CHAPTER 3
RESULTS

3.1 Gas Chromatographic Conditions

3.1.1 Results of GC-FID conditions

In this study, extraction of BTEX compounds from water was performed using a poly(dimethylsiloxane) solid-phase microextraction fiber assembly with separation and quantification by HP-FFAP capillary column gas chromatography and flame ionization detection. All components of BTEX were resolved and the chromatograms obtained from direct SPME sampling and both headspace SPME sampling by saturating an aqueous solution with salt and without salt addition were compared. Retention times of BTEX are shown in Table 3.1, and three chromatograms of BTEX are shown in Figure 3.1 3.2 and 3.3.

Table 3.1 Retention times (min) of BTEX in aqueous standard solution by SPME-GC-FID and HSSPME-GC-FID

Order of elution	Compound	Retention time (min)		
		SPME	HSSPME[a]	HSSPME[b]
1	Benzene	3.706	3.708	3.703
2	Toluene	5.232	5.234	5.225
3	Ethylbenzene	7.563	7.530	7.756
4	p-Xylene	7.902	7.870	8.081
5	m-Xylene	8.178	8.146	8.349
6	o-Xylene	10.330	10.306	10.467

[a] 10-ml of aqueous solution was not add with salt before extraction.

[b] 10-ml of aqueous solution was saturated with 3 g NaCl before extraction.

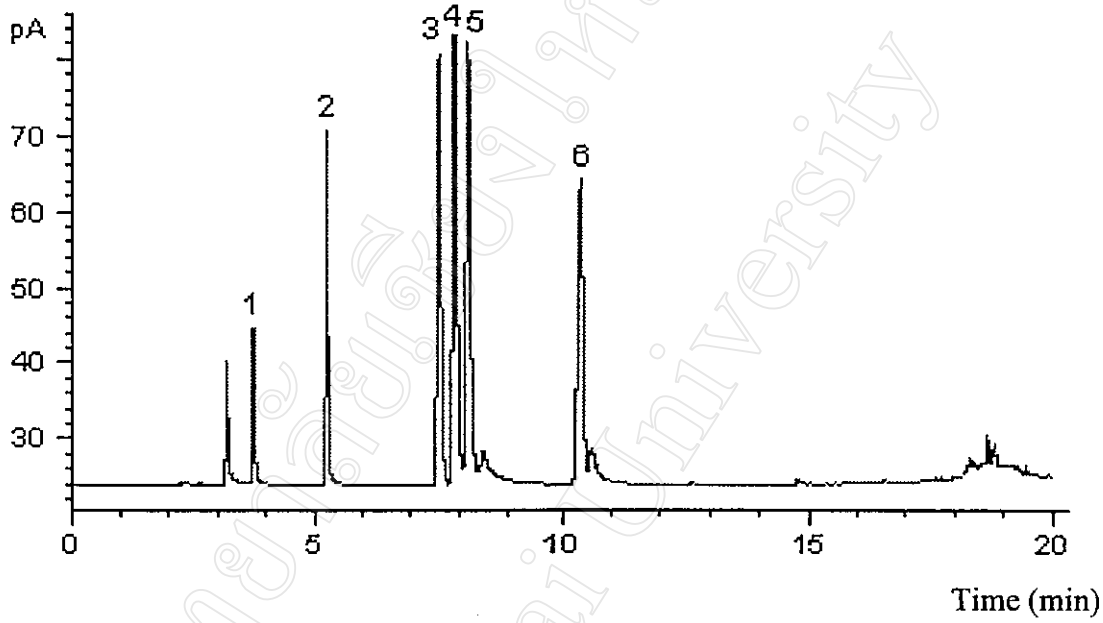

Figure 3.1. GC-FID chromatogram of mixed BTEX in aqueous standard solution obtained with the direct SPME sampling under optimum conditions of SPME-GC-FID listed in Table 2.8. Peak identification: (1) benzene (200 µg/L) ,(2) toluene (100 µg/L) (3) ethylbenzene (100 µg/L), (4) p-xylene (100 µg/L), (5) m-xylene (100 µg/L) and (6) o-xylene (100 µg/L).

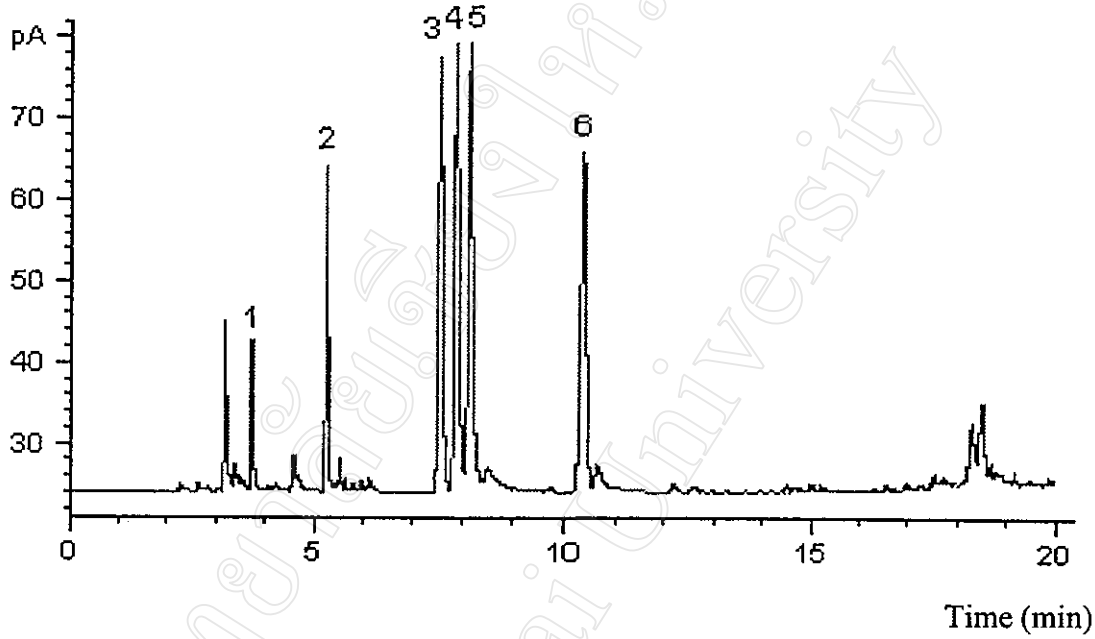

Figure 3.2 GC-FID chromatogram of mixed BTEX in aqueous standard solution obtained with the HSSPME sampling under optimum conditions of HSSPME-GC-FID listed in Table 2.8.
Peak identification : 1) benzene (200 µg/L), (2) toluene (100 µg/L) (3) ethylbenzene (100 µg/L), (4) p-xylene (100 µg/L), (5)m-xylene (100 µg/L) and (6) o-xylene (100 µg/L).

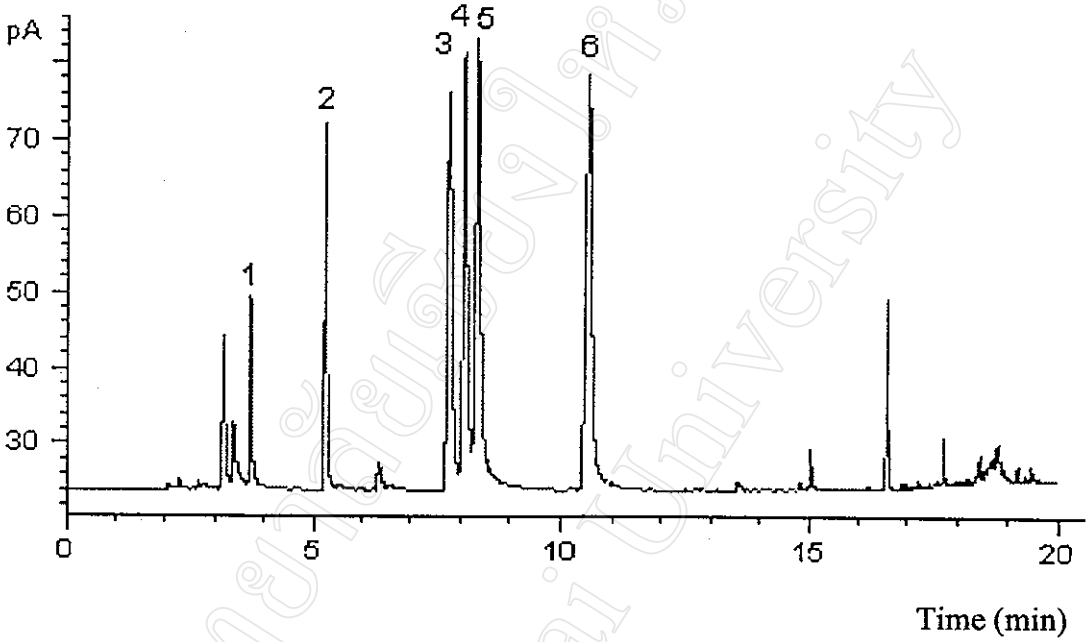

Figure 3.3 GC-FID chromatogram of mixed BTEX in aqueous standard solution obtained with the HSSPME sampling were performed with an aqueous solution was saturated with 3 g NaCl before extraction under optimum conditions of HSSPME-GC-FID are listed in Table 2.8. Peak identification : 1) benzene (200 µg/L), (2) toluene (100 µg/L), (3) ethylbenzene (100 µg/L), (4) p-xylene (100 µg/L), (5)m-xylene (100 µg/L) and (6) o-xylene (100 µg/L).

3.1.2 Confirmatory Identification of BTEX by HSSPME-GC-FID

Initial confirmation of the of BTEX peaks was obtained by HSSPME. The extract was performed with BTEX stock standard solutions of single-component and mixed BTEX in methanol. The assigning of peaks in GC-FID chromatograms was by means of comparing the retention times with those of standard compounds chromatographed under the same conditions. It was found that the elution order for confirmation of BTEX was the following: benzene< toluene<ethylbenzene< p-xylene < m-xylene< o-xylene. GC-FID chromatographic patterns of the HSSPME extracts of mixed BTEX and single-components of BTEX in methanol are shown in Figure 3.4.

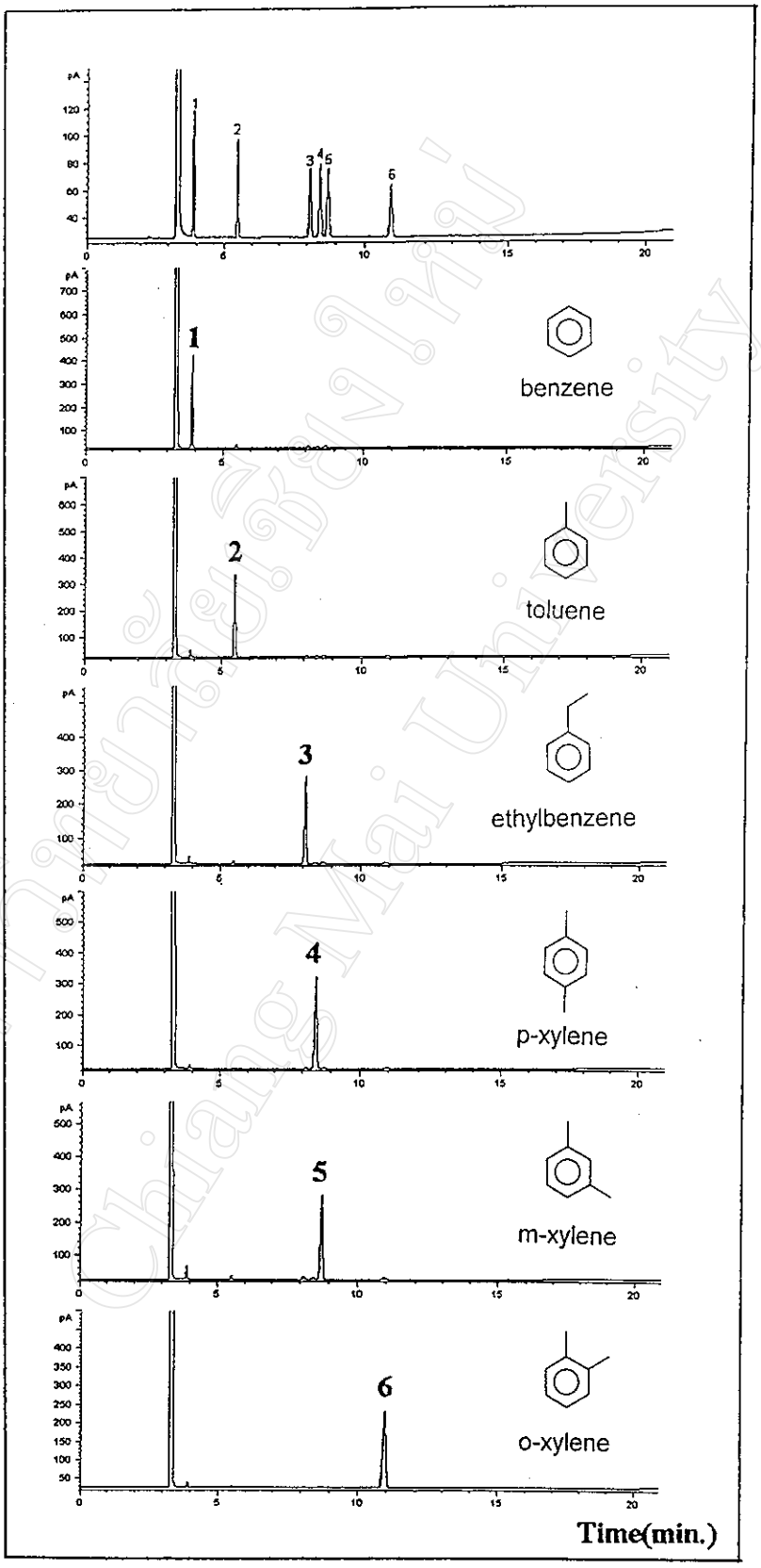

Figure 3.4 GC-FID chromatographic patterns of the HSSPME extracts of mixed BTEX and single- component of BTEX in methanol.

3.1.3 Confirmatory Identification of BTEX by HS-SPME-GC-MS

After assigning peaks of BTEX in the GC-FID chromatograms, identification process was further carried out in order to obtain a high degree of confidence with the aid of GC-MS data. HSSPME extracted of both the saturating an aqueous solution with salt and without salt addition, and analysis was obtained under optimum conditions of HS-SPME-GC-MS as described in Section 2.4.6.2 and table 2.9. Identification of each BTEX component was by means of comparison of the mass spectral data with those of the available standards. The spectral library available in this study was that based on the Wiley mass spectral database. Similarity in the mass spectral data greater than 95% was assumed as correct identification in this study.

Comparison results for the retention time at scan number on apex peak of BTEX with elution order are summarized in table 3.2. GC/MS chromatogram with the single ion mass-chromatograms and mass spectra of BTEX from HSSPME extracted of an aqueous solution without salt addition before extraction are showed in Figure 3.5, 3.6 and 3.7. GC/MS chromatogram with the single ion mass-chromatograms and mass spectra of BTEX from HSSPME extracted of the saturating an aqueous solution with 3 g NaCl before extraction are showed in Figure 3.8, 3.9 and 3.10.

