J. Braz. Chem. Soc., Vol. 22, No. 7, S1-S3, 2011.
Printed in Brazil - ©2011 Sociedade Brasileira de Química
0103 - 5053 $6.00+0.00

Supplementary Information

HPLC-FLD Simultaneous Determination of 13 Polycyclic Aromatic Hydrocarbons: Validation of an Analytical Procedure for Soybean Oils

Mônica C. R. Camargo,*[,a] Paula R. Antoniolli[b] and Eduardo Vicente[a]

[a]Centro de Ciência e Qualidade de Alimentos, Instituto de Tecnologia de Alimentos, Av. Brasil, 2880, 13070-178 Campinas-SP, Brazil

[b]Instituto de Química, Universidade Estadual de Campinas, 13083-970 Campinas-SP, Brazil

Table S1. Comparison of the evaluated solid phase extraction (SPE) cartridges

Sorbent	Producer	Sorbent mass / mg, column volume / mL	Porous / Å	Endcapped	Carbon loading / %
BondElut	Varian	500, 3	60	yes	17.0
AccuBond^{II} ODS C18	Agilent	500, 3	60	yes	19.0
AccuBond^{II} ODS C18	Agilent	1000, 6	60	yes	18.0
Strata C18-E	Phenomenex	500, 3	64	yes	17.1
Chromabond ec C18	Macherey-Nagel	500, 3	60	yes	14.0

Table S2. Parameters tested during the optimization of the extraction conditions

Parameters	Variable	Best condition
Conditioning solvent	methanol, dimethylformamide, water	dimethylformamide
Rinse solvent	methanol, methanol-water, water, dimethylformamide-water	dimethylformamide-water
Elution solvent	hexane, cyclohexane	hexane
Flow rate	6, 4 and 2 mL min^{-1}	2 mL min^{-1}
Cartridge drying step	10, 15 and 20 min	20 min
Eluent volume	4, 8 and 10 mL	10 mL

*e-mail: monicarojo@uol.com.br

Table S3. Recoveries (%) of the studied PAHs (mean value ± standard deviation) from soybean oils

PAH	Recovery / % (mean ± RSD, n = 3)		
	Spiked level: 0.5 μg kg^{-1}	Spiked level: 1.0 μg kg^{-1}	Spiked level: 5.0 μg kg^{-1}
B[*a*]A	91 ± 3.1	94 ± 5.4	93 ± 3.8
Chy	82 ± 6.3	88 ± 11.2	85 ± 2.8
5MeChy	115 ± 8.4	109 ± 1.5	107 ± 2.1
B[*j*]F	81 ± 2.5	85 ± 2.7	88 ± 2.9
B[*b*]F	85 ± 4.7	82 ± 1.2	84 ± 3.7
B[*k*]F	81 ± 3.6	84 ± 1.6	81 ± 2.2
B[*a*]P	102 ± 3.8	102 ± 2.5	98 ± 6.5
D[*al*]P	71 ± 11.9	88 ± 4.7	87 ± 3.9
D[*ah*]A	106 ± 1.9	97 ± 3.2	95 ± 4.1
Indeno	73 ± 8.4	77 ± 5.9	71 ± 3.3
D[*ae*]P	94 ± 3.1	91 ± 6.7	98 ± 1.0
D[*ai*]P	61 ± 4.7	66 ± 1.2	63 ± 4.2
D[*ah*]P	92 ± 2.0	96 ± 6.3	94 ± 1.1

B[*a*]A: benzo[*a*]anthracene; Chy: chrysene; 5MeChy: 5-methylchrysene; B[*j*]F: benzo[*j*]fluoranthene; B[*b*]F: benzo[*b*]fluoranthene; B[*k*]F: benzo[*k*]fluoranthene; B[*a*]P: benzo[*a*]pyrene; D[*al*]P: dibenzo[*a,l*]pyrene; D[*ah*]A: dibenzo[*a,h*]anthracene; indeno: indeno[1,2,3-*cd*]pyrene; D[*ae*]P: dibenzo[*a,e*]pyrene; D[*ai*]P: dibenzo[*a,i*]pyrene; D[*ah*]P: dibenzo[*a,h*]pyrene; RSD: relative standard deviation.

Table S4. Intra- and inter-day precision data for the extraction of PAHs from soybean oils

