Application Note

Food

Agilent

Trusted Answers

Fast Analysis of Pesticide Residues in Food Samples Using GC/MS/MS

Authors

Amadeo R. Fernandez-Alba, Samanta Uclés, and Elena Hakme
Pesticide Residue Research Group
Department of Hydrogeology and Analytical Chemistry
University of Almería
Almería, Spain

Joerg Riener
Agilent Technologies, Inc.
Waldbronn, Germany

Abstract

This Application Note describes the evaluation and validation of a fast, 12.4-minute method for the multiresidue pesticide analysis of various fruits and vegetables using an Agilent Intuvo 9000 GC system and an Agilent 7010B triple quadrupole mass spectrometer. It also describes its use with reference materials and routine samples. The 203 compounds targeted were the main pesticides recommended for GC/MS analysis by the EURL for fruits and vegetables. Satisfactory sensitivity results were obtained by achieving a limit of quantitation (LOQ) of 2 µg/kg for a wide variety of fruits and vegetables. The speed of the method was possible because of direct heating GC technology, which ensures that the separation power of the chromatography and robustness in day-to-day operation are maintained at a high level. This method enables increased sample throughput, and represents a significant benefit for control laboratories.

Introduction

Interest in fast gas chromatography (GC) methods continues to increase. Laboratories are looking for ways to shorten analysis times to increase sample throughput and reduce analysis costs, without compromising results. Classic conventional ovens used in most GC systems first heat the air inside the oven, then transfer the heat to the GC column. Run times with this approach tend to be 20 to 41 minutes. The Intuvo 9000 GC, with a planar column design, uses efficient direct-contact conduction heating, enabling faster thermal gradients with excellent control. This means the oven can be cooled faster, and that the system uses less than half of the power of a conventional air-bath oven.

In a recent publication[1], the results of a development project were outlined looking at different speed settings for Intuvo-based GC/MS analyzing key pesticides. The publication concluded that satisfactory data for control work could be achieved with a run time of only 12.4 minutes. This Application Note focuses specifically on this 12.4-minute method.

Validation data are shown at 2, 10, and 50 µg/kg for 203 multiclass pesticides in three food matrices (apple, orange, and tomato) in terms of linearity, recoveries, limits of detection (LODs) and LOQs, matrix effects, plus intraday and interday precision.

Experimental

GC/MS method

An Intuvo 9000 GC was configured with a midcolumn backflush chip configured with two 15 Intuvo HP-5ms Ultra Inert (UI) columns. The GC was interfaced to a 7010 mass spectrometer with a high-efficiency ion source. Table 1 lists the instrument conditions used during the study. Analyses were performed using dynamic multireaction monitoring (dMRM) instead of time segments. Two transitions per analyte were set with a retention time window range of 0.2 minutes. The dMRM function automatically adjusts the dwell times according to the number of transitions needed at any given time in the method. Table 2 shows the compounds covered in the study with the two transitions for each analyte, the collision energy, and the retention time.

Table 1. 9000 Intuvo GC and 7010B GC/TQ instrument conditions.

Parameter	Value
9000 Intuvo GC	
Inert flowpath configuration	Midcolumn backflush chip (p/n G4588-60721)
Syringe	10 µL (p/n G4513-80204)
Solvent washes	**Preinjection:** 2× solvent A, ethyl acetate (8 µL) 2× solvent B, ethyl acetate (8 µL) **Post injection:** 4× solvent A, ethyl acetate (8 µL) 4× solvent B, ethyl acetate (8 µL)
Sample wash	1 × 1 µL
Sample pumps	6
Carrier gas	Helium
Inlet	Multimode Injector (MMI) in pulsed splitless mode, 300 °C
Purge flow to split vent	60 mL/min at 0.75 minutes
Septum purge flow	2 mL/min, switched
Gas saver	20 mL/min after 3 minutes
Intuvo Guard Chip	Track oven temperature
Column	Two Agilent J&W HP-5ms Ultra Inert columns (15 m × 0.25 mm id, 0.25 µm film thickness, p/n 19091S-431UI-INT)
Column flow	Column 1: 1.611 mL/min Column 2: 1.811 mL/min
Column temperature program	80 °C initial temperature 20 °C/min to 170 °C 20 °C/min to 310 °C (3.5 minutes)
7010B Series triple quadrupole GC/MS	
Transfer line	280 °C
Source temperature	280 °C
Quadrupole temperature	150 °C
Solvent delay	3.1 minutes
Tune file	atunes.eihs.tune
Acquisition method	Two transitions per analyte, dynamic MRM (time window 0.2 minutes)

Table 2. List of compounds analyzed with their corresponding acquisition parameters (retention times, transitions, and collision energies).

