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# Determination of aliphatic amines by gas chromatography–mass spectrometry after in-syringe derivatization with pentafluorobenzoyl chloride

Deepesh Kumar Singh [a], Sunil Kumar Sanghi [a,*], Sorna Gowri [a], Navin Chandra [a], Shail Bala Sanghi [b]

[a] Advanced Materials and Processes Research Institute, Bhopal 462026, India
[b] Government M. L. B. Girls College, Bhopal, India

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ABSTRACT

A simple and highly sensitive gas chromatographic method has been developed for the determination of low molecular weight short-chain aliphatic amines (SCAAs) after their simultaneous extraction and in-syringe derivatization with pentafluorobenzoyl chloride (PFBOC). Derivatization of the low molecular weight aliphatic amines in bicarbonate buffer of pH 10.5 with PFBOC was followed by immersed solvent microextraction. Derivatization conditions, including reagent concentration, reaction pH, ionic concentration of buffer, reaction time, stirring rate, reaction temperature and extraction solvent, have been investigated for method optimization. Linearity was studied within range of 0.15 pg ml$^{-1}$–50 ng ml$^{-1}$. The correlation coefficients were between 0.9934 and 0.9999. Detection limit of derivatized amines proved to be in the range of 0.117–1.527 pg ml$^{-1}$, and the intraday and interday relative standard deviation (RSD) values were less than 8% with respect to peak area. The method was applied for analysis of lake, river and industrial waste water. The recoveries of extraction from lake, river and industrial waste water samples, which have been spiked with different levels of aliphatic amines, were in the range of 68–99%, 63–102% and 62–105%, respectively.



## 1. Introduction

Aliphatic amines find wide industrial applications as chemical intermediates or as raw materials in polymers, pharmaceuticals, pesticides, corrosion protection, etc. [1]. Due to toxic nature their estimation and control are of prime importance to protect flora and fauna. Their high basicity, reactivity and polar nature pose problems in extraction from water matrix and hence create difficulty in chromatographic analysis [2].

Several methods have been developed for the analysis of low molecular weight short-chain aliphatic amines (SCAAs) in water and other samples involving derivatization followed by liquid chromatography (LC) coupled with UV [3,4], electrochemical (ECD) [5–7] and fluorescence detection [4,8–11]. Several reagents for the determination of aliphatic amines by LC with fluorescence detection and post-column online identification by atmospheric pressure chemical ionization mass spectrometry (APCI/MS) [12–20] and APCI/MS–MS [21] have been reported. Application of capillary electrophoresis (CE) with UV- [22] and laser-induced fluorescence detection (LIF) [23,24] was also reported for the analysis of SCAAs. Gas chromatography (GC) with flame ionization detector (FID) [25], MS [26–30] and tandem MS [31] is often preferred over LC in environmental samples due to its superior selectivity and sensitivity. Simultaneous determination of aliphatic and aromatic amines was also performed using GC–MS [32,33]. The sensitivity of all the above-reported methods for aliphatic amines was at pg ml$^{-1}$.

Efforts have also been made to detect amines at low level (ng ml$^{-1}$) in nonderivatized form using CE-UV [2] and GC–FID techniques after pre-concentrating the aliphatic amine using different extraction techniques such as solid-phase microextraction [34], head space single-drop microextraction (HS-SDME) [35] and solvent bar microextraction [36].

Derivatization of amines after extraction using isobutyl chloroformate [32,33], 2,4-dinitrofluorobenzene and benzenesulfonyl chloride [27], *N*-hydroxylsuccinimidyl phenylacetate (SIPA) [37], *N*-succinimidyl benzoate (SIBA) [38] has been carried out to make them less polar, improve their gas chromatographic characteristics and thus lower the detection limits in some cases down to pg ml$^{-1}$. However, the derivatization procedure requires more time and efforts. In order to resolve the above-mentioned problem, in situ derivatization was developed, which simply adds a reagent into a liquid sample. In situ derivatization combining the pre-concentration step under immersed condition and derivatization steps in buffered media is expected to further improve the sensitivity of estimation of aliphatic amines by GC.

