Evaluation of Liquid−Liquid Extraction and Liquid−Solid Extraction with a New Sorbent for the Determination of Polycyclic Aromatic Hydrocarbons in Raw and Finished Drinking Waters

Manuel J. Fernandez, César Garcia, Rafael J. Garcia-Villanova,* and Juan A. Gomez

Departamento de Química Analítica, Nutrición y Bromatología, Facultad de Farmacia, Universidad de Salamanca, Avenida Campo Charro s/n, E-37007 Salamanca, Spain

A comparative study has been made of the application of the liquid−solid extraction (LSE) technique using a new sorbent solid, tC18, and liquid−liquid extraction (EPA Method 610) of 11 polycyclic aromatic hydrocarbons (PAHs) considered by the EPA as priority pollutants. The experimental conditions of both extraction techniques are evaluated and applied to the determination of 11 PAHs using a simple HPL chromatograph with fluorometric detection. The recovery yields obtained were greater in LLE. However, for most of the PAHs the yields were found to be greater than 80% with LSE. The detection limits do not differ significantly between either technique and vary between 0.007 μg·L^{-1} (benzo[*k*]fluoranthene) and 1.3 μg·L^{-1} (naphthalene). The absolute standard deviations and variation coefficients of both techniques are slightly higher with LSE and vary between 0.007 and 0.37 μg·L^{-1} and between 8 and 19%, respectively. The method was applied to analyze raw and finished drinking waters from four towns supplied with different water sources and qualities.

Keywords: *Liquid−solid extraction; PAH enrichment; PAHs in water; tC18 cartridges*

INTRODUCTION

The presence of polycyclic aromatic hydrocarbons (PAHs) in the environment arises from natural sources and, mainly, human activity. PAHs constitute a group of compounds characterized by their ubiquity (Cerniglia and Heitkamp, 1989; National Research Council, 1985), and interest in their study derives from the mutagenicity and carcinogenicity of most such compounds (Fazio and Howard, 1983; Miyashita et al., 1987).

About two-thirds of the PAHs found in raw water appears to be bound to particulate matter which is removed by flocculation, sedimentation, and filtration processes. The remaining third may be significantly removed by oxidation. Contact with coal-tar-based pipe linings during distribution is known to lead to elevated concentrations of some PAHs, particularly fluoranthene. However, estimates of exposure to PAHs indicate that drinking water is a very minor source, with a typical contribution of less than 1% (Fawell and Hunt, 1988).

Studies by the Water Research Center (Marlow, U.K.) have identified some 20 PAHs in drinking water in the United Kindom (Fawell and Hunt, 1988).

Determination of PAHs is a difficult analytical problem owing to the large number of compounds and because the levels they generally reach in raw and treated water are very low; that is, at nanograms per liter scale. Accordingly, the analytical procedure consists of a phase involving the isolation and preconcentration of PAHs in water samples before their analysis by chromatography.

Different techniques have been used to isolate PAHs in water samples, the most frequently employed preconcentration techniques being liquid−liquid extraction (LLE) (Nuñez et al., 1990) and liquid−solid extraction (LSE) (López et al., 1992). A recent technique employs

micellar chromatography to concentrate them (Rodriguez et al., 1994; Kaneta et al., 1995).

Concerning the quantitation of PAHs, the most suitable techniques are gas chromatography coupled with mass spectrometry (Langfeld et al., 1993) and high-performance liquid chromatography (HPLC) (Bo-Xing and Yu-Zhi, 1988) with fluorescence detection.

Finally, two official methods were established several years ago employing HPLC/UV-fluorescence, one based on LLE and another on LSE, corresponding, respectively, to the EPA Methods 610 and 550.1.

The present work reports on a comparative study of a LLE method and a LSE one, with a new sorbent, employing a very simple HPL chromatograph to the determination of 11 PAHs considered by the EPA as priority pollutants in surface raw and finished drinking waters.

