Accelerated Solvent Extraction and Gas Chromatography/Mass Spectrometry for Determination of Polycyclic Aromatic Hydrocarbons in Smoked Food Samples

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Accelerated solvent extraction (ASE) is a new sample extraction method offering a number of advantages such as low pe -extraction cost, reduced solvent and time consumption, and simplified extraction protocols. In this study, the ASE method was applied to the extraction of polycyclic aromatic hydrocarbons (PAHs) from biological samples. For recovery studies, fish tissues and ground pork were used as sample matrices. Sample aliquots fortified with 16 PAHs were extracted by ASE, and the extracts were treated with sulfuric acid and Florisil, followed by gas chromatography−mass spectrometry analysis. The PAH recoveries by the ASE method were found to be comparable with or better than those by Soxhlet extraction. The extraction and quantitation method was then applied to the determination of PAHs in several smoked meat samples obtained from a local market. Up to 12 PAHs were found to be present at concentrations ranging from 3 to 52 ng/g wet sample.

Keywords: *Accelerated solvent extraction; polycyclic aromatic hydrocarbons; GC/MS; smoked food*

INTRODUCTION

Polycyclic aromatic hydrocarbons (PAHs) are a major class of environmentally hazardous organic compounds due to their known or suspected carcinogenicity. PAHs are ubiquitous in the environment, largely due to the extensive use of fossil fuels (petroleum and coal). In addition to a wide distribution of PAHs in air particulates and soil, PAHs are also introduced into waters and marine sediments by storm runoff, factory discharge, and petroleum spills. PAH contamination in foods and the effects on human health have been the subject of major concern (Lawrence and Weber, 1984). Several sources are responsible for the occurrence of PAHs in foodstuffs: processing of food including smoking and cooking, natural sources, and environmental contamination (Joe et al., 1980).

Determination of PAHs in biological samples such as foods has traditionally relied on prolonged extraction and cleanup procedures such as Soxhlet extraction and saponification extraction. These procedures are time-consuming and require large quantities of organic solvents that are usually toxic and are expensive to dispose. The continued investigation into alternative methods that are more efficient and generate little additional laboratory wastes has led to the introduction of supercritical fluid extraction (SFE) (Hawthorne, 1990) and, more recently, accelerated solvent extraction (ASE) (Richter et al., 1996). ASE enhances extraction efficiency by operating automatically at elevated pressure and temperature using small volumes of traditional organic solvents. Extraction of multiple sample aliquots can be achieved within 5−15 min/aliquot, and the typical solvent consumption is 15−30 mL/aliquot. ASE recoveries of PAHs from several types of environmental samples such as sewage sludge and marine particulate matter have been reported to be comparable with or better than conventional extraction methods (Richter et al., 1996; Heemken et al., 1997; Saim et al., 1998). This paper applies ASE to the extraction of PAHs from smoked food samples. Gas chromatography/mass spectrometry will be employed to quantify the extracted PAHs. A post-extraction cleanup method for removing high lipid contents present in the ASE extracts will also be described. PAH recoveries in the accelerated solvent extraction will be investigated followed by determination of PAHs in selected smoked food samples.

EXPERIMENTAL PROCEDURES

Materials and Reagents. PAH standard mixtures, 4,4-difluorobiphenyl, and *p*-terphenyl-d_{14} were obtained from ChemService (West Chester, PA). The mixture contains 16 PAHs, which are naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, benz[*a*]anthracene, chrysene, benzo[*k*]fluoranthene, benzo[*j*]-fluoranthene, benzo[*a*]pyrene, indeno[1,2,3-*cd*]pyrene, dibenz-[*a*,*h*]anthracene, and benzo[*g*,*h*,*i*]perylene, each at 100 μg/mL in methanol. The calibration standard solutions were prepared by diluting the mixture to concentrations ranging from 0.1 to 2.0 μg/mL with methylene chloride. 4,4-Difluorobiphenyl and *p*-terphenyl-d_{14} were used as internal standards, and they were added to the final extracts prior to GC/MS analysis. GC grade solvents (hexane, methylene chloride, acetone, and acetonitrile), concentrated sulfuric acid, sodium sulfate, and Ottawa Sand Standard were all purchased from Fisher Scientific (Fair Lawn, NJ). Florisil, 100−200 mesh size, and Supelclean LC-18 adsorbents were from Supelco (Bellefonte, PA). All fresh and smoked fish and pork samples were obtained from local food stores.