This paper reports the results of our studies on the development of highly sensitive method for determination of aliphatic amines in

* Corresponding author. Tel.: +91 755 2489402; fax: +91 755 2457042.
*E-mail address:* sanghisk@gmail.com (S.K. Sanghi).

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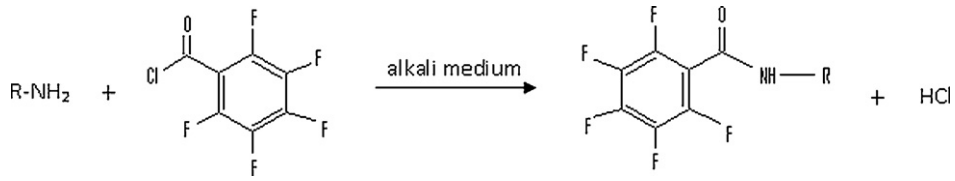


**Fig. 1.** The derivatization reaction of short-chain aliphatic amines with pentafluorobenzoylchloride.

water. In situ derivatization and immersed solvent microextraction of SCAAs using PFBOC is reported for the first time. The aliphatic amines present in water react with PFBOC, in alkaline medium, to form thermally stable volatile derivative, which is quantitatively analyzed and characterized by GC–MS. The influence of various parameters on the efficiency of extraction and derivatization is described in detail in this paper.

## 2. Experimental

### 2.1. Chemicals

Propylamine and pentylamine (99.5%) were purchased from Fluka, Buchs, Switzerland, while hexyl, heptyl, *n*-octylamine (99%), pentafluorobenzoylchloride (98%,) and boric acid (99%) were procured from ACROS Organics, New Jersey, USA. Sodium hydroxide (98%), methanol (99.8%) and nitrobenzene (98%) were procured from RANKEM, Delhi, India. Sodium bicarbonate (99.5%) was procured from CDH, New Delhi, India. Methylamine (40% solution in water), ethylamine (70% solution in water), butylamine (98%), sodium hydrogen phosphate and disodium hydrogen phosphate (98%) were procured from E. Merck, Mumbai, India. Stock solutions of amines were prepared in methanol and diluted to desired concentration in bicarbonate buffer (pH 10.5). Milli-Q water (Millipore, MA, USA) was used for dilutions and to prepare buffer solutions. Stock solution ($1.53 \times 10^{-3}$ mol $l^{-1}$) of pentafluorobenzoylchloride was prepared in nitrobenzene and diluted to desired concentration for use as extraction and derivatization media.

### 2.2. Apparatus

Trace GC Ultra (Thermo Electron Corporation, Milan, Italy) coupled with a Trace DSQ-Mass Spectrometer (Thermo Electron Corporation, TX, USA) fitted with quadruple detector was used for identification of derivatives and evaluating the completion of derivatization process. GC was fitted with capillary column BPX-5 (30 m × 0.25 mm i.d., 0.25 μm film thickness, 5% diphenyl – 95% dimethyl polysiloxane) from Supelco, PA, USA. The inlet pressure of carrier gas, helium, was set to 4 kg/$cm^2$. The analysis was performed with an initial temperature of 80 °C, held for 1 min and followed by heating to 120 °C at 20 °C $min^{-1}$ (held 2 min), which is further raised to 250 °C at 15 °C $min^{-1}$ (held 2 min). To protect the source of the mass spectrometer 6.4 min solvent delay time was opted. The temperatures of the injector port and transfer line were maintained at 250 °C and 280 °C, respectively. Samples were introduced in the splitless injection mode. The electron impact (EI) source (electron energy of 70 eV) was maintained at 200 °C. The mass range scanned was 50–450. A 10 μl syringe with bevel-shaped needle tip (Hamilton, Boanaduz, Switzerland) was used to form micro-droplet to be used for extraction and derivatization.

### 2.3. Immersed single-drop microextraction procedure

For immersed SDME, 3 μl of extraction media $4.6 \times 10^{-4}$ mol $l^{-1}$ (PFBOC in nitrobenzene) was drawn into the GC syringe and the sample vial septum was pierced with the needle. The syringe was clamped into place such that the tip of the needle was dipped in a consistent position in the 4 ml buffered (pH 10.5) water sample (containing dissolved SCAAs, 50 ng $ml^{-1}$ of each) from which the extraction was performed. The syringe plunger was then pressed to expose a micro drop of 2 μl solvent in the sample vial. The simultaneous pre-concentration and derivatization step was carried out for 25 min under continual stirring at 300 rpm using magnetic stirrer.

