

Application Data Sheet

No. 71

GC-MS

Gas Chromatograph Mass Spectrometer

Simultaneous Analysis of Residual Pesticides in Foods via the QuEChERS Method Utilizing GC-MS/MS

Analytical standards (0.001 mg/L to 0.1 mg/L), as well as samples (0.01 mg/L) created by pretreating paprika with the QuEChERS method and then adding pesticides to the resulting solution, were measured using the analysis conditions shown in Table 1.

Experimental

The European Union Reference Laboratory (EURL) has reported their results on evaluating the validity of residual pesticide analysis utilizing GC-MS/MS and LC-MS/MS¹⁾. In their report, the measurement of 66 pesticides using GC-MS/MS was recommended. This data sheet presents selected results of analysis of these pesticides using the triple quadrupole GCMS-TQ8030.

Table 1 Analytical Conditions

GC-MS	:GCMS-TQ8030		
Column	:Rxi-5Sil MS (30 m length, 0.25 mm I.D., df=0.25 μm)		
Glass Liner	:Sky Liner, Splitless Single Taper Gooseneck w/Wool (Restek Corporation, catalog # 567366)		
[GC]			
Injection Temp.	:250 °C		
Column Oven Temp.	:70 °C (2 min) → (25 °C/min) → 150 °C → (3 °C/min) → 200 °C → (8 °C/min) → 280 °C (10 min)		
Injection Mode	:Splitless		
Flow Control Mode	:Linear velocity (58.1 cm/sec.)		
Injection volume	:1 μL		
		[MS]	
		Interface Temp.	:250 °C
		Ion Source Temp.	:230 °C
		Data Acquisition Mode	:MRM (See the below.)

