

# Disposable ionic liquid coating for headspace solid-phase microextraction of benzene, toluene, ethylbenzene, and xylenes in paints followed by gas chromatography–flame ionization detection

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Received 15 November 2004; received in revised form 5 January 2005; accepted 14 January 2005  
Available online 1 February 2005

## Abstract

Solid-phase microextraction (SPME) with a disposable ionic liquid (IL) coating was developed for headspace extraction of benzene, toluene, ethylbenzene, and xylenes (BTEX) in paints. The SPME fiber was coated with IL prior to every extraction, then the analytes were extracted and desorbed on the injection port of gas chromatography, and finally the IL coating on the fiber was washed out with solvents. The coating and washing out of IL from the fiber can be finished in a few minutes. This disposable IL-coated fiber was applied to determine BTEX in water-soluble paints with results in good agreement with that obtained by using commercially available SPME fibers. For all the four studied paints samples, the benzene contents were under the detection limits, but relatively high contents of toluene, ethylbenzene and xylenes (56–271  $\mu\text{g g}^{-1}$ ) were detected with spiked recoveries in the range of 70–114%. Compared to the widely used commercially available SPME fibers, this proposed disposable IL-coated fiber has much lower cost per determination, comparable reproducibility (RSD < 11%), and no carryover between each determination. Considering that IL possess good extractability for various organic compounds and metals ions, and that task-specific IL can be designed and synthesized for selective extraction of target analytes, this disposable IL coating SPME might have great potential in sample preparation.

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**Keywords:** Ionic liquid; Solid-phase microextraction; Disposable coating; Benzene; Toluene; Ethylbenzene; Xylenes; Paint analysis

## 1. Introduction

Volatile organic compounds such as benzene, toluene, ethylbenzene, and xylenes (BTEX) have become of particular interest in the field of indoor air quality during the past several years. BTEX pollution in indoor air results from smoking and burning processes, as well as from emission from building or furnishing materials, paints, adhesives, other consumer products [1,2]. Since paints are one of the main sources of BTEX in indoor air, it has great significance to strictly control the

contents of BTEX in paints to decrease its impact on the environment. Therefore, development of analytical method for determination of BTEX in paints has an important relevance. To our knowledge, however, studies on the determination of BTEX in paints are very limited.

Due to the recent trends of using water-soluble paints instead of organic solvent-soluble paints, it is necessary to develop appropriate sample handling methods that are compatible with the determination of BTEX by gas chromatography (GC) in these matrices.

Since its introduction by Berladi and Pawliszyn in 1989 [3], solid-phase microextraction (SPME) has developed rapidly and it has been widely applied in sample analysis [4].

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This is because this simple and solvent-free sample preparation technique, which combines extraction, concentration and sample introduction in one step, is portable, sensitive and convenient to couple with various analytical instruments, especially GC. Almost a dozen different fiber coatings have been commercialized. Among them, the polydimethylsiloxane (PDMS) shows excellent selectivity for non-polar compounds while the polyacrylate (PA) coating facilitates the extraction of relatively polar analytes. To date, most of these fiber coatings suffered from limited life span (50–100 times of use), relatively low operating temperatures (200–270 °C) in GC applications, and incompatibility with the organic solvents in HPLC mobile phases. Several approaches have been proposed to overcome these drawbacks. While the sol-gel technology was adopted to prepare SPME fibers [5–7], also disposable usage of technical grade optical fibers as SPME fiber was proposed [8]. Another alternative is to employ liquid-phase microextraction (LPME) performed by using a single drop of solvent [9–11] or a small length of porous hollow fiber protecting the solvent [12]. It was reported that LPME shows comparable extraction efficiency and reproducibility to the widely used SPME, in addition to the advantage of very low cost and freedom of selection of appropriate solvent for extraction of selected analytes. Unfortunately, LPME suffers from relatively low sensitivity and inconvenient in performance compared with SPME.

