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Trace analysis by combined use of off-line solid-phase extraction, on-column sample focusing and U-shape flow cell in capillary liquid chromatography

Y. He, H.K. Lee*

Department of Chemistry, National University of Singapore, Kent Ridge, Singapore 119260, Singapore

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Abstract

The potential of capillary liquid chromatography for routine analysis of trace environmental pollutants was demonstrated in the determination of four polycyclic aromatic hydrocarbons (PAHs) at sub-ppb levels in natural water. The method involved the quick conversion of conventional high-performance liquid chromatography capillary LC using a commercially available upgrading kit. Focus was placed on enhancing concentration sensitivity by the combined use of off-line solid-phase extraction (SPE), on-column sample focusing and a U-shaped capillary flow cell. Influencing factors in SPE (sample volume, sorbent, eluent and elution temperature) and sample focusing (injection volume and sample solvent) were investigated together for optimizing overall enrichment. Under optimal conditions, the detection limit was in the range of 0.04–0.2 μg/l for the four PAHs. Sample volume was reduced, evaporation of organic eluent was eliminated, and therefore total analysis time was reduced; recoveries for the PAHs were >84%. © 1998 Elsevier Science B.V. All rights reserved.

Keywords: Trace analysis; Polynuclear aromatic hydrocarbons

1. Introduction

The evolution of analytical chemistry over the last 30 years has produced a systematic and rational trend toward miniaturization. This is particularly true for chromatographic techniques [1,2], such as capillary gas chromatography (GC) [3], capillary liquid chromatography (LC) [4,5] and capillary electrophoresis (CE) [6,7]. Capillary GC has been widely used in the analysis of complex environmental samples, and has ranked as one of the established methods in routine environmental analysis [8–12]. Recently, the potential of CE has also been actively investigated in the determination of various pollutants in the environment [13–15]. In contrast, capillary LC has captured little attention in the field of environmental analysis.

In recent years, there has been increasing concern over environmental pollution and tight operational budgets in the analytical laboratory. Moreover, environmental samples are becoming increasingly complex due to the fact that more than one thousand chemicals are released into the aqueous environment every year. Although capillary LC has advantages over conventional LC in terms of significant reduction of the consumption and disposal of organic solvents, minimization of exposure to toxic reagents, easy coupling with secondary separation system to

*Corresponding author.

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permit multidimensional chromatography, and with mass spectrometry for analyte characterization in complex environmental samples, its application in environmental analysis has so far been rarely reported [16]. The reluctance of environmental analysts to adopt this technique may be due to two problems with capillary LC. The first problem is the lack of reliable instrumentation and the limited availability of a wide range of packed capillary columns, which probably prevent faster development of this technique and its routine use [17]. The second problem is the limited concentration sensitivity, although capillary LC has good mass sensitivity.

The first problem may be addressed by the conversion of conventional high-performance liquid chromatography (HPLC) to capillary LC so that capillary LC can be easily performed on the basis of conventional HPLC [16,17]. Some manufacturers, such as LC-Packings, have offered the accessories to conveniently upgrade conventional HPLC to capillary LC [17]. The second problem may be addressed in two ways: (1) by improvement in the function of the detector, (2) by enriching the sample with off- or on-line solid-phase extraction (SPE), and on-column sample focusing [16–20]. These techniques have their own features: off-line SPE as a general sample enrichment technique has the advantage that selectivity and enrichment can be easily adjusted using a wide variety of sorbents in cartridge or disk formats, which are commercially available, but it is not easily automated without additional expense; on-line SPE facilitates the automation of sample treatment, but usually requires additional pump and a specially designed micro-precolumn which is available from very few manufacturers; on-column sample focusing has the merit of simplicity, but in some cases suffers the problem of peak broadening, low recovery and compromises between separation and enrichment. In previous works [16–20], these approaches were usually applied individually. Only recently has Dolezel et al. [21] reported an on-line arrangement of a precolumn, capillary mixer and fluorescence detector for determination of trace analytes. However, the method required a rather complicated and a specially designed set-up.