Apparatus. A Dionex (Sunnyvale, CA) accelerated solvent extractor (ASE 200) was used for all sample extractions. A Waring blender (New Hartfort, CT) with a stainless steel container was used to homogenize all food samples. A Hewlett-Packard model 5890 Series II Plus gas chromatograph equipped with an HP 5989B MS engine detector (Palo Alto, CA) was used for all quantitative determination of PAHs. The instrument was controlled by the HP G1034C ChemStation software.

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Table 1. Retention Times, Detection Limits, and Representative Ions for 16 PAHs Used in SIM GC/MS Quantification

PAH	retention time (min)	ions assigned for SIM (*m*/*z*)	detection limit (μg/mL)
naphthalene	9.43	128.2, 127.2, 64.1	0.002
acenaphthylene	12.53	152.2, 151.2, 76.1	0.005
acenaphthene	12.90	153.2, 154.2, 152.2	0.005
fluorene	13.91	166.2, 165.2, 163.2	0.005
phenanthrene	15.79	178.2, 176.2, 76.1	0.002
anthracene	15.88	178.2, 176.2, 76.1	0.010
fluoranthene	18.16	202.2, 200.2, 101.2	0.010
pyrene	18.59	202.2, 101.2, 100.2	0.010
benz[*a*]anthracene	21.42	228.3, 226.3, 113.2	0.025
chrysene	21.53	228.3, 226.3, 113.2	0.025
benzo[*k*]fluoranthene	24.55	252.3, 126.2, 125.2	0.100
benzo[*j*]fluoranthene	24.62	252.3, 126.2, 125.2	0.100
benzo[*a*]pyrene	25.51	252.3, 250.3, 126.2	0.100
indeno[1,2,3-*cd*]pyrene	28.89	276.3, 138.2, 137.2	0.100
dibenz[*a*,*h*]anthracene	29.02	278.3, 276.3, 138.2	0.100
benzo[*g*,*h*,*i*]perylene	29.62	276.1, 138.2, 137.2	0.100
internal standard[a]			
4,4-difluoro-1,1-biphenyl	11.68	190.2, 188.2, 89.2	
p-terphenyl-d_{14}	19.05	244.4, 243.3, 122.2	

[a] The internal standard, 4,4′-difluoro-1,1-biphenyl, was used for the eight PAHs that eluted before 20 min, and *p*-terphenyl-d_{14} was used for the other eight PAHs that eluted after 20 min.

The GC column used was an HP-5MS, cross-linked 5% phenyl methyl siloxane capillary column, 30 m × 0.25 mm i.d., with film thickness of 0.25 μm.

Sample Preparation. Tissue samples were cut into small pieces and mixed with dry ice at approximately 1:1 ratio (by weight). Fish and pork samples were then homogenized in a Waring stainless steel blender. For PAH recovery studies in fish, 1.0-g aliquots of homogenized catfish tissues were spiked with 100 μg/mL PAH standard mixture to yield concentration levels of 0.3, 0.6, and 1.0 μg/g wet sample. For recovery studies in pork, 1.0-g aliquots of ground pork were prepared and fortified with 10 μL of standard PAH solution to give a tissue concentration level of 1.0 μg/g wet sample. The fortified sample aliquots were then mixed with 1.0 g of Supelclean LC-18 and 1.0 g of anhydrous Na_2SO_4. The mixtures were ground gently to a semi-dry state and transferred into ASE extraction cells. Three aliquots were prepared for each concentration level. Smoked pork tasso, smoked sausage, and smoked salmon were homogenized as described above, and 5-g aliquots were mixed with 5 g of Na_2SO_4 and 5 g of C-18 before ASE extraction.