The aim of this paper was to assess the validity of this new sorbent (tC18), slightly different from the C18 of the EPA Method 550.1, which has a trifunctional bonding chemistry, and a smaller particle size−however with similar pore size. This sorbent, according to manufacturer specifications, permits submission to prolonged exposures of acidic solutions without the risk of releasing the C18 functional group; this might be particularly interesting when there is a need to process large volumes of water for measuring parts per trillion levels of contaminants and/or when the extracts are to be kept for a certain time before eluting them.

As indicated above, many studies have been performed for the C18 cartridges; however none has been found in the literature on tC18.

The method proposed for LSE is simple and rapid and requires only conventional instruments; it was applied to the determination of the following 11 PAHs in samples of surface raw and finished drinking waters: naphthalene, acenaphthene, anthracene, fluoranthene, pyrene, benzo[*a*]anthracene, benzo[*b*]fluoranthene, benzo[*k*]fluoranthene, benzo[*a*]pyrene, indeno[1,2,3-*cd*]pyrene and benzo[*g,h,i*]perylene.

* Author to whom correspondence should be addressed (fax 34 + 23-294515; e-mail nutr@gugu.usal.es).
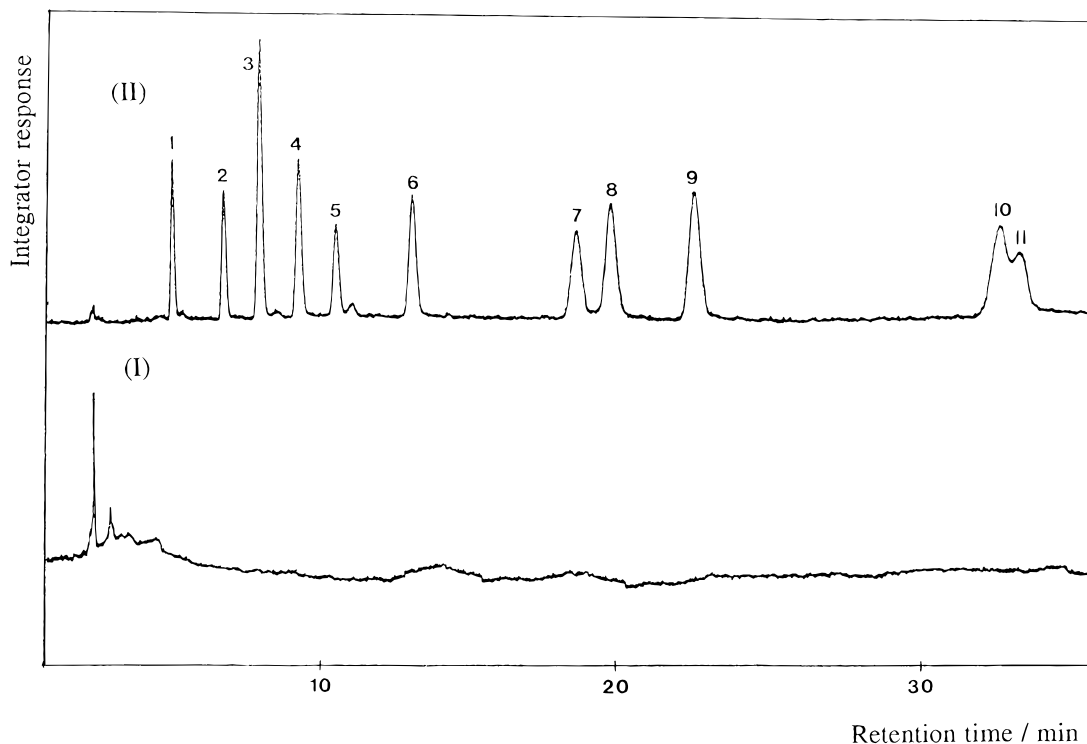
Figure 1. (I) Liquid chromatograph of blank in UHQ water with LSE. (II) Liquid chromatograph of a working solution of the 11 PAHs: (1) 4.9 $\mu g \cdot L^{-1}$ naphthalene; (2) 4.5 $\mu g \cdot L^{-1}$ acenaphthene; (3) 0.15 $\mu g \cdot L^{-1}$ anthracene; (4) 0.20 $\mu g \cdot L^{-1}$ fluoranthene; (5) 0.50 $\mu g \cdot L^{-1}$ pyrene; (6) 0.12 $\mu g \cdot L^{-1}$ benzo[*a*]anthracene; (7) 0.09 $\mu g \cdot L^{-1}$ benzo[*b*]fluoranthene; (8) 0.07 $\mu g \cdot L^{-1}$ benzo[*k*]fluoranthene; (9) 0.15 $\mu g \cdot L^{-1}$ benzo[*a*]pyrene; (10) 0.18 $\mu g \cdot L^{-1}$ indeno[1,2,3-*cd*]pyrene; (11) 0.40 $\mu g \cdot L^{-1}$ benzo[*g,h,i*]perylene.