PAH	Intra-day precision (n = 3, mean)						Inter-day precision (n = 9, mean)	
	Day 1		Day 2		Day 3		Days 1, 2 and 3	
	Content / μg kg^{-1}	RSD / %	Content / μg kg^{-1}	RSD / %	Content / μg kg^{-1}	RSD / %	Content / μg kg^{-1}	RSD / %
B[*a*]A	1.23	2.15	1.17	1.30	1.22	2.87	1.21	2.66
Chy	1.11	6.76	1.12	6.82	1.06	4.32	1.10	2.92
5MeChy	1.39	4.31	1.34	4.16	1.42	1.40	1.38	2.93
B[*j*]F	1.12	5.45	1.23	1.62	1.10	2.41	1.15	6.09
B[*b*]F	1.02	5.39	0.96	3.13	1.04	2.54	1.01	4.12
B[*k*]F	1.04	1.47	1.11	2.90	1.01	1.98	1.05	4.88
B[*a*]P	1.22	1.42	1.22	2.06	1.23	1.24	1.22	0.47
D[*al*]P	1.08	8.83	1.06	4.99	1.06	3.81	1.07	1.08
D[*ah*]A	1.18	2.13	1.15	3.98	1.13	1.35	1.15	2.19
Indeno	0.95	3.79	1.02	2.99	0.94	3.73	0.97	4.49
D[*ae*]P	1.08	5.78	1.15	5.24	1.09	4.61	1.11	3.41
D[*ai*]P	0.88	3.68	0.90	5.19	0.98	7.52	0.92	5.75
D[*ah*]P	1.19	3.97	1.22	2.06	1.16	4.42	1.19	2.52

B[*a*]A: benzo[*a*]anthracene; Chy: chrysene; 5MeChy: 5-methylchrysene; B[*j*]F: benzo[*j*]fluoranthene; B[*b*]F: benzo[*b*]fluoranthene; B[*k*]F: benzo[*k*]fluoranthene; B[*a*]P: benzo[*a*]pyrene; D[*al*]P: dibenzo[*a,l*]pyrene; D[*ah*]A: dibenzo[*a,h*]anthracene; indeno: indeno[1,2,3-*cd*]pyrene; D[*ae*]P: dibenzo[*a,e*]pyrene; D[*ai*]P: dibenzo[*a,i*]pyrene; D[*ah*]P: dibenzo[*a,h*]pyrene; RSD: relative standard deviation.

Table S5. Statistical parameters of the calibration equations of individual PAHs

PAH	Concentration range / μg mL^{-1}	Slope (a) × 10^{8a}	Intercept (b) × 10^{4a}	R^{2a}
B[*a*]A	0.0005-0.05	2.83	-1.06	0.9994
Chy	0.0005-0.05	2.47	-5.21	0.9994
5MeChy	0.0005-0.05	3.66	-2.54	0.9998
B[*j*]F	0.001-0.25	0.06	-0.04	0.9999
B[*b*]F	0.0005-0.05	1.18	-3.41	0.9993
B[*k*]F	0.0005-0.05	5.85	-4.80	0.9990
B[*a*]P	0.0005-0.05	5.24	8.28	0.9998
D[*al*]P	0.0005-0.05	1.59	-1.56	0.9993
D[*ah*]A	0.0005-0.05	1.68	-8.29	0.9984
Indeno	0.001-0.25	0.28	-3.28	0.9997
D[*ae*]P	0.0005-0.05	3.60	-9.43	0.9999
D[*ai*]P	0.0005-0.05	1.23	-0.52	0.9967
D[*ah*]P	0.0005-0.05	1.20	-1.31	0.9993

B[*a*]A: benzo[*a*]anthracene; Chy: chrysene; 5MeChy: 5-methylchrysene; B[*j*]F: benzo[*j*]fluoranthene; B[*b*]F: benzo[*b*]fluoranthene; B[*k*]F: benzo[*k*]fluoranthene; B[*a*]P: benzo[*a*]pyrene; D[*al*]P: dibenzo[*a,l*]pyrene; D[*ah*]A: dibenzo[*a,h*]anthracene; Indeno: indeno[1,2,3-*cd*]pyrene; D[*ae*]P: dibenzo[*a,e*]pyrene; D[*ai*]P: dibenzo[*a,i*]pyrene; D[*ah*]P: dibenzo[*a,h*]pyrene; [a]y = ax + b: regression equation for each curve, where y = peak area, x = analyte concentration (μg mL^{-1}); a = slope; b = intercept and R^2 = coefficient of determination.

Table S6. Limits of detection (LOD) and quantification (LOQ) of the studied PAHs

PAH	LOD	LOQ
B[*a*]A	0.03	0.05
Chy	0.02	0.03
5MeChy	0.16	0.27
B[*j*]F	0.39	0.65
B[*b*]F	0.20	0.35
B[*k*]F	0.16	0.25
B[*a*]P	0.16	0.25
D[*al*]P	0.12	0.20
D[*ah*]A	0.15	0.24
Indeno	0.54	0.90
D[*ae*]P	0.07	0.12
D[*ai*]P	0.10	0.25
D[*ah*]P	0.76	0.96

B[*a*]A: benzo[*a*]anthracene; Chy: chrysene; 5MeChy: 5-methylchrysene; B[*j*]F: benzo[*j*]fluoranthene; B[*b*]F: benzo[*b*]fluoranthene; B[*k*]F: benzo[*k*]fluoranthene; B[*a*]P: benzo[*a*]pyrene; D[*al*]P: dibenzo[*a,l*]pyrene; D[*ah*]A: dibenzo[*a,h*]anthracene; indeno: indeno[1,2,3-*cd*]pyrene; D[*ae*]P: dibenzo[*a,e*]pyrene; D[*ai*]P: dibenzo[*a,i*]pyrene; D[*ah*]P: dibenzo[*a,h*]pyrene; LOD and LOQ in μg kg^{-1}.