MRM Monitoring *m/z*

Compound Name	Quantitative Transition		Qualitative Transition		Compound Name	Quantitative Transition		Qualitative Transition					
	Precursor>Product	CE (V)	Precursor>Product	CE (V)		Precursor>Product	CE (V)	Precursor>Product	CE (V)				
Diphenylamine	169.10>77.00	26	169.10>115.10	30	Buprofezin	172.10>57.10	18	105.10>104.10	4				
Ethoprophos	200.00>157.90	6	200.00>114.00	14	200.00>97.00	26	273.10>193.20	8	273.10>108.00	18			
Chlorpropham	213.10>171.10	6	213.10>127.10	18	beta-Endosulfan	240.90>205.90	14	238.90>203.90	14				
Trifluralin	306.10>264.00	8	264.10>206.10	8	264.10>160.10	18	Oxadixyl	163.10>132.10	10	163.10>117.10	24		
Dicloran	206.00>176.00	12	206.00>124.00	26	176.00>148.00	12	Ethion	231.00>174.90	14	231.00>128.90	26		
Propyzamide	172.90>144.90	16	172.90>109.00	26	172.90>109.00	26	Triazophos	161.10>134.10	8	161.10>106.10	14		
Chlorothalonil	265.90>230.90	14	265.90>167.90	24	263.90>167.90	24	Endosulfan sulfate	386.90>252.90	10	386.90>216.90	26		
Diazinon	304.10>179.10	12	179.20>137.20	18			Propiconazole-1	259.10>190.90	8	259.10>172.90	18	259.10>69.10	12
Pyrimethanil	199.10>184.10	14	199.10>158.10	14			Propiconazole-2	259.10>190.90	8	259.10>172.90	18	259.10>69.10	12
Tefluthrin	197.10>141.10	26	177.10>127.10	32			Tebuconazole	252.10>127.00	24	250.10>125.10	24		
Pirimicarb	238.20>166.10	10	166.10>96.00	14			Iprodione	314.10>244.90	12	314.10>56.10	24		
Chlorpyrifos-methyl	285.90>270.90	12	285.90>93.00	22			Bromopropylate	340.90>184.90	18	182.90>154.90	16		
Vinclozolin	212.10>172.00	14	212.10>144.90	26	212.10>109.00	30	Bifenthrin	181.10>166.10	16	181.10>165.10	22	181.10>153.10	10
Parathion-methyl	263.10>109.00	18	263.10>81.00	26			Fenpropathrin	265.10>210.10	12	181.10>152.10	24	181.10>127.10	26
Tolclofos-methyl	265.00>249.90	12	265.00>93.00	24			Fenazaquin	160.20>145.10	8	145.20>115.10	24	145.20>91.10	24
Metalaxyl	206.20>162.10	8	206.20>132.10	18			Tebufenpyrad	333.20>276.10	8	333.20>171.00	22		
Fenitrothion	277.10>125.00	18	277.10>109.00	18			Tetradifon	355.90>158.90	12	353.90>159.00	12	228.90>200.90	14
Pirimiphos-methyl	305.10>290.10	12	290.10>125.00	24			Phosalone	182.00>138.00	8	182.00>111.00	18	182.00>102.10	18
Dichlofuanid	332.00>167.10	6	224.00>123.00	12			Pyriproxyfen	136.10>96.00	12	136.10>78.00	24		
Malathion	173.10>117.00	12	173.10>99.00	18			Cyhalothrin	181.10>152.10	24	163.10>127.00	14	163.10>91.00	22
Chlorpyrifos	196.90>168.90	14	196.90>107.00	26			Fenarimol	251.00>139.00	18	139.10>111.00	16		
Fenthion	278.10>125.00	22	278.10>109.00	18			Acrinathrin	289.10>93.10	12	181.10>152.10	24	208.10>181.10	8
Parathion	291.10>109.00	14	291.10>81.00	26			Permethrin-1	183.10>168.10	12	183.10>153.10	18	183.10>115.10	24
Tetraconazole	336.10>218.00	18	336.10>204.00	26			Pyridaben	147.20>132.10	14	147.20>117.10	22		
Pendimethalin	252.20>162.10	12	252.20>161.10	12			Permethrin-2	183.10>168.10	12	183.10>153.10	18	183.10>115.10	24
Cyprodinil	225.20>224.10	6	224.20>208.10	18			Cyfluthrin-1	206.10>151.20	24	163.10>127.10	6	163.10>91.00	14
(E)-Chlorfenvinphos	323.10>266.90	14	267.00>159.00	18			Cyfluthrin-2	206.10>151.20	24	163.10>127.10	6	163.10>91.00	14
Tolyfluanid	137.10>91.00	18	137.10>65.00	26			Cyfluthrin-3	206.10>151.20	24	163.10>127.10	6	163.10>91.00	14
Fipronil	367.00>227.90	26	367.00>212.90	26			Cyfluthrin-4	206.10>151.20	24	163.10>127.10	6	163.10>91.00	14
Captan	79.00>77.00	8	79.00>51.00	22			Cypermethrin-1	181.10>152.10	24	163.10>127.10	6	163.10>91.00	14
(Z)-Chlorfenvinphos	323.10>266.90	14	267.00>159.00	18			Cypermethrin-2	181.10>152.10	24	163.10>127.10	6	163.10>91.00	14
Phenthoate	274.10>125.00	18	274.10>121.10	12			Cypermethrin-3	181.10>152.10	24	163.10>127.10	6	163.10>91.00	14
Folpet	147.10>103.10	10	147.10>76.00	26			Cypermethrin-4	181.10>152.10	24	163.10>127.10	6	163.10>91.00	14
Procymidone	283.10>96.10	12	283.10>67.10	24			Ethofenprox	163.20>135.00	10	163.20>107.10	18		
Methidathion	145.10>85.00	8	145.10>58.00	18			Fenvalerate-1	125.10>99.00	22	125.10>89.00	22		
alpha-Endosulfan	240.90>205.90	14	238.90>203.90	16			tau-Fluvalinate-1	250.10>200.10	16	250.10>55.00	18		
Mepanipyrim	222.20>220.10	8	222.20>193.10	26			Fenvalerate-2	125.10>99.00	22	125.10>89.00	22		
Profenofos	337.10>266.80	16	207.90>63.00	26			tau-Fluvalinate-2	250.10>200.10	16	250.10>55.00	18		
Myclobutanil	179.10>152.00	8	179.10>125.00	16			Deltamethrin-1	252.90>93.10	18	181.10>152.10	24		
Flusilazole	233.10>165.10	18	233.10>152.10	18			Deltamethrin-2	252.90>93.10	18	181.10>152.10	24		

Results

Calibration curves for each pesticide obtained by analyzing six calibration standards (0.001 mg/L to 0.1 mg/L), the mass chromatograms for the 0.01 mg/L samples, and the area repeatability (n=6) for each pesticide obtained from the pesticide-spiked samples (0.01 mg/L) are shown below.