Compound	RT (min)	SRM 1	CE1 (V)	SRM 2	CE2 (V)
2,4'-DDE	7.083	246 → 211	20	246 → 176	30
2-Phenylphenol	4.412	170 → 141	30	170 → 115	40
4,4'-DDD	7.758	235 → 199	15	235 → 165	20
4,4'-DDE	7.364	246 → 211	20	246 → 176	30
4,4'-DDT	8.11	235 → 199	20	235 → 165	20
Acrinathrin	9.076	289 → 93	5	208 → 181	5
Alachlor	6.103	188 → 160	10	188 → 130	40
Aldrin	6.506	293 → 257	8	293 → 186	40
Ametryn	6.09	227 → 212	8	227 → 185	5
Atrazine	5.349	215 → 173	5	215 → 58	10
Azoxystrobin	11.449	344 → 329	10	344 → 156	40
Benalaxyl	8.021	204 → 176	2	148 → 105	20
Bifenox	8.707	311 → 279	14	311 → 216	25
Bifenthrin	8.527	181 → 166	10	181 → 115	50
Biphenyl	3.894	154 → 126	40	154 → 102	40
Bixafen	10.301	413 → 159	12	159 → 139	15
Boscalid	10.069	140 → 112	10	140 → 76	25
Bromopropylate	8.564	341 → 185	20	341 → 155	20
Bupirimate	7.441	273 → 193	5	273 → 108	15
Buprofezin	7.44	305 → 172	5	119 → 91	5
Butralin	6.613	266 → 190	12	266 → 174	20
Butylate	4.013	174 → 146	3	156 → 57	5
Cadusafos	5.09	213 → 73	10	158 → 97	15
Carbofuran	5.319	164 → 149	12	164 → 122	12
Carbophenothion	7.997	342 → 157	10	199 → 143	10
Chinomethionate	7.079	234 → 206	10	206 → 148	15
Chlorbromuron	3.94	233 → 205	12	233 → 124	25
Chlordane	7.16	373 → 301	10	373 → 266	20
Chlorfenapyr	7.563	247 → 227	15	247 → 200	25
Chlorfenvinphos	6.848	295 → 267	5	267 → 81	40
Chlorobenzilate	7.649	139 → 111	15	139 → 75	30
Chlorothalonil	5.755	266 → 231	20	266 → 133	40
Chlorpropham	4.902	213 → 171	5	213 → 127	5
Chlorpyrifos	6.474	314 → 286	5	314 → 258	5
Chlorpyrifos-methyl	6.045	288 → 93	26	286 → 271	26
Chlorthal-dimethyl	6.535	330 → 299	12	330 → 221	12
Chlozolinate	6.791	331 → 216	5	259 → 188	5
Coumaphos	9.606	362 → 109	15	210 → 182	15
Cyfluthrin	9.813	226 → 206	10	263 → 127	10
Cypermethrin	10.022	209 → 141	20	163 → 127	20
Cyproconazole	7.595	222 → 125	18	139 → 111	18
Cyprodinil	6.719	224 → 208	20	224 → 197	20