Accelerated Solvent Extraction. Extraction parameters were adapted from Dionex application note 313 (Sunnyvale, CA) with some modifications. A cellulose paper was placed at the bottom of each 11-mL Dionex extraction cell before the sample homogenates were loaded. The extra space in the cell was filled up with Ottawa Sand Standard before sealing off the top. ASE conditions were set up as follows: 5 min heating-up time, 5 min first static period followed by a second 5 min static time, 1500 psi cell pressure, 100 °C oven temperature, 60% flush volume, 1.5 min purge time, and 20 mL of extraction solvent. The ASE extracts were collected in 40-mL glass vials with Teflon septum caps.

Post-Extraction Cleanup Procedures. Concentrated or 9 M sulfuric acid was used to remove the high level lipids present in the ASE extracts. For hexane extracts of 1.0-g sample aliquots, 1.0 mL of 9 M H_2SO_4 was added to each extract, and the mixture was shaken vigorously for 2 min. When other solvents were used in ASE, the extracts were first evaporated to near dryness under a nitrogen stream and then reconstituted with 10 mL of hexane before H_2SO_4 cleanup. The two layers were separated, and the yellow-brown aqueous layer was discarded. The above procedure was repeated twice followed by rinsing the extracts with water. The extracts were then evaporated to 4 mL under a nitrogen stream before being passed through a Florisil column.

The Florisil column was prepared by filling a 1 cm i.d. chromatographic column with 6 g of Florisil. After rinsing the Florisil system with 10 mL of deionized water, the column was conditioned with 10 mL of methanol and methylene chloride at 3 mL/min. The column was dried, and the ASE extract was loaded slowly (2 mL/min) and allowed to dry for 1 min in the air. PAHs were eluted from the column with 10 mL of methylene chloride (2 mL/min). The collected eluent was evaporated down to 1 mL followed by the addition of internal standands prior to GC/MS analysis. All evaporation and cleanup procedures were carried out in the vented hood to minimize PAH and organic solvent contamination in the laboratory.

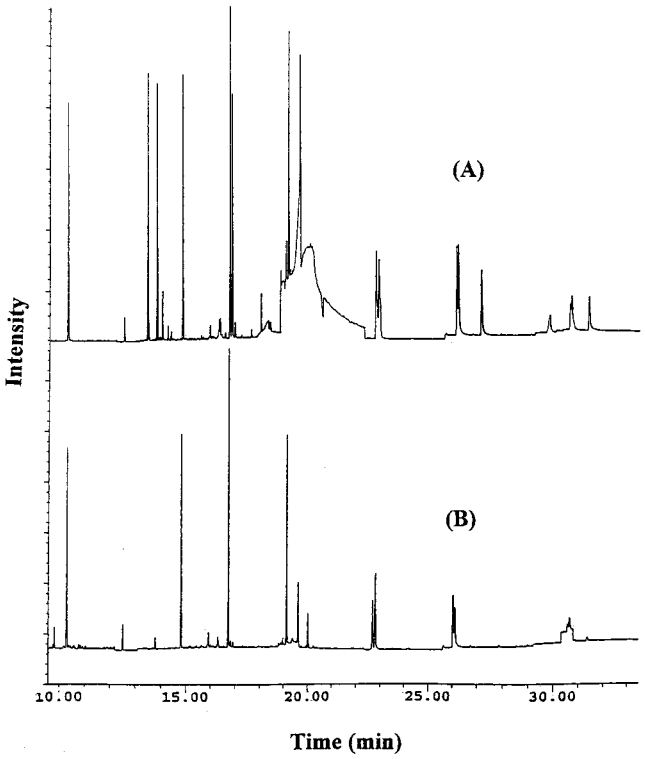

Figure 1. SIM chromatograms of the extract from PAH-spiked fish tissue (5 μg/g): (A) before and (B) after rinsing with concentrated H_2SO_4.

