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Application Data Sheet

No.78

GC-MS
Gas Chromatograph Mass Spectrometer

Analysis of PCBs and Organochlorinated Pesticides in River Water Using Simultaneous Scan/MRM Measurement in GC-MS/MS (1)

PCBs and some chlorinated pesticides are resistant to decomposition in the environment, and tend to accumulate in living organisms. As a result, they are designated as POPs (Persistent Organic Pollutants) under the Stockholm Convention. In monitoring these environmental pollutants in river water and sludge, sensitivity for the detection of trace quantities and separation from impurities in the samples become important issues. In addition, in recent years, incidents of the discharge of chemical substances have increased on a global scale, heightening demands for the identification of unanticipated pollutants in order to investigate causes and take necessary measures swiftly.

The GCMS-TQ8030 GC-MS/MS system achieves simultaneous scan/MRM measurements using high-speed scan and high-speed MRM data acquisition technology.

This Application Data Sheet reports on MRM sensitivity and analytical repeatability when using simultaneous scan/MRM measurements with respect to PCBs and chlorinated pesticides. Application Data Sheet No. 79 presents an example of the quantification of PCBs and chlorinated pesticides by using the MRM data acquired in simultaneous scan/MRM measurements of a river water sample. In addition, Application Data Sheet No. 80 features an example of screening for environmental pollutants not suited to MRM measurement by applying Compound Composer Database Software to the scan data acquired in simultaneous scan/MRM measurements.

Experimental

The Solution/Mixture of Chlorinated Biphenyl Congeners (BP-MS and BP-MS2, Wellington Laboratories) was used as the PCB standard mixture solution. Pesticide Mix 1037 (Dr. Ehrenstorfer GmbH) was used as the chlorinated pesticide sample, and Custom Internal Standard (P/N: 560294, Restek Corporation) was used as the internal standard. Table 1 shows the analysis conditions. Using the simultaneous scan/MRM measurement mode, 70 PCBs, 22 chlorinated pesticides, and 5 internal standard compounds were subjected to MRM measurement.

Table 1 Analytical Conditions

GC-MS	:GCMS-TQ8030
Column	:DB-5MS (30 m long, 0.25 mm I.D., df=0.25 µm)
Glass Liner	:Deactivated splitless liner without wool (PN: 221-48876-05)

[GC]		[MS]	
Injection Temp.	:250 °C	Interface Temp.	:300 °C
Column Oven Temp.	:40 °C (2 min) → (8 °C /min) → 310 °C (5 min)	Ion Source Temp.	:200 °C
Injection Mode	:Splitless	Acquisition Mode	:Scan/MRM
Flow Control Mode	:Linear velocity (40.0 cm/sec)	Scan Event Time	:0.15 sec
Injection Volume	:1 µL	Scan Mass Range	:*m/z* 45 – 600
		Scan Speed	:5,000 u/sec

MRM Monitoring *m/z*

Internal Standards

Compound Name	Quantitative Transition Precursor > Product	CE (V)	Qualitative Transition Precursor > Product	CE (V)
Acenaphthene-D10	164.1>162.1	31	164.1>164.1	25
Phenanthrene-D10	188.1>186.1	28	188.1>160.1	31
Fluoranthene-D10	212.2>210.2	37	212.2>208.2	46
Chrysene-D12	240.2>238.2	26	240.2>236.2	41
Perylene-D12	264.2>260.2	47	264.2>262.2	44

PCBs

Compound Name	Quantitative Transition Precursor > Product	CE (V)	Qualitative Transition Precursor > Product	CE (V)
Chlorobiphenyl	188.0>152.0	24	190.0>152.0	24
Dichlorobiphenyl	222.0>152.0	24	224.0>152.0	24
Trichlorobiphenyl	256.0>186.0	24	258.0>186.0	24
Tetrachlorobiphenyl	289.9>219.9	24	291.9>221.9	24
Pentachlorobiphenyl	323.9>253.9	24	325.9>255.9	24
Hexachlorobiphenyl	357.9>287.9	27	359.9>289.9	27
Heptachlorobiphenyl	391.9>321.9	30	393.9>323.9	30
Octachlorobiphenyl	427.8>355.8	30	429.8>357.8	30
Nonachlorobiphenyl	461.8>391.8	30	463.8>393.8	30
Decachlorobiphenyl	495.7>425.7	30	497.7>427.7	30