## 3. Results and discussion

Optimization of the derivatizing conditions and chromatographic separation of the derivatives formed were done by solvent selection, derivatizing reagent concentration, stirring rate, reaction time, temperature, buffer pH and ionic concentration. The aliphatic amines present in water react with PFBOC, in alkaline medium, to form thermally stable volatile derivatives as shown in Fig. 1. We selected five compounds as representatives of the aliphatic amine and showed their behavior under these extractions and derivatization conditions.

Several solvents, i.e. toluene, chlorobenzene, carbon tetrachloride, isooctane, nitrobenzene and dichloromethane, were tried to extract the derivative formed; in case of toluene and isooctane, the microdroplet was not stable and reproducible results were not found due to its lower density than water. Higher solubility of dichloromethane in water caused a significant loss of sensitivity of the analyte; also, the droplet was not stable to perform extraction. The splitting of amines peaks with identical mass fragmentation patterns was observed in case of toluene and chlorobenzene. This must be due to the formation of geometric isomers of derivatized amines that were partially separated [28]. Low extraction efficiency with carbon tetrachloride was observed because of its high vapor pressure. Low solubility, higher density and low vapor pressure make nitrobenzene an ideal solvent for the extraction of SCAAs. The low vapor pressure of nitrobenzene leads to the stable microdroplet for longer duration. Stirring rate plays a major role in the derivatization as well as extraction. The sample solution is agitated with a magnetic stirrer by means of a 10 mm × 2 mm stir bar. The stirring rate was studied between 100 and 400 rpm. To get the stabilized microdroplet and reproducible peak area, 300 rpm was selected for extraction. The optimal immersed SDME parameters were applied to derivatize five aliphatic amines in a single step as explained in Section 2.3. The derivatives were identified by MS operating in full-scan mode in the range of 50–450 *m*/*z*. The SCAAs were separated according to the retention times and mass spectra of the corresponding derivatives. The retention times of methyl, ethyl, propyl, butyl, pentyl, hexyl, heptyl and octylamine derivatives are observed to be at 6.63, 7.15, 8.13, 8.66, 10.01, 10.84, 11.61 and 12.32 min, respectively. The peak corresponding to PFBOC occurs at 9.51 min. The mass spectra of the PFBOC derivative of amines showed typical fragments ions at *m*/*z* 195, 225, 252 and 266. The base peak corresponding to $[C_6F_5–C{=}O]^+$ appears at *m*/*z* 195 in all the amine derivates and is in accordance with the molecular structure of the compounds. The fragment ion at *m*/*z* 225 corresponding to $[C_6F_5–CO–NH–CH_2]^+$ appears in all the mass fragmentation patterns except methyl, ethyl and propylamine due to unstable nature of $[CH_3–CH_2]^+$, $[CH_3]^+$ cations.

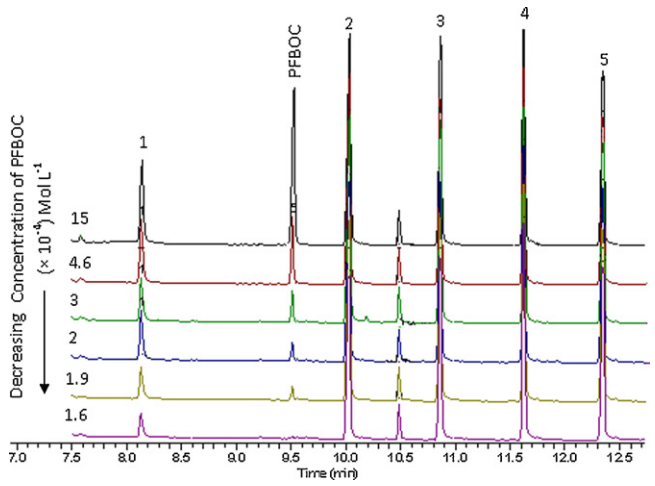


**Fig. 2.** Optimization of derivatizing reagent concentration. Extraction solvent: nitrobenzene; derivatization and extraction time: 25 min; reaction buffer pH: 10.2; stirring rate: 300 rpm; reaction temperature: 30 °C; standard mixture of each aliphatic amines: 50 ng ml$^{-1}$ to which SDME performed.

