.12	28.3	5
Fluchloralin	306	264, 158	8.32	13.1	10	Captan	149	70	11.12	30.1	15
Pyrimethanil	198	118	8.43	24.9	35	Captan	117	82	11.12	30.1	30
Tefluthrin	197	161	8.46	36.8	5	Methodathion	145	85, 58	11.12	15.2	5
Tefluthrin	177	127, 87	8.46	36.8	20	Methodathion	85	58	11.12	16.4	5
Chlorothalonil	266	133	8.49	17.7	40	Clorbensid	270	127	11.19	11.4	10
Chlorothalonil	264	168	8.49	15.5	25	Clorbensid	268	125	11.19	11.4	10
Isazophos	257	162	8.52	9.2	5	Chlordane-Cis	372.9	265.9, 236.9	11.20	31.5	20
Isazophos	257	161, 119	8.52	9.1	10	Chlordane-trans (gamma)	375	266	11.43	31.8	20
Terbacil	161	88	8.53	31.9	20	Chlordane-trans (gamma)	373	301, 266	11.43	31.8	10
Terbacil	117	76	8.53	28.5	5	Bromfenvinphos	268.9	161.1	11.44	29.1	15
Triallate	268	226, 184	8.66	32.5	10	Bromfenvinphos	266.9	159.1	11.44	29.1	15
Hexachlorocyclohexane-delta	219	183, 109	8.69	9.5	5	Fenamiphos	303	180	11.45	15.3	20
Hexachlorocyclohexane-delta	217	181	8.69	10.4	5	Fenamiphos	303	154	11.45	14.9	15
Pirimicarb	238	166.2	8.71	13.1	10	Endosulfan alfa	336.8	125	11.46	13.9	52
Pirimicarb	166	96, 55.1	8.71	13.1	15	Endosulfan alfa	241	206	11.46	14	16
Pirimicarb desmethyl	224	152, 92	8.82	15.3	15	Endosulfan alfa	194.9	160, 159	11.46	14	5
Pirimicarb desmethyl	224	96	8.82	15.3	20	Flutriafol	219	123, 95	11.48	12	15
Dimethachlor	209.9	134.1, 132.1	8.96	13.1	10	Nonachlor, cis-	406.8	299.8, 108	11.48	13	15
Dimethachlor	198.9	148.2	8.96	13.1	10	Flutolanil	323	281, 173	11.50	12.2	5
Dimethachlor	196.9	148.2	8.96	13.1	10	Fludioxonil	248	154, 127	11.57	13.1	20
Pentachloraniline	265	230, 194	9.00	12.8	10	Iodofenphos	377	250, 157	11.58	10.4	25
Pentachloraniline	263	192	9.00	12.8	20	Chlorfenson	302	175, 111	11.61	22.9	5



Table 1. MRM Transitions and Retention Profile for Multi-Pesticide Analysis, Continued

Compound Name	Precursor ion	Product ion	RT (min)	Dwell (ms)	CE (V)	Compound Name	Precursor ion	Product ion	RT (min)	Dwell (ms)	CE (V)
Pretilachlor	262	202, 145	11.61	15.2	20	Resmethrin	171	128	13.64	26.5	15
Prothiofos	309	239, 221	11.63	39.8	15	Iprodione	314	245	13.91	9.2	10
Oxyfluorfen	361	300	11.65	13.7	15	Iprodione	314	56	13.91	9.2	20
Oxyfluorfen	300	223	11.65	13.5	15	Pyridaphenthion	340	203	13.92	24.9	30
Profenofos	339	269, 251	11.70	18.6	15	Pyridaphenthion	340	199	13.92	23.7	5
Bupirimate	272.9	193.1, 108	11.70	28.4	5	Pyridaphenthion	199	92	13.92	23.7	15
Oxadiazone	174.9	112, 76	11.73	13	15	Bifenthrin	181	165	14.051	32.4	25
DDE-p,p'	318	248, 176	11.79	13.1	15	Bifenthrin	181	115	14.05	32.4	55
Myclobutanil	179	125, 90	11.83	13	10	Endrin ketone	317	281	14.10	17.2	5