Compound	RT (min)	SRM 1	CE1 (V)	SRM 2	CE2 (V)
Deltamethrin	11.16	253 → 172	5	253 → 93	5
Desmethyl-pirimicarb	5.886	224 → 152	8	152 → 96	8
Diazinon	5.554	304 → 179	15	137 → 84	15
Dichlofluanid	6.384	224 → 123	8	167 → 124	8
Dichloran	5.318	206 → 176	5	206 → 148	5
4,4'-Dichlorobenzophenone	6.531	250 → 139	8	139 → 111	8
Dichlorvos	3.385	185 → 109	15	185 → 93	15
Diclobutrazol	7.497	270 → 201	8	270 → 159	8
o,p´-dicofol and *p,p´*-dicofol Dieldrin	6.531/9.197 7.454	251 → 139 345 → 263	15 8	139 → 111 279 → 243	15 8
Diethofencarb	6.353	207 → 151	10	151 → 123	10
Dimethenamid	5.984	230 → 154	10	154 → 111	10
Dimethipin	5.385	124 → 76	5	118 → 58	10
Diphenylamine	4.832	169 → 77	35	168 → 140	40
Disulfoton	5.651	142 → 109	5	142 → 81	12
Disulfoton-sulfoxide	3.667	212 → 153	15	125 → 97	3
Dodemorph	6.73	154 → 97	10	154 → 82	20
Endosulfan sulfate	8.134	387 → 289	5	272 → 237	15
Endosulfan-*alpha*	7.211	239 → 204	15	195 → 160	5
Endosulfan-*beta*	7.745	207 → 172	15	195 → 159	10
Endrin	7.66	263 → 193	35	245 → 173	30
EPN	8.58	157 → 110	15	157 → 77	25
Epoxiconazole	8.389	192 → 138	10	192 → 111	35
Ethion	7.772	231 → 175	5	231 → 129	25
Ethofenprox	10.165	163 → 135	5	163 → 107	15
Ethofumesate	6.288	207 → 161	5	207 → 137	10
Ethoprophos	5.09	158 → 114	5	158 → 97	15
Ethoxyquin	5.3	202 → 174	15	202 → 145	30
Etrimfos	5.7	292 → 181	5	292 → 153	20
Fenamidone	8.679	268 → 180	20	238 → 103	20
Fenarimol	9.197	219 → 107	10	139 → 111	15
Fenazaquin	8.712	160 → 145	5	160 → 117	20
Fenbuconazole	9.828	198 → 129	5	129 → 102	15
Fenchlorphos	6.181	285 → 270	15	285 → 240	30
Fenhexamid	8.125	177 → 113	10	177 → 78	20
Fenitrothion	6.282	277 → 260	5	277 → 109	20
Fenpropathrin	8.604	265 → 210	10	181 → 152	25
Fenpropidin	6.219	273 → 98	3	98 → 55	12
Fenpropimorph	6.437	128 → 110	10	128 → 70	12
Fenthion	6.455	278 → 169	20	278 → 109	20
Fenvalerate	10.624/ 10.772	167 → 125	12	125 → 89	20
Fipronil	6.824	213 → 178	10	213 → 143	20

Compound	RT (min)	SRM 1	CE1 (V)	SRM 2	CE2 (V)
Fipronil sulfone	7.399	452 → 383	8	383 → 255	20
Fipronil-desulfinil	6.055	388 → 333	20	333 → 281	15
Flamprop-isopropyl	7.695	276 → 105	5	276 → 77	40
Flamprop-methyl	7.403	276 → 105	8	230 → 170	15
Fluacrypyrim	7.813	145 → 115	15	145 → 102	30
Fluazifop-p-butyl	7.512	282 → 238	20	282 → 91	15
Flucythrinate	10.16	199 → 157	5	157 → 107	15
Fludioxonil	7.308	248 → 154	25	248 → 127	30
Fluopicolide	8.115	209 → 182	20	173 → 109	25
Fluopyram	6.821	223 → 196	15	173 → 145	15
Fluquinconazole	9.608	340 → 298	20	340 → 286	30
Flusilazole	7.435	233 → 165	20	233 → 152	20
Flutolanil	7.217	323 → 281	5	323 → 173	15
Flutriafol	7.212	219 → 123	12	219 → 95	20
Fluvalinate-*tau*	10.75	250 → 200	20	250 → 55	15
Fonofos	5.566	246 → 137	5	137 → 109	5
Formothion	5.841	224 → 125	20	170 → 93	5
Fosthiazate	6.67	195 → 139	5	195 → 103	5
HCB	5.315	284 → 249	25	284 → 214	40
HCH-*alpha*	5.235	219 → 183	5	219 → 145	25
HCH-*beta*	5.449	219 → 183	5	219 → 145	25
Heptachlor	6.174	272 → 237	10	272 → 143	40
Heptachlor endo-epoxide	6.904	183 → 155	15	183 → 119	30
Heptachlor exo-epoxide	6.865	217 → 182	22	183 → 119	25
Heptenophos	4.614	215 → 200	10	124 → 89	15
Hexaconazole	7.281	214 → 172	20	214 → 159	20
Indoxacarb	11.105	264 → 148	25	203 → 134	10
Iprodione	8.14	314 → 245	10	314 → 56	20
Iprovalicarb	7.38	158 → 116	5	158 → 98	10
Isazofos	5.696	257 → 162	5	161 → 119	5
Isocarbophos	6.541	230 → 212	8	136 → 108	8
Isofenphos	6.842	213 → 185	3	213 → 121	3
Isofenphos-methyl	6.709	199 → 167	10	199 → 121	10
Isoprothiolane	7.29	162 → 134	5	162 → 85	5
Isopyrazam	9.303	359 → 303	8	159 → 139	8
Kresoxim-methyl	7.432	206 → 131	10	206 → 116	10
lambda-Cyhalothrin	9.023	197 → 161	5	197 → 141	5
Lindane	5.52	219 → 183	5	219 → 145	5
Malaoxon	5.986	195 → 125	15	127 → 99	15
Malathion	6.343	173 → 99	15	158 → 125	15
Mecarbam	6.837	329 → 160	3	131 → 74	3
Mepanipyrim	7.12	222 → 207	30	222 → 158	30
Merphos	7.337	169 → 113	3	169 → 57	3