Table 3.2 Scan number and retention time of BTEX in aqueous standard solution by HSSPME-GC-MS.

Order of elution	compound	HSSPME extraction			
		Without salt addition		With salt addition	
		Scan no.	t_R (min)	Scan no.	t_R (min)
1	Benzene	713	2.119	720	2.134
2	Toluene	1,175	3.109	1,180	3.119
3	Ethylbenzene	1,878	4.614	1,945	4.758
4	p-Xylene	1,973	4.818	2,036	4.953
5	m-Xylene	2,065	4.996	2,116	5.124
6	o-Xylene	2,701	6.377	2,751	6.485

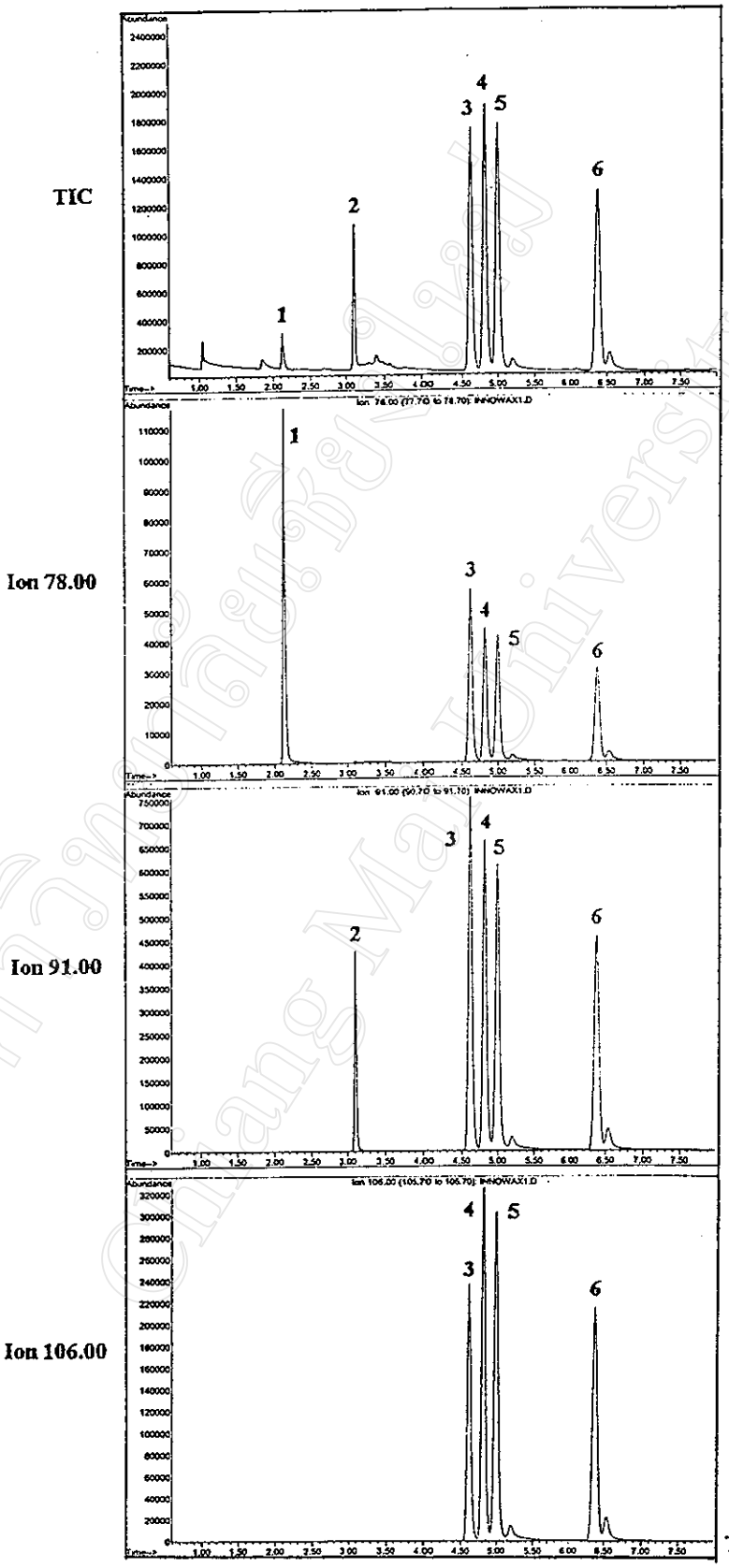

Figure 3.5 The total and single ion chromatograms of BTEX in aqueous solution without salt addition.

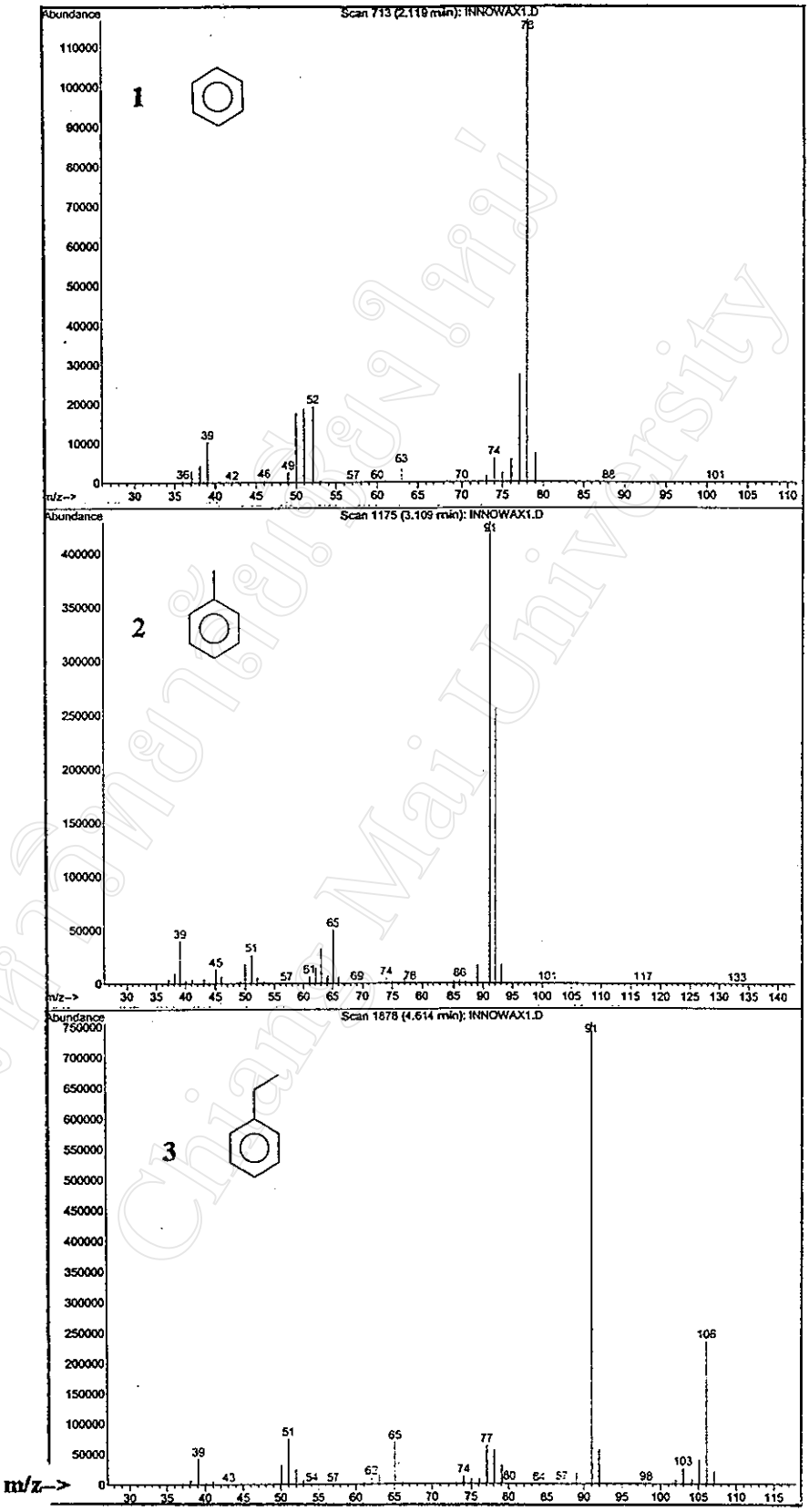

Figure 3.6 Mass spectra of 1) benzene 2) toluene and 3) ethylbenzene in aqueous solution without salt addition.

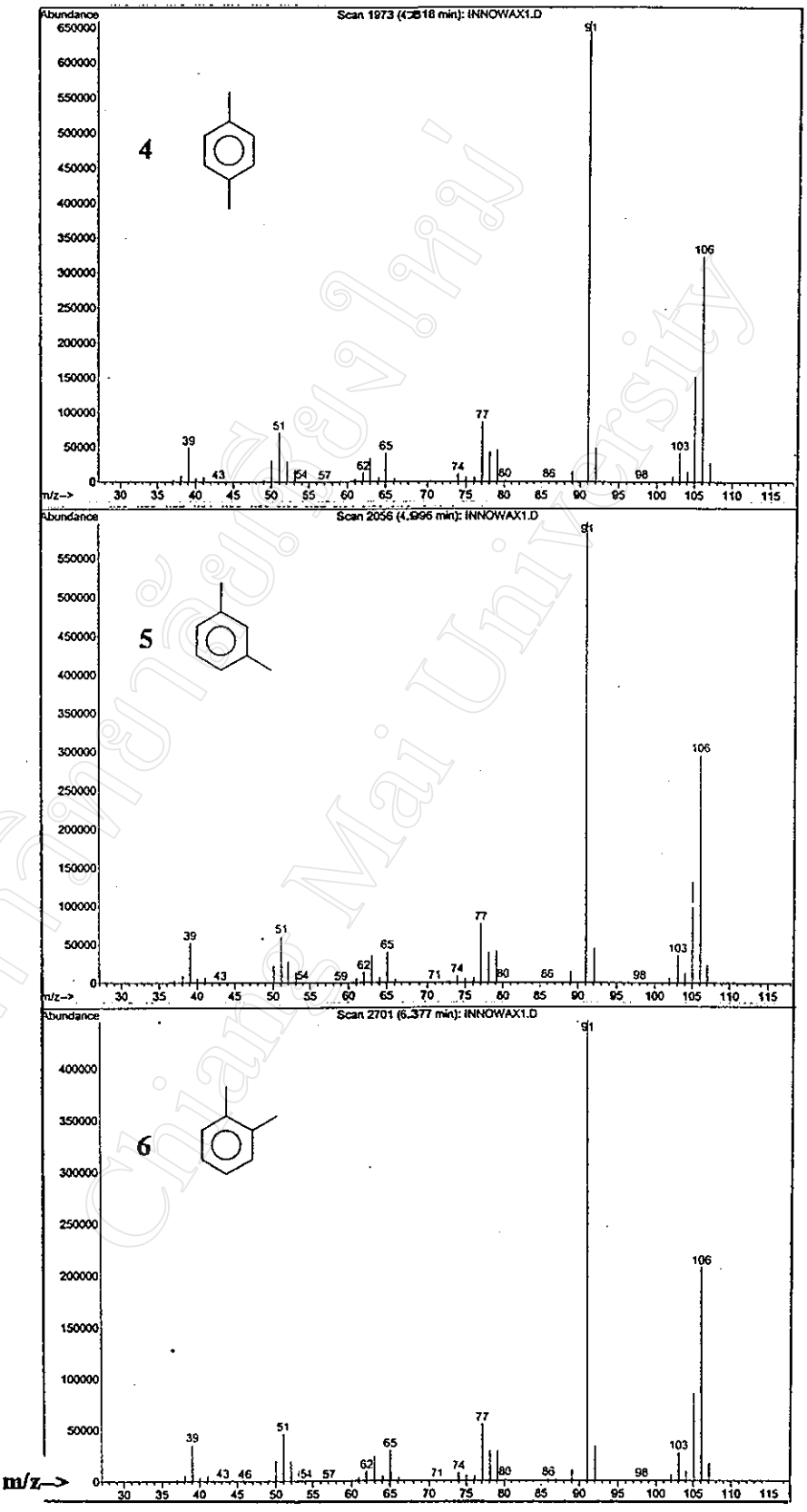

Figure 3.7 Mass spectra of 1) p-xylene 2) m-xylene and 3) o-xylene in aqueous solution without salt addition.

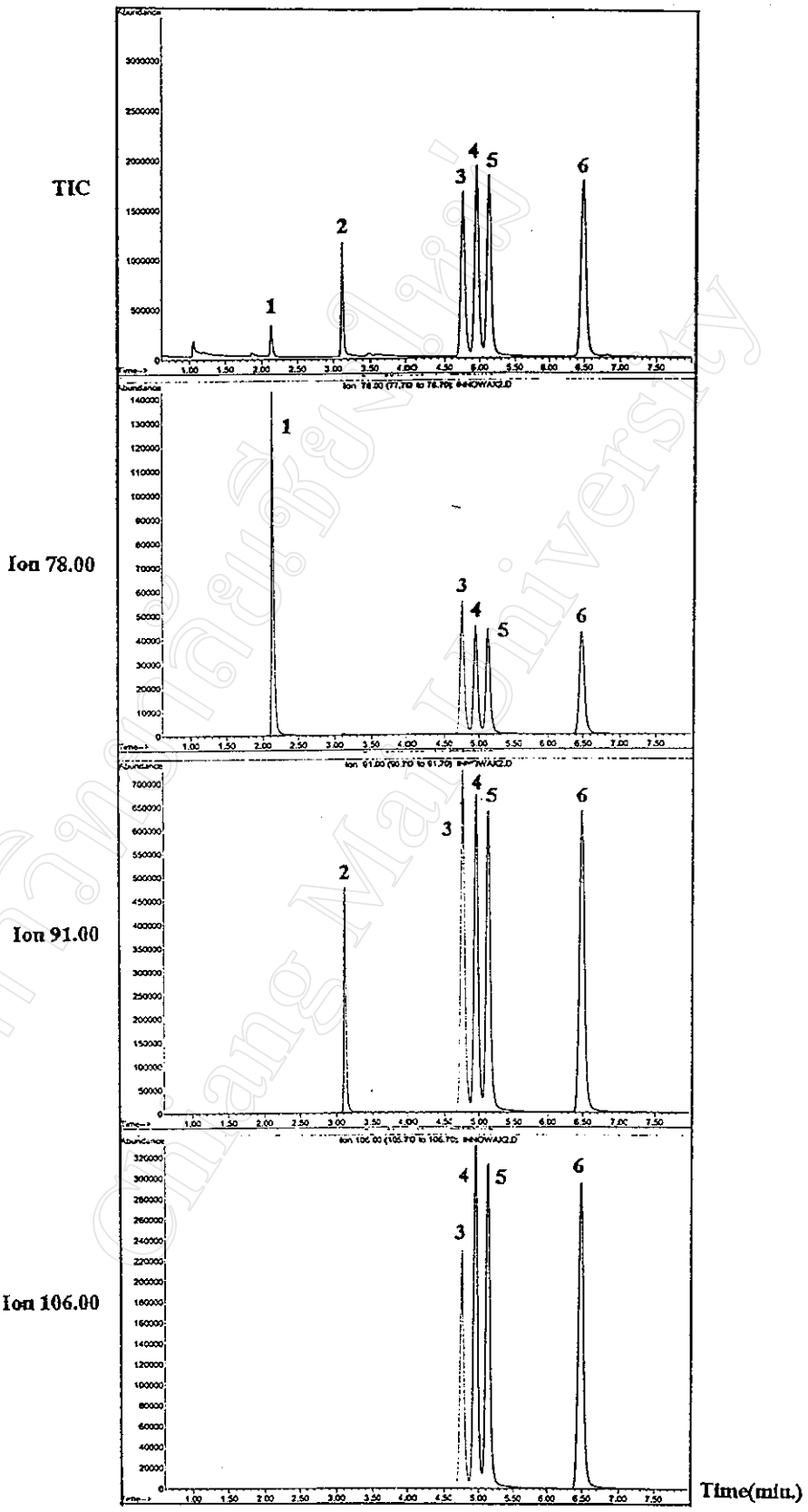

Figure 3.8 The total and single ion chromatograms of BTEX in aqueous solution with salt addition.