In this article, the potential of capillary LC for trace environmental analysis was demonstrated in the determination of trace amount of polycyclic aromatic hydrocarbons (PAHs) in lake water. PAHs are ubiquitous environmental contaminants that are suspected to be carcinogenic to mammals even in trace quantities [22]. The allowable maximum level of PAHs in natural water by national and international regulations is usually at the sub-ppb levels [23]. Hence, sensitive techniques were required for monitoring trace amounts of PAHs in the environment. In the present study, focus was placed on the combined use of off-line SPE, on-column sample focusing, and a U-shaped capillary flow-cell in capillary LC to enhance detection sensitivity. In addition, capillary LC was set up by converting a conventional HPLC with a commercially available upgrading kit for enhanced practicality in routine analysis.

2. Experimental

2.1. Apparatus

Conversion of a conventional HPLC instrumental set-up into a capillary LC system for isocratic separations was made in a similar way as shown in the work by Chervet et al. [17]. The HPLC pump (Model PU-980, Jasco, Tokyo, Japan) was converted to deliver a microflow by interfacing the pump with a microflow processor for isocratic elution (Model IC-70-CAP, LC-Packings, Amsterdam, The Netherlands). The processor uses split-flow techniques for delivering a reproducible and stable microflow, and was connected between the pump and the injector. A high-pressure in-line filter (0.5 μm) (Upchurch Scientific, Oak Harbor, WA, USA) was placed inside the inlet of the microflow processor. The filter was used to trap particles from the mobile phase or pump. A conventional Rheodyne injector (Model 8125, Rheodyne, Cotati, CA, USA) with a 20-μl sample loop (steel tubing) was used for large volume injection. For comparison, a Model 3493 Rheodyne injector with a 5-μl sample loop was also used. Separations were performed using a 150×0.18 mm, 3-μm d_p C_{18} Fusica column (LC-Packings). Detection was performed at 254 nm using a UV detector (Model 785A, Applied Biosystems, Foster City, CA, USA) equipped with a 8-mm, 35-nl, U-shaped microflow cell. Data acquisition was performed on a HP

integrator (Model 3396 Series II, Hewlett-Packard, Avondale, PA, USA).

Conventional HPLC was performed on a standard column (250×4.6 mm I.D., 5 μm, C_{18}) bought from Phenomenex (Torrance, CA, USA). Detection was performed at 254 nm using the above UV detector equipped with a 8-μl flow cell.

Two-hundred mg C_{18} cartridges were bought from Supelco (Bellefonte, PA, USA). Two-hundred mg C_8 cartridges were obtained from Whatman (Clifton, NJ, USA). The circulation water bath (Model F25) was from Julabo Labortechnik (Seelbach, Germany).

2.2. Materials

Naphthalene, fluorene, anthracene and fluoranthene were bought from Fluka (Buchs, Switzerland). All standards and mobile phase were prepared in ultra-pure water obtained from a ultra-pure water system (Model D4700, Barnstead, Dubuque, IA, USA). HPLC-grade acetonitrile and analytical-reagent grade tetrahydrofuran (THF) were obtained from J.T. Baker (Phillipsburg, NJ, USA), and analytical-reagent grade propanol was bought from Fisher Scientific (Fair Lawn, NJ, USA).

2.3. Standard and sample preparation

A stock solution with 1000 μg/l of naphthalene, fluorene, fluoranthene and 250 ppm of anthracene was prepared in methanol. Appropriate dilution of this stock solution was made in acetonitrile–water (40:60) to obtain the standard solutions.

Samples of natural water were collected from a small lake near the Botanical Gardens of Singapore, and stored in a glass bottle. Samples were stored in the refrigerator at ca. 5°C.