Compound	RT (min)	SRM 1	CE1 (V)	SRM 2	CE2 (V)
Metalaxyl	6.141	206 → 162	8	206 → 132	20
Metazachlor	6.789	209 → 133	10	133 → 117	25
Metconazole	8.744	125 → 99	20	125 → 89	20
Methidathion	7.042	145 → 85	5	145 → 58	15
Methiocarb	6.273	168 → 153	10	153 → 109	10
o,p´-methoxychlor and *p,p´*-methoxychlor Metolachlor	8.213/8.610 6.452	227 → 169 238 → 162	25 8	227 → 115 162 → 133	40 10
Mevinphos	3.988	127 → 109	10	127 → 95	15
Molinate	4.492	187 → 126	3	126 → 55	12
Myclobutanil	7.415	179 → 152	5	179 → 125	10
Napropamide	7.257	271 → 128	3	128 → 72	3
Nuarimol	8.253	235 → 139	12	203 → 107	10
Ofurace	7.989	232 → 186	5	232 → 158	20
Oxadixyl	7.795	163 → 132	15	163 → 117	25
Paclobutrazol	7.099	236 → 167	20	236 → 125	10
Paraoxon-methyl	5.645	230 → 200	5	109 → 79	5
Parathion	6.485	291 → 109	10	139 → 109	10
Parathion-methyl	6.046	263 → 109	10	233 → 124	10
Penconazole	6.798	248 → 192	15	248 → 157	25
Pendimethalin	6.775	252 → 191	10	252 → 162	10
Pentachloroaniline	5.945	263 → 227	15	263 → 192	25
Permethrin	9.478	183 → 153	15	163 → 127	5
Phenothrin	8.75	183 → 153	15	123 → 81	8
Phenthoate	6.887	274 → 246	5	274 → 121	10
Phorate	5.12	231 → 175	20	231 → 129	20
Phorate sulfone	6.44	199 < 143	8	153 → 97	10
Phosmet	8.573	160 → 133	15	160 → 77	30
Phthalimide	4.161	147 → 103	5	147 → 76	30
Picolinafen	8.56	376 → 238	25	238 → 145	25
Picoxystrobin	7.156	335 → 173	10	303 → 157	15
Pirimicarb	5.804	238 → 166	10	166 → 96	20
Pirimiphos-methyl	6.267	305 → 180	5	290 → 151	15
Procymidone	6.951	283 → 255	8	283 → 96	8
Profenofos	7.32	337 → 309	5	337 → 267	15
Prometon	5.29	225 → 183	3	225 → 168	10
Prometryn	6.113	241 → 226	8	241 → 184	12
Propaphos	6.997	220 → 140	12	220 → 125	25
Propazine	5.377	229 → 187	3	214 → 172	8
Propiconazole	8.08	259 → 191	8	259 → 173	10
Propyzamide	5.507	173 → 145	15	173 → 109	30
Prosulfocarb	6.177	251 → 128	5	128 → 86	3
Prothiofos	7.295	309 → 239	15	309 → 221	25
Pyraclostrobin	10.666	164 → 132	10	132 → 77	20