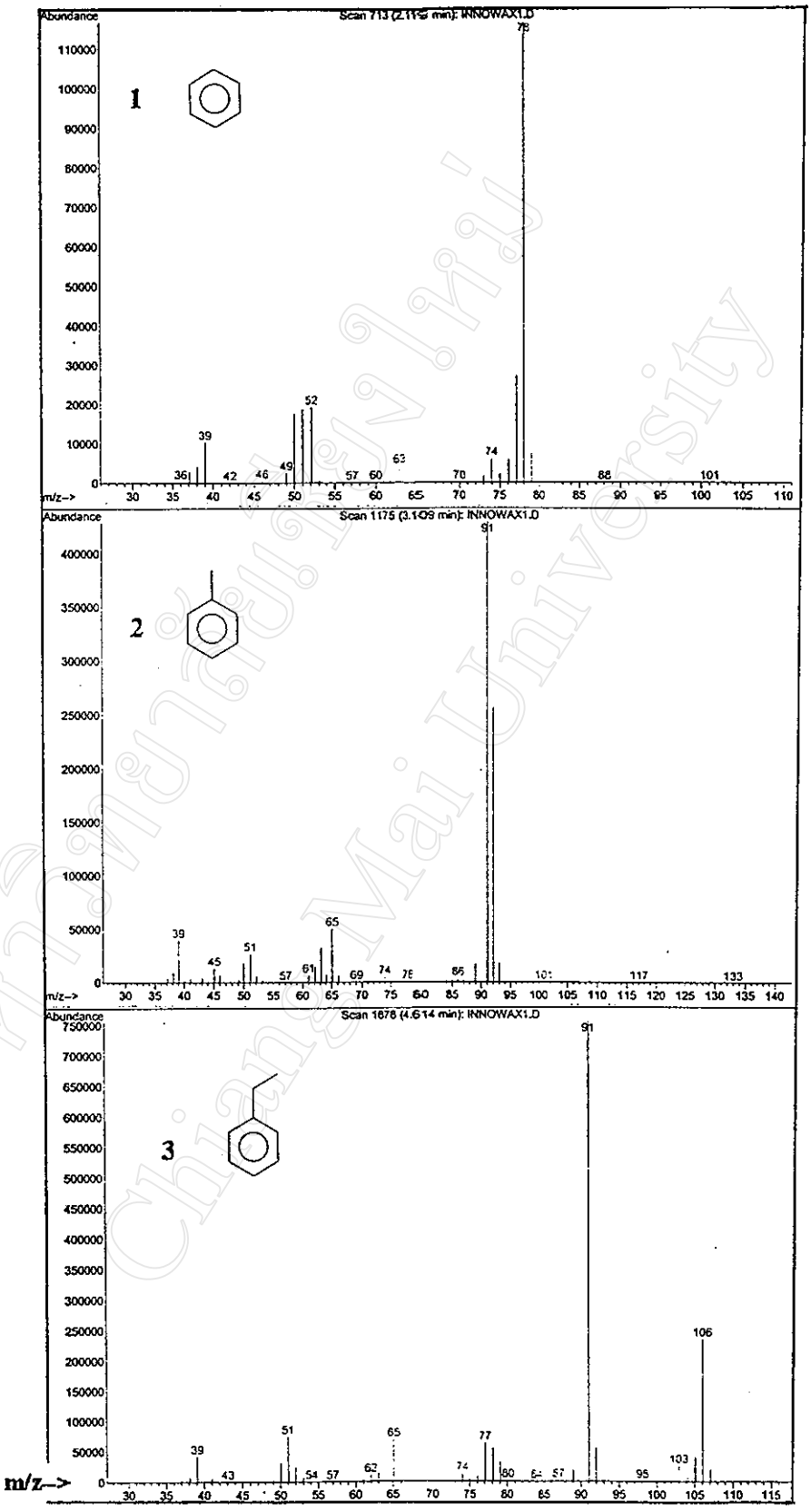

Figure 3.9 Mass spectra of 1) benzene 2) toluene and 3) ethylbenzene in aqueous solution with salt addition.

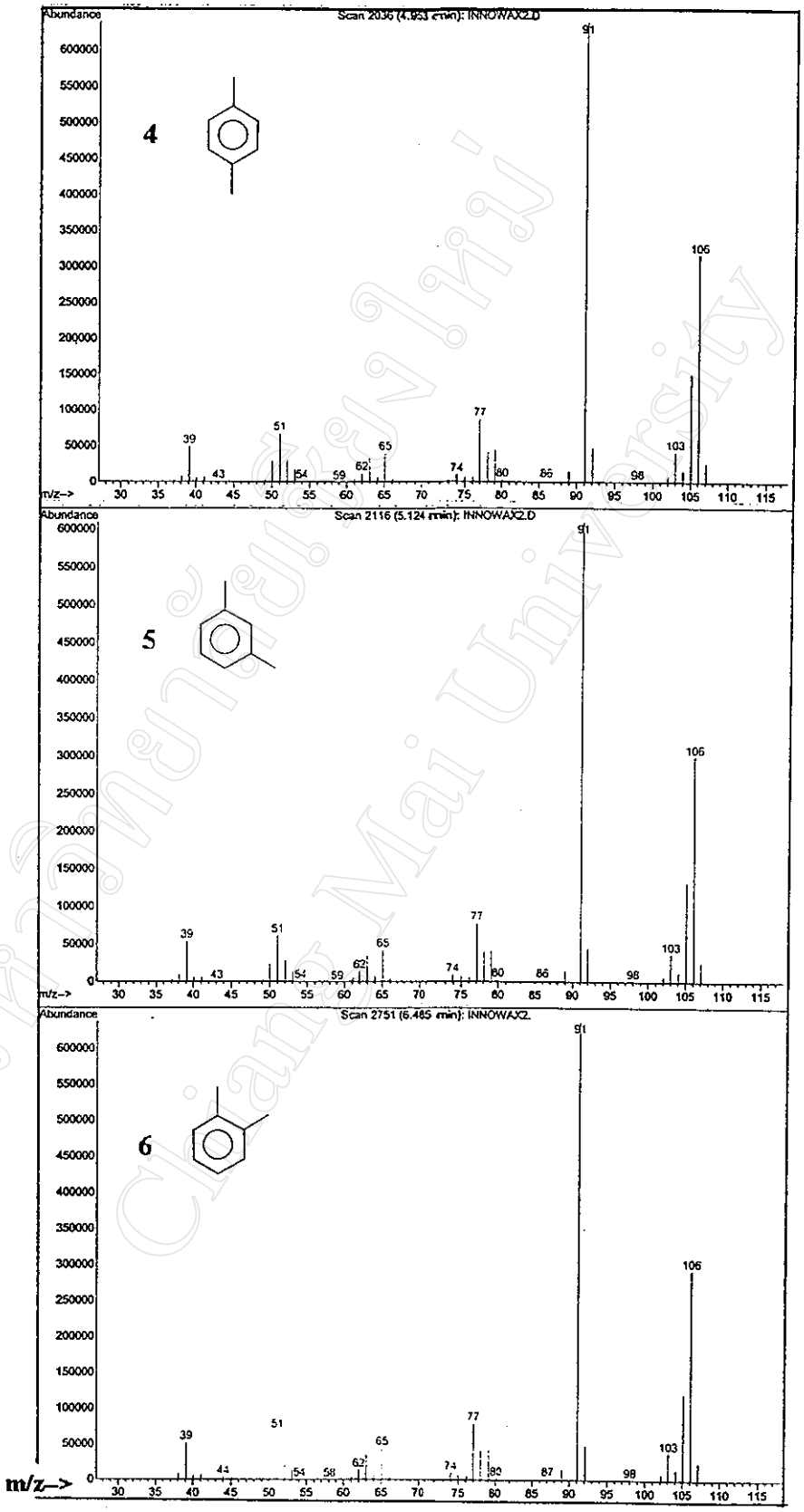

Figure 3.10 Mass spectra of 1) p-xylene 2) m-xylene and 3) o-xylene in aqueous solution without salt addition.

3.2 Optimization of Solid-Phase Microextraction

3.2.1 Comparison between SPME and HSSPME technique

The first step in development of the SPME method for determination of BTEX in aqueous sample, two exposure techniques in aqueous BTEX standard solution were investigated. The results of peak areas of BTEX obtained from comparison of headspace and direct immersion SPME procedures are shown in Table 3.3 and the histograms of these results are shown in Figure 3.11.

Table 3.3 Peak areas of BTEX obtained by SPME-GC FID and HSSPME-GC-FID (n=3)

Compound	Peak area (pA)	
	SPME	HSSPME
Benzene	62.2	62.2
Toluene	133.1	125.0
Ethylbenzene	312.2	312.7
p-Xylene	335.0	343.7
m-Xylene	359.7	366.5
o-Xylene	289.1	304.5

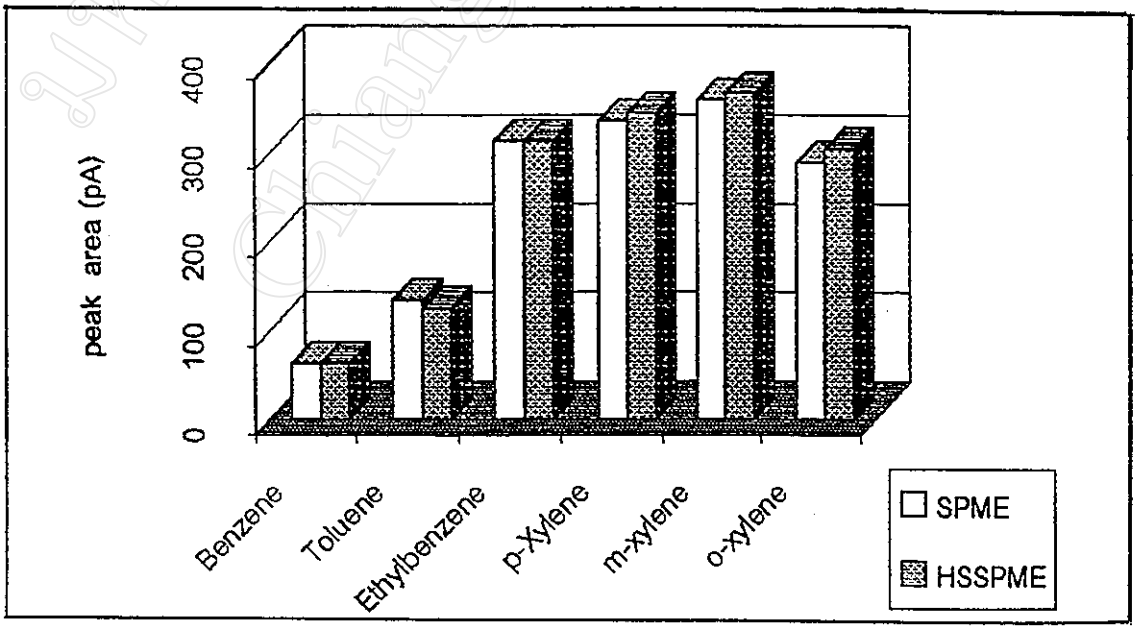

Figure 3.11 Comparison between the responses obtained by SPME-GC-FID

3.2.2 Determination of the optimal conditions for HSSPME

3.2.2.1 Study of stirring speed

Table 3.4 Effect of the stirring speed on BTEX determination by HSSPME-GC-FID (n=2).

Compound	Peak area (pA)					
	0%	10%	30%	50%	70%	80%
Benzene	24.2	46.4	63.0	66.9	72.1	75.8
Toluene	36.3	68.9	96.0	101.7	113.0	119.0
Ethylbenzene	81.3	158.1	223.7	244.0	280.0	301.3
p-Xylene	88.6	174.0	248.3	270.0	307.0	327.0
m-Xylene	90.8	177.0	250.0	274.1	311.1	334.7
o-Xylene	94.2	173.00	230.0	247.4	275.3	288.6

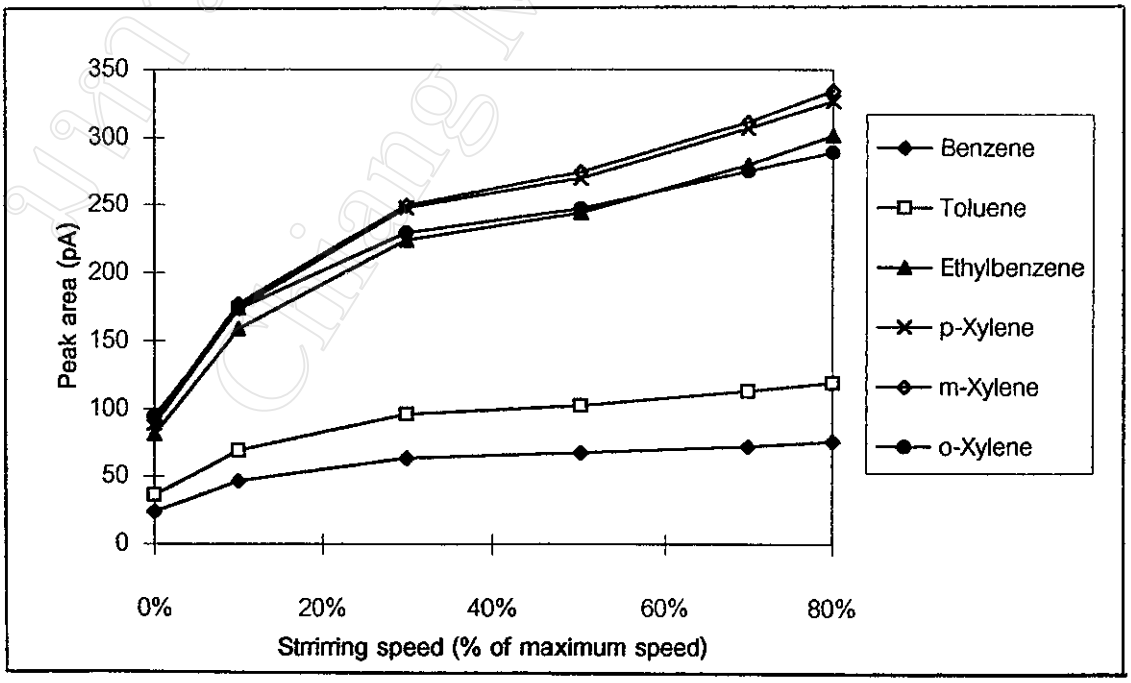

Figure 3.12 Effect of the strring speed on BTEX determination in water by HSSPME-GC-FID.

