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Application Data Sheet

No.109

GC-MS
Gas Chromatograph Mass Spectrometer

Analysis of Polycyclic Aromatic Hydrocarbons (PAHs) in Foods by GC-MS/MS

In recent years, there has been a trend toward viewing high concentrations of polycyclic aromatic hydrocarbons (PAHs) in foods (particularly smoked products) as a problem. In 2009, the Codex Alimentarius Commission issued standards for reducing the PAH contamination level in smoked and directly dried foods. In addition, Europe, Canada, South Korea, China, and others established food standards for benzo[a]pyrene. This Application Data Sheet describes using a GC-MS/MS system to analyze the PAHs in "katsuobushi" (dried, smoked, and fermented skipjack tuna, which is very popular in Japanese cuisine).

Experiment

An extract solution from pretreated katsuobushi was injected into the GC-MS/MS system. The analysis conditions are shown in Table 1. MS/MS measurement program was created by Smart MRM in GCMSsolution automatically.

Table 1 Analysis Conditions

GC-MS:	GCMS-TQ8040
Column:	Rxi-PAH (60 m long, 0.25 mm I.D., df = 0.1 μm) (RESTEK, P/N: 49317)
Glass Insert:	Splitless insert with wool (Shimadzu, P/N: 221-48876-03)
[GC]	
Injection Volume:	2 μL
Injection Unit Temp.:	330 °C
Column Oven Temp.:	180 °C (2 min) → (5 °C /min) → 260 °C → (15 °C /min) → 350 °C (12 min)
Carrier Gas Control:	Constant linear velocity (40.0 cm/sec)
Injection Mode:	Splitless
Sampling Time:	2 min
Carrier Gas:	Helium

[MS]	
Interface Temp.:	330 °C
Ion Source Temp.:	230 °C
Measurement Mode:	MRM
Loop Time:	0.3 sec

MRM monitoring m/z:

Compound Name	Quantitative Transition Precursor>Product	CE(V)	Qualitative Transition Precursor>Product	CE(V)
Benzo[c]fluorene	216.10>215.10	22	216.10>189.10	30
Benzo[a]anthracene	228.10>226.10	28	228.10>202.10	26
Cyclopenta[c,d]pyrene	226.10>224.10	38	226.10>200.10	30
Chrysene	228.10>226.10	28	228.10>202.10	26
5-Methylchrysene	242.10>239.10	32	242.10>215.10	22
Benzo[b]fluoranthene	252.10>250.10	28	252.10>226.10	30
Benzo[k]fluoranthene	252.10>250.10	30	252.10>226.10	30
Benzo[j]fluoranthene	252.10>250.10	30	252.10>226.10	30
Benzo[a]pyrene	252.10>250.10	30	252.10>226.10	24
Dibenzo[a,h]anthracene	278.10>276.10	30	278.10>252.10	30
Indeno[1,2,3-cd]pyrene	276.10>274.10	34	276.10>250.10	30
Benzo[g,h,i]perylene	276.10>274.10	32	276.10>275.10	28
Dibenzo[a,l]pyrene	302.10>300.10	36	302.10>298.10	60
Dibenzo[a,e]pyrene	302.10>300.10	36	302.10>276.10	28
Dibenzo[a,i]pyrene	302.10>300.10	36	302.10>276.10	28
Dibenzo[a,h]pyrene	302.10>300.10	36	302.10>276.10	28
D12-Benzo[a]anthracene	240.20>236.20	28		
D12-Chrysene	240.20>236.20	28		
D12-Benzo[b]fluoranthene	264.20>260.20	32		
D12-Benzo[k]fluoranthene	264.20>260.20	32		
D12-Benzo[a]pyrene	264.20>260.20	32		
D14-Dibenzo[a,h]anthracene	292.20>288.20	34		
D12-Indeno[1,2,3-cd]pyrene	288.20>284.20	38		
D12-Benzo[g,h,i]perylene	288.20>284.20	38		
D14-Dibenzo[a,i]pyrene	316.20>312.20	40		

Analysis Results

A standard sample was measured to calculate the lower limit of detection (see Table 2). For comparison purposes, the lower limit of detection values for a GC/MS (SIM) system are also indicated to the left. MRM allows achieving the lower limit of detection lower than SIM.
Fig. 1 shows chromatograms obtained from analyzing the katsuobushi extract. These results demonstrate that for actual samples containing contaminants, MRM, which offers superior separation, can provide better peak detection.