Flusilazole	233	165, 152	11.86	12.2	15	Endrin ketone	317	101	14.10	17.2	15
Kresoxim Methyl	206	132, 131, 116	11.87	9.4	10	Fosmet	160	133	14.10	11.3	10
DDD-o,p'	235	199, 165	11.91	12	15	Fosmet	160	105	14.10	10.8	15
Chlorfenapyr	328	247, 227	12.02	25.8	15	Fosmet	160	77	14.10	10.8	20
Fluzifop-P-butyl	383	282, 254	12.17	13.1	10	EPN	157	110	14.13	17	15
Ethylan	223.1	179.1, 167.1	12.23	16	20	EPN	157	63	14.13	17	10
Perthane	223	193, 165	12.23	12.4	30	Tetramethrin	164	135	14.13	28.6	15
Endrin	278.8	242.9, 227.9	12.34	15.5	10	Tetramethrin	164	107	14.13	28.6	10
Endrin	262.8	227.9	12.34	16.6	20	Tetramethrin	164	77	14.13	28.6	25
Endrin	244.8	173	12.34	16.6	30	Bromopropylate	341	183	14.16	28.4	20
Endrin	242.8	173	12.34	17.6	30	Bromopropylate	341	157	14.16	28.4	45
Chlorobenzilate	251	139, 111	12.37	17.7	15	Methoxychlor	227	169	14.23,	16.5	25
Endosulfan beta	338.8	125	12.53	15.5	52	Methoxychlor	227	141	14.23	16.5	40
Endosulfan beta	336.8	125	12.53	15.5	52	Tetradifon	354	227	14.65	36.2	10
Endosulfan beta	241	206	12.53	15.5	16	Tetradifon	354	159	14.65	36.2	10
Endosulfan beta	239	204	12.53	15.5	16	Phosalone	367	182	14.75	12.2	5
Chlorthiophos	325	205	12.56	12.6	30	Phosalone	367	111	14.75	12.2	35
Chlorthiophos	269	205	12.56	12.6	15	Leptophos	377	362, 269	14.77	11.4	20
DDD-p,p'	320	237, 165	12.56	12.3	3	Leptophos	171	77	14.77	10	15
DDD-p,p'	235	199, 165	12.56	12.3	15	Azinphos-methyl	160	132, 77	14.85	22.2	0
DDT-o,p'	354	235	12.56	12.9	15	Azinphos-methyl	132	51	14.85	22.5	30
DDT-o,p'	235	199, 165	12.56	12.9	15	Pyriproxifen	226	186	14.90	24.9	15
Ethion	231	175, 129	12.57	16.3	10	Pyriproxifen	136	96, 41	14.90	24.9	15
Nonachlor, trans-	406.8	299.8, 108.8	12.58	13	15	Lambda-Cyhalothrin	208	181	15.02	9.4	5
Endrin aldehyde	345	281, 245	12.78	17.6	5	Lambda-Cyhalothrin	199	161	15.02	9.6	5
Triazophos	257	162, 134	12.80	35.5	5	Lambda-Cyhalothrin	197	161	15.02	9.6	5
Sulprophos	322	156, 139	12.84	33.1	10	Acrinathrin	289	93	15.20	34.8	10
Carfentrazone ethyl	312	195, 151	12.91	30	15	Acrinathrin	247	68	15.20	34.8	30
Benalaxyl	266	148	12.97	24.6	5	Mirex	272	237	15.27	13.91	15
Benalaxyl	234	146	12.97	24.6	20	Mirex	237	143	15.27	13.9	30
Trifloxystrobin	222	190	12.98	35.5	5	Fenarimol	330	139, 111	15.30	14.9	5
Trifloxystrobin	222	130	12.98	35.5	15	Azinphos-ethyl	160	132, 77	15.43	22.1	0
4-4'Methoxychlor Olefin	308		13.00	37.7	20	Azinphos-ethyl	132	51	15.43	22.2	30
4-4'Methoxychlor Olefin	238	223.1	13.00	37.7	15	Pyraclofos	360	194, 138.8	15.54	63.6	10
Norflurazon	303	173	13.03	13	10	Pyraclofos	194	138	15.54	37.4	15