3.2.2.2 Study of the effect of added salt

Table 3.5 Effect of ionic strength on the determination of BTEX in waters by HSSPME-GC-FID (n=3).

Compound	Peak area (pA)	
	Without NaCl	30% NaCl
Benzene	62.2	84.9
Toluene	125.0	153.2
Ethylbenzene	312.7	361.5
p-Xylene	343.7	395.9
m-Xylene	366.5	469.8
o-Xylene	304.5	445.9

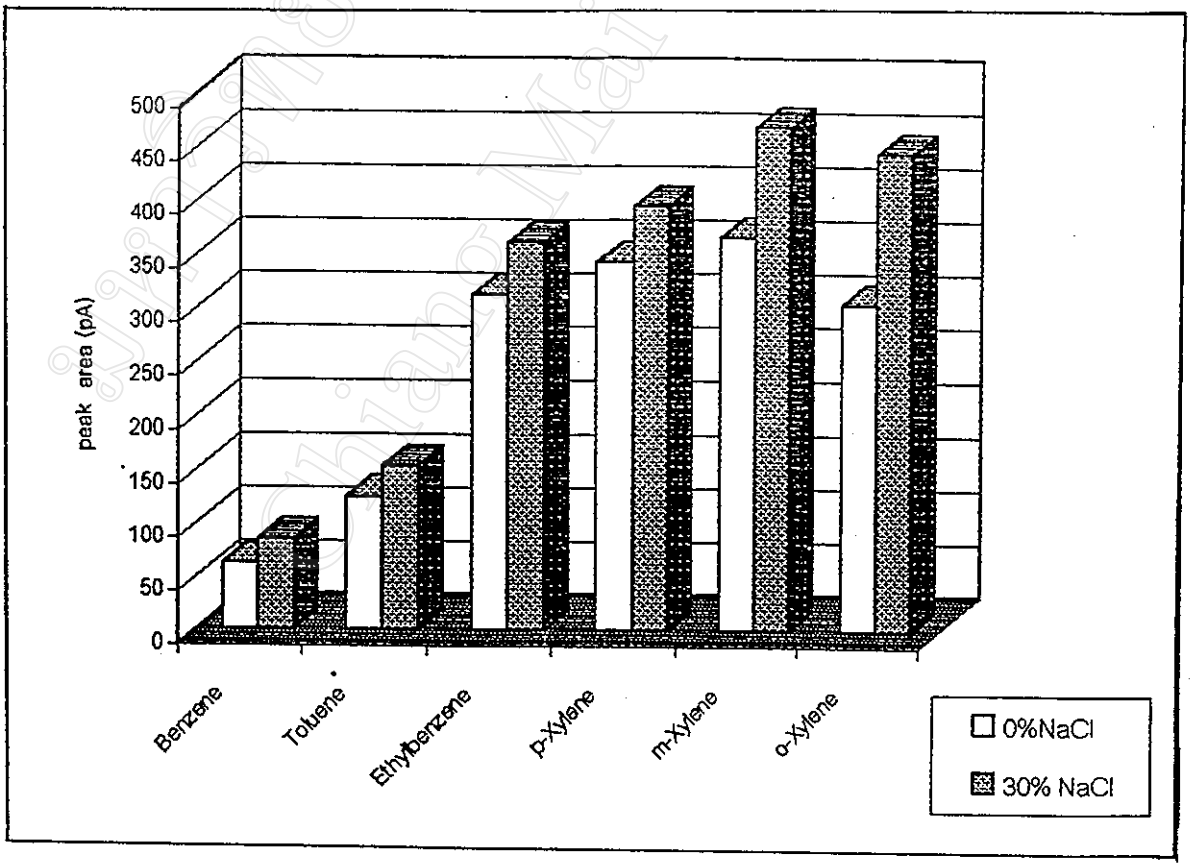

Figure 3.13 Effect of ionic strength on the determination of BTEX in waters by HSSPME-GC-FID (n=3).

3.2.2.3 Determination of adsorption-time profile

Table 3.6 Determination of adsorption-time profile for BTEX into a 100 µm polydimethylsiloxane HSSPME fiber by HSSPME-GC-FID (n=2).

Time (min)	Peak area (pA)					
	Benzene	Toluene	Ethylbenzene	p-Xylene	m-Xylene	o-Xylene
3	44.1	63.0	155.2	170.7	170.2	186.7
6	57.3	83.8	195.3	214.9	216.4	250.1
8	72.7	107.8	243.4	266.8	271.6	321.5
10	75.5	112.6	250.0	275.9	282.8	345.2
12	90.2	135.6	301.9	328.7	337.2	408.3
15	96.5	143.4	315.9	347.0	359.2	447.3
20	106.7	158.8	345.7	379.1	393.8	497.2

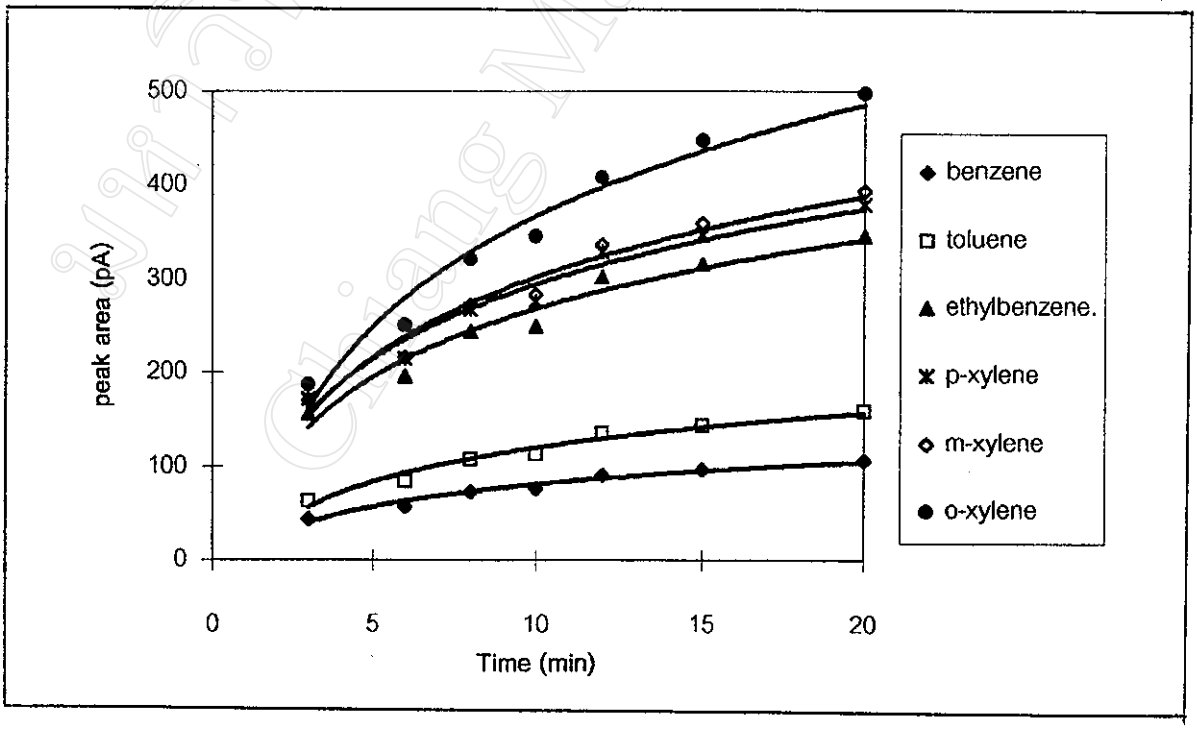

Figure 3.14 Adsorption time profile for BTEX into a 100 µm polydimethylsiloxane HSSPME fiber by HSSPME-GC-FID (n=2).

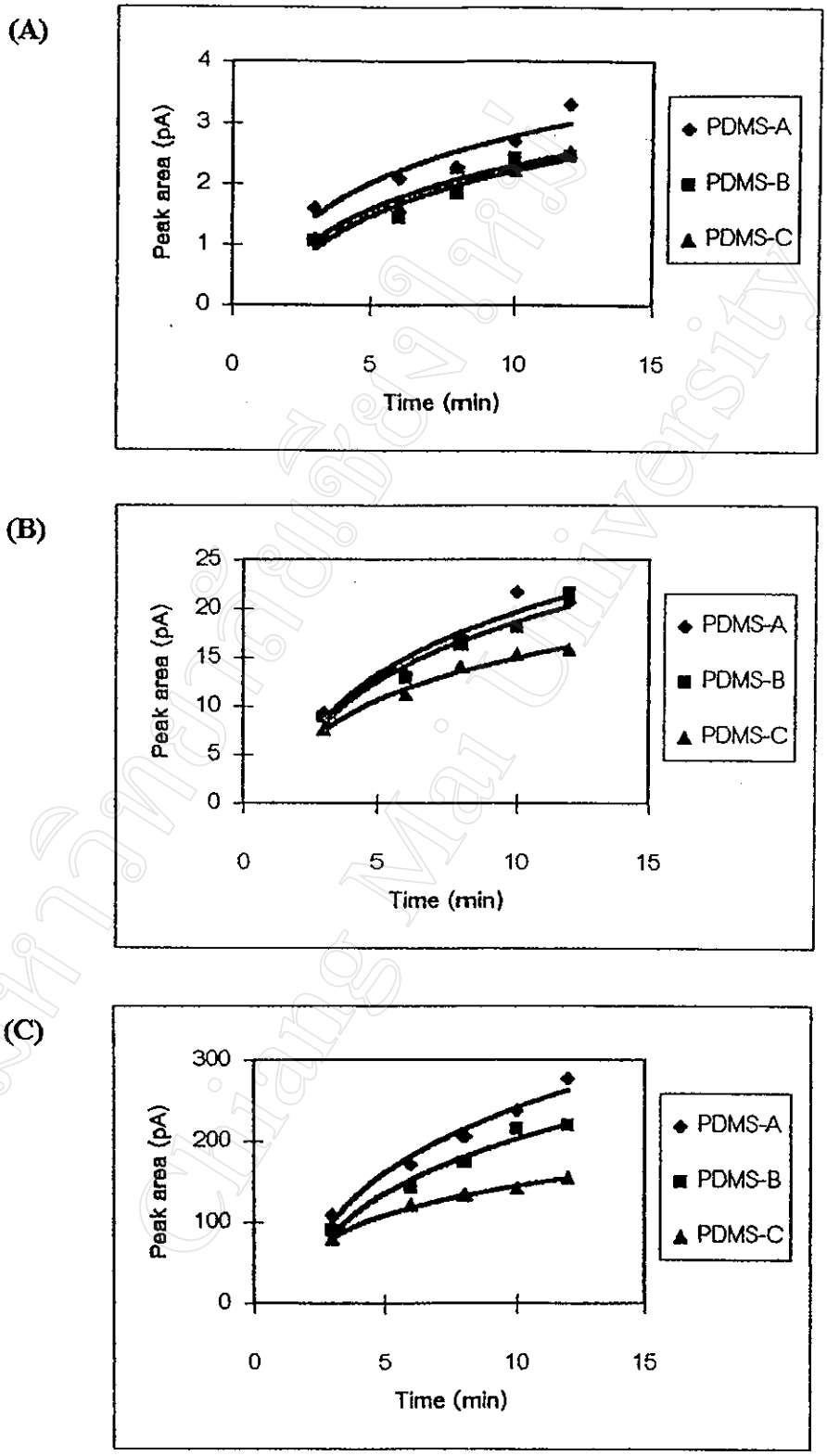

Figure 3.15 Adsorption time profile for benzene at concentration level of (A) 4 µg/L, (B) 40 µg/L and (C) 400 µg/L using 100 µm PDMS-A, 100 µm PDMS-B and 100 µm PDMS-C by HSSPME-GC-FID.

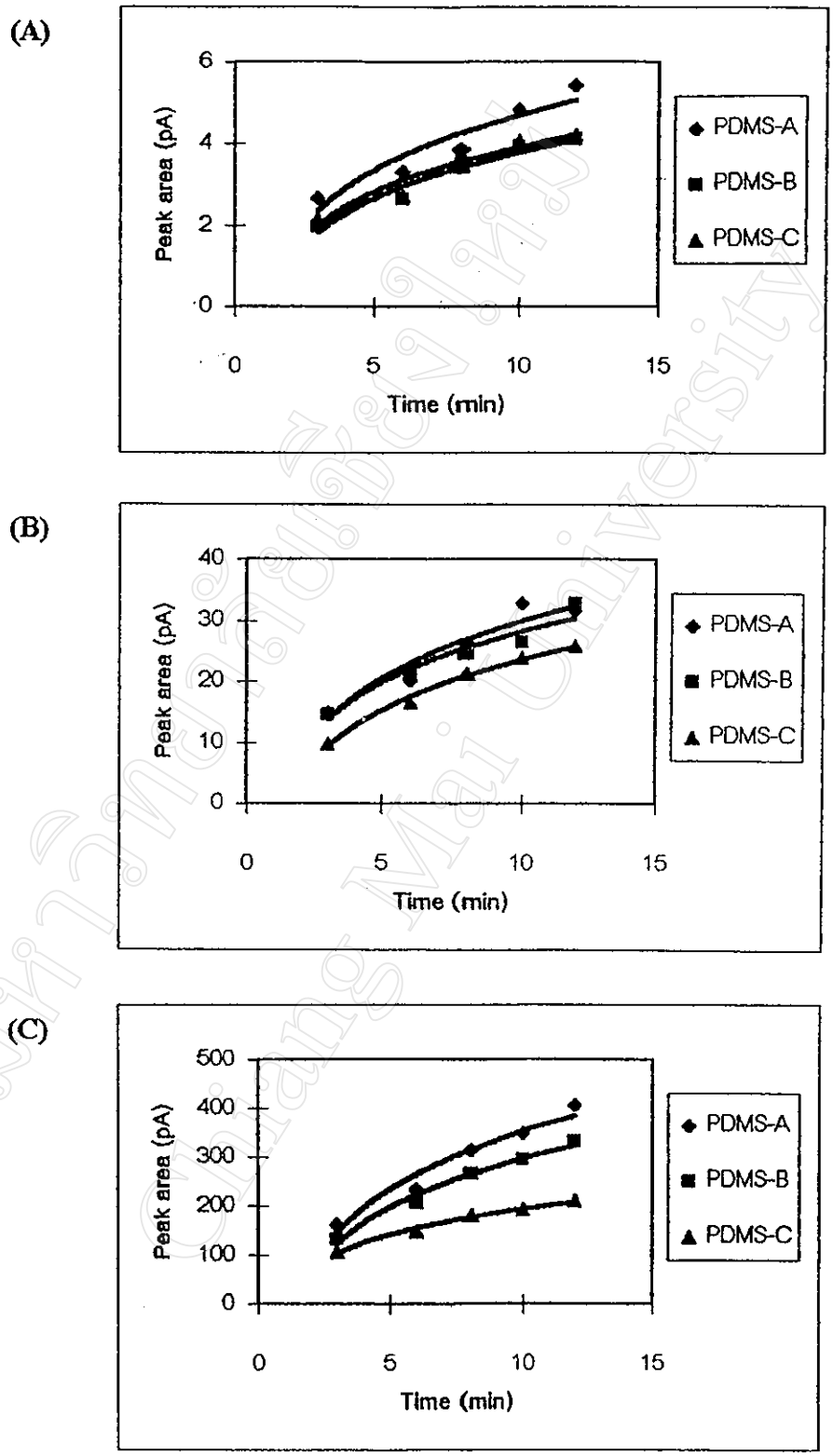

Figure 3.16 Adsorption time profile for toluene at concentration level of (A) 4 µg/L, (B) 40 µg/L and (C) 400 µg/L using 100 µm PDMS-A, 100 µm PDMS-B and 100 µm PDMS-C by HSSPME-GC-FID.