Room temperature ionic liquids (IL), resulting from the combination of organic cations and various anions that are liquids at room temperature, are gaining wide recognition as potential environmentally benign solvents. The several unique properties of IL such as negligible vapor pressure, good thermal stability, tunable viscosity and miscibility with water and organic solvents [13], as well as good extractability for various organic compounds [14–19] and metals ions [20–23], merit their consideration as very potential absorbents for SPME/LPME. Additionally, IL were successfully used as stationary phases for gas chromatography by Armstrong and co-workers [15,20]. Our previous study also demonstrated that IL are attractive solvents for drop-based LPME of polycyclic aromatic hydrocarbons (PAHs) [24]. However, this drop-based LPME is incompatible with GC due to the nonvolatility of the IL.

In this present study, a new microextraction technique based on IL and coupled with GC was developed for the analysis of BTEX in paints. The proposed technique, a hybrid of SPME and LPME, for the first time suggests IL as disposable coating for microextraction. IL was coated on the fiber of a SPME device before every extraction, the analytes were extracted and desorbed on the injection port of GC, and finally the IL-coating on the fiber was washed out with solvents. It was expected that the relatively large viscosity of IL should improve their coating on the SPME fiber, while the liquid state of the of coating should facilitate easy washing out after sample injection and fast extraction as diffusion rates of analytes in liquid phase are much larger than that in

solid-phase. On the other hand, the nonvolatility and good thermal stability of IL permit them to stay on the fiber when the analytes were desorbed on the injection port of GC.

## 2. Experimental

### 2.1. Reagents

Reagents used for synthesis of IL were obtained from Acros Organics. BTEX standards were obtained from Institute for Reference Materials of SEPA (Beijing, China). HPLC-grade methanol and dichloromethane were purchased from Scharlau Chemie SA, Barcelona, Spain. All other chemicals were of analytical grade (Beijing Chemicals Corporation, Beijing, China) and ultrapure water (EASYpure LF, Barnstead International, Dubuque, IA, USA) was used throughout.

### 2.2. Synthesis of IL

The synthesis and physicochemical properties of 1-octyl-3-methylimidazolium hexafluorophosphate ( $[C_8MI-M][PF_6]$ ) IL was described in our previous study [24].

### 2.3. Apparatus

GC analyses were performed using a Hewlett-Packard 6890A gas chromatograph equipped with a split-splitless injector and the technique of flame ionization detection (FID) (Agilent, Palo Alto, CA, USA). The system was controlled by computer and Chemstation software (Agilent, Ver A.07.01). A HP-5 fused-silica column (25 m  $\times$  0.25 mm I.D.) with 95% dimethylsiloxane and 5% diphenylsiloxane (film thickness 0.25  $\mu$ m) was used. The injector port was operated with splitless mode and temperature was kept at 200 °C, while the detector temperature was maintained at 250 °C. The column oven temperature was programmed as follows: initial temperature of 40 °C for 3 min, then rising to 200 °C at a rate of 12 °C  $min^{-1}$  and hold at 200 °C for 3 min. High purity nitrogen was used as carrier gas (1.0 ml  $min^{-1}$ ) and make-up gas (30 ml  $min^{-1}$ ). Hydrogen and air were used as detector gases at 30 ml  $min^{-1}$  and 400 ml  $min^{-1}$ , respectively. Under this GC condition, the three xylenes isomers were separated into two peaks (one is the mixture of *m*-, *p*-xylene, another is *o*-xylene). In quantification, however, we integrated the area of these two peaks together and calculated the whole concentration of xylenes.