Quantitation. The GC/MS was operated under the following conditions. The temperature of the injection port and the detector was held at 290 °C. The oven temperature was set at 40 °C initially (1 min hold), increased to 250 °C at 12 °C/min, and increased to 310 °C at a rate of 5 °C/min (3 min hold). The temperatures of the ion source and the quadrupole mass analyzer were kept at 250 and 100 °C, respectively. Helium gas was used as the carrier gas at a constant flow rate of 0.8 mL/min. An automatic sample injector (HP 6890) was used to introduce 1.0 μL of each sample extract in an intermittent standard injection sequence. The retention time of each PAH component was determined by injecting individual PAH solutions under constant GC/MS instrumental conditions. The SIM mode (selected ion monitoring) was then used for quantitation in which three ions were selected for calculating the chromatographic peak area of each PAH (see Table 1). For standard calibration curves, the response factors of PAHs relative to the two internal standard compounds were determined at five PAH concentration levels (from 0.10 to 5.0 μg/mL). For PAH determination in fortified and smoked food samples, internal standards were added to the final extracts before GC/MS analysis.

RESULTS AND DISCUSSION

Table 1 lists the retention times, the detection limits, and three *m*/*z* values used in SIM for determination of the peak areas corresponding to individual PAHs. Because elution lasted for about 30 min, two internal

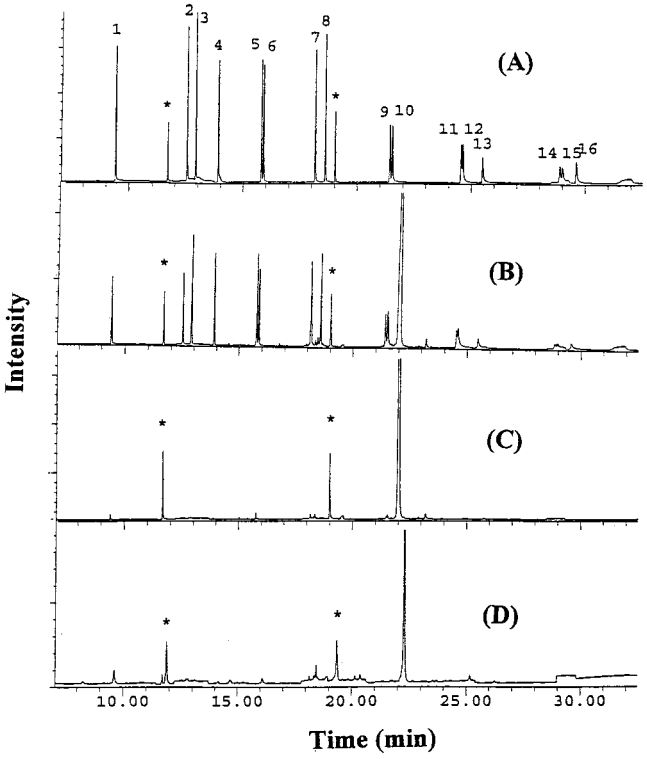

Figure 2. SIM chromatograms of the following: (A) Standard solution containing 1.0 μg/mL PAHs with corresponding peaks labeled as 1, naphthalene; 2, acenaphthylene; 3, acenaphthene; 4, fluorene; 5, phenanthrene; 6, anthracene; 7, fluoranthene; 8, pyrene; 9, benz[*a*]anthracene; 10, chrysene; 11, benzo[*k*]-fluoranthene; 12, benzo[*j*]fluoranthene; 13, benzo[*a*]pyrene; 14, indeno[1,2,3-*cd*]pyrene; 15, dibenz[*a*,*h*]anthracene; 16, benzo-[*g*,*h*,*i*]perylene; and with internal standards labeled with an asterisk (*). (B) Extract from fish tissue spiked at a PAH level of 1.0 μg/g sample. (C) Extract from fish tissue without spiking. (D) Extract from smoked salmon tissue.

standards were used to calculate the relative response factors of the PAHs. The internal standard 4,4′-difluoro-1,1-biphenyl was used for the eight PAHs that eluted before 20 min, and *p*-terphenyl-d_{14} was used for the other eight PAHs that eluted after 20 min. Also included in Table 1 are the GC/MS detection limits for the PAHs under SIM mode. The larger PAHs have increasingly higher detection limits (up to 0.1 μg/mL), which sets the lower end of PAH concentration range that can be employed in this study.