C_8 cartridges were conditioned with 5 ml of methanol, followed by 2×4 ml of water–2-propanol (9:1, v/v). The 100-ml water samples were loaded onto the cartridge under vacuum within half an hour. Two ml of water–methanol (50:50) was applied to wash the cartridge and remove the residual water. After air drying the cartridge for 2 min, it was immersed in the circulation water bath (50°C) as shown in Fig. 1. Three ml of acetonitrile was applied. Three min were allowed for acetonitrile to soak through and thermally equilibrate with the cartridge bed before elution. A syringe plunger was manipulated to force acetonitrile through the cartridge within ca. 2 min. The temperature variation of the water bath was ±2°C.

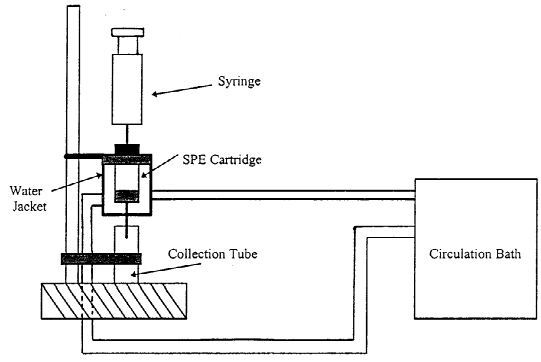

Fig. 1. Schematic diagram of temperature control in SPE.

3. Results and discussion

3.1. Off-line SPE

A number of works have investigated the use of SPE for clean-up and concentration of PAHs in soil and water samples [24–27]. Kootstra et al. [25] developed a SPE method for the analysis of 16 PAHs in soil samples. In that paper, C_8 was used as sorbent and THF was used as eluent. In the present paper, we followed a similar procedure to optimize SPE for enrichment of four PAHs from water samples. However, some modifications were made so that the final extract could be easily reconstituted to meet the requirements by the subsequent on-column sample focusing.

Since PAHs are strongly retained on the C_8 sorbent, it was expected that enrichment could be enhanced by increasing the sample volume loaded on the SPE cartridge. Our studies showed that a 500-ml sample could be preconcentrated without significant losses. However, more than 3 h were required for processing the 500-ml sample. The analysis of relatively small volumes of sample has many advantages. The time needed for processing the sample decreased dramatically and the manipulation is also simplified. In this study, 100 ml of water sample was passed though cartridge within half an hour.

Table 1
Recoveries of PAHs in SPE using C_8 (200 mg) and C_{18} (200 mg) cartridges ($n=3$) at room temperature (25°C)

	C_{18}		C_8	
PAH	Recovery (%)	R.S.D. (%)	Recovery (%)	R.S.D. (%)
Naphthalene	83	5.2	87	4.2
Fluorene	67	4.8	84	5.4
Anthracene	54	8.9	69	3.2
Fluoranthene	52	4.1	60	6.4

According to Kootstra et al. [25], C_8 material is better than C_{18} for extracting PAHs in both recoveries and reproducibilities. In our experiments, these two sorbents were compared. Recoveries with 3 ml of acetonitrile as eluent are presented in Table 1. As can be seen, C_8 did provide better recoveries than C_{18}. However, the recoveries were inadequate for quantitative analysis even when C_8 was used.

Kootstra et al. [25] used THF instead of acetonitrile to improve recoveries of PAHs. However, THF has a much stronger absorbance than acetonitrile at 254 nm [28], and it is much more difficult to flush it from the column than the other solvent [29], hence it is expected that the detection of early eluted PAHs would be probably strongly interfered with by the huge solvent peak especially when a large volume injection was performed for on-column sample focusing.