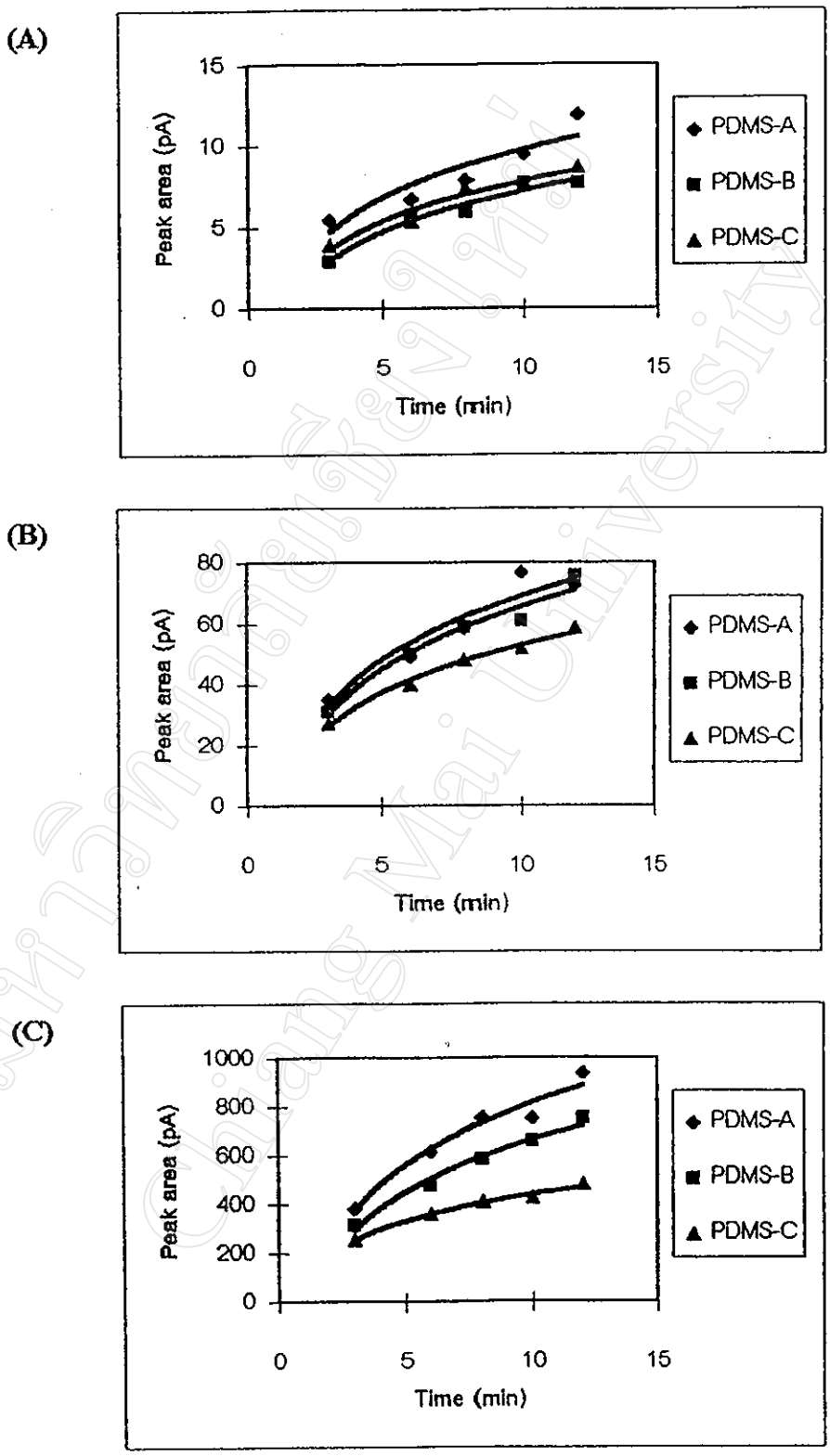

Figure 3.17 Adsorption time profile for ethylbenzene at concentration level of (A) 4 µg/L, (B) 40 µg/L and (C) 400 µg/L using 100 µm PDMS-A, 100 µm PDMS-B and 100 µm PDMS-C by HSSPME-GC-FID.

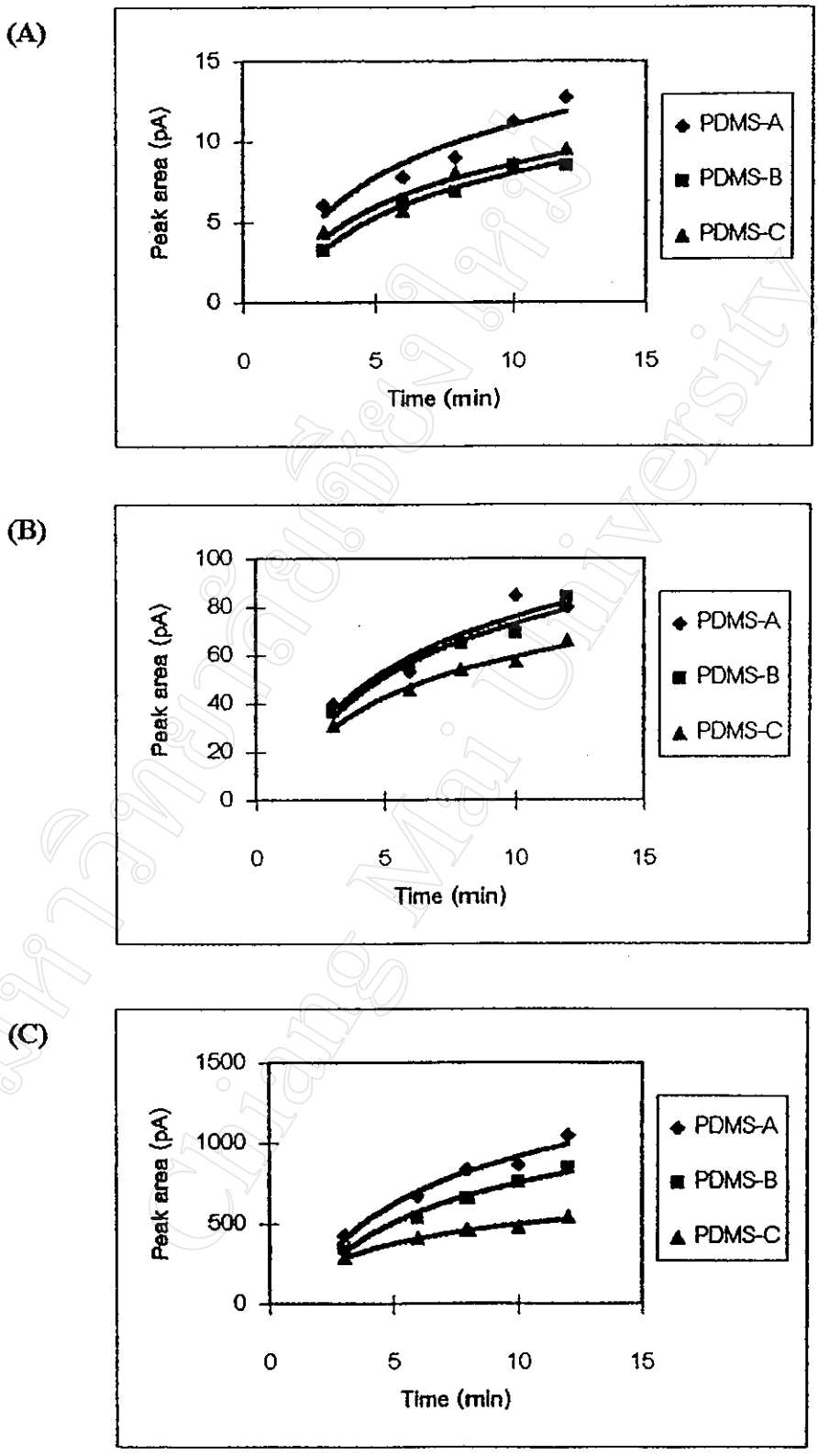

Figure 3.18 Adsorption time profile for p-xylene at concentration level of (A) 4 µg/L, (B) 40 µg/L and (C) 400 µg/L using 100 µm PDMS-A, 100 µm PDMS-B and 100 µm PDMS-C by HSSPME-GC-FID.

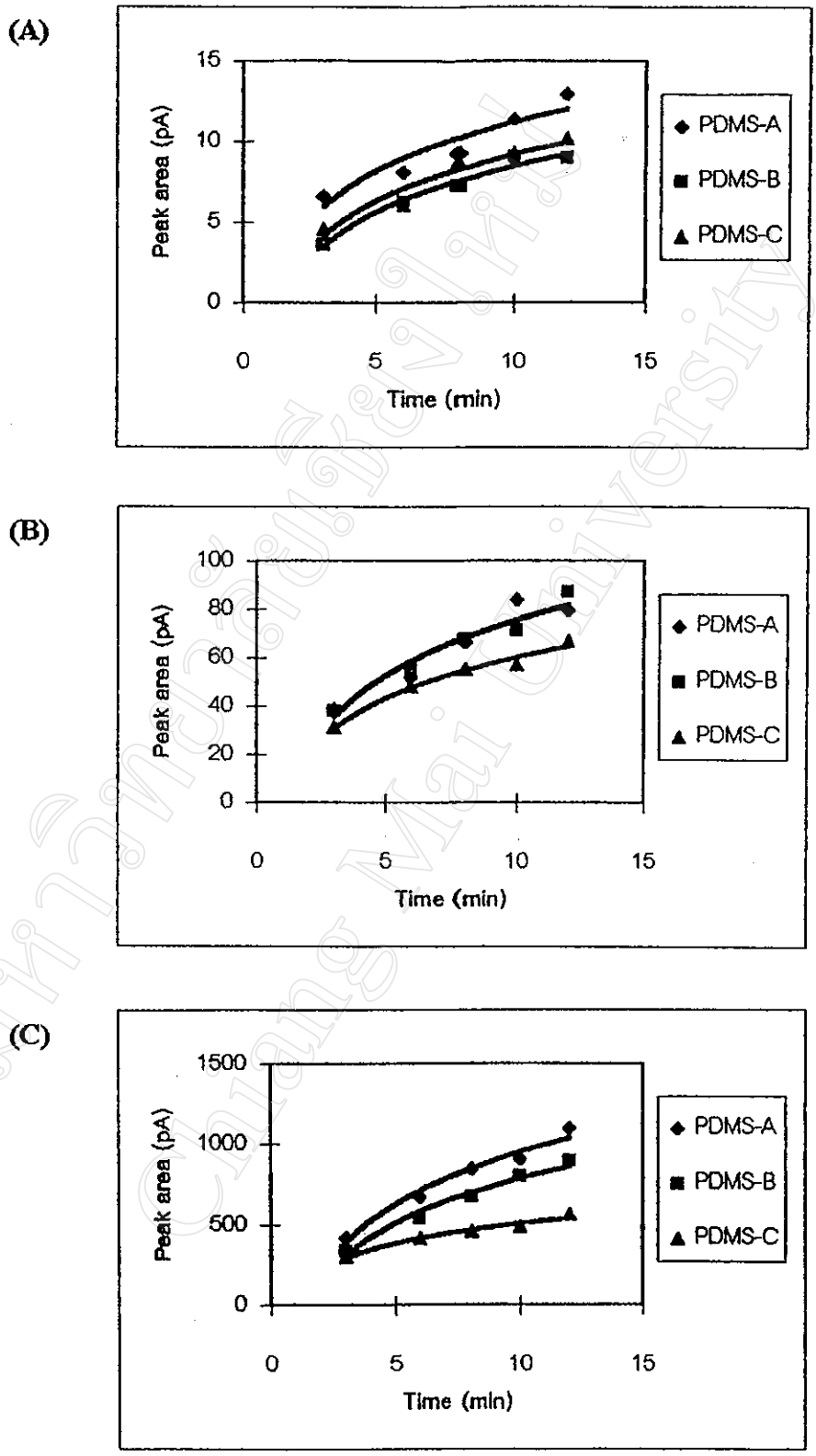

Figure 3.19 Adsorption time profile for m-xylene at concentration level of (A) 4 µg/L, (B) 40 µg/L and (C) 400 µg/L using 100 µm PDMS-A, 100 µm PDMS-B and 100 µm PDMS-C by HSSPME-GC-FID.