EXPERIMENTAL PROCEDURES

Apparatus. The liquid chromatograph used was a Perkin-Elmer Model Tridet fitted with a fluorescence detector and connected to a Varian 4400 integrator for peak-area measurement. A 1 mL manual glass syringe was used to inject through a loop a 50 μL sample into a chromatographic column (Superspher 100 C18 column; 25 cm × 4.6 mm i.d.; 4 μm particles, manufactured by Teknokroma). Solid-phase extraction was performed with Sep-pak tC18 cartridges provided with trifunctional bonding chemistry (1000 mg of C18-bonded porous silica) obtained from Waters and a Gilson Peristaltic Pump Minipuls 3 was employed to assist passing the water samples. Finally, a Zymark Turbovap concentrator was used for concentrating the sample extracts.

Reagents. High-purity methylene chloride (Carlo Erba), methanol (Aldrich), acetonitrile (Merck), and UHQ (ultra high quality) reagent water from Elgastat were used.

Anhydrous sodium sulfate, sodium thiosulfate, and hydrochloric acid were of reagent grade quality and supplied by Carlo Erba.

Standard solutions (10 ng·μL^{-1} in toluene) of fluoranthene, benzo[*b*]fluoranthene, benzo[*k*]fluoranthene, benzo[*a*]pyrene, indeno[1,2,3-*cd*]pyrene, and benzo[*g,h,i*]perylene were supplied by Promochem GmbH D 4230 Wesel. Solid standards— naphthalene, acenaphthene, anthracene, pyrene, and benzo[*a*]anthracene (purity > 98%)—were supplied by Aldrich.

The working solutions used for the qualitative and quantitative analyses were prepared by appropriate dilution of the standards with acetonitrile.

Procedure. Water samples were collected in 2500 mL amber glass bottles, previously washed with Mucasol (Merz + Co) and rinsed with methylene chloride. Raw water samples were filtered through glass wool, and to the treated drinking water samples was added 80 mg·L^{-1} $Na_2S_2O_3$ as dechlorinating agent. To inhibit biological activity, all the samples were acidified to pH 2 with 6 N HCl and extracted as soon as possible to avoid adsorption of PAHs in the sampling bottles (López et al., 1992).

Liquid—Liquid Extraction. A sample of 1000 mL was extracted successively with 3 volumes of 60 mL of methylene chloride. The extract, 180 mL, was passed through anhydrous Na_2SO_4 and concentrated to dryness with the help of a

