SHIMADZU

Application Data Sheet

No.57

GC-MS

Gas Chromatograph Mass Spectrometer

Analysis of Residual Pesticides in Processed Foods Using GC-MS/MS

When analyzing residual pesticides in processed foods, the same analysis method for vegetables cannot be used due to the interference by a large quantity of substances co-extracted from the sample. In such cases, a different sample-preparation method must be considered to remove or minimize the co-extracted substances. However, it is time consuming to develop a optimal sample-preparation method.
This application datasheet presents a study of an analysis of residual pesticides in retort-pouched curry (curry packaged in a sealed pouch) using QuEChERS and GC-MS/MS. QuEChERS method is commonly used for extraction and cleanup of food products prior to GC/MS/MS analysis, due to its speed and simplicity. While GC-MS/MS is able to selectively separate residual pesticides from a large quantity of co-extracted substances.

Experimental

The curry was extracted and prepared for analysis using the QuEChERS method and then spiked with a mixture of 39 pesticides at 0.01 mg/L of all pesticides. The spiked sample was then analyzed by GC-MS and GC-MS/MS using the analytical conditions indicated in Table 1. The SIM (selected ion monitoring) mode was used for the GC-MS and MRM (multiple reaction monitoring) mode was used for the GC-MS/MS.

Table 1 Analysis Conditions

GC-MS :GCMS-TQ8030
Column :Rxi-5Sil MS (30 m length, 0.25 mm I.D., df = 0.25 μm)
Glass Insert :Splitless insert with wool (P/N: 221-48876-03)
[GC]
Injection Temp. : 250 °C
Column Oven Temp. : 50 °C (1 min) → (25 °C/min) → 125 °C → (10 °C/min) → 300 °C (15 min)
Injection Mode : Splitless (high pressure injection at 250 kPa for 1.5 min)
Carrier Gas Control : Linear velocity (47.2 cm/sec)
Injection Volume : 1 μL

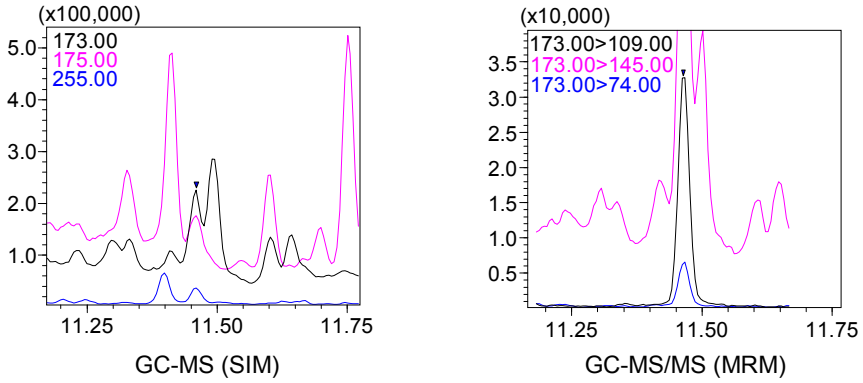
[MS]
Interface Temp. : 250 °C
Ion Source Temp. : 230 °C
Acquisition Mode : MRM (see below)