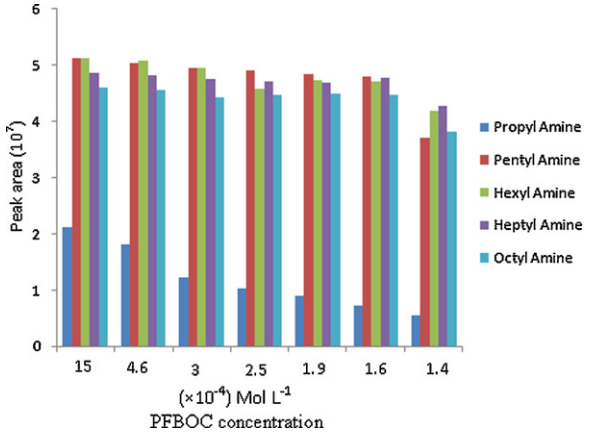


**Fig. 3.** Effect of PFBOC on derivatization efficiency. All the conditions as in Fig. 2.

### 3.1. Effect of derivatizing reagent concentration

The PFBOC concentration was studied for the derivatization of 50 ng ml$^{-1}$ solution of aliphatic amine in between $1.44 \times 10^{-4}$ mol l$^{-1}$ and $15 \times 10^{-4}$ mol l$^{-1}$. The in situ derivatization and extraction time was 25 min. It is clear from the chromatogram shown in Fig. 2 that PFBOC elute out at 9.52 min without interfering with the amine derivative. It was also observed from Fig. 2 that no peaks for derivatizing reagent were observed at $1.6 \times 10^{-4}$ mol l$^{-1}$ of PFBOC. The maximum sensitivity was obtained at $\geq 4.6 \times 10^{-4}$ mol l$^{-1}$ of PFBOC as observed from Fig. 3. No significant difference of the PFBOC concentration was observed on the derivatization efficiency from $15 \times 10^{-4}$ mol l$^{-1}$ to $4.6 \times 10^{-4}$ mol l$^{-1}$. Therefore, $4.6 \times 10^{-4}$ mol l$^{-1}$ of PFBOC was selected as optimum for further analysis.

### 3.2. Effect of pH

The derivatization of aliphatic amines is known to be pH sensitive, since the aliphatic amine forms nonvolatile salts in acidic medium. Therefore, reaction was performed only in alkaline medium. The pH optimized for the reaction was slightly below the p$K_a$ of the analyte; therefore, derivatization reaction was performed in between pH 8.5 and 12. For its optimization, solutions comprising a mixture of propyl, pentyl, hexyl, heptyl and octylamines (50 ng ml$^{-1}$ each) with pH values 8.5–12.0 were analyzed (Fig. 4). The peak area and in turn sensitivity of detection of each of the five amines studied are observed to be the highest at pH 10.5. Also, the effect of ionic strength of the buffer, varied between 10 and 40 mM, is observed to be negligible. Hence, further studies were conducted in solutions buffered using $NaHCO_3$-buffer (pH 10.5) of 30 mM ionic strength.

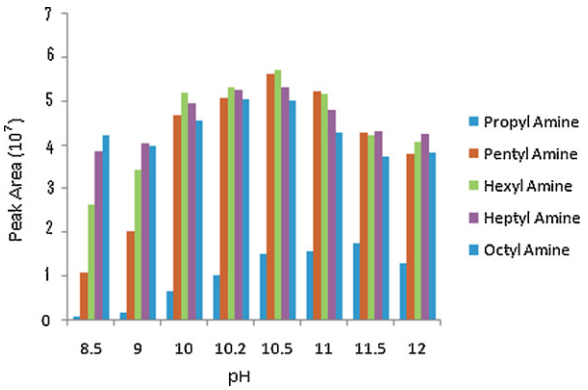


**Fig. 4.** Influence of pH on derivatization. PFBOC concentration was $4.6 \times 10^{-4}$ mol l$^{-1}$. All other conditions as in Fig. 2.