Table 1. Chromatographic Conditions

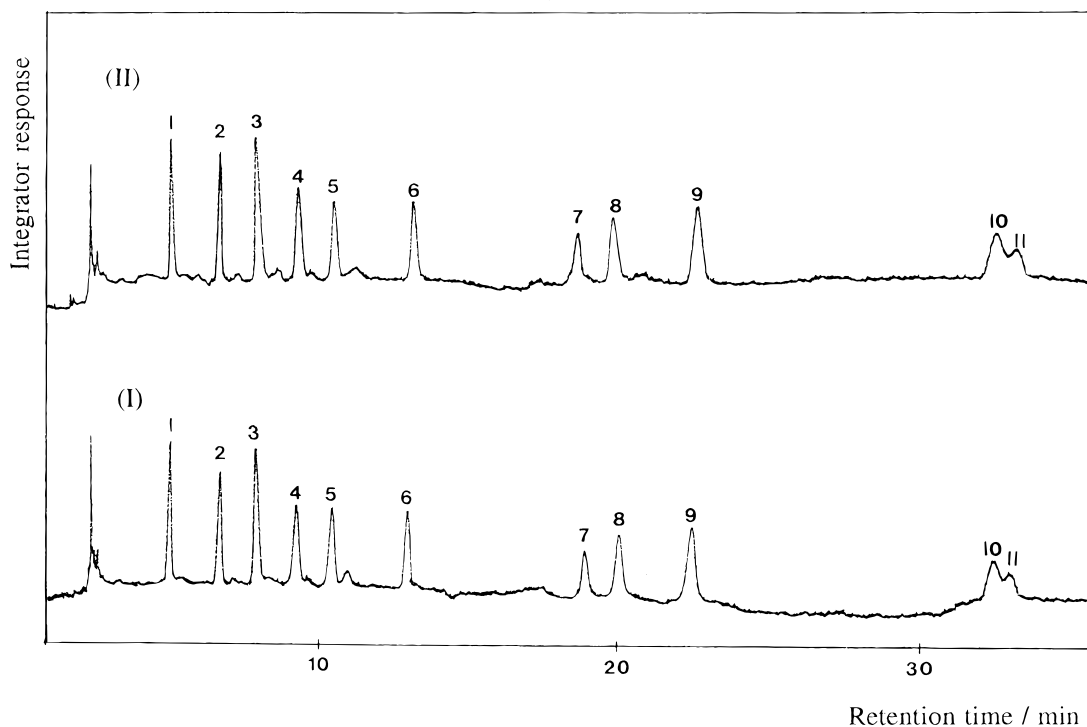|	
mobile phase	$CH_3CN:H_2O$ (80/20)
flow rate	1.2 mL·min^{-1}
regimen	isocratic
column	Superspher 100 (Teknokroma) C18, 25 cm × 4.6 mm i.d., 4 μm particles
detector	fluorescence (Perkin-Elmer, Model Tridet): excitation wavelength 254 nm (fixed) emission wavelength above 280 nm
temperature	20 °C
sample	50 μL

Turbovap concentrator. The dry residue was redissolved in 3 mL of acetonitrile and concentrated again to 0.5 mL.

Liquid—Solid Extraction. The Sep-pak tC18 cartridges were preactivated with methylene chloride, methanol, and reagent water at pH 2 (20 mL each). The sample, 1000 mL, was passed through at a flow rate of 8–10 mL·min^{-1}. Analytes were then eluted with 15 mL of methylene chloride; the extract was passed through anhydrous Na_2SO_4 and concentrated to dryness with a Turbovap concentrator. The dry residue was redissolved in 3 mL of acetonitrile and concentrated again to 0.5 mL.

Chromatographic Determination. Aliquots (50 μL) of extract and standard of suitable concentration were injected into the liquid chromatograph using the chromatographic conditions indicated in Table 1. Analyte concentrations were determined by used of an external standard. For control of results, periodic calibration graphs were obtained of the compounds identified in the samples in the working concentration zone.

RESULTS AND DISCUSSION

Figure 1 shows the chromatogram, obtained under the described conditions, of a working solution of the 11 PAHs: 4.9 $\mu g \cdot L^{-1}$ naphthalene; 4.5 $\mu g \cdot L^{-1}$ acenaphthene; 0.15 $\mu g \cdot L^{-1}$ anthracene; 0.20 $\mu g \cdot L^{-1}$ fluoranthene; 0.50 $\mu g \cdot L^{-1}$ pyrene; 0.12 $\mu g \cdot L^{-1}$ benzo[*a*]anthracene; 0.09 $\mu g \cdot L^{-1}$ benzo[*b*]fluoranthene; 0.07 $\mu g \cdot L^{-1}$ benzo[*k*]fluoranthene; 0.15 $\mu g \cdot L^{-1}$ benzo[*a*]pyrene; 0.18 $\mu g \cdot L^{-1}$ indeno[1,2,3-*cd*]pyrene; 0.40 $\mu g \cdot L^{-1}$ benzo[*g,h,i*]perylene.