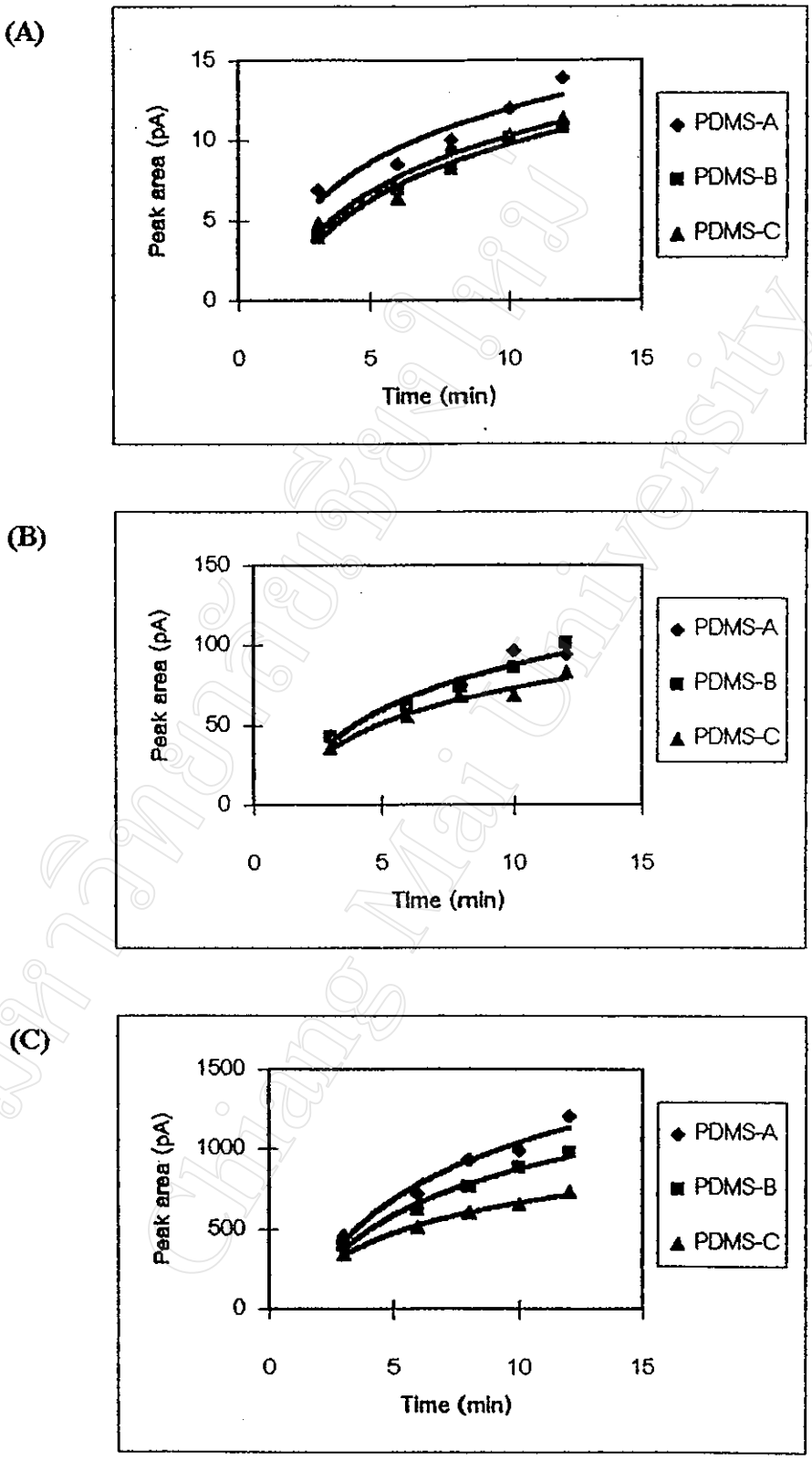

Figure 3.20 Adsorption time profile for o-xylene at concentration level of (A) 4 µg/L, (B) 40 µg/L and (C) 400 µg/L using 100 µm PDMS-A, 100 µm PDMS-B and 100 µm PDMS-C by HSSPME-GC-FID.

3.2.2.4 Determination of extraction-temperature profile

Table 3.7 Effect of temperature in the adsorption of BTEX into a 100 μm polydimethylsiloxane SPME fiber by HSSPME-GC-FID (n=2).

Temperature (°c)	Peak area (pA)					
	Benzene	Toluene	Ethylbenzene	p-Xylene	m-Xylene	o-Xylene
2	216.3	379.3	935.2	1025.7	1131.4	1126.1
20	150.0	245.2	598.5	658.6	715.3	769.7
24	128.8	212.6	493.2	546.0	597.3	671.3
31	110.3	178.6	408.8	456.0	505.6	568.0
40	86.6	136.2	272.0	326.2	329.8	351.8

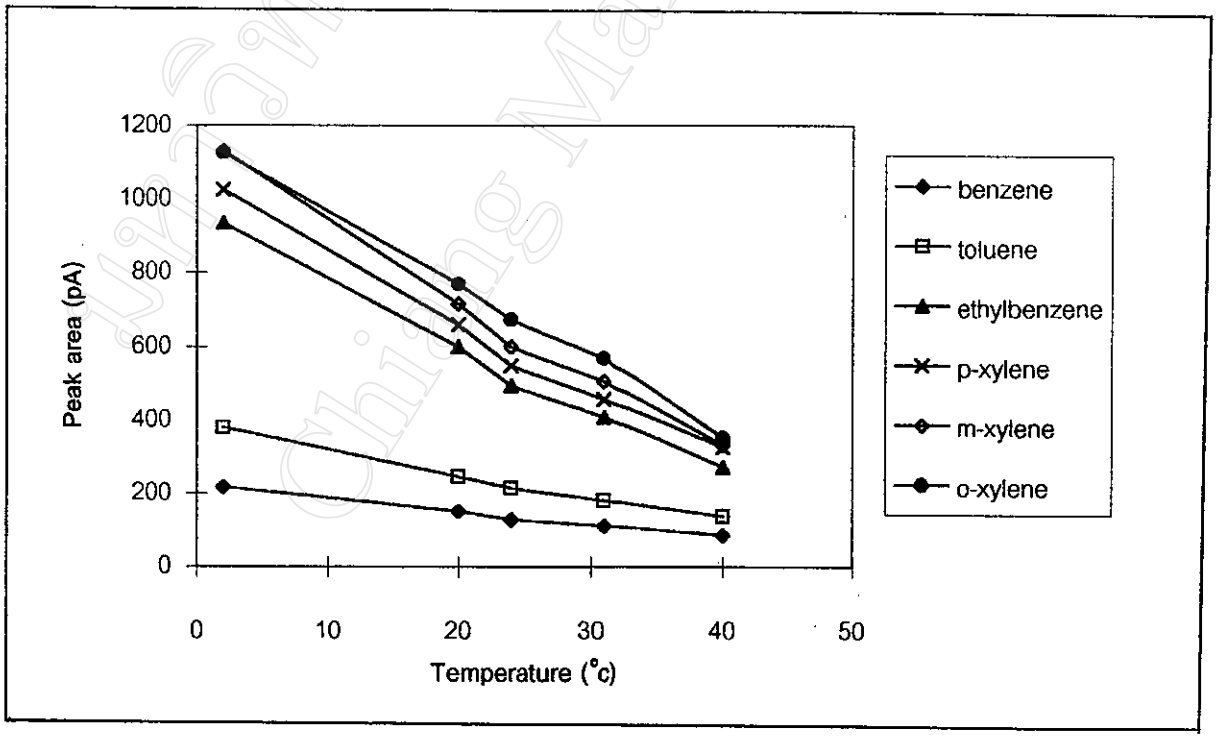

Figure 3.21 Effect of temperature in the adsorption of BTEX into a 100 μm polydimethylsiloxane SPME fiber by HSSPME-GC-FID.

3.2.3 Summary of the optimized Solid-Phase Microextraction conditions

The optimized solid-phase microextraction conditions established in this work are summarized in table 3.8.

Table 3.8 Optimized solid-phase microextraction conditions for analysis of BTEX in water samples.

Operation	Optimal extraction conditions
1. Extraction sampling mode	Headspace SPME mode (HSSPME)
2. Headspace vial size	22 ml
3. Sample Volume	10 ml
4. Sample preparation	3.0 g NaCl was added to 10 ml of sample solution
5. Speed of agitation system	70% of maximum speed
6. Extraction temperature	At ambient (24-25 °C)
7. Adsorption time	10 minutes
8. Desorption time	2 minutes
9. Desorption temperature	200 °C

3.3 Detection Limits , Linearity and Precision Study

3.3.1 Detection limits

The detection limits for BTEX compounds in waters were selected based on the signal-to-noise (S/N) ratio of a minimum 3:1 in three replicates. The results are shown in table 3.9.

Table 3.9 The detection limits of BTEX compounds in water analysis.

Compound	LOD (μg/L)	Mean S/N
Benzene	1.0	3.0
Toluene	0.5	3.3
Ethylbenzene	0.5	3.3
p-Xylene	0.5	3.6
m-Xylene	0.5	3.3
o-Xylene	0.5	3.8