Compound	RT (min)	SRM 1	CE1 (V)	SRM 2	CE2 (V)
Pyrazophos	9.175	221 → 193	10	221 → 149	15
Pyridaben	9.553	147 → 132	10	147 → 117	20
Pyrifenox	6.820/ 7.068	262 → 227	10	262 → 200	20
Pyrimethanil	5.575	198 → 156	25	198 → 118	25
Pyriproxyfen	8.913	136 → 96	10	136 → 78	20
Quinalphos	6.89	157 → 129	15	146 → 91	30
Quinoxyfen	8.056	307 → 272	5	307 → 237	25
Quintozene	5.558	295 → 265	10	295 → 237	15
Secbumeton	5.643	225 → 196	5	225 → 169	5
Spirodiclofen	9.496	312 → 259	10	312 → 109	20
Spiromesifen	8.451	272 → 254	3	272 → 209	12
Sulfotep	5.053	238 → 146	10	202 → 146	10
Sulprofos	7.894	322 → 156	10	156 → 141	15
Tebuconazole	8.233	250 → 153	12	250 → 125	20
Tebufenpyrad	8.638	333 → 276	5	333 → 171	20
Tecnazene	4.796	215 → 179	10	203 → 143	20
Tefluthrin	5.617	177 → 137	15	177 → 127	15
Terbufos	5.505	231 → 175	10	231 → 129	25
Terbumeton	5.391	225 → 169	3	169 → 154	5
Terbutryn	6.244	241 → 185	3	241 → 170	10
Tetrachlorvinphos	7.108	329 → 109	25	329 → 79	35
Tetraconazole	6.517	336 → 218	30	336 → 204	30
Tetradifon	8.831	356 → 229	10	356 → 159	10
Tetrahydrophthalimide	4.237	151 → 122	8	151 → 80	5
Tetramethrin	8.500	164 → 107	15	164 → 77	30
Thiobencarb	6.389	125 → 89	15	100 → 72	3
Tolclofos-methyl	6.096	265 → 250	15	265 → 220	25
Tolylfluanid	6.844	238 → 137	10	137 → 91	20
Triadimefon	6.503	208 → 181	5	208 → 127	15
Triazophos	7.887	161 → 134	5	161 → 106	10
Trifloxystrobin	8.003	222 → 190	3	222 → 130	15
Trifluralin	4.966	306 → 264	10	264 → 160	15
Vinclozolin	6.02	212 → 172	15	212 → 109	40

Method validation

This method was validated according to EU quality control procedures[2]. The analytical parameters evaluated were:

- Selectivity
- Sensitivity
- Linearity
- Recovery
- Repeatability
- Matrix effect
- Inter- and intraday precisions

The linearity of the instrumental method was evaluated by establishing three matrix-matched calibration curves with three different matrices of apple, orange, and tomato. Seven calibration levels of 1, 2, 5, 10, 50, 100, and 200 µg/kg were prepared by spiking the corresponding blank extracts of apple, orange, and tomato, previously prepared by citrate QuEChERS extraction. The LOD for all compounds was also studied by checking the lowest calibration level of 1 µg/kg, with correct accuracy, by checking the two transitions and the ion ratio (<30 %).

Precision of the instrumental method was evaluated with two concentration levels at 2 and 5 µg/kg, in tomato, apple, and orange. Five replicate injections were carried out for each sample.

Interday precision of the overall method was studied by performing spiking experiments of 2, 10, and 50 µg/kg on five different days. Intraday precision was studied by performing five spiking experiments on the same day.

Accuracy of the overall method was studied by comparing the average of the five intraday spiking experiments to the calibration on the same day of analysis.

Spiking procedure

A 35 g sample of homogenized blank of tomato was spiked with a mix of 203 pesticides standards. To ensure homogenization, the sample was stirred for 30 minutes, then left to stand for a further 30 minutes at room temperature, prior to extraction. The spiked sample was split into three portions. The final spiking concentrations were 2, 10, and 50 µg/kg. The same procedure was repeated for the apple and orange matrices. The subsequent steps of sample preparation were applied for each concentration level.

Sample preparation

When considering the merits of developing and validating a routine GC method, the time for sample preparation should be considered; otherwise, the benefits derived from speeding up the separation on the GC instrument become less significant. Citrate QuEChERS[4] was adopted, and was further simplified by excluding the cleanup step. Accordingly, a 10 g portion of sample was weighted into a 50-mL PTFE centrifuge tube. Ten milliliters of acetonitrile were added. A 10 µL aliquot of a 10 mg/kg mixture of three procedural standards (dichlorvos-D_6, malathion-D_{10}, and triphenyl phosphate) was added, and the tubes were shaken in an automatic axial agitator (AGYTAX, Cirta lab. S.L., Spain) for four minutes. Afterwards, 4 g of magnesium sulfate, 1 g of sodium chloride, 1 g of trisodium citrate dihydrate, and 0.5 g of disodium hydrogencitrate sesquihydrate were added, and the samples were again shaken in the automatic axial agitator for four minutes. The extract was then centrifuged at 3,500 rpm for five minutes. Prior to injection into the GC instrument, a solvent exchange took place in which 50 µL of the extract was evaporated and reconstituted with 50 µL of ethyl acetate. An injection standard of lindane-D_6 was also added, at 50 µg/kg.