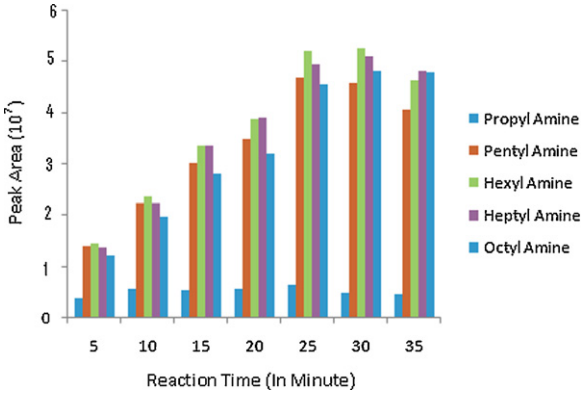


**Fig. 5.** Effect of extraction and derivatization time on derivative yield. pH 10.5. All other conditions as in Fig. 4.

### 3.3. Effect of reaction time

The effect of reaction time on peak area for the five amines studied is shown in Fig. 5. With an increase in the time of reaction, the peak area increases at 25 min for all the amines. Thus, 25 min reaction time was considered as optimum. After 25 min the

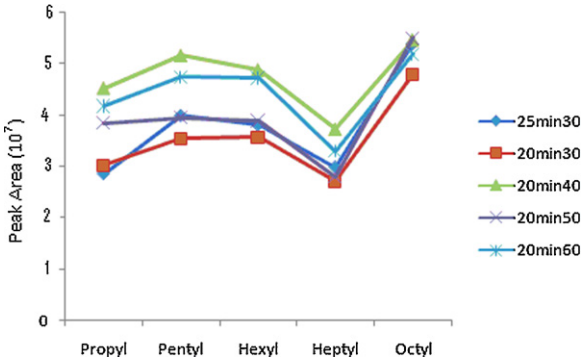


**Fig. 6.** Effect of variation in reaction temperature compared with the room temperature reaction (room temperature 30 °C). All other conditions as in Fig. 5.

**Table 1**
Calibration curve, detection limits and limit of quantification.

| Amines | $y = mx + c$ | | | | |
|---|---|---|---|---|---|
| | $C$ | $m$ | $r^2$ | LOD (pg ml$^{-1}$) | LOQ (pg ml$^{-1}$) |
| 1-Propylamine | 362525 | 706085 | 0.9934 | 0.117 | 0.391 |
| 1-Hexylamine | 669415 | 143406 | 0.9993 | 1.33 | 4.44 |
| 1-Heptylamine | 495948 | 124002 | 0.9998 | 1.46 | 4.83 |
| 1-Octylamine | 336293 | 979190 | 0.9999 | 1.527 | 5.08 |

$x$: concentration (pg ml$^{-1}$); $y$: peak area; $c$: intercept; $m$: slope.

derivative concentration decreases rapidly due to a loss in microdroplet volume. The longer reaction time was selected to get the maximum sensitivity, which is assisted by the low vapor pressure of nitrobenzene.

### 3.4. Effect of reaction temperature

From Fig. 6, it is clearly observed that the extraction rate can be improved by increasing temperature. The reaction proceeds efficiently at 40 °C for 20 min, but due to volatility of SCAAs, reproducibility was affected; therefore, extraction was performed at room temperature for 25 min.

### 3.5. Validation

Calibration curves were found to be rectilinear over the range 0.15 pg ml$^{-1}$–50 ng ml$^{-1}$ of amines. The $r^2$ values, limit of detection (LOD) and limit of quantification (LOQ) are given in Table 1. LOD was taken as concentration that gave a signal to noise ratio of 3. For the determination of amines in lake, river and industrial waste water samples, three replicate analyses of samples spiked at concentrations of 1 ng ml$^{-1}$, 2.5 ng ml$^{-1}$ and 10 ng ml$^{-1}$ were carried out. The LOD was between 0.117 pg ml$^{-1}$ and 1.527 pg ml$^{-1}$ of amines, and the intraday and interday relative standard deviation (RSD) values are less than 8% with respect to peak area. The LOQ was defined as average of background plus 10 standard deviations.