Norflurazon	303	145	13.03	13	20	Permethrin	182.9	168.1, 155.1	15.90	12.7	10
Carbophenothion	342	157	13.04	28.8	10	Fluquinconazole	340	298, 286	16.00	12.2	15
Carbophenothion	199	47	13.04	28.8	20	Prochloraz	310	70	16.05	17	15
Edifenphos	310	173	13.12	13.3	10	Prochloraz	308	70	16.05	17	15
Edifenphos	173	109	13.12	13.9	5	Prochloraz	180	138, 169	16.05	17	10
Lenacil	153	136	13.18	9.8	15	Pyridaben	309	147, 132	16.06	24.9	15
Lenacil	153	110	13.18	9.8	15	Cyfluthrin	226	206	16.48	11.1	15
Lenacil	153	82	13.18	11.4	20	Cyfluthrin	199	170	16.48	11.1	30
Endosulfan sulfate	387	289	13.21	15.5	5	Cyfluthrin	163	127, 135.1	16.48	10.9	5
Endosulfan sulfate	272	237	13.21	15.5	15	Etofenprox	163	107.1	16.80	15.5	20
Endosulfan sulfate	270	235	13.21	15.5	15	Cypermethrin	181	127	16.89	10.9	35
DDT-p,p'	235	199	13.25	12.7	15	Cypermethrin	165	127	16.89	10.9	5
DDT-p,p'	235	165	13.25	13.9	20	Cypermethrin	163	127, 91	16.89	11	5
Hexazinone	171	85	13.32	10.4	10	Flucythrinate	451	225, 199	16.95	13	5
Hexazinone	171	71	13.32	10.4	10	Flucythrinate	451	199	16.95	13	5
Methoxychlor, o,p'-	227.1	121.1	13.33	16.3	15	Flucythrinate	225	147, 119	16.95	12.9	10
Methoxychlor, o,p'-	227.1	77	13.33	16.3	45	Flucythrinate	199	107	16.95	12.9	25
Methoxychlor, o,p'-	227.1	65	13.33	15.4	45	Flucythrinate	157	107	16.95	12.9	15
2-4'Methoxychlor	227.1	121.1	13.34	40.3	10	Fluvalinate tau	252	200	17.55	12	15
2-4'Methoxychlor	227.1	91.1	13.34	40.3	35	Fluvalinate tau	250.1	208	17.55	12	15
Diflufenican	394	266	13.47	13.7	10	Fluvalinate tau	250	200	17.55	11.8	15
Diflufenican	266	246	13.47	13.7	15	Fluvalinate tau	250	55	17.55	11.8	15
Propargite	350	201	13.50	19.6	5	Fenvalerate	419	225	17.70	13.5	10
Propargite	350	173	13.50	19.6	20	Fenvalerate	225	119	17.70	13.5	15
Propargite	173	135	13.50	21.5	15	Fenvalerate	169	127	17.70	13.1	10
Tebuconazole	250	153	13.50	33.1	10	Fenvalerate	167	125	17.70	13.1	10
Tebuconazole	250	125	13.50	34.5	20	Difenoconazole	324.8	266.8	18.20	14.5	15
Piperonylbutoxide	338	176	13.59	12.6	5	Difenoconazole	322.8	264.8	18.20	13.7	15
Piperonylbutoxide	338	118	13.59	12.6	35	Difenoconazole	264.9		18.20	13.7	20
Piperonylbutoxide	176	131	13.59	13.1	15	Deltamethrin	253	174	18.38	13.9	5
Piperonylbutoxide	176	117	13.59	13.1	20	Deltamethrin	251	172	18.38	14.4	5
Triphenylphosphate (TPP)	325	231	13.60	50.7	20	Deltamethrin	172	93	18.38	14.4	10
Triphenylphosphate (TPP)	325	169	13.60	50.7	20	Azoxystrobin	344	172, 156	18.56	22.5	40
Resmethrin	171	143	13.64	26.5	5						



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