In the present work, an alternative approach was developed to improve recoveries of PAHs. Acetonitrile was still used as eluent, but the cartridge and eluent temperatures were elevated. Temperature as an important influencing parameter has been used to effect extraction in traditional liquid–liquid extraction (LLE) [30–32], supercritical fluid extraction [33,34], microwave assisted extraction and solid-phase microextraction [35,36]. However, the use of temperature (pertaining to both cartridge and eluent) in SPE has rarely been attempted. High temperature increases solvent strength, reduces retention factor and retention volume, and therefore might improve recoveries of strongly retained solutes. In our experiments, a 200-mg rather than 500-mg cartridge was used because of smaller mass, lower heat capacity, and hence faster thermal response. Experiments were carried out at two temperatures: 25°C (room temperature) and 50°C. In Table 2, results are summarized. It is clear that the recoveries were improved by 10–30% for four PAHs at 50°C. However, the standard deviation at 50°C was not as good as that at 25°C, which may be due to the temperature variation around 50°C. Experiment was also performed at 60°C, which showed no significant improvement of recoveries as compared to those at 50°C, implying that quantitative recovery of PAHs was attained at 50°C. The maximum temperature that could be used was limited by the boiling point of acetonitrile (81.6°C).

3.2. On-column sample focusing and the use of a U-shaped microflow cell

In capillary LC, injection, separation and detection are dimensionally much more closely related to one another. On the one hand, large-volume injection and flow cell are required in order to improve con-

Table 2
Recoveries of four PAHs using a C_8 (200 mg) at two different temperatures

	25°C		50°C	
PAH	Recovery (%)	R.S.D. (%)	Recovery (%)	R.S.D. (%)
Naphthalene	89	4.2	92	7.5
Fluorene	84	2.1	95	5.2
Anthracene	69	4.7	87	5.9
Fluoranthene	60	5.8	91	3.7

centration sensitivity. On the other, small-volume injection and flow cell are necessary in order to maintain the resolution obtained on the capillary column.

One of the approaches for large-volume injection without compromising resolution is to dissolve the solute in a solvent whose elution strength is weaker than that of mobile phase (similar to sample stacking in CE, where the solutes are dissolved in a solvent whose ionic strength is lower than that of the separation buffer) as has been shown by many articles [16–19]. However, some limitations may arise when very large injection volumes of several hundred microlitres are applied. This can result in increased analysis time, reduced column lifetime, peak dispersion, sample breakthrough or frontal analysis [17,19]. In non-selective focusing, enrichment is not only limited to the targeted compounds, but also to the contaminants [17]. Last but not least, injection of a very large volume sample (200 μl or more) requires a specially designed injection loop made of fused-silica tubing [16]. In order to make capillary LC more acceptable for routine applications, large volume injections should be more conveniently performed in a conventional injection loop made of stainless steel tubing. According to Brunmark et al. [16], the allowable injection volume into a conventional sample loop (I.D. 0.2 and 0.25 mm) without compromising efficiency obtained on capillary column (200×0.32 mm I.D., d_p 5 μm) was about 20 μl. In our experiments, the effects of injection volume on separation and detection using a conventional injection loop were investigated on a smaller capillary column (150×0.18 mm I.D., d_p 3 μm). The sample was dissolved in acetonitrile–water (40:60), and the mobile phase was acetonitrile–water (70:30). The results (Fig. 2) show that the peak height response (peak corresponding to fluorene) increased linearly with injection volume below 10 μl, and the loss in resolution (2.3%, resolution at 0.5 μl was used as reference) of the critical pair (fluorene and anthracene) was negligible. However, loss in resolution (>5%) became significant when the injection volume was 15 μl. Hence, injection of 10 μl sample was preferable in detection and considered acceptable in separation.