3.3.2 Results on linearity study

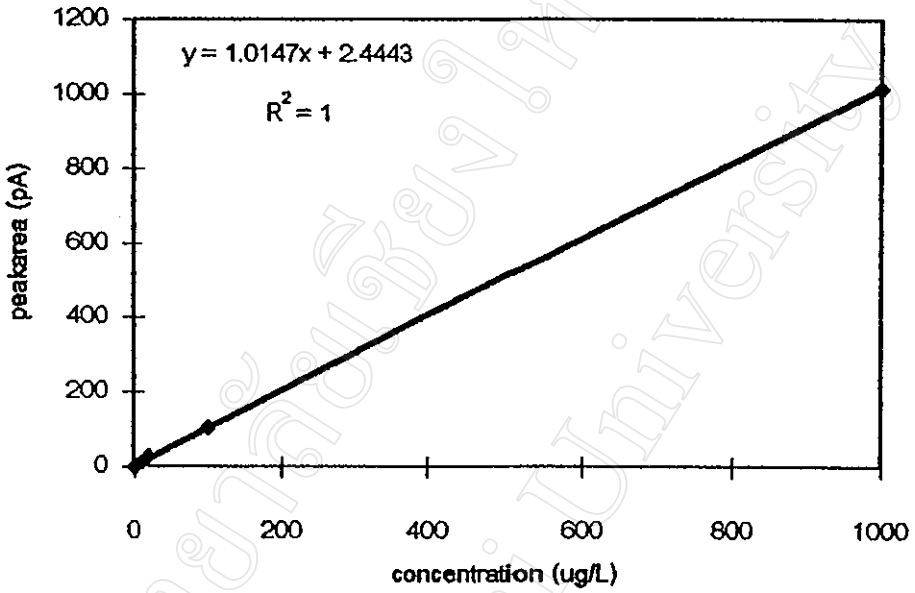

Figure 3.22 Calibration curve of benzene in the range 2-2000 µg/L

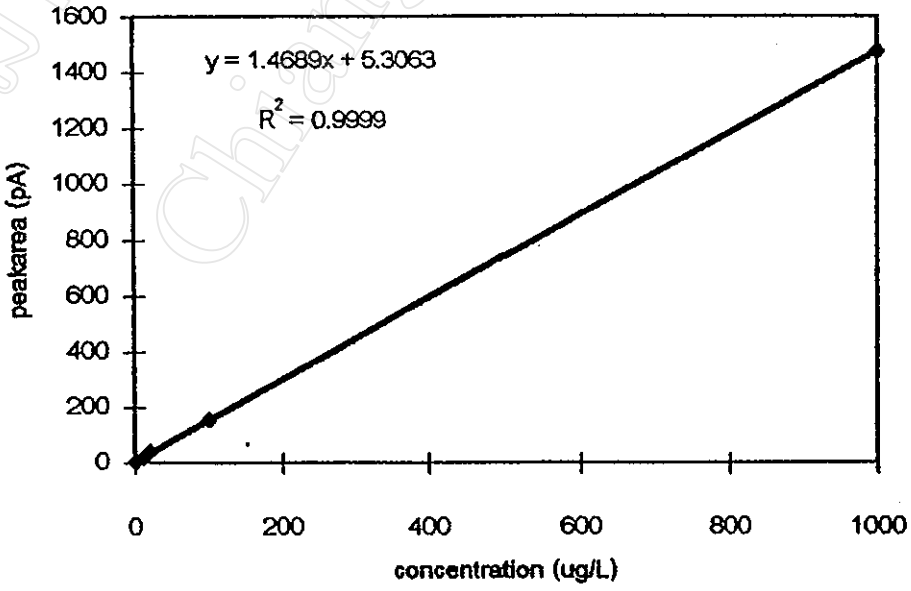

Figure 3.23 Calibration curve of toluene in the range 1-1,000 µg/L

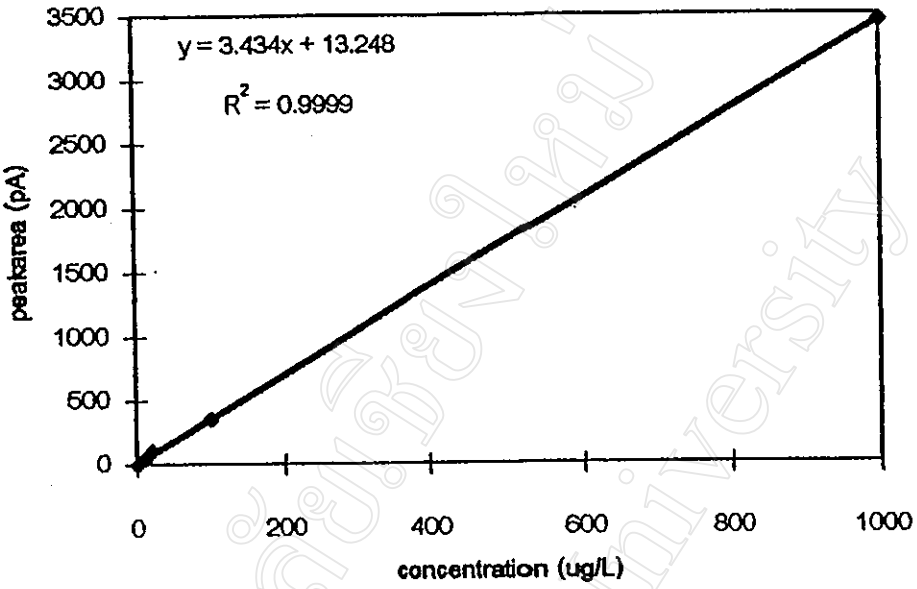

Figure 3.24 Calibration curve of ethylbenzene in the range 1-1,000 µg/L

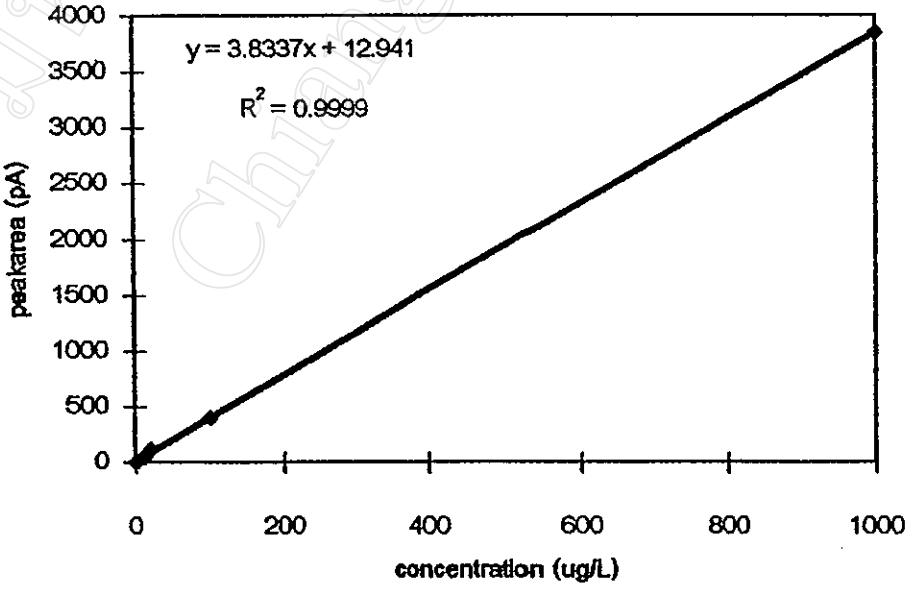

Figure 3.25 Calibration curve of p-xylene in the range 1-1,000 µg/L

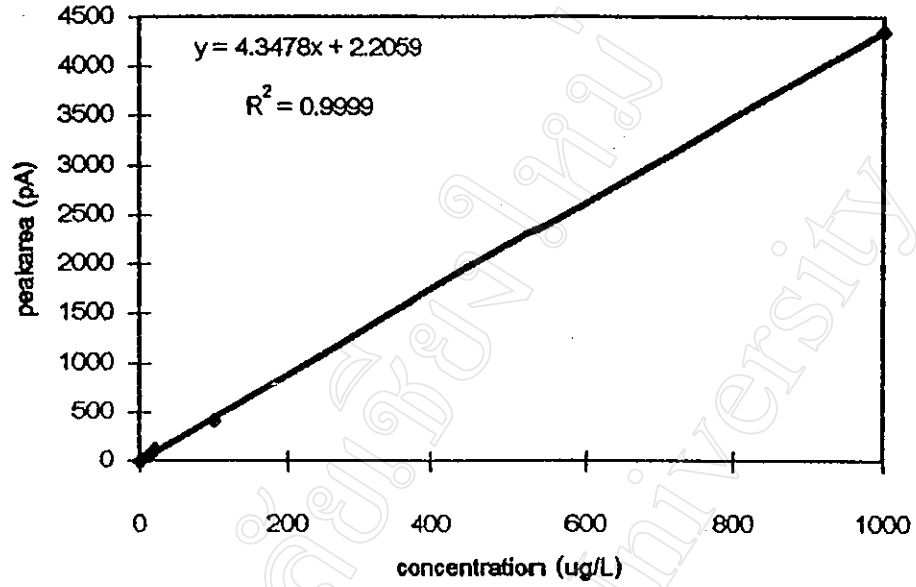

Figure 3.26 Calibration curve of m-xylene in the range 1-1,000 µg/L

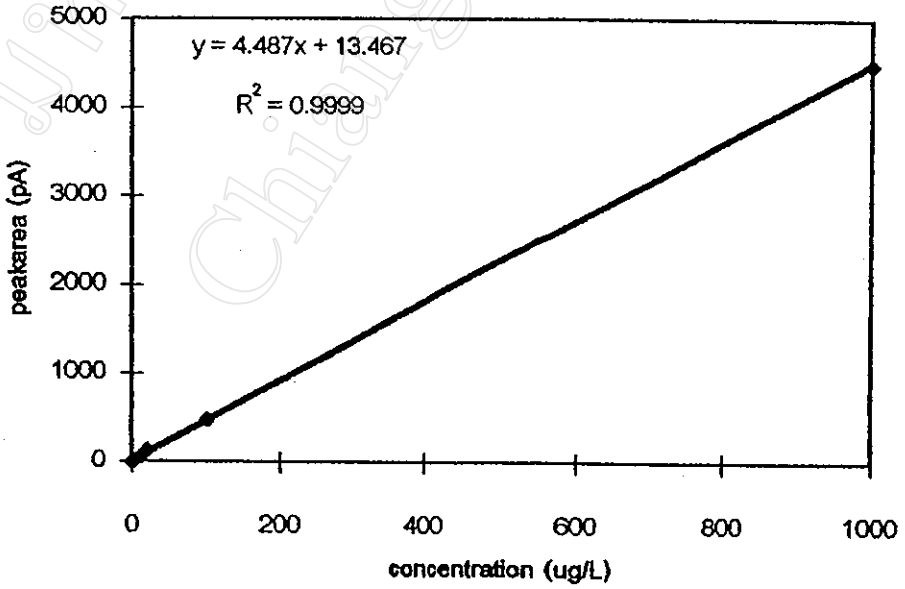

Figure 3.27 Calibration curve of o-xylene in the range 1-1,000 µg/L

Table 3.10 Summary of detection limits, the linearity regression and precision for the determination of BTEX compounds in ultrapure water.

Compounds	Regression equation	Correlation coefficient (R^2)	Concentration(µg/L)	Limit of detection (µg/L)	Precision (%R.S.D.)
Benzene	Y=1.0147X+2.4443	1.0000	2-2,000	1.0	[a]3.7
Toluene	Y=1.4689X+5.3063	0.9999	1-1,000	0.5	[b]7.6
Ethylbenzene	Y=3.434X+13.248	0.9999	1-1,000	0.5	[b]8.0
p-Xylene	Y=3.8337X+12.941	0.9999	1-1,000	0.5	[b]9.3
m-Xylene	Y=4.3478X+2.2059	0.9999	1-1,000	0.5	[b]8.0
o-Xylene	Y=4.487X+13.467	0.9999	1-1,000	0.5	[b]6.4

[a]Six replicates at 4.0 µg/L level

[b]Six replicates at 2.0 µg/L level

3.4 Recovery Assay

Table 3.11 Spiking at low level, estimated concentrations, % recoveries with standard deviations and relative standard deviations of spiked water from the gasoline station, based on three determinations.

Compound	Spiked (µg/L)	Extracted (µg/L)	Recovery (% ± S.D)	% R.S.D
Benzene	6.50	6.57	102 ± 0.07	3.8
Toluene	3.50	2.90	83 ± 0.48	8.8
Ethylbenzene	3.50	3.41	97 ± 0.36	3.8
p-Xylene	3.50	3.45	99 ± 0.15	1.5
m-Xylene	3.50	3.41	97 ± 0.33	2.9
o-Xylene	3.50	3.47	99 ± 0.22	2.0

Table 3.12 Spiking at low level, estimated concentrations, % recoveries with standard deviations and relative standard deviations of spiked industrial effluent, based on three determinations.

Compound	Spiked (µg/L)	Extracted (µg/L)	Recovery (% ± S.D)	% R.S.D
Benzene	6.50	6.52	101 ± 0.01	0.4
Toluene	3.50	2.72	78 ± 0.48	9.6
Ethylbenzene	3.50	3.48	100 ± 0.37	3.8
p-Xylene	3.50	3.55	102 ± 0.43	4.2
m-Xylene	3.50	3.54	101 ±1.08	9.0
o-Xylene	3.50	3.69	106 ± 0.36	3.1

Table 3.13 Spiking at low level, estimated concentrations, % recoveries with standard deviations and relative standard deviations of spiked domestic wastewater, based on three determinations.

Compound	Spiked (µg/L)	Extracted (µg/L)	Recovery (% ± S.D)	% R.S.D
Benzene	6.50	7.50	116 ± 0.28	12.9
Toluene	3.50	2.15	61 ± 0.63	6.7
Ethylbenzene	3.50	3.24	92 ± 0.41	2.8
p-Xylene	3.50	3.22	92 ± 0.92	6.2
m-Xylene	3.50	3.11	89 ± 0.33	1.9
o-Xylene	3.50	3.58	102 ± 0.58	4.2

Table 3.14 Spiking at low level, estimated concentrations, % recoveries with standard deviations and relative standard deviations of spiked natural surface water, based on three determinations.

Compound	Spiked (µg/L)	Extracted (µg/L)	Recovery (% ± S.D)	% R.S.D
Benzene	6.50	6.55	101 ± 0.12	6.5
Toluene	3.50	2.68	77 ± 0.64	13.1
Ethylbenzene	3.50	3.63	104 ± 0.49	4.9
p-Xylene	3.50	3.66	104 ± 0.51	4.9
m-Xylene	3.50	3.69	106 ± 0.45	3.6
o-Xylene	3.50	3.67	106 ± 0.78	6.8

Table 3.15 Spiking at medium level, estimated concentrations, % recoveries with standard deviations and relative standard deviations of spiked industrial effluent, based on three determinations.

Compound	Spiked (µg/L)	Extracted (µg/L)	Recovery (% ± S.D)	% R.S.D
Benzene	100	93	93 ± 0.48	1.3
Toluene	50	46	91 ± 1.55	2.8
Ethylbenzene	50	47	95 ± 4.48	3.3
p-Xylene	50	47	94 ± 4.38	2.9
m-Xylene	50	47	94 ± 5.31	3.5
o-Xylene	50	46	92 ± 3.79	2.3

Table 3.16 Spiking at medium level, estimated concentrations, % recoveries with standard deviations and relative standard deviations of spiked domestic wastewater, based on three determinations.

Compound	Spiked (µg/L)	Extracted (µg/L)	Recovery (% ± S.D)	% R.S.D
Benzene	100	72	72 ± 1.97	6.9
Toluene	50	35	70 ± 3.84	9.3
Ethylbenzene	50	37	73 ± 6.84	6.6
p-Xylene	50	37	73 ± 8.43	7.5
m-Xylene	50	37	73 ± 11.8	10.2
o-Xylene	50	36	73 ± 10.3	8.1

Table 3.17 Spiking at high level, estimated concentrations, % recoveries with standard deviations and relative standard deviations of spiked industrial effluent, based on three determinations.

Compound	Spiked (µg/L)	Extracted (µg/L)	Recovery (% ± S.D)	% R.S.D
Benzene	1,000	732	73 ± 12.0	3.7
Toluene	500	378	76 ± 21.7	4.4
Ethylbenzene	500	388	78 ± 30.6	2.5
p-Xylene	500	392	78 ± 40.3	3.0
m-Xylene	500	405	81 ± 53.5	3.8
o-Xylene	500	401	80 ± 71.0	4.6

Table 3.18 Spiking at high level, estimated concentrations, % recoveries with standard deviations and relative standard deviations of spiked domestic wastewater, based on three determinations.

Compound	Spiked (µg/L)	Extracted (µg/L)	Recovery (% ± S.D)	% R.S.D
Benzene	1,000	831	83 ± 7.27	2.0
Toluene	500	411	82 ± 6.70	1.2
Ethylbenzene	500	420	84 ± 23.8	1.8
p-Xylene	500	425	85 ± 19.1	1.3
m-Xylene	500	430	86 ± 45.3	3.0
o-Xylene	500	438	88 ± 65.5	3.9

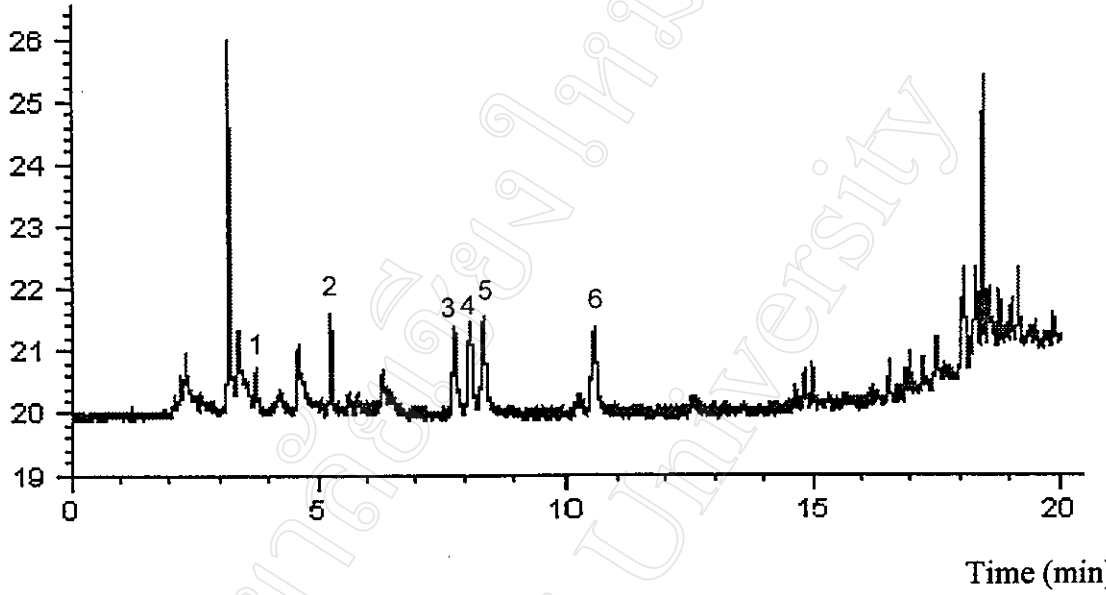

Figure 3.28 GC-FID chromatogram of spiked water from the gasoline station at low level concentration. Peak as in Figure 3.3.

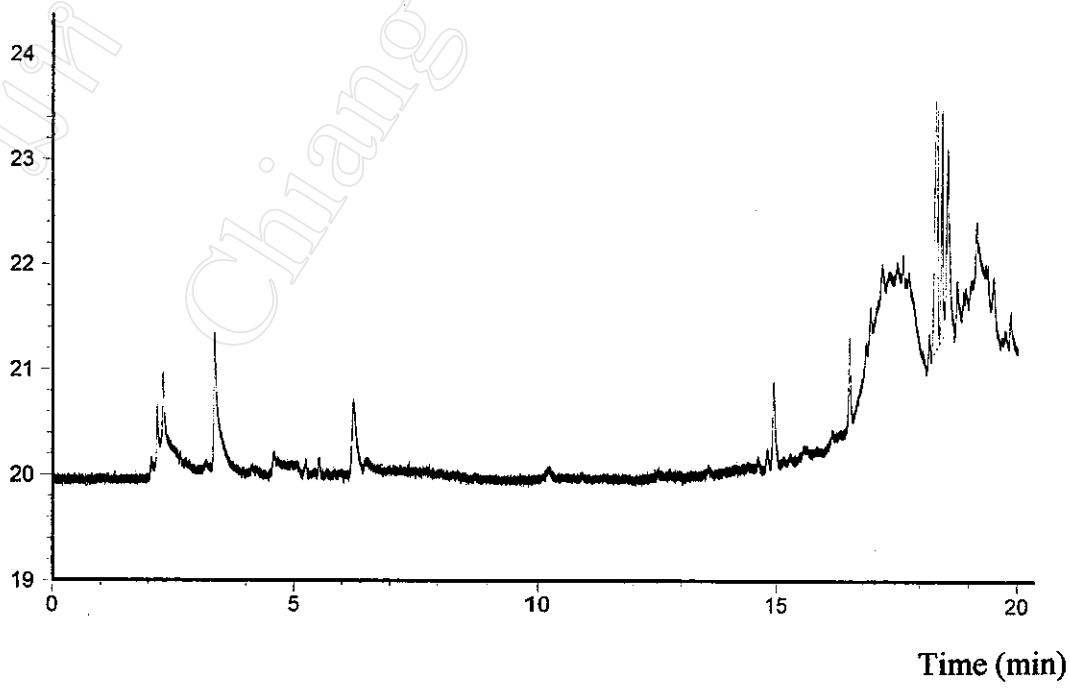

Figure 3.29 GC-FID chromatogram of nonspiked water from the gasoline station at low level concentration.