Because ASE employs organic solvents that are the same as those used in conventional methods (e.g., Soxhlet), it is essentially a nonselective extraction method. An effective cleanup procedure for removing co-extracted interfering substances is thus a crucial step before quantitative analysis of PAHs that are present only at trace levels in the crude extracts. As evidenced in Figure 1A, even under selected ion monitoring mode, a catfish sample extract treated only with Florisil column cleanup gives rise to a wide unresolved peak centered around 20 min. This peak is attributed to the presence of co-extracted lipids from fish sample. Methanolic saponification has been one of the most commonly used treatments to remove the fats (Chen et al., 1996) by means of hydrolysis of the fatty acid esters. However, it has been reported that saponification in the cleanup of the Soxhlet extracts of smoked chicken samples was not adequate (Chiu et al., 1997) without further partition procedure. Initial results obtained in our laboratory also indicated that removal of the fat contents in the ASE extracts was incomplete with saponification.

To develop an alternative cleanup method, the hexane extracts of fortified fish tissues by ASE were rinsed three times with sulfuric acid at two concentration levels: concentrated (18 M) and 9 M H_2SO_4. The sulfuric acid treatment has been used in cleaning up food sample

Table 2. ASE and Soxhlet Recoveries of 16 PAHs from Fish Tissues Fortified with PAHs at 0.3, 0.6, and 1.0 μg/g Tissue[a]

	Soxhlet recovery %	ASE recovery %		
	0.3 μg/g	0.3 μg/g	0.6 μg/g	1.0 μg/g
naphthalene	51 ± 6	46 ± 4	35 ± 3	58 ± 5
acenaphthylene	84 ± 7	93 ± 9	86 ± 9	88 ± 9
acenaphthene	68 ± 4	70 ± 3	65 ± 3	67 ± 6
fluorene	77 ± 6	84 ± 3	80 ± 4	81 ± 7
phenanthrene	91 ± 12	84 ± 16	73 ± 12	71 ± 11
anthracene	66 ± 5	61 ± 2	57 ± 3	61 ± 5
fluoranthene	71 ± 9	74 ± 7	70 ± 6	73 ± 9
pyrene	69 ± 7	66 ± 4	65 ± 2	68 ± 6
benz[*a*]anthracene	70 ± 5	61 ± 11	64 ± 12	68 ± 4
chrysene	44 ± 3	51 ± 6	51 ± 5	53 ± 4
benzo[*k*]fluoranthene	45 ± 10	46 ± 7	53 ± 4	49 ± 11
benzo[*j*]fluoranthene	71 ± 17	63 ± 9	76 ± 14	91 ± 11
benzo[*a*]pyrene	74 ± 14	70 ± 18	62 ± 5	57 ± 6
indeno[1,2,3-*cd*]pyrene	56 ± 6	66 ± 4	60 ± 8	56 ± 12
dibenz[*a*,*h*]anthracene	55 ± 8	53 ± 12	58 ± 10	48 ± 8
benzo[*g*,*h*,*i*]perylene	61 ± 11	56 ± 16	60 ± 9	67 ± 10

[a] Each value is the average of four measurements. Recoveries were determined from calibration curves of standard PAH solutions.