### 3.6. Application

The total ion chromatogram of river water sample, collected from Betawa River, Bhojpur, Bhopal, India, after derivatization with PFBOC is shown in Fig. 7. The curve A shows the presence of excess PFBOC, pentylamine and dibutyl phthalate, which are identified by mass spectrometry. The other seven aliphatic amines are observed to be absent in the river water. On spiking the river water with 10.0 ng ml$^{-1}$ of each of eight amines (Fig. 7, curve B), the chromatogram shows an enhanced intensity of the peak corresponding to pentylamine, along with the peaks of methyl, ethyl, propyl, butyl, hexyl, heptyl and octylamines. The presence of propylamine and butylamines were observed in lake water and industrial waste water, respectively. The recoveries were determined by comparing the peak area of derivatized amines in spiked water samples with derivatized standards of amines in ultra pure water. The river, lake and industrial waste water samples were spiked with the

**Table 2**
Recoveries of aliphatic amines from spiked lake, river and industrials waste water samples.

| Amines | Added (ng ml$^{-1}$) | Lake water | | River water | | Industrial waste water | |
|---|---|---|---|---|---|---|---|
| | | Found (SD, $n = 3$) (ng ml$^{-1}$) | Recovery (%) | Found (SD, $n = 3$) (ng ml$^{-1}$) | Recovery (%) | Found (SD, $n = 3$) (ng ml$^{-1}$) | Recovery (%) |
| Methylamine | 1 | 0.68 (0.034) | 68 | 0.67 (0.022) | 67 | 0.69 (0.027) | 69 |
| | 2.5 | 1.85 (0.075) | 74 | 1.67 (0.041) | 77 | 1.7 (0.041) | 68 |
| | 10 | 7.0 (0.27) | 70 | 6.3 (0.21) | 63 | 6.2 (0.18) | 62 |
| Ethylamine | 1 | 0.77 (0.032) | 77 | 0.81(0.029) | 81 | 0.82 (0.031) | 82 |
| | 2.5 | 1.97 (0.059) | 79 | 2.1 (0.049) | 84 | 1.9 (0.048) | 76 |
| | 10 | 7.4 (0.20) | 74 | 7.8 (0.34) | 78 | 8.0 (0.26) | 80 |
| 1-Propylamine | 1 | 0.72 (0.015) | 72 | 0.81 (0.023) | 81 | 0.78 (0.039) | 78 |
| | 2.5 | 1.97 (0.043) | 79 | 2.12 (0.039) | 85 | 2.02 (0.056) | 81 |
| | 10 | 7.4 (0.17) | 74 | 8.9 (0.20) | 89 | 7.8 (0.21) | 78 |
| 1-Butylamine | 1 | 0.79 (0.019) | 79 | 0.81(0.04) | 81 | 0.97 (0.037) | 97 |
| | 2.5 | 2.22 (0.038) | 89 | 2.2 (0.07) | 88 | 2.45 (0.067) | 98 |
| | 10 | 9.2 (0.15) | 92 | 8.6 (0.17) | 86 | 10.5 (0.45) | 105 |
| 1-Pentylamine | 1 | 0.89 (0.04) | 89 | 0.94 (0.035) | 94 | 0.88 (0.034) | 88 |
| | 2.5 | 2.1 (0.048) | 84 | 2.42 (0.069) | 97 | 2.05 (0.054) | 82 |
| | 10 | 9.9 (0.37) | 99 | 10.2 (0.38) | 102 | 9.8 (0.27) | 98 |
| 1-Hexylamine | 1 | 0.93 (0.023) | 93 | 0.84 (0.037) | 84 | 0.91(0.027) | 91 |
| | 2.5 | 2.17 (0.049) | 87 | 1.97 (0.05) | 79 | 2.22 (0.06) | 89 |
| | 10 | 9.9 (0.40) | 99 | 8.9 (0.16) | 89 | 9.2 (0.21) | 92 |
| 1-Heptylamine | 1 | 0.86 (0.011) | 86 | 0.81(0.041) | 81 | 0.86 (0.017) | 86 |
| | 2.5 | 2.12 (0.029) | 85 | 2.2 (0.089) | 88 | 2.12 (0.034) | 85 |
| | 10 | 8.0 (0.22) | 80 | 8.6 (0.19) | 86 | 8.9 (0.11) | 89 |
| 1-Octylamine | 1 | 0.93 (0.011) | 93 | 0.91 (0.019) | 91 | 0.89 (0.025) | 89 |
| | 2.5 | 1.97 (0.027) | 79 | 2.22 (0.041) | 89 | 2.3 (0.059) | 92 |
| | 10 | 9.7 (0.16) | 97 | 9.6 (0.13) | 96 | 9.0 (0.18) | 90 |