Results and discussion

Shortening the method run time

The full study and optimization has been published previously[1]. dMRM proved a helpful tool, because evaluation of different methods did not require different MRM acquisition segments. This saved a lot of manual effort in method development.

Linearity and repeatability for the instrumental method

Good linearity was achieved in all cases, with residuals lower than 20 % and correlation coefficients (R^2) higher than 0.99. All compounds were linear up to a concentration level of 200 µg/kg, the highest concentration level examined. However, linearity concentration ranges were different for some pesticides. For 99 % of compounds analyzed in tomato samples, the linearity range was between 1 and 200 µg/kg. Phenothrin and buprofezin had a narrow range of linearity, between 2 and 200 µg/kg. Propaphos had a linear range of 1 to 100 µg/kg. As for the linearity study with apple samples, 98 % of the compounds had a linearity range between 1 and 200 µg/kg. Quinalphos, metconazole, fipronil, and fenhexamid showed a linear range from 2 to 200 µg/kg because of low accuracy at 1 µg/kg. Chlordane showed linearity results between 5 and 200 µg/kg. The picture is different for orange samples due to the difficulty of the acidic matrix. In addition, 94 % of compounds showed good linearity between 1 and 200 µg/kg. Sulfotep, paraoxon methyl, merphos, mercabam, isazofos, chlordane, disulfoton, diethofencarb, malaoxon, quinalphos, and secbumeton showed a narrowed linear range due to compound sensitivity. Ethoxyquin was the only compound that showed a linear range, between 1 and 10 µg/kg.

Repeatability assessment at 2 and 5 µg/kg showed RSDs <10 % obtained for all compounds and in all matrices.

Limits of identification for the instrumental method

All compounds could be identified at 1 µg/kg with tomato samples. Identification is demonstrated with two transitions and good peak shapes. This instrumental limit was also checked when studying the linearity of the method with the concerned matrix studied.

Inter- and intraday precision of the method overall

For interday precision (five days), 97 % of the compounds studied showed satisfactory results (RSD <20 %). Some compounds, such as biphenyl and butylate, showed higher RSDs values. For intraday precision, 97 % of pesticides in all matrices showed an RSD below 20 % except for biphenyl, butylate, chlozolinate, and pyrifenox. Chlozolinate and pyrifenox showed different behavior with apple samples. The intraday precision study included different matrices, which explains the high RSDs obtained for these two compounds.

Accuracy of the method overall

Recovery data showed that 97 % of the compounds had good recovery rates in the acceptance range, between 70 and 120 %, at the concentration levels of 2, 5, and 10 µg/kg in apple, orange, and tomato samples. Figure 1 presents the values obtained.

According to EU analytical quality control procedures, the LOQ is the lowest concentration tested at which recoveries and repeatability values were satisfactory[5]. Therefore, the LOQ for 97 % of the compounds is 2 µg/kg, the lowest validated level with acceptable accuracy and precision.

This was significant for the analysis of baby food, which often has special MRLs lower than the default MRL of 10 µg/kg.

Real-life samples

After the validation study was completed, and to prove the effectiveness of the 12.4-minute run time method and its suitability in routine analysis, the method was applied first to EUPT-FV18 (spinach) and EUPT-FV19 (lemon) samples[6]. Pesticides included in the EUPT-FV18 sample that were suitable for GC analysis were fluopyram, indoxacarb, and metalaxyl. Pesticides included in the EUPT-FV18 sample were fluopyram, boscalid, chlorpyrifos, diazinon, fipronil, iprodione, and pyraclostrobin. Z scores were calculated for each compound, and the results were acceptable. The Z scores obtained were between −0.9 and +0.1 for EUPT-FV18, and between −1.49 and +0.84 for EUPT-FV19.