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Figure 2. Comparison of the chromatograms obtained of 11 PAHs after application, for the same concentration level, of (I) LSE and (II) LLE.

Table 2. Recovery (%) of PAHs in Reagent Water

compound	mean level (μg·L^{-1})	LLE[a]	LSE[a]
naphthalene	10.4	75 ± 7	72 ± 8
acenaphthene	9.60	84 ± 6	80 ± 9
anthracene	0.26	90 ± 8	87 ± 10
fluoranthene	0.25	88 ± 10	90 ± 11
pyrene	1.40	97 ± 9	91 ± 8
benzo[*a*]anthracene	0.20	103 ± 11	97 ± 15
benzo[*b*]fluoranthene	0.10	91 ± 14	90 ± 11
benzo[*k*]fluoranthene	0.06	89 ± 12	92 ± 13
benzo[*a*]pyrene	0.10	93 ± 10	82 ± 12
indeno[1,2,3-*cd*]pyrene	0.25	80 ± 9	84 ± 11
benzo[*g,h,i*]perylene	0.50	83 ± 11	80 ± 10

[a] $n = 3$. Mean recovery ± SD (%).

It may be seen that this can be resolved and eluted within a reasonable time. A blank was also performed with reagent water, for which no response to the fluorescence detector can be observed during the time of elution of the PAHs (Figure 1).

Extraction Yields. The efficiency of liquid−liquid extraction (LLE) was determined by adding to 1000 mL of reagent water acidified with HCl to pH 2 the 11 PAHs at three different concentrations. Table 2 shows the results obtained for the intermediate concentration level. The upper and lower levels are, respectively, double and half the intermediate level. The recovery yields—the mean of three determinations—are above 75%, and the standard deviations range from 6 and 14%. These values are acceptable for obtaining reproducible results in quantitative analysis of the 11 PAHs.

The efficiency of liquid−solid extraction (LSE) was determined by passing 1000 mL of reagent water, to pH 2, containing the 11 PAHs at the same concentration levels as those used for LLE through previously activated Sep-pak tC18 cartridges. Table 2 shows the results obtained for the intermediate concentration level.

The mean recovery yields for LSE are slightly lower than those observed for LLE, although the standard

Table 3. Detection Limits of PAHs Analyzed

compound	detection limits (μg·L^{-1})	
	LLE	LSE
naphthalene	1.1	1.3
acenaphthene	0.93	1.2
anthracene	0.019	0.015
fluoranthene	0.033	0.037
pyrene	0.14	0.18
benzo[*a*]anthracene	0.022	0.019
benzo[*b*]fluoranthene	0.013	0.019
benzo[*k*]fluoranthene	0.007	0.011
benzo[*a*]pyrene	0.019	0.026
indeno[1,2,3-*cd*]pyrene	0.048	0.044
benzo[*g,h,i*]perylene	0.16	0.14

deviations (between 8 and 15%) are similar to those obtained with the LLE method. The mean yields of LSE are above 80%, except in the case of naphthalene, for which the yield is of the order of 72%. Accordingly, these yields are acceptable for obtaining reproducible results in their application to quantitative analysis.

Figure 2 compares the chromatograms of the 11 PAHs after application of LLE and LSE for the same concentration level.

Detection Limits. In order to calculate the detection limits (DL) of the 11 PAHs studied, the LLE and the LSE methods were applied to reagent solutions whose concentration in each of the analytes ranged from 2 to 5 times the estimated DL. Seven determinations were performed, and the DLs were calculated according to the expression (Glaser et al., 1981)

$$DL = 3.707(Sc)$$

where 3.707 is Student's *t* for six degrees of freedom and 99% probability and Sc is the standard deviation of the seven determinations.