		Quantitative Transition		Qualitative Transition 1		Qualitative Transition 2	
Compound Name	Retention Time	Precursor>Product	CE	Precursor>Product	CE	Precursor>Product	CE
Mevinphos	7.621	192.0>164.0	5	192.0>127.0	10	192.0>109.0	26
Carbofuran	10.971	164.0>149.0	10	164.0>131.0	16	164.0>103.0	25
Simazine	11.007	201.0>173.0	6	201.0>186.0	6	201.0>138.0	12
gamma-HCH (Lindane)	11.298	219.0>183.0	6	219.0>145.0	20	219.0>181.0	8
Propyzamide	11.464	173.0>145.0	15	173.0>109.0	26	173.0>74.0	30
Diazinon	11.521	304.0>179.0	12	304.0>162.0	6	304.0>195.0	10
delta-HCH	11.838	219.0>183.0	10	219.0>145.0	22	219.0>147.0	22
Ametryn	12.748	227.0>185.0	8	227.0>170.0	12	227.0>212.0	10
Fenitrothion	13.070	277.0>260.0	6	277.0>109.0	18	277.0>125.0	16
Malathion	13.246	173.0>127.0	6	173.0>99.0	15	173.0>145.0	6
Thiobencarb	13.405	257.0>100.0	8	257.0>72.0	24	257.0>224.0	4
Fenthion	13.464	278.0>109.0	20	278.0>125.0	20	278.0>169.0	16
Parathion	13.532	291.0>109.0	15	291.0>137.0	6	291.0>142.0	5
Phthalide	13.726	243.0>215.0	18	243.0>179.0	26	243.0>144.0	40
Fosthiazate-1	13.824	195.0>103.0	8	195.0>139.0	5	195.0>167.0	5
Fosthiazate-2	13.873	195.0>103.0	8	195.0>139.0	5	195.0>167.0	5
Isofenphos	14.200	213.0>121.0	16	213.0>185.0	6	213.0>93.0	28
Procymidone	14.416	283.0>96.0	12	283.0>68.0	24	283.0>255.0	12
Dimepiperate	14.419	145.0>112.0	8	145.0>69.0	16	145.0>84.0	20
Tetrachlorvinphos	14.740	329.0>109.0	20	329.0>314.0	16	329.0>79.0	28
Flutolanil	15.080	173.0>145.0	16	173.0>125.0	26	173.0>95.0	28
Isoprothiolane	15.174	290.0>204.0	6	290.0>118.0	14	290.0>162.0	18
Myclobutanil	15.393	179.0>125.0	15	179.0>152.0	10	179.0>90.0	30
Chlorfenapyr	15.638	247.0>227.0	16	247.0>200.0	26	247.0>177.0	25
Triazophos	16.381	161.0>134.0	8	161.0>106.0	14	161.0>91.0	18
Bifenthrin	17.713	181.0>166.0	12	181.0>165.0	25	181.0>179.0	12
Fenpropathrin	17.892	265.0>210.0	12	265.0>172.0	14	265.0>89.0	26
Pyridaben	19.647	147.0>117.0	22	147.0>119.0	10	147.0>132.0	14
Cypermethrin-1	20.345	163.0>127.0	6	163.0>91.0	15	163.0>109.0	20
Cypermethrin-2	20.448	163.0>127.0	6	163.0>91.0	15	163.0>109.0	20
Cypermethrin-3	20.506	163.0>127.0	6	163.0>91.0	15	163.0>109.0	20
Flucythrinate-1	20.507	451.0>225.0	6	451.0>199.0	15	451.0>157.0	28
Cypermethrin-4	20.546	163.0>127.0	6	163.0>91.0	15	163.0>109.0	20
Flucythrinate-2	20.702	451.0>225.0	6	451.0>199.0	15	451.0>157.0	28
Fenvalerate-1	21.233	419.0>225.0	6	419.0>167.0	14	419.0>125.0	28
Fluvalinate-1	21.351	250.0>200.0	18	250.0>55.0	18	250.0>145.0	26
Fluvalinate-2	21.415	250.0>200.0	18	250.0>55.0	18	250.0>145.0	26
Fenvalerate-2 (Esfenvalerate)	21.435	419.0>225.0	6	419.0>167.0	14	419.0>125.0	28
Imibenconazole	23.008	375.0>260.0	22	375.0>306.0	8	375.0>271.0	18

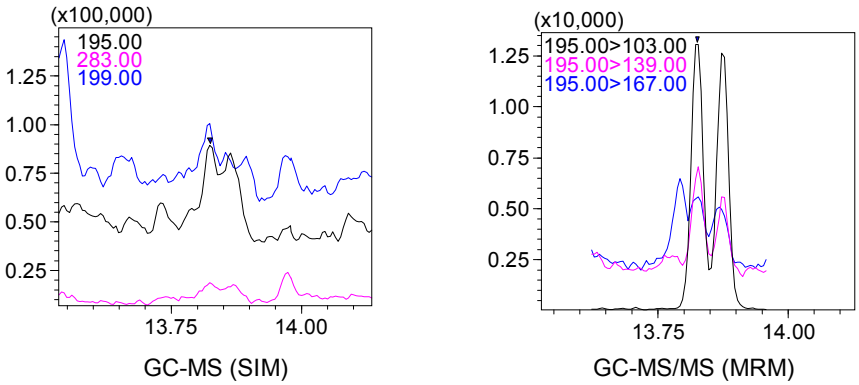
Analysis Results

Fig. 1 shows mass chromatograms of the spiked sample extract (0.01 mg/L) using the SIM mode of GC-MS and the MRM mode of GC-MS/MS. Some of the pesticides could not be detected or identified in the SIM of GC-MS because they overlap with co-extracted substances. By contrast, it was possible to selectively detect and identify those same pesticides using the Multiple Reaction Monitoring technique of GC-MS/MS.
Therefore, GC-MS/MS provided an effective means of analyzing residual pesticides in processed foods that contain a large quantity of substances co-extracted from the sample, such as retort-packed curry.

Propyzamide



Fosthiazate-1,2



Isofenphos

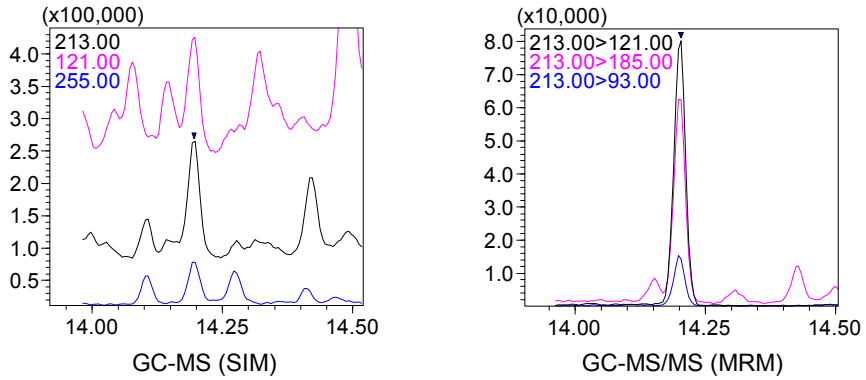


Fig. 1 Mass Chromatograms of Sample Extracts Spiked a mixture of 39 pesticides at 0.01 mg/L

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