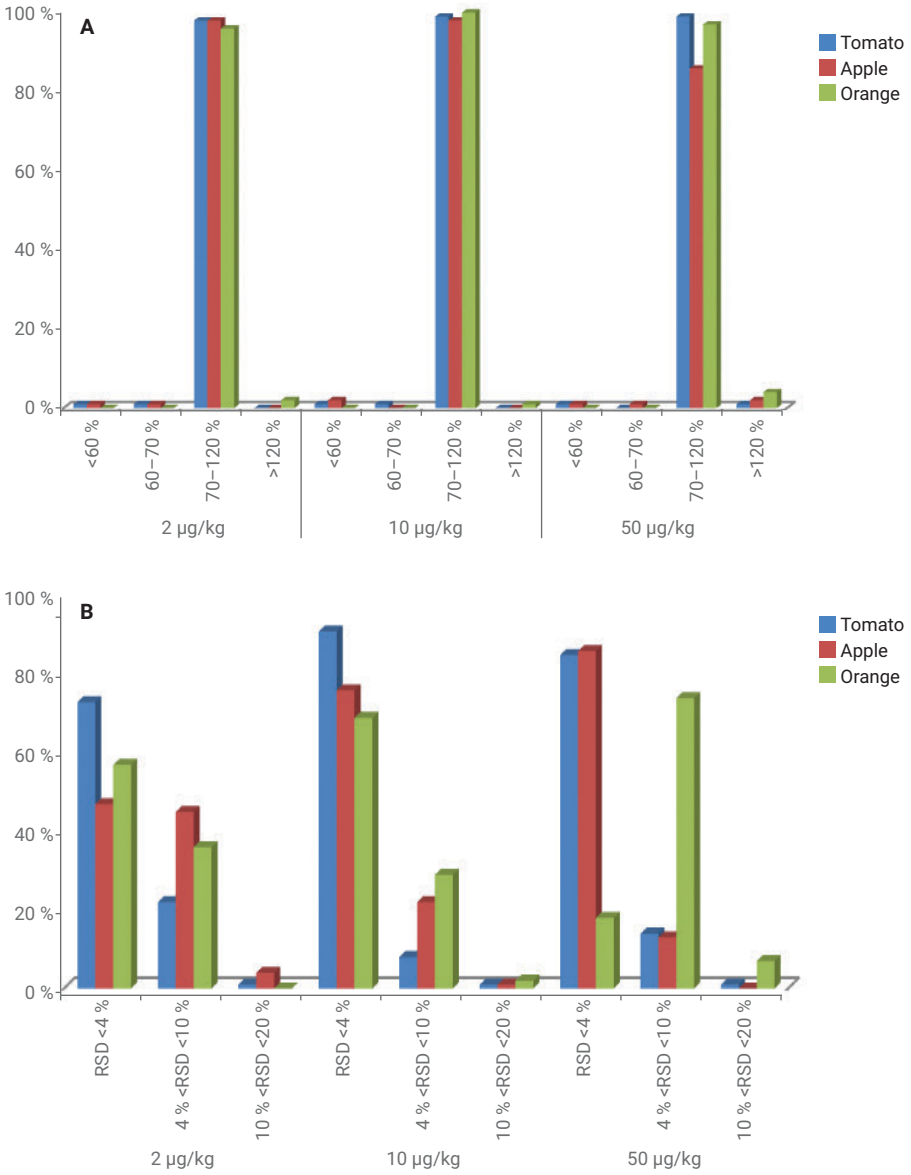

Figure 1. Recovery values (A) and RSDs (B) obtained with the 12.4-minute runtime method with apple, orange, and tomato samples.