The values obtained are shown in Table 3. For the same analyte, no significant differences are seen in the DLs calculated with the LLE method and the LSE method. Concerning individual values, the DLs range

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Table 4. Recovery of PAHs from Fortified Surface Water by LLE

compound	added (μg·L^{-1})	present (μg·L^{-1})	found (μg·L^{-1})	rec[a] (%)	mean rec[b] (%)
naphthalene	10.4		7.5	72	71 ± 8
acenaphthene	9.6		7.1	74	75 ± 9
anthracene	0.13	0.026	0.13	82	84 ± 13
fluoranthene	0.25		0.21	85	89 ± 12
pyrene	0.70		0.58	83	85 ± 12
benzo[a]anthracene	0.20		0.18	92	95 ± 16
benzo[b]fluoranthene	0.05	0.029	0.068	86	86 ± 14
benzo[k]fluoranthene	0.06	0.022	0.072	88	94 ± 18
benzo[a]pyrene	0.10		0.086	86	84 ± 11
indeno[1,2,3-cd]pyrene	0.25		0.21	84	81 ± 12
benzo[g,h,i]perylene	0.50		0.39	78	79 ± 13

[a] Recovery of intermediate concentration level. [b] Mean recovery (%) ± SD of the three concentration levels of added PAHs.

Table 5. Recovery of PAHs from Fortified Surface Water by LSE

compound	added (μg·L^{-1})	present (μg·L^{-1})	found (μg·L^{-1})	rec[a] (%)	mean rec[b] (%)
naphthalene	10.4		7.1	68	69 ± 9
acenaphthene	9.6		6.6	69	72 ± 10
anthracene	0.13	0.031	0.14	89	87 ± 12
fluoranthene	0.25		0.22	88	85 ± 13
pyrene	0.70		0.56	80	83 ± 11
benzo[a]anthracene	0.20		0.17	85	94 ± 21
benzo[b]fluoranthene	0.05	0.027	0.062	81	88 ± 16
benzo[k]fluoranthene	0.06	0.014	0.063	85	91 ± 15
benzo[a]pyrene	0.10		0.084	84	83 ± 10
indeno[1,2,3-cd]pyrene	0.25		0.19	76	79 ± 12
benzo[g,h,i]perylene	0.50		0.37	74	76 ± 11

[a] Recovery of intermediate concentration level. [b] Mean recovery (%) ± SD of the three concentration levels of added PAHs.

Table 6. Concentrations (ng·L^{-1}) of PAHs in Raw Water of Four Different Towns[a]

compound	town 1 LLE	town 1 LSE	town 2 LLE	town 2 LSE	town 3 LLE	town 3 LSE	town 4 LLE	town 4 LSE
anthracene	26	29	24	22	35	26	33	28
fluoranthene	−	−	49	53	−	−	43	40
benzo[b]fluoranthene	25	30	−	−	−	−	−	−
benzo[k]fluoranthene	19	15	−	−	−	−	−	−
benzo[a]pyrene	−	−	41	35	−	−	−	−

parameter	town 1	town 2	town 3	town 4
conductivity (μS·cm^{-1})	454	202	108	166
DQO (KMnO$_4$) (mg O$_2$·L^{-1})	5.6	4.1	3.4	2.2

[a] −, lower than detection limit.

from 1.3 μg·L^{-1} for naphthalene to 0.007 μg·L^{-1} for benzo[k]fluoranthene.

Precision. The precision of the LLE and LSE techniques was determined by application to five identically prepared synthetic samples containing the 11 PAHs at the same concentrations as those indicated in the legend to Figure 1.

The standard deviations and variation coefficients (VC) are slightly higher in the LSE method and vary from 0.007 to 0.37 μg·L^{-1} and from 8 to 19%, respectively. These values are acceptable according to the VC criterion of Horwitz (1982).