### 2.4. SPME device

The SPME device was adapted from a used 0.5- $\mu$ l microsyringe as shown in Fig. 1. A stainless steel wire was inserted into a movable stainless steel tubing held in the glass body of the microsyringe, and the wire pierces a piece of silicone rubber and gets into the hollow stainless steel

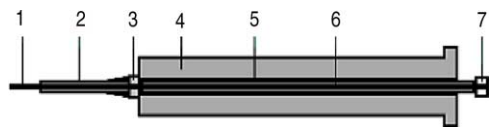


Fig. 1. Schematic diagram of the home-made SPME device: (1) IL-coated fiber tip; (2) stainless steel needle; (3) silicone rubber O-ring; (4) glass syringe body; (5) moveable stainless steel tubing; (6) stainless steel fiber or polyimide coated fused-silica fiber; (7) localizer.

needle. The silicone rubber provides an effective seal during the movement of the wire. A plastic localizer is fixed at the up end of the wire to make sure that the lower side of the wire can be moved 1 cm out of the stainless steel needle for coating and extraction. When fused-silica fiber was used, the stainless steel wire was replaced by a piece of fused-silica optical fiber. A SPME fiber holder for manual use and a 100  $\mu\text{m}$  polydimethylsiloxane (PDMS) fiber (Supelco, St. Louis, MO, USA) were used in this study for comparing study.

### 2.5. Fiber preparation

An aliquot of 900  $\mu\text{l}$  of  $[\text{C}_8\text{MIM}][\text{PF}_6]$  and 100  $\mu\text{l}$  of dichloromethane was added to a vial with a cap and mixed, and the obtained mixture was ready for the coating of an SPME fiber. When a stainless steel wire was coated, the 1-cm tip of the wire was washed with methanol and dichloromethane successively, and then conditioned at 200  $^\circ\text{C}$  under nitrogen for approximately 3 min in the GC injection port. After it was removed from the injector, the wire was cooled to room temperature and dipped vertically into the coating solution and held for 1 min, and then removed from the coating solution and kept in air for another 1 min for the dichloromethane to evaporate. This dipping and evaporating process was repeated three times to obtain relatively thick coating and better repeatability of the coating. When a fused-silica fiber was coated, the 1-cm tip of protective polyimide layer was removed firstly by dipped into dichloromethane. Then, the fused-silica fiber was pretreated and coated according to the same procedure as coating the stainless steel wire.

### 2.6. Solid-phase microextraction

A headspace SPME procedure was adopted. For each sample (20 ml water mixed with 3 ml methanol) spiked with 17  $\mu\text{g ml}^{-1}$  of benzene and toluene, and 8.5  $\mu\text{g ml}^{-1}$  of ethylbenzene and xylenes, the SPME fiber was exposed to the headspace for 30 min. The vial cap was sealed with aluminium foil and the foil was used only once. The sample was agitated with a magnetic stirring bar at room temperature (20  $^\circ\text{C}$ ) during the extraction process to allow the equilibration of analytes between the aqueous phase and the headspace, then the fiber was immediately inserted into the GC port for thermal desorption of the extracted analytes. In the experiments for optimization of analytical conditions,

each determination was repeated three times. The mean values were adopted and the standard deviations were shown as error bar in these figures. For sample analysis, determination was repeated five times.

### 2.7. Sample analysis

To 100 ml of flask were added 0.20 g paints, 20 ml water and 3 ml methanol and mixed. Headspace SPME was conducted as the above-described procedure for standard solutions. Spiked recoveries were determined by spiking standards into 0.20 g sample at concentration levels at 500  $\mu\text{g g}^{-1}$  of benzene, 450  $\mu\text{g g}^{-1}$  toluene, 250  $\mu\text{g g}^{-1}$  of ethylbenzene, and 350  $\mu\text{g g}^{-1}$  xylenes, respectively.

## 3. Results and discussion

### 3.1. Optimization of the coating procedure

Since the relatively high viscosity of  $[\text{C}_8\text{MIM}][\text{PF}_6]$  makes it inconvenient for direct coating, a mixture of dichloromethane and  $[\text{C}_8\text{MIM}][\text{PF}_6]$  (1 + 9, v + v) was used as coating solution. Experiments showed that both stainless steel wire and fused-silica fiber could be coated equably by IL, and the stainless steel wire had the advantage of long life span as it is a strong material. Unfortunately, the stainless steel wire available in our laboratory was relatively thick which made it difficult to move it in the hollow needle after coating and this provided worse repeatability. Therefore, a fused-silica fiber was adopted in the following studies.