Table 3. Solvent Effect on the PAH Recoveries in Fish Tissues Fortified at 1.0 μg/g Wet Sample[a]

	recovery %		
PAH	hexane	CH_2Cl_2:ACN (9:1)	CH_2Cl_2:ACN (7:3)
naphthalene	58 ± 5	62 ± 6	66 ± 5
acenaphthylene	88 ± 9	76 ± 5	73 ± 8
acenaphthene	67 ± 6	69 ± 4	66 ± 5
fluorene	81 ± 7	77 ± 10	78 ± 9
phenanthrene	71 ± 11	93 ± 8	90 ± 10
anthracene	61 ± 5	81 ± 7	80 ± 7
fluoranthene	73 ± 9	101 ± 7	90 ± 6
pyrene	68 ± 6	92 ± 5	90 ± 7
benz[*a*]anthracene	68 ± 4	96 ± 11	91 ± 9
chrysene	53 ± 4	93 ± 9	88 ± 6
benzo[*k*]fluoranthene	54 ± 11	89 ± 5	88 ± 6
benzo[*j*]fluoranthene	91 ± 11	88 ± 4	86 ± 3
benzo[*a*]pyrene	57 ± 6	79 ± 8	83 ± 5
indeno[1,2,3-*cd*]pyrene	56 ± 12	81 ± 3	89 ± 6
dibenz[*a*,*h*]anthracene	48 ± 8	84 ± 8	87 ± 9
benzo[*g*,*h*,*i*]perylene	67 ± 10	85 ± 7	84 ± 5

[a] Each value is the average of four replicate extracts.

Table 4. PAH Recoveries from Ground Pork Fortified with Each PAH at 1.0 μg/g Wet Sample, Using 9:1 (vol) Methylene Chloride:Acetonitrile as Extraction Solvent[a]

PAH	recovery %	PAH	recovery %
naphthalene	54 ± 8	benz[*a*]anthracene	83 ± 4
acenaphthylene	81 ± 6	chrysene	84 ± 4
acenaphthene	62 ± 4	benzo[*k*]fluoranthene	79 ± 12
fluorene	67 ± 9	benzo[*j*]fluoranthene	85 ± 10
phenanthrene	102 ± 6	benzo[*a*]pyrene	77 ± 8
anthracene	76 ± 6	indeno[1,2,3-*cd*]pyrene	72 ± 11
fluoranthene	75 ± 7	dibenz[*a*,*h*]anthracene	62 ± 8
pyrene	98 ± 5	benzo[*g*,*h*,*i*]perylene	64 ± 4

[a] Each value is the average of four measurements.

Table 5. PAH Concentrations[a] in Commercial Smoked Meat Samples

PAH	smoked pork tasso 1	smoked pork tasso 2	smoked sausage 1	smoked sausage 2	smoked salmon
naphthalene	8.6 ± 0.8	20.6 ± 2.8	13.1 ± 0.6	12.0 ± 0.8	27.0 ± 2.0
acenaphthylene	ND[b]	ND	ND	ND	ND
acenaphthene	ND	12.4 ± 1.4	ND	4.8 ± 1.4	ND
fluorene	ND	4.4 ± 1.1	ND	5.0 ± 1.7	ND
phenanthrene	10.2 ± 3.4	24.8 ± 1.2	22.0 ± 2.0	ND	11.1 ± 1.0
anthracene	ND	28.0 ± 2.2	ND	15.6 ± 1.8	ND
fluoranthene	7.2 ± 2.4	39.6 ± 5.4	5.0 ± 1.3	42.2 ± 5.6	13.4 ± 6.2
pyrene	6.3 ± 3.9	33.8 ± 6.4	5.4 ± 1.6	52.0 ± 4.4	ND
benz[*a*]anthracene	11.7 ± 4.3	ND	9.4 ± 1.0	2.5 ± 1.0	ND
chrysene	9.9 ± 3.3	15.4 ± 4.8	12.3 ± 0.5	ND	ND
benzo[*k*]fluoranthene	ND	5.2 ± 1.2	ND	ND	ND
benzo[*j*]fluoranthene	ND	ND	ND	ND	ND
benzo[*a*]pyrene	ND	ND	ND	ND	ND
indeno[1,2,3-*cd*]pyrene	ND	8.4 ± 1.0	ND	ND	ND
dibenz[*a,h*]anthracene	ND	8.5 ± 1.2	ND	ND	ND
benzo[*g,h,i*]perylene	ND	ND	ND	3.0 ± 1.2	ND

[a] Concentration in ng/g wet sample. [b] ND, not detected.

extracts for pesticide determinations (Bernal et al., 1992; Kipcic and Vukusic, 1991). The method was reported to offer several advantages including simplicity and economy, but the method also suffered from partial or complete loss of some organochlorine pesticides during the acid treatment process (Bernal et al., 1992).