Chlorinated Pesticides

Compound Name	Quantitative Transition Precursor > Product	CE (V)	Qualitative Transition Precurcer > Product	CE (V)
BHC (alpha, beta, gamma, delta)	218.9>182.9	8	218.9>145.0	20
Hexachlorobenzene	283.9>248.8	24	283.9>213.9	28
Heptachlor	271.8>236.8	20	271.8>117.0	32
Aldrin	262.9>192.9	28	262.9>202.9	26
Heptachlor-exo-epoxide	352.9>262.9	14	352.9>281.9	12
Oxychlordane	386.9>286.9	26	386.9>322.9	18
Heptachlor-endo-epoxide	352.9>288.9	6	352.9>252.9	26
Chlordane (cis, trans)	372.9>336.9	10	372.9>265.9	22
DDE (o,p'-, p,p'-)	246.0>176.0	30	246.0>211.0	22
Nonachlor (cis, trans)	408.9>373.9	16	408.9>145.0	24
Dieldrin	276.9>240.9	8	276.9>170.0	38
DDD (o,p'-, p,p'-)	235.0>165.0	24	235.0>199.0	14
Endrin	262.9>190.9	30	262.9>227.9	22
DDT(o,p'-, p,p'-)	235.0>165.0	24	235.0>199.0	16

Results

The MRM mass chromatogram from the measurement of a 1 ng/mL standard mixture containing 70 PCBs is shown in Fig. 1. The MRM mass chromatograms of typical chlorinated pesticides from the measurement of a 1 ng/mL standard sample are shown in Fig. 2. At a concentration of 1 ng/mL, the PCBs and chlorinated pesticides were detected with sufficient sensitivity. Table 2 shows the analytical repeatability (n=8) at a concentration of 5 ng/mL (at 10 ng/mL for some of the chlorinated pesticides).

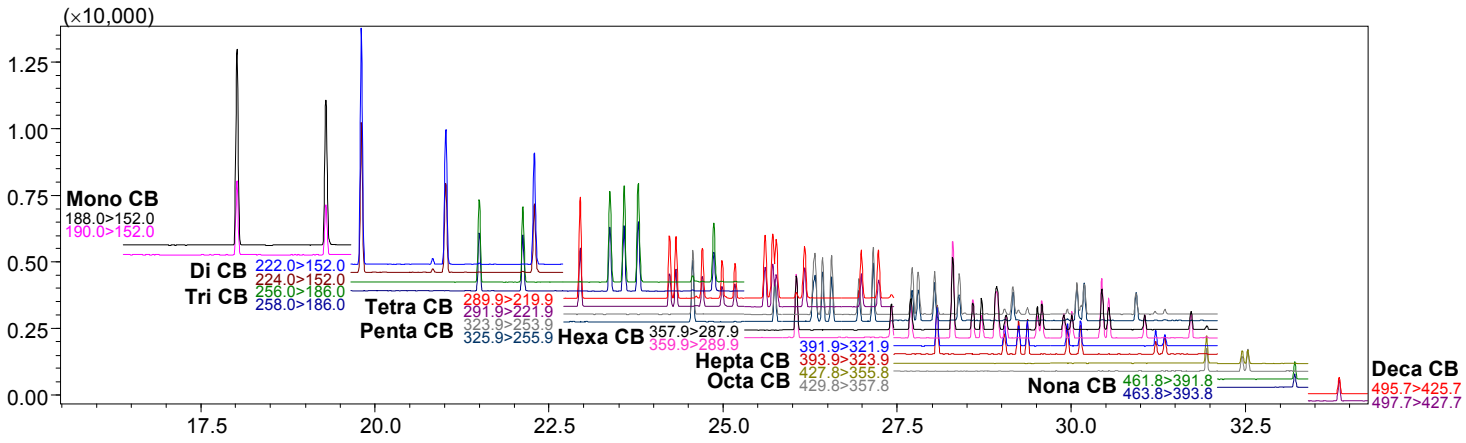

Fig. 1 MRM Mass Chromatogram from the Measurement of a 70 PCB Standard Mixture (1 ng/mL)

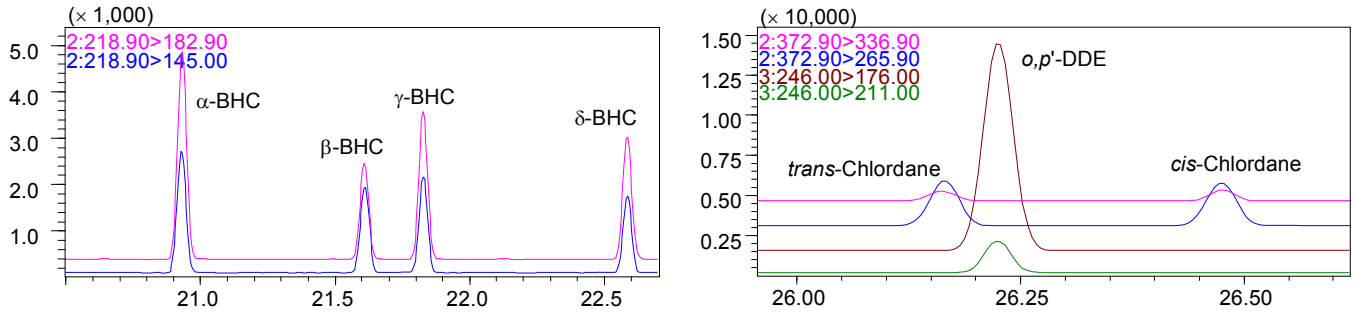

Fig. 2 MRM Mass Chromatograms for Typical Chlorinated Pesticides (1 ng/mL)

