Waters
Lab Highlights
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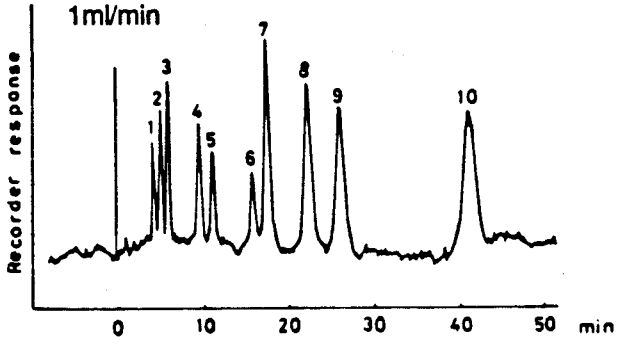
LIQUID CHROMATOGRAPHIC DETERMINATION OF POLYCYCLIC AROMATIC HYDROCARBONS IN FISH AND SHELLFISH WITH RADIAL COMPRESSION

A simple and accurate analytical method has been reported by Takatsuki and co-workers (1) for the determination of polycyclic aromatic hydrocarbons (PAH's) in fish and shellfish. This procedure is considered by the authors to be useful for routine analyses and for screening purposes and involves alkaline digestion, extraction with n-hexane, silica gel column chromatography, and LC determination with fluorometric detection.

Figure 1 shows the liquid chromatogram of PAH standards and Figure 2 shows the liquid chromatogram of PAH's in mussels using the developed analytical method. Table I shows the levels of PAH's detected in several lots of various shellfish from Miyagi Prefecture, Japan.

FIGURE 1

Column: Radial-PAK™ PAH Cartridge (5mm X 10cm)
Holder: Z-Module™
Detection: Fluorescence, 370nm (Ex.), 410nm (Em)
Mobile Phase: Acetonitrile/Water (80:20)
Flow Rate: 1ml/min



1. anthracene
2. fluoranthene
3. pyrene
4. benz[a]anthracene
5. chrysene
6. benzo[e]pyrene
7. benzo[b]fluoranthene
8. benzo[k]fluoranthene
9. benzo[a]pyrene
10. benzo[g,h,i]perylene

During development of the analytical method, it was found that benzo[a]pyrene, [the most intensively investigated PAH, known for its carcinogenicity and consistently used as a representative of PAH's] decomposed under alkaline conditions, exposure to light, oxygen and peroxides (in aged ethyl ether), and by oxygen when adsorbed onto silica gel. Therefore, the analytical methodology developed uses the following precautions: protection from light through all analytical steps; addition of Na_2S to alkaline digestion mixture as an antioxidant; complete removal of peroxides from ethyl ether just before use; quick column chromatography on silica gel; and prevention of air from contact with sorbent. When this method was applied to fish and shellfish samples, recoveries greater than 90% for all PAH's investigated were obtained, and no serious interferences were observed in the chromatograms.

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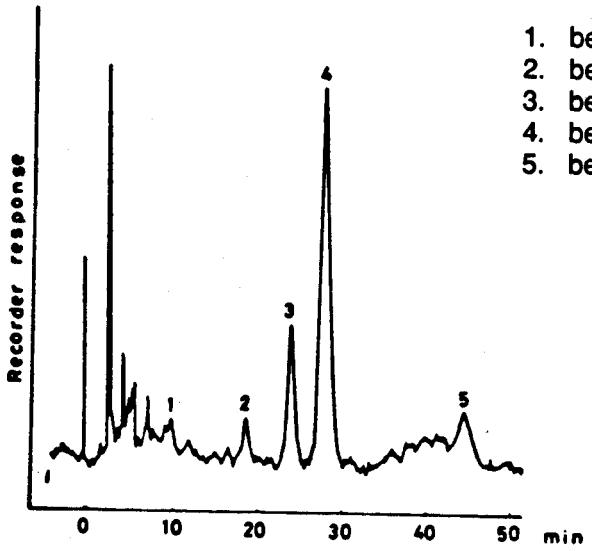
FIGURE 2



1. benz[a]anthracene
2. benzo[b]fluoranthene
3. benzo[k]fluoranthene
4. benzo[a]pyrene
5. benzo[g,h,i]perylene

Table I

POLYCYCLIC AROMATIC HYDROCARBONS IN SHELLFISH FROM MIYAGI PREFECTURE, JAPAN

	PAH,ppb				
Sample	B[a]A	B[b]F	B[k]F	B[a]P	B[ghi]Per
Mussel	-	0.83	0.32	0.40	0.83
	2.28	1.94	1.16	2.64	2.28
	0.95	1.51	0.64	0.86	1.44
Oyster	1.13	2.17	1.21	0.78	0.87
Corb-shell	1.11	1.37	0.38	0.21	0.95
Shortnecked clam	1.81	0.82	0.29	0.41	0.53
	1.18	1.44	0.54	1.05	1.13
	1.02	1.87	0.80	1.79	1.89

1. Takatsuki, K., S. Suzuki, N. Sata, and I. Ushizawa, J. Assoc. Off. Anal. Chem. ,68 (1985), 945-949.