Figure 1B illustrates the effect of this treatment when compared with Figure 1A. The peaks corresponding to lipid impurities are virtually gone, but so are several PAHs. Notably, acenaphthylene, anthracene, and benzo[*a*]pyrene are completely absent in the chromatogram, whereas the peak intensities of acenaphthene, indeno[1,2,3-*cd*]pyrene, dibenz[*a,h*]anthracene, and benzo[*g,h,i*]perylene were reduced considerably after rinsing with concentrated sulfuric acid. This may be attributable to the strong oxidizing nature of 18 M H_2SO_4, resulting in the decomposition of some PAHs.

When the concentration of sulfuric acid was reduced to 9 M during the cleanup process, no appreciable loss of PAHs was observed. This is illustrated in Figure 2 and in the recovery data in Table 2 obtained for fortified catfish tissue for which 9 M H_2SO_4 was used as the cleanup reagent. The ASE recoveries for the PAHs at a spiked concentration of 0.3 μg/g of sample are compared to those achieved by Soxhlet extraction using the same sulfuric acid cleanup procedure (Table 2).

To find a solvent system that gives better PAH recoveries and a minimum amount of co-extracted lipids, three solvent systems [hexane, methylene chloride: acetonitrile (9:1 v/v), and methylene chloride:acetonitrile (7:3 v/v)] were tested for extraction of PAHs from fish tissues fortified to contain PAHs at 1.0 μg/g. Results are summarized in Table 3. Except acenaphthylene, fluorene, and benzo[*j*]fluoranthene for which the recoveries with hexane extraction are slightly bettter than with the methylene:acetonitrile mixed solvents, PAH recoveries achieved by the latter solvents are considerably higher than by hexane (Table 3). The mixed solvent system consisting of 90% methylene chloride and 10% acetonitrile was later used in the recovery studies for ground pork and in all food sample extractions. It was noted that with the methylene chloride/acetonitrile solvent systems, the separation of the aqueous acid layer from the organic layer was somewhat difficult during sulfuric acid cleanup process. The problem was solved by first drying the extracts and then reconstituting them with hexane before the sulfuric acid cleanup. Presented in Table 4 are PAH recoveries in fresh ground pork fortified at a concentration of 1.0 μg/g.

The results obtained from analysis of PAHs in smoked food samples are summarized in Table 5 using the extraction and cleanup protocols developed for fortified samples. Three PAHs were detected in a commercial smoked salmon sample: naphthalene, phenanthrene, and fluoranthene with concentrations from 11 to 27 ng/g wet sample. A blank sample of fresh salmon was also analyzed in which the only PAH detected was naphthalene present at 5 ng/g wet sample (see also Figure 2D). With smoked sausage and smoked pork tasso samples, the number of PAHs detected varied from 6 for one tasso sample to 12 for another. PAH concentration levels ranged from 3 to 52 ng/g wet sample (Table 5).

CONCLUSION

Accelerated solvent extraction was applied to the determination of PAHs in selected smoked food samples. Significant reductions in extraction time and solvent consumption were achieved when compared with traditional extraction methods. With ASE, the amount of solvent consumption was reduced to 20−30 mL/aliquot, and the extraction time was between 10 and 20 min. A modified cleanup procedure using sulfuric acid was also developed. It was found that the use of concentrated sulfuric acid resulted in the complete loss of three PAHs and partial loss of four other PAHs. However, use of 9 M sulfuric acid as a cleanup reagent yielded satisfactory results.

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