In traditional LLE of PAHs, evaporation after extraction commonly causes loss of volatile PAHs,

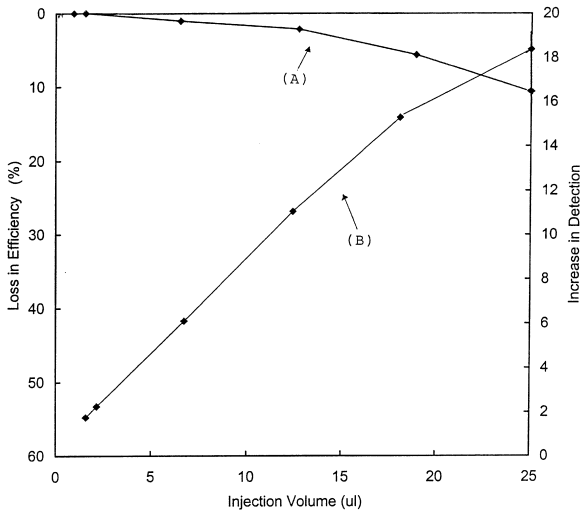

Fig. 2. Influence of injection volume on (A) efficiency (fluorene) and (B) peak height response (fluorene) in on-column sample stacking. Column: 150×0.18 mm I.D. fused-silica column packed with 3-μm C_{18}. Mobile phase: acetonitrile–water (70:30). Flow-rate: 2.0 μl/min. Sample solvent: acetonitrile–water (40:60). Detection: 254 nm.

such as naphthalene and fluorene. In the present case, evaporation of organic eluent after SPE was eliminated in order to improve recoveries of volatile PAHs, and simplify and speed up the overall enrichment process. The collected organic eluent was simply reconstituted by adding a certain amount of water to reduce the solvent strength to lower than that of the mobile phase. In Section 3.1, it was shown that THF showed better recoveries for the PAHs than acetonitrile did at room temperature. Therefore, THF should be chosen as suitable eluent if only off-column SPE was considered. However, it was uncertain whether the mixture of THF and water was suitable for the subsequent on-column sample focusing. Comparison was thus made between THF–water (30:70) and acetonitrile–water (40:60) (these two mixtures have similar solvent strength [29]) with regards to on-column sample focusing. The results (Fig. 3) show that the PAHs were completely or partly masked by the huge solvent peak when 5 μl of sample in THF–water was applied. This was due to the much stronger absorbance of THF at 254 nm than that of acetonitrile [28] and the difficulty of flushing THF from the column [29]. In contrast, all