The coating procedure was optimized to obtain better repeatability and higher sensitivity by using BTEX as model extracts. As can be seen in Fig. 2, the peak areas obtained when the fiber was dipped in the coating solution for 3 min and evaporated in air for 1 min were significantly smaller

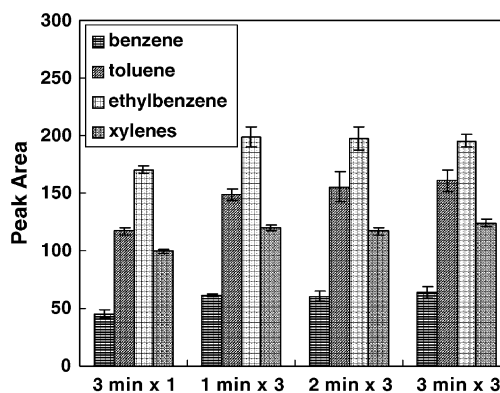


Fig. 2. Peak areas obtained by home-made disposable fiber coated with IL under different coating conditions: 3 min  $\times$  1, dipped in coating solution for 3 min followed by holding in air for 1 min to evaporated solvent; 1 min  $\times$  3, 2 min  $\times$  3, 3 min  $\times$  3, dipped in coating solution for 1 min, 2 min, 3 min, respectively, and followed by holding in air for 1 min to evaporated solvent, repeating this procedure three times.

than that when the fiber was coated by three cycles of dipping in the coating solution for 1 min and evaporated in air for 1 min. If the coating was kept to be three cycles, however, an increased dipping time from 1 min to 3 min did not increase the peak area. This is because the extracted amount of BTEX is governed by the amount of IL (thickness of IL film) coated on the fiber. The thickness of IL film is dependent on the number of coating cycle but not the duration of coating time. Therefore, the fiber was coated by three cycles of dipping in the coating solution for 1 min in the following studies.

### 3.2. Desorption time and temperature

To reach the highest sensitivity and to avoid carryover, the desorption time and temperature (GC injector temperature) were also evaluated to ensure that the analytes were completely desorbed from the fiber. Experiments showed that for all the studied four analytes, the desorption was almost complete after 3 min at 200 °C. Thus, these conditions were chosen for routine analysis. It should be addressed that as the IL film was physically coated on the fiber and the viscosity of IL decreased with the increasing of temperature, higher temperature than 200 °C resulted in significant loss of IL film. Thus, this present procedure is applicable for high volatile analytes such as BTEX, while it may be unsuitable for less volatile compounds such as polychlorinated biphenyls or PAHs.

### 3.3. Extraction temperature

It is well known that the extraction temperature has a double impact on HS-SPME. High temperature increases diffusion coefficients of the analytes in water and from liquid sample to the headspace, which efficiently shortens the extraction time; but also decreases the partition coefficients of analytes in the fiber coating, thereby decreasing extraction efficiencies. The effect of temperature on extraction of BTEX was studied in the range of 0–50 °C. Fig. 3 clearly shows that the peak area increased with temperature in the range of 0–20 °C and then decreased at higher temperatures. Room temperature (20 ± 2 °C) was adopted in the following studies.