Recovery. Recovery was studied in six samples of surface raw water: the LLE technique was applied to three samples and the LSE method to the other three, adding the 11 PAHs at three different concentration levels. The upper and lower PAH concentrations were double and half, respectively, the intermediate concentrations.

Tables 4 and 5 show the results obtained in the recovery of the 11 PAHs for the intermediate concentration and the mean recovery values for the three concentration levels.

The recovery yields using LLE and LSE for the intermediate concentration level and the mean yields

for the three concentration levels are slightly lower than those obtained on applying both techniques to reagent water. The mean recovery yields for LLE are greater than 75%, except in the case of naphthalene (71%), and the standard deviations range between 8 and 18%. The mean recovery yields with LSE are above 70%, except in the case of naphthalene (69%), and the standard deviations range between 9 and 21%.

APPLICATION OF THE METHOD

The LLE and LSE methods were applied to the determination of the 11 PAHs in water samples, both raw and finished, of different mineralization (conductivity) and organic matter (DQO) values selected from four different towns (Tables 6 and 7).

A comparison of both LLE and LSE methods was performed by using an analysis of variance (ANOVA) for four factors, namely, extraction method, compound, town, and class of water (raw or drinking water); concentration was considered as the dependent variable. Table 8 shows no statistically significant difference for the concentration between both extraction methods, since the *p*-value was 0.3639—the null hypothesis is accepted for a $p \geq 0.05$.

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Table 7. Concentrations (ng·L^{-1}) of PAHs in Finished Drinking Water of Four Different Towns[a]

compound	town 1 LLE	town 1 LSE	town 2 LLE	town 2 LSE	town 3 LLE	town 3 LSE	town 4 LLE	town 4 LSE
anthracene	23	22	–	–	–	–	26	30
fluoranthene	–	–	43	41	–	–	–	–
benzo[*b*]fluoranthene	–	–	–	–	–	–	–	–
benzo[*k*]fluoranthene	16	14	–	–	–	–	–	–
benzo[*a*]pyrene	–	–	33	31	–	–	–	–

parameter	town 1	town 2	town 3	town 4
conductivity (μS·cm^{-1})	479	216	117	173
DQO (KMnO$_4$) (mg O$_2$·L^{-1})	2.4	1.3	1.1	0.7

[a] –, lower than detection limit.

Table 8. Analysis of Variance (ANOVA) for Comparison of both LLE and LSE Methods, Using Concentration as the Dependent Variable

source	degrees of freedom	sum of squares	mean squares	*F*-value	*P*-value
method	1	14.286	14.286	0.868	0.3639
compound	4	1081.273	270.318	16.419	0.0001
town	3	21.523	7.174	0.436	0.7301
class of water	1	51.000	51.000	3.098	0.0954
residual	18	296.339	16.463		

From the results obtained in duplicate analysis of the samples of raw and finished drinking waters (Tables 6 and 7) it may be deduced that of the 11 PAHs studied, 5 were detected in the raw water: anthracene in 4 samples, fluoranthene in 2, and benzo[*b*]fluoranthene, benzo[*k*]fluoranthene, and benzo[*a*]pyrene in 1 sample. Only four PAHs were detected in the samples of finished drinking water: anthracene in two samples and fluoranthene, benzo[*k*]fluoranthene, and benzo[*a*]pyrene in one sample. Additionally, the concentration levels are close to the DL of the method.

The levels found when LLE and LSE methods were applied do not differ to a significant extent.

CONCLUSION

This study reveals that even with a simple instrument the new sorbent tC18 tested for LSE with HPLC fluorescence quantitation reaches DL values very similar to those of EPA Method 550.1 for the analytes studied. The results obtained with this proposed LSE method do not differ significantly from those obtained with the LLE method. In addition, it is suitable for automation and employs much lower amounts of usually toxic organic solvents. Concerning the different mineralization and organic matter of the waters assayed, no influence on the extraction efficiency and analytical results has been observed.

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