Table 2 Analytical Repeatability (5 ng/mL, n=8) for PCBs and Chlorinated Pesticides

Compound Name	%RSD	Compound Name	%RSD	Compound Name	%RSD	Compound Name	%RSD
PCB #1 (Mono CB)	3.28	PCB #60 (Tetra CB)	4.51	PCB #183 (Hepta CB)	9.70	*alpha*-BHC	3.82
PCB #3 (Mono CB)	2.56	PCB #101 + PCB #90 (Penta CB)	5.48	PCB #128 (Hexa CB)	13.06	Hexachlorobenzene	7.73
PCB #4 & #10 (Di CB)	2.78	PCB #99 (Penta CB)	8.12	PCB #167 (Hexa CB)	8.34	*beta*-BHC	9.20
PCB #8 (Di CB)	4.94	PCB #119 (Penta CB)	4.10	PCB #177 (Hepta CB)	9.98	*gamma*-BHC	5.68
PCB #19 (Tri CB)	2.56	PCB #87 (Penta CB)	6.82	PCB #202 (Octa CB)	8.07	*delta*-BHC	11.21
PCB #18 (Tri CB)	1.72	PCB #81 (Tetra CB)	5.61	PCB #171 (Hepta CB)	9.44	Heptachlor	8.10
PCB #15 (Di CB)	3.31	PCB #110 (Penta CB)	3.26	PCB #156 (Hexa CB)	5.40	Aldrin	11.79
PCB #54 (Tetra CB)	3.89	PCB #77 (Tetra CB)	5.65	PCB #201 (Octa CB)	9.84	Heptachlor epoxide *	7.70
PCB #28 (Tri CB)	4.10	PCB #151 (Hexa CB)	6.19	PCB #157 (Hexa CB)	6.97	*trans*-Chlordane	7.87
PCB #33 (Tri CB)	5.57	PCB #149 (Hexa CB)	8.17	PCB #180 (Hepta CB)	2.39	*o,p'*-DDE	4.10
PCB #22 (Tri CB)	2.97	PCB #123 (Penta CB)	7.15	PCB #191 (Hepta CB)	5.23	*cis*-Chlordane	7.78
PCB #52 (Tetra CB)	4.82	PCB #118 (Penta CB)	7.62	PCB #169 (Hexa CB)	7.90	*trans*-Nonachlor *	12.77
PCB #49 (Tetra CB)	3.45	PCB #114 (Penta CB)	6.11	PCB #170 (Hepta CB)	5.14	*p,p'*-DDE	4.92
PCB #104 (Penta CB)	3.60	PCB #188 (Hepta CB)	5.58	PCB #199 (Octa CB)	10.65	Dieldrin *	13.96
PCB #44 (Tetra CB)	5.65	PCB #153 & PCB #168 (Hexa CB)	6.61	PCB #203 (Octa CB)	5.49	*o,p'*-DDD	5.40
PCB #37 (Tri CB)	3.28	PCB #105 (Penta CB)	11.19	PCB #189 (Hepta CB)	10.58	Endrin *	15.85
PCB #41 (Tetra CB)	5.65	PCB #141 (Hexa CB)	9.20	PCB #208 (Nona CB)	6.75	*cis*-Nonachlor *	16.23
PCB #40 (Tetra CB)	4.44	PCB #137 (Hexa CB)	7.29	PCB #194 (Octa CB)	3.40	*p,p'*-DDD	6.80
PCB #74 (Tetra CB)	4.16	PCB #138 & PCB #129 (Hexa CB)	7.55	PCB #205 (Octa CB)	4.37	*o,p'*-DDT	7.59
PCB #70 (Tetra CB)	5.35	PCB #158 (Hepta CB)	8.63	PCB #206 (Nona CB)	7.81	*p,p'*-DDT	8.26
PCB #95 (Penta CB)	6.28	PCB #178 (Hexa CB)	12.04	PCB #209 (Deca CB)	4.97		
PCB #66 (Tetra CB)	3.19	PCB #126 (Penta CB)	7.60				
PCB #155 (Hexa CB)	3.33	PCB #187 (Hepta CB)	4.25				

For compounds marked with an asterisk (*), analytical repeatability was calculated at 10 ng/mL.

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