### 3.4. Extraction time

To obtain extraction equilibrium in a reasonable analysis time, the extraction time was optimized in the range of 5–60 min. The results illustrated in Fig. 4 show that the peak areas of BTEX increased significantly in the range of 5–20 min and then only slowly with the prolonged extraction time. The equilibrium times were somewhat different for different analytes. Extraction equilibrium was reached in 40 min for benzene and toluene. For ethylbenzene and xylenes, however, extraction equilibrium was not reached even after 60 min. In regard of reasonable extraction effi-

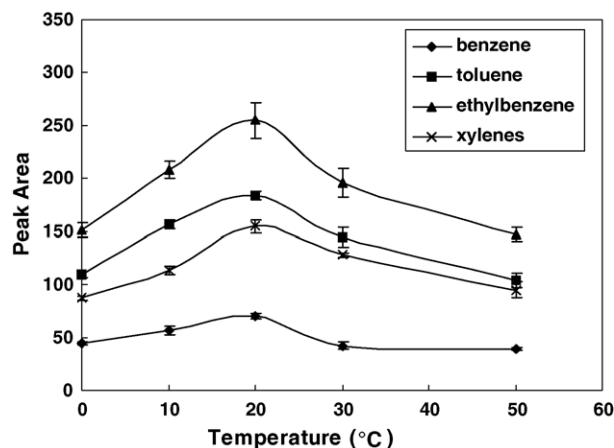


Fig. 3. Effect of temperature on the extraction of BTEX by a home-made disposable IL-coated fiber. Water (20 ml) spiked with 17  $\mu\text{g ml}^{-1}$  of benzene and toluene, and 8.5  $\mu\text{g ml}^{-1}$  of ethylbenzene and xylenes, headspace SPME for 30 min.

ciency and appropriate analysis time, 30 min was ultimately chosen as the preferred extraction time.

### 3.5. Salt effect

For some compounds, the addition of salts might decrease their solubility in the aqueous phase and thus increase their concentration in headspace. The influence of salt on the studied system was investigated by adding various amounts of NaCl in a series of concentrations (0, 10%, 20%, 30%, w/v), and results shown in Fig. 5 illustrate that the extraction efficiencies of BTEX decreased with the increase of NaCl content in the tested range. The reason is not clear, probably it is because the addition of salt in sample makes more methanol evaporating from sample solution to the headspace and then extracted into the IL film, which might decrease the extraction efficiency of BTEX. In this present study, no NaCl was added in the subsequent experiments.

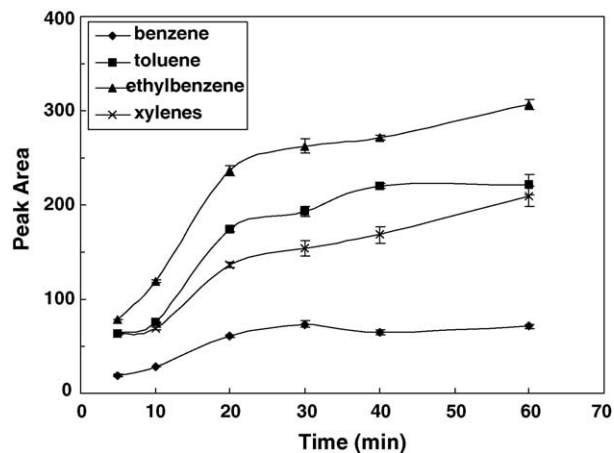


Fig. 4. Effect of extraction time on the extraction of BTEX by a home-made disposable IL-coated fiber. Sample solution and conditions as in Fig. 3.

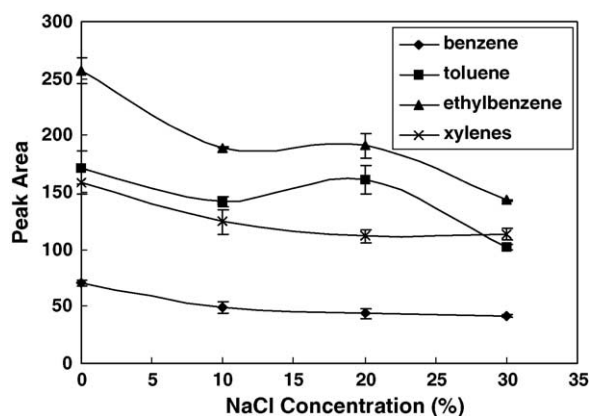


Fig. 5. Effect of salt on extraction of BTEX by home-made disposable IL-coated fiber. Sample solution and conditions as in Fig. 3.