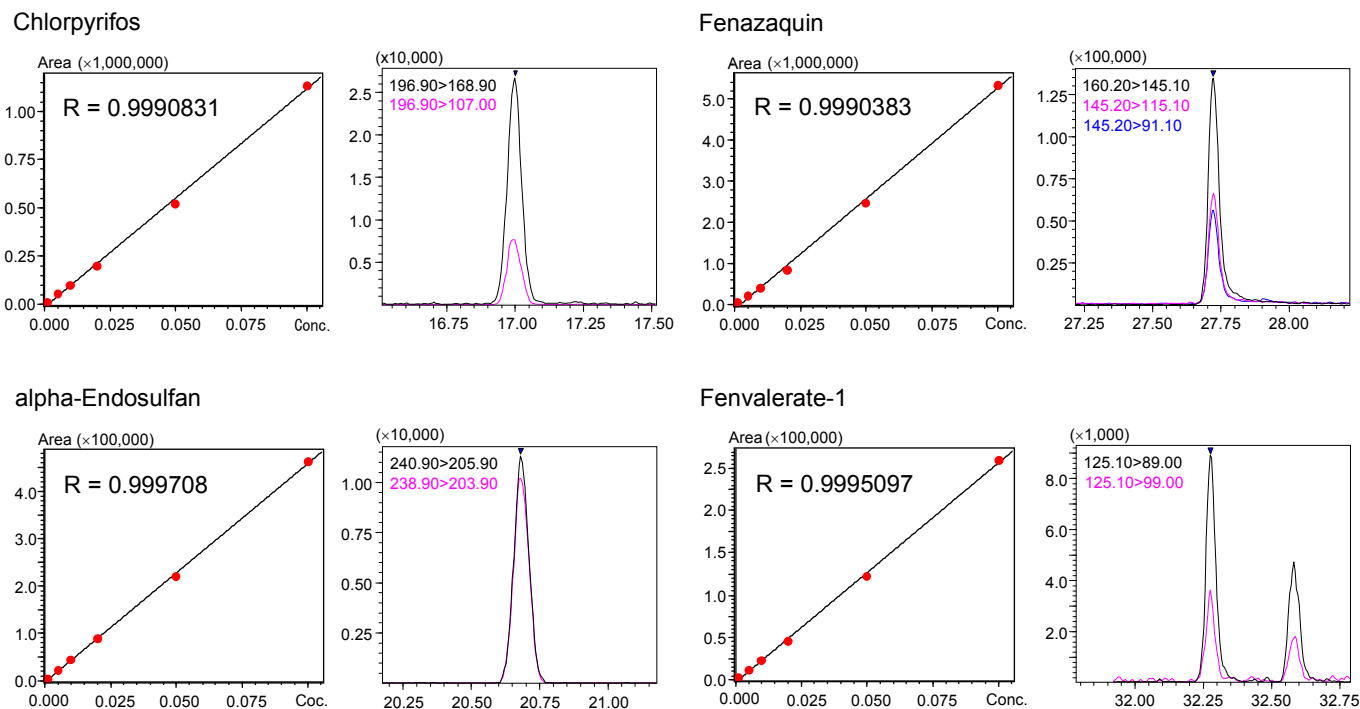


Fig. 1 Calibration Curves for Each Pesticide and the Mass Chromatograms for the 0.01 mg/L Samples

Table 2 Area Repeatability for Each Pesticide (n=6)

Compound Name	%RSD	Compound Name	%RSD	Compound Name	%RSD	Compound Name	%RSD
Diphenylamine	4.99	Chlorpyrifos	5.23	Buprofezin	4.92	Fenarimol	5.16
Ethoprophos	4.95	Fenthion	5.75	Bupirimate	5.47	Acrinathrin	2.03
Chlorpropham	6.26	Parathion	6.93	beta-Endosulfan	6.29	Permethrin-1	6.34
Trifluralin	5.33	Tetraconazole	6.96	Oxadixyl	5.74	Pyridaben	7.11
Dicloran	6.49	Pendimethalin	6.29	Ethion	6.18	Permethrin-2	6.24
Propyzamide	5.52	Cyprodinil	5.21	Triazophos	3.45	Cyfluthrin-1	4.44
Chlorothalonil	4.46	(E)-Chlorfenvinphos	5.35	Endosulfan sulfate	4.26	Cyfluthrin-2	3.77
Diazinon	5.45	Tolyfluanid	4.81	Propiconazole-1	6.02	Cyfluthrin-3	7.35
Pyrimethanil	3.18	Fipronil	6.76	Propiconazole-2	5.56	Cyfluthrin-4	8.19
Tefluthrin	5.13	Captan	5.74	Tebuconazole	7.59	Cypermethrin-1	8.58
Pirimicarb	5.00	(Z)-Chlorfenvinphos	5.52	Iprodione	1.72	Cypermethrin-2	3.71
Chlorpyrifos-methyl	5.27	Phenthoate	6.40	Bromopropylate	5.71	Cypermethrin-3	8.08
Vinclozolin	6.33	Folpet	6.56	Bifenthrin	5.29	Cypermethrin-4	2.48
Parathion-methyl	5.81	Procymidone	6.40	Fenpropathrin	4.00	Ethofenprox	5.03
Tolclofos-methyl	4.89	Methidathion	6.17	Fenazaquin	4.84	Fenvalerate-1	4.20
Metalaxyl	5.43	alpha-Endosulfan	6.27	Tebufenpyrad	5.62	tau-Fluvarlinate-1	2.16
Fenitrothion	5.10	Mepanipyrim	6.41	Tetradifon	6.09	Fenvalerate-2	5.65
Pirimiphos-methyl	5.35	Profenofos	5.92	Phosalone	5.90	tau-Fluvarlinate-2	2.14
Dichlofluanid	4.04	Myclobutanil	5.46	Pyriproxyfen	5.16	Deltamethrin-1	7.58
Malathion	6.31	Flusilazole	5.63	Cyhalothrin	5.38	Deltamethrin-2	7.32

Reference

1) EURL-FV Multiresidue Method using QuEChERS followed by GC-QqQ/MS/MS and LC-QqQ/MS/MS for Fruits and Vegetables (European Reference Laboratory, 2010-M1)

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