All results are the average of three triplicate analyses.

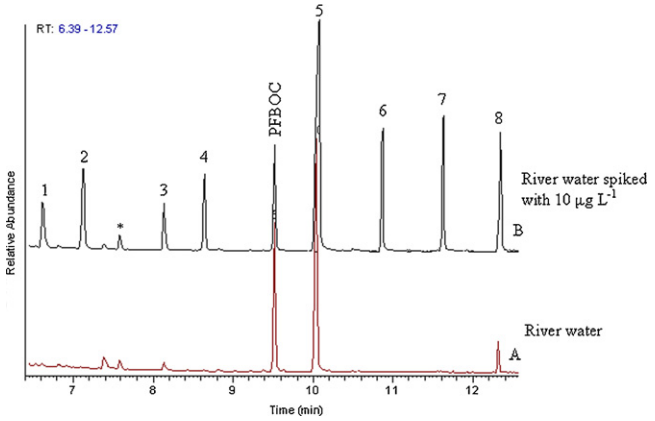


**Fig. 7.** The chromatogram of river water (A) spiked with eight aliphatic amines at 10 ng ml$^{-1}$ each. (B) River water sample. Peaks: * unknown peak; 1, methylamine; 2, ethylamine; 3, propylamine; 4, butylamine; 5, pentylamine; 6, hexylamine; 7, heptylamine; 8, octylamine.

standards of SCAAs at three different concentration levels. The average recoveries for lake, river and industrial waste water samples were found in between 68–99%, 63–102% and 62–105%, respectively, as shown in Table 2.

The interferences studies were performed separately with the aromatic amines (2-chloro aniline; 2, 5-dimethyl aniline; 2,4-dichloro aniline; 2,6-diethyl aniline) and phenols (2,3-dichlorophenol; 4-chlorophenol; 3,5-dimethyl phenol) under optimized conditions but no interferences of aromatic amines and phenols were observed. This may be due to aromatic amines that have p$K_a$ value less than 5 and show maximum reactivity below this pH [39]. Phenols will be derivatized at pH higher than 11 but their extraction recovery will be low enough at high pH [40,41].

The proposed method can be applied to the trace-level identification and quantification of SCAAs in different kind of real water samples. The sensitivity attainable is comparable to or even better than that of reported methods that employ fluorescent derivatization coupled with CE-LIF [23,24], LC–APCI/MS [12–20] and other GC methods.

## 4. Conclusions

The developed method of immersed solvent microextraction combined with in situ derivatization of aliphatic amine was shown to be comparatively fast, simple and sensitive for analysis of low molecular weight short-chain aliphatic amines in water at sub-pg ml$^{-1}$ levels. Insitu derivatization and immersed solvent microextraction of SCAAs using PFBOC has been reported for the first time. The most important parameter involved in the derivatization and extraction processes was pH. Solvent consumption and waste generation are much lower than those of methods involving conventional forms of analyte enrichment such as liquid–liquid extraction and separation. However, nitrobenzene is toxic to the environment but the extraction solvent used in microliter volume is justifiable. The developed method has low detection limit, good linearity and precision. The experimental results show that the proposed method is a rapid, simple and low-cost tool for quantitative analysis of low molecular weight short-chain aliphatic amines in aqueous samples.

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