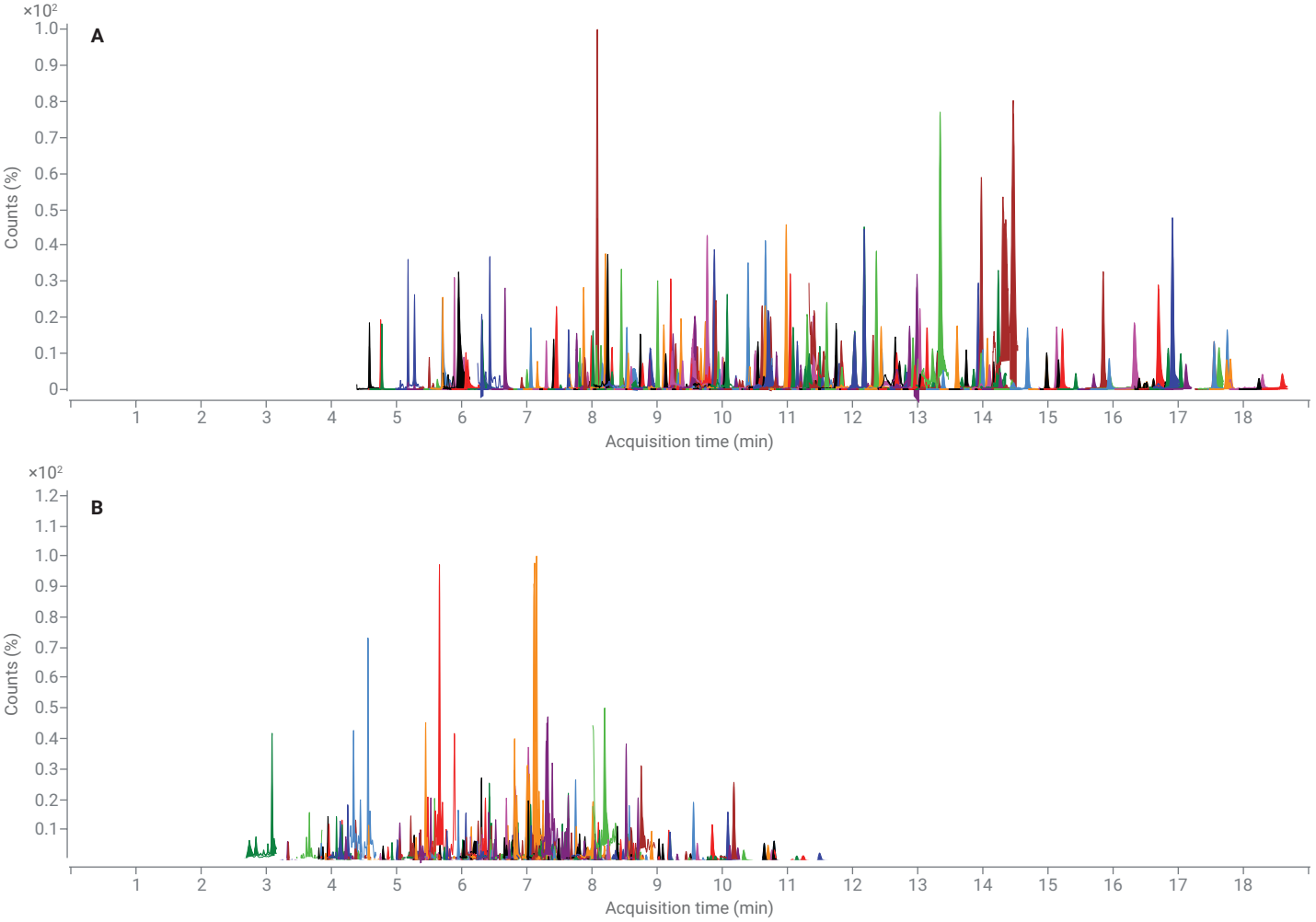

Figure 2. Comparison of standard (A) and fast Agilent Intuvo 9000 GC method (B) (from 21 to 12 minutes).

Conclusions

Using a fast GC program temperature enabled the reduction of total GC analysis time by a factor of 1.5 without compromising the quality of results or the sensitivity of the method. The main benefit of a fast GC method was the increase of laboratory throughput while maintaining necessary separations. This lead to satisfactory method validation parameters (recovery, repeatability, linearity, and matrix effect) even down at 2 µg/kg in most of the cases. The proper quantitation of the method was evaluated by the analysis of two EUPT-FV samples.

References

1. Hakme, E.; *et al.* Further improvements in pesticide residue analysis in food by applying gas chromatography triple quadrupole mass spectrometry (GC-QqQ-MS/MS) technologies, *Analytical and Bioanalytical Chemistry* **2018**, *410/22*, 5491–5506, https://doi.org/10.1007/s00216-017-0723-x.
2. European Commission DG-SANTE (**2015**) Guidance document on analytical quality control and method validation procedures for pesticides residues analysis in food and feed, No SANTE 11945/2015.
3. EU pesticides database available at http://www.ec.europa.eu
4. Anastassiades, M.; *et al.* Fast and easy multiresidue method using acetonitrile extraction/partitioning and "dispersive solid-phase extraction" for the determination of pesticide residues in produce, *J. AOAC Int.* **2003**, *86*, 412–31.
5. Maštovská, K.; Hajšlová, J.; Lehotay, S.J. Ruggedness and other performance characteristics of low-pressure gas chromatography-mass spectrometry for the fast analysis of multiple pesticide residues in food crops. *J Chromatogr A*. **2004**, *1054(1–2)*, 335–49.
6. Ferrer, C.; *et al.* European Union proficiency tests for pesticide residues in fruit and vegetables from 2009 to 2016: Overview of the results and main achievements, *Food Control* **2017**, *82*, 101–113.

www.agilent.com/chem

Printed in the USA, January 9, 2019
5994-0496EN

Agilent
Trusted Answers