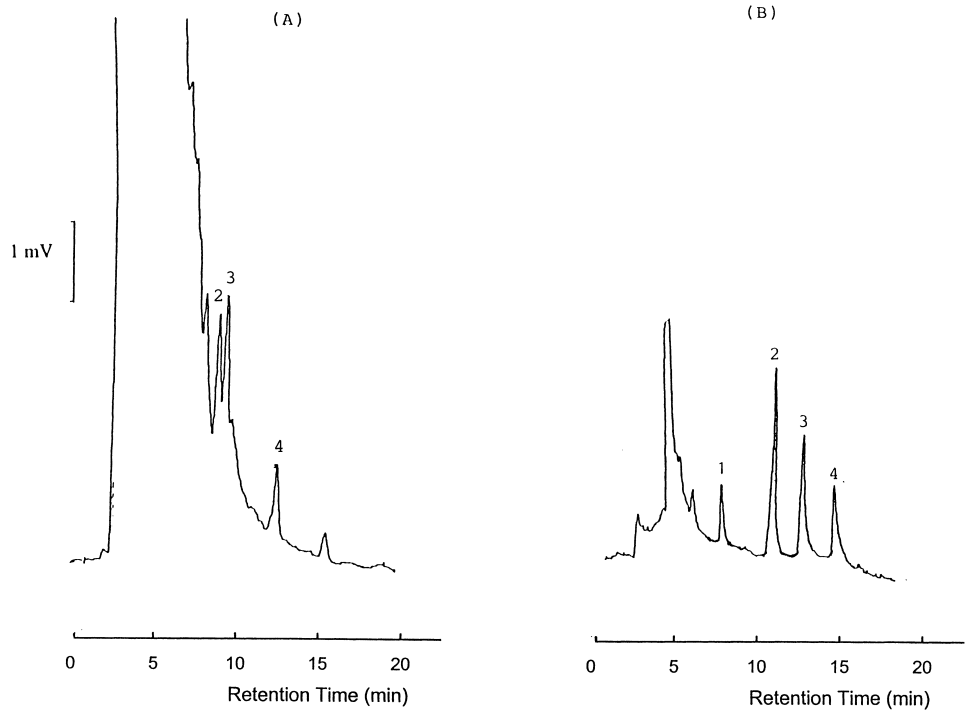

Fig. 3. Comparison of (A) THF–water (30:70) and (B) acetonitrile–water (40:60) as sample solvent in on-column sample stacking. Injection volume: 5 μl. Peaks: 1=naphthalene, 2=fluorene, 3=anthracene, 4=fluoranthene. Other conditions as in Fig. 2.

PAHs were well isolated from the much smaller solvent peak when 5 μl of sample in acetonitrile–water was injected.

Detection sensitivity can be further enhanced by the use of a U- or Z-shaped capillary flow cell [17,37]. In our experiment, a U-shaped capillary flow cell was used. In contrast to conventional on-column detection with very short path length determined by the I.D. (ca. 75 μm) of the fused-silica capillary, the bending of the capillary into a U-shape yields a much longer path length of 8 mm while maintaining an extremely small cell volume of 35 nl. This significant increase in path length boosts the sensitivity by a factor of nearly 100, whereas the extremely low cell volume guarantees the maintenance of the resolution attained on the capillary column.

With the injection of a 10 μl sample in acetonitrile–water (40:60), and the use of the U-shaped flow cell, the detection limits, calculated at $S/N=3$, were in the range of 0.4 (anthracene)–2 (naphthalene) μg/l.

It is well known that capillary LC is advantageous over conventional HPLC in terms of mass sensitivity due to the much lower dilution factor. However, this issue is more complicated when comparison is made from the point of view of concentration sensitivity due to the fact that the volumes of the injection and detector cell used are different in the two systems [38].

For practical purposes, the present paper compares the concentration sensitivity attained on capillary LC using a large volume injection technique (10 μl) and a U-shaped microflow cell with that attained on a conventional HPLC using standard injection volume (10 μl), a column with the dimensions (250×4.6 mm I.D.), and a normal detector cell (8 μl). It was found that the detection limit of four PAHs in conventional HPLC is in the range of 10–30 μg/l, which is about 20-times higher than that (0.4–2 μg/l) obtained in capillary LC. In this respect, the presented configuration of capillary LC is superior to conventional LC in terms of concentration sensitivity.

3.3. Combined use of off-line SPE, on-column sample focusing and U-shaped microflow cell for the analysis of natural water

As investigated above, enrichment could be enhanced by simply increasing the sample volume loaded onto the SPE cartridge (Vs-spe). This, however, resulted in an unacceptably long time for sample processing. Enrichment could also be improved by injecting very large volume of sample (Vs-inj), which has its disadvantages as noted above. The problem was addressed by the combined use of off-line SPE and on-column sample focusing. This combination offered sufficient enrichment with a reasonably small sample volume (100 rather than 500 or 1000 ml) loaded on to SPE cartridge and a moderate injection volume (10 instead of 100 or 200 μl) for on-column sample focusing. In practice, Vs-spe and Vs-inj can also be flexibly adjusted according to the particular requirements of time and detection sensitivity.

In the present study, the solvent evaporation step, commonly used in traditional LLE, was eliminated. The small-volume organic eluent (3 ml acetonitrile) after SPE was simply reconstituted by adding 4.5 ml distilled water to make up the proper 40:60 proportion prior to the injection for on-column sample focusing. Recoveries of the volatile PAHs, such as naphthalene and anthracene, were good (92–95%), and overall time for sample enrichment was reduced.