### 3.6. Analytical performance

This proposed procedure was evaluated by the determination of calibration curves (based on peak area), detection limits and precision for BTEX by using standard solutions. The results shown in Table 1 demonstrate that the linear range could reach as wide as  $10^2$  or more. The detection limits ( $\mu\text{g ml}^{-1}$ ), defined as three times of base-line noise, were somewhat higher than those obtained using commercially available PDMS fibers due to the relative thin film of IL that can be coated on the fiber. Fortunately, it could offer sufficient detection sensitivities for analysis of BTEX in paints. The precision (RSD) of the system, measured by five repeated determinations of a standard solution containing  $17 \mu\text{g ml}^{-1}$  of benzene and toluene and  $8.5 \mu\text{g ml}^{-1}$  of ethylbenzene and xylenes, were in the range of 8–10%.

Table 1  
Some analytical performance data of the proposed method

Compounds	Linear range ( $\mu\text{g ml}^{-1}$ )	Regression equation	Correlation coefficient ( <i>R</i> )	Detection limit ( $\mu\text{g ml}^{-1}$ )	RSD (%)
Benzene	0.8–120	$y = 4.5754x - 15.27$	0.9979	0.2	8.3
Toluene	0.8–120	$y = 11.876x - 25.717$	0.9996	0.1	6.9
Ethylbenzene	0.4–60	$y = 27.821x - 5.9263$	0.9996	0.8	3.5
Xylenes	0.4–60	$y = 17.317x - 7.3716$	0.9985	0.8	10

Table 2  
Contents of BTEX ( $\mu\text{g g}^{-1}$ , mean  $\pm$  s,  $n=5$ ) in paints determined by SPME-GC with home-made IL-coated fiber and commercially available PDMS coated fiber

Paints	Benzene			Toluene			Ethylbenzene			Xylenes		
	PDMS <sup>a</sup>	IL <sup>b</sup>	Rec <sup>c</sup> (%)	PDMS	IL	Rec (%)	PDMS	IL	Rec (%)	PDMS	IL	Rec (%)
1	n.d. <sup>d</sup>	n.d.	88	214 $\pm$ 4	200 $\pm$ 5	70	84 $\pm$ 4	97 $\pm$ 4	96	164 $\pm$ 4	164 $\pm$ 6	99
2	n.d.	n.d.	107	261 $\pm$ 6	243 $\pm$ 6	92	85 $\pm$ 3	83 $\pm$ 3	72	168 $\pm$ 5	152 $\pm$ 6	77
3	n.d.	n.d.	114	264 $\pm$ 4	271 $\pm$ 5	91	61 $\pm$ 2	56 $\pm$ 3	80	127 $\pm$ 2	106 $\pm$ 4	78
4	n.d.	n.d.	100	263 $\pm$ 7	250 $\pm$ 6	109	91 $\pm$ 5	77 $\pm$ 5	93	149 $\pm$ 4	135 $\pm$ 2	78

<sup>a</sup> Determined with commercially available PDMS coated fiber.

<sup>b</sup> Determined with IL-coated fiber.

<sup>c</sup> Recoveries determined with IL-coated fiber at spiked levels of  $500 \mu\text{g g}^{-1}$  of benzene,  $450 \mu\text{g g}^{-1}$  toluene,  $250 \mu\text{g g}^{-1}$  of ethylbenzene, and  $350 \mu\text{g g}^{-1}$  xylenes, respectively.

<sup>d</sup> Not detected.