Table 2 Detection Limits

Compound Name	Detection Limit (concentration: pg/µl)	
	GC/MS(SIM)	GC/MS/MS(MRM)
Benzo[c]fluorene	0.028	0.036
Benz[a]anthracene	0.137	0.134
Cyclopenta[cd]pyrene	0.109	0.105
Chrysene	0.209	0.068
5-Methylchrysene	0.323	0.098
Benzo[b]fluoranthene	0.143	0.058
Benzo[k]fluoranthene	0.158	0.080
Benzo[j]fluoranthene	0.172	0.075
Benzo[a]pyrene	0.159	0.029
Indeno[1,2,3-cd]pyrene	0.075	0.011
Dibenzo[a,h]anthracene	0.063	0.032
Benzo[ghi]perylene	0.086	0.050
Dibenzo[a,l]pyrene	0.271	0.035
Dibenzo[a,e]pyrene	0.017	0.017
Dibenzo[a,i]pyrene	0.178	0.086
Dibenzo[a,h]pyrene	0.076	0.035

Note: Detection limits (3.3 σ) were calculated based on the standard deviation from consecutive analyses (n = 3) of 0.5 pg/µL standard samples.

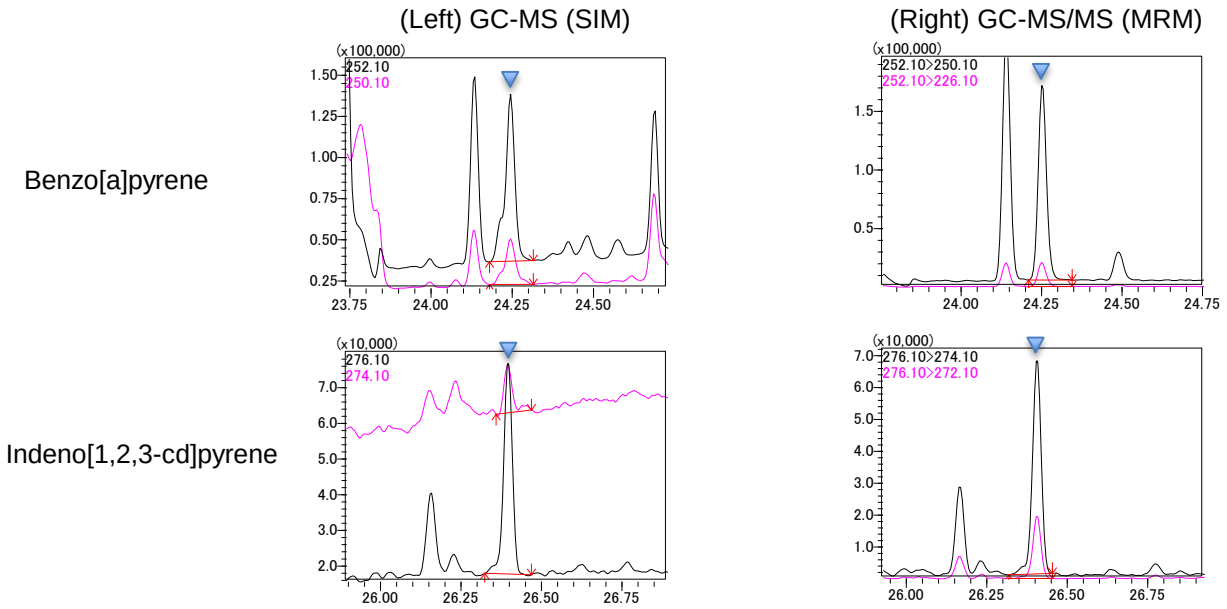

Fig. 1 SIM and MRM Chromatograms of PAHs in Katsuobushi Extract

Summary

This Application Data Sheet describes using a GC-MS/MS system to analyze polycyclic aromatics in food. The results showed that the GC-MS/MS system provided better sensitivity and peak separation than conventional GCMS methods.

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