To resolve the conflict of THF being preferred for off-line SPE as against acetonitrile for on-column sample focusing, we used the latter as eluent at elevated temperature (50°C) to improve recoveries of strongly retained PAHs, and employed acetonitrile–water (40:60) as sample solvent for on-column sample focusing.

The method thus described was applied to the analysis of the four PAHs in natural water. Fig. 4 shows the chromatogram corresponding to lake water spiked with the PAHs at 0.2–0.8 μg/l. Analyte identification was based on retention times. To confirm the identification, single subsequent fortifications of the water extract were made with PAHs at 5–20 μg/l, and reanalyzed. Recovery of each PAH was over 83% (Table 3). In addition, there was little interference caused by other constituents in lake water samples. This implied that most interferences had been removed by the combination of off-line SPE and on-column sample focusing. Since off-line SPE using C_8 sorbent and on-column sample focusing using C_{18} sorbent provide different extraction selectivity, the combination of the two may therefore provide improved extraction selectivity, especially in the analysis of real samples.

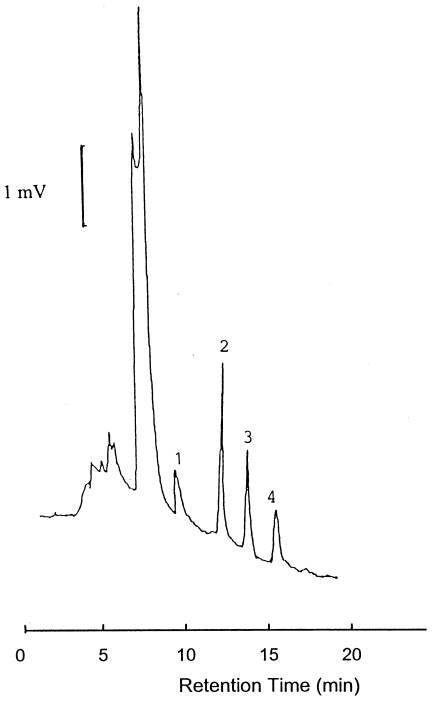

Fig. 4. Chromatogram of the extract of lake water sample fortified with anthracene at 0.2 μg/l, naphthalene, fluorene and fluoranthene at 0.8 μg/l. Peak identifications as in Fig. 3.

The linearity of the method was evaluated by analyzing calibration standards at concentrations of 0.2–5 μg/l for anthracene, and 0.8–20 μg/l for naphthalene, fluorene and fluoranthene. Peak area measurements were used for quantification. Good linearity was obtained with correlation coefficient

Table 3
Recoveries after duplicate SPE from lake water samples spiked with PAHs at concentrations of 0.2–0.8 μg/l

PAH	Recovery (%)	R.S.D. (%)
Naphthalene (0.8 μg/l)	94	7.8
Fluorene (0.8 μg/l)	89	6.3
Anthracene (0.2 μg/l)	92	4.7
Fluoranthene (0.8 μg/l)	84	4.2

values ≥0.995. Overall precision (coefficient of variations of repeated analysis of five samples) was determined by using a set of five lake water samples spiked with 0.2 μg/l of anthracene and 0.8 ppb of naphthalene, fluorene and fluoranthene. The precision was <7.8% for the four PAHs. The detection limit, calculated at $S/N=3$, was in the range of 0.03 (anthracene)–0.2 (naphthalene) μg/l.

4. Conclusion

The reported data show that capillary LC can be conveniently used for routine monitoring of PAHs at sub-ppb level in natural water. The major drawback of capillary LC, i.e., the limited concentration sensitivity, has been overcome by combined use of off-line SPE, on-column sample focusing and a U-shaped capillary flow cell. In addition, the capillary LC system used here, except for the column and the detector cell, consisted largely of conventional HPLC components, making the method easily applicable in a routine HPLC laboratory. Finally, temperature was shown to be a valuable parameter for SPE that should be further investigated.

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