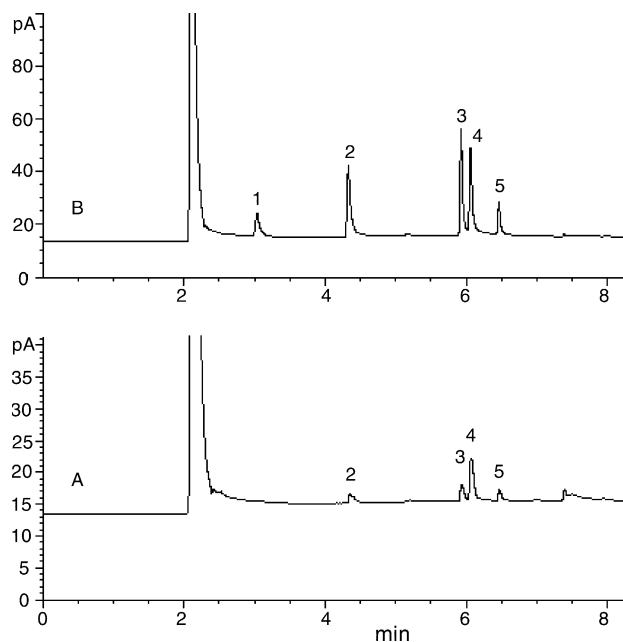


Fig. 6. Typical chromatograms of BTEX in a paint sample, extracted with home-made disposable IL-coated fiber: (A) paint blank; (B) addition of  $500 \mu\text{g g}^{-1}$  of benzene,  $450 \mu\text{g g}^{-1}$  toluene,  $250 \mu\text{g g}^{-1}$  of ethylbenzene, and  $350 \mu\text{g g}^{-1}$  xylenes, respectively. Peak symbols: (1) benzene; (2) toluene; (3) ethylbenzene; (4) *m*-, *p*-xylene; (5) *o*-xylene.

### 3.7. Sample analysis

By using the linear regressions shown in Table 1, the proposed method was successfully applied to determine BTEX in paints and the results obtained by five repeated measurements were shown in Table 2. For all four studied paints samples, the benzene contents were under the detection limits, but relatively high contents of toluene, ethylbenzene and

xylenes ( $56\text{--}271\ \mu\text{g g}^{-1}$ ) were detected. Good spike recoveries were achieved for all these four samples at spiking levels shown in Table 2. To further evaluate the credibility of the proposed IL-coated fiber, these samples were analyzed by using the widely used commercially available PDMS fibers under the same sampling and analytical conditions. Table 2 demonstrated that the results obtained by these two kinds of fibers were in very good agreement, which indicates the reliability of this proposed IL-based disposable coating for microextraction determination of volatile organic compounds (VOCs). Fig. 6 shows typical chromatograms of a sample obtained by using the proposed IL-coated SPME fiber. The large peak just after 2 min came from the methanol added into the sample solution.

#### 4. Conclusion

Microextraction with a disposable IL coating, a hybrid of solid-phase microextraction and liquid-phase microextraction (LPME), was developed as a sample handling technique for routine analysis of BTEX in paints. It was demonstrated that the relatively large viscosity of IL aided its coating on the SPME fiber and permitted easy washing out after sample injection. On the other hand, the involatility and good thermal stability of the IL will keep it staying on the fiber when the analytes were thermally desorbed in the injection port of GC, though loss of some of the IL film occurred due to the simple coating procedure. This proposed disposable IL-coated fibers show reproducibility comparable with the widely used commercially available SPME fibers, but its sensitivity is lower due to the relatively thin coating. The major advantage of the disposable IL-coated fiber is the very low cost because only a very low amount of IL is consumed for each determination. Another advantage of this disposable coating is the carry-over was avoided as new fiber coating is used for every determination. However, the detection limits were higher than those obtained using commercially available PDMS fibers due to the relative thin film of IL that can be coated on the fiber. Therefore, this proposed method is only applicable to samples with relatively high levels of BTEX such as paints. Considering that IL possess good extractability for various organic compounds and metals ions, and task-specific IL can be designed and synthesized for selective extraction of target

analytes, this disposable IL-coating for SPME/LPME might has great potential in sample preparation.

#### Acknowledgements

This work was jointly supported by the National Natural Science Foundation of China (20477052), and the National Key Project for Basic Research (2002CB412308).

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