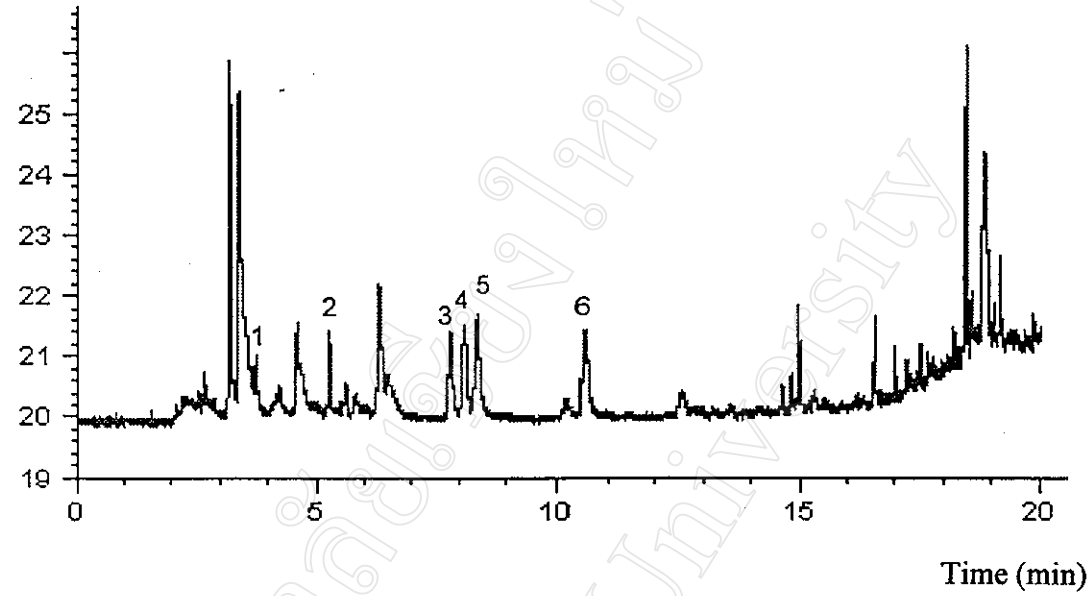

Figure 3.30 GC-FID chromatogram of spiked industrial effluent at low level concentration. Peak as in Figure 3.3.

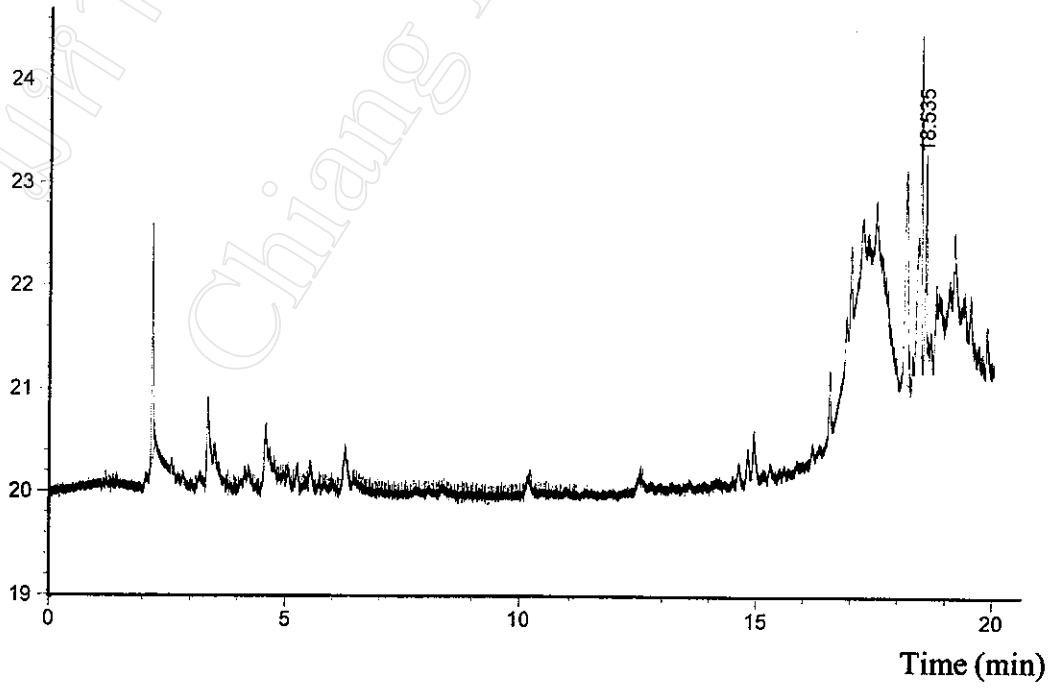

Figure 3.31 GC-FID chromatogram of nonspiked industrial effluent at low level concentration. Peak as in Figure 3.3.

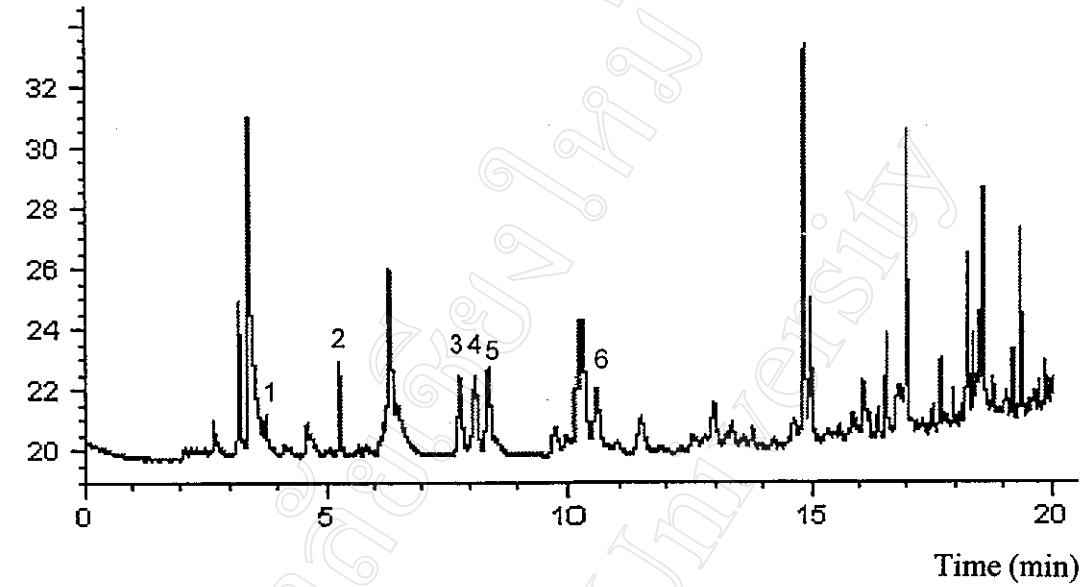

Figure 3.32 GC-FID chromatogram of spiked domestic wastewater at low level concentration. Peak as in Figure 3.3.

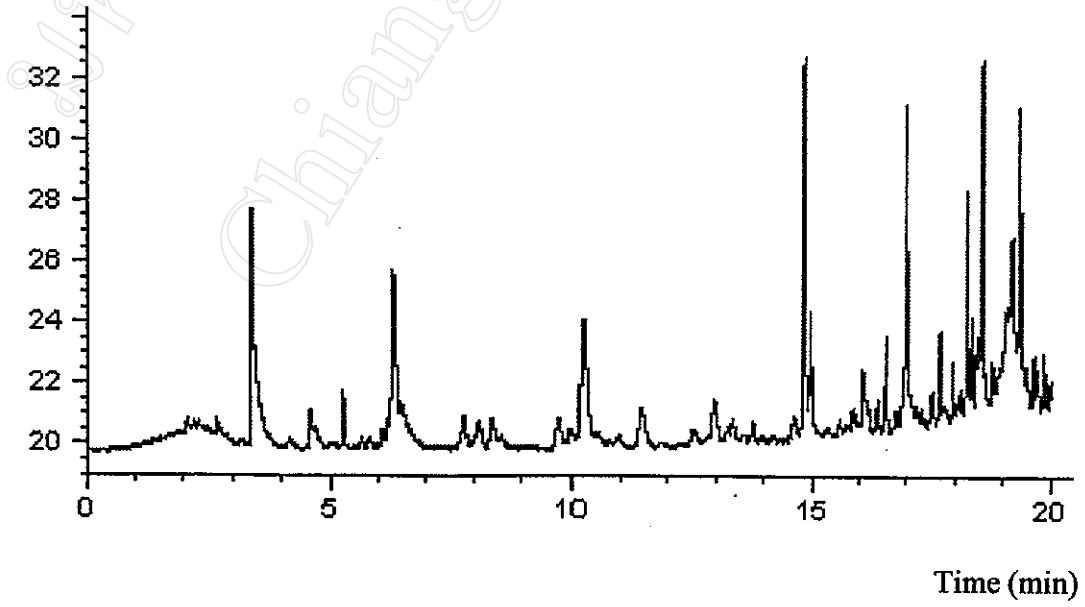

Figure 3.33 GC-FID chromatogram of nonspiked domestic wastewater at low level concentration. Peak as in Figure 3.3.

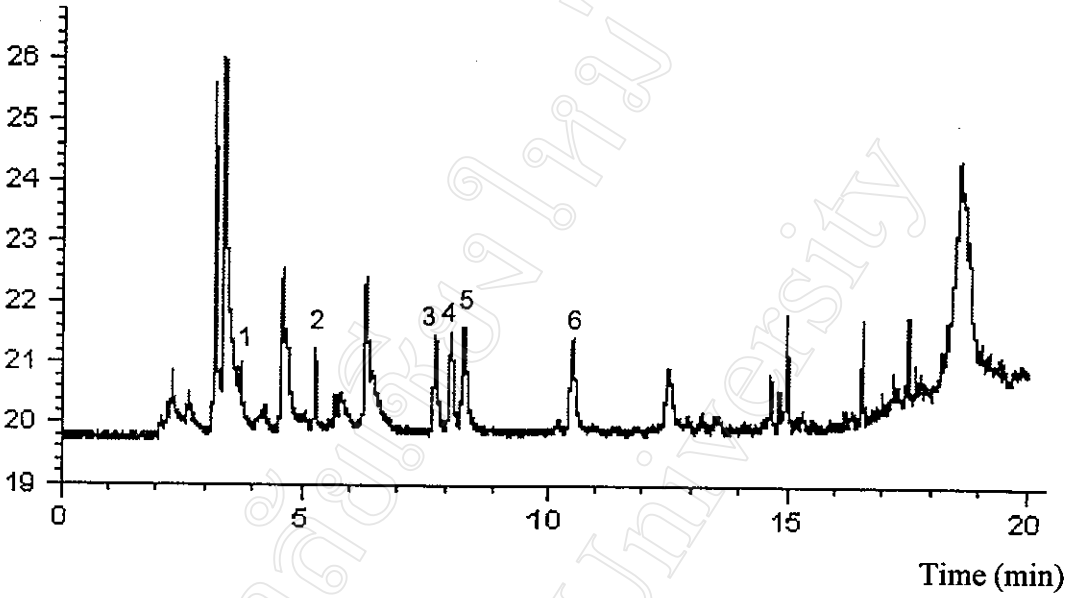

Figure 3.34 GC-FID chromatogram of spiked natural surface water at low level concentration. Peak as in Figure 3.3.

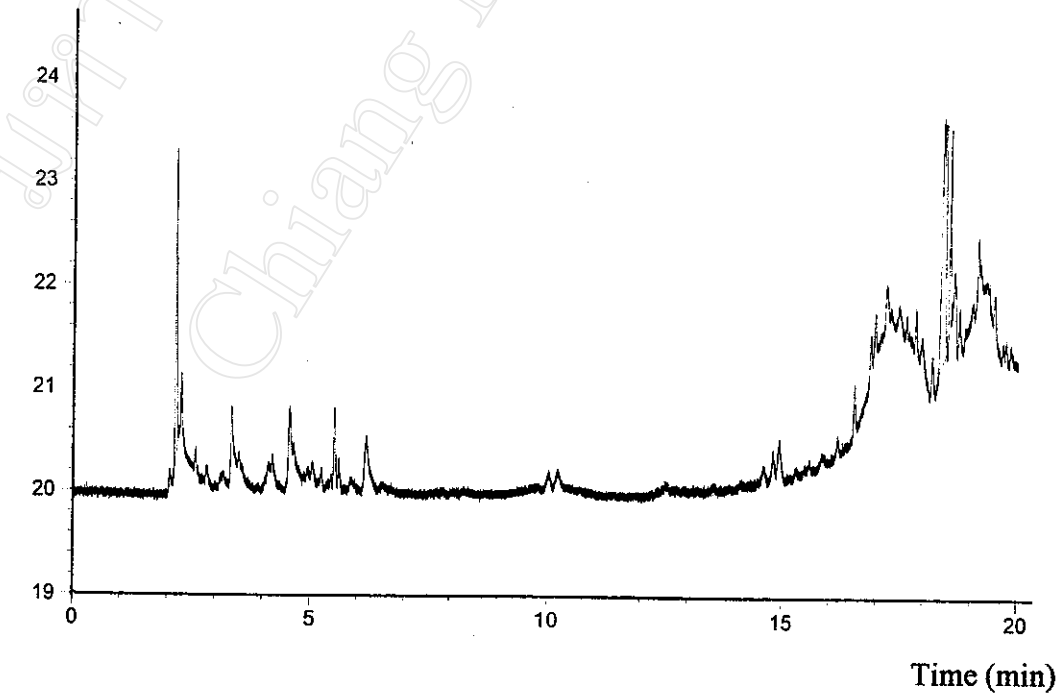

Figure 3.35 GC-FID chromatogram of nonspiked natural surface water at low level concentration.

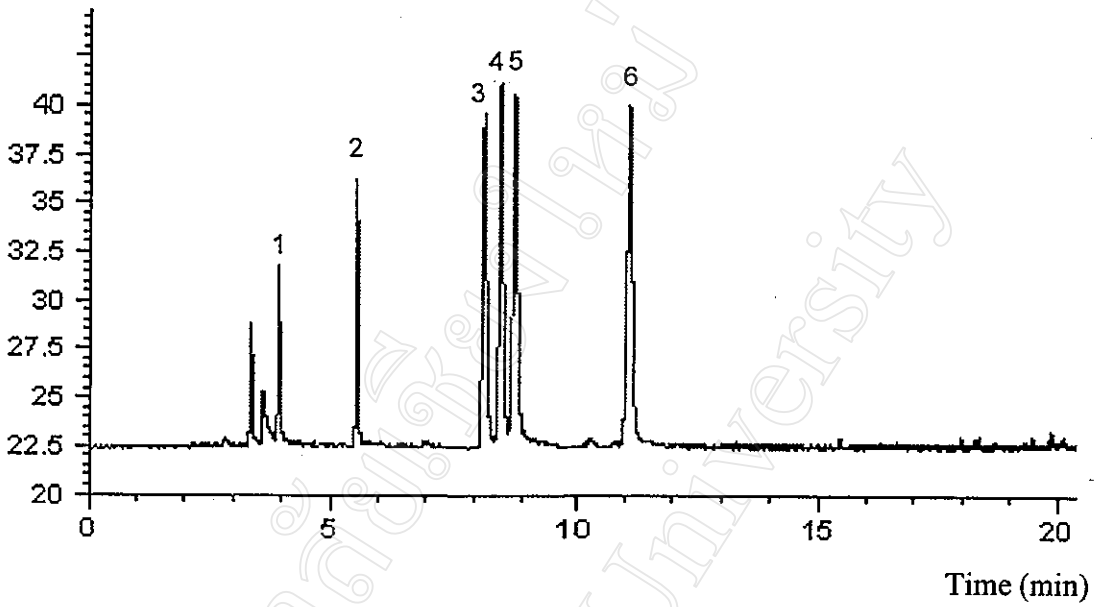

Figure 3.36 GC-FID chromatogram of spiked industrial effluent at medium level concentration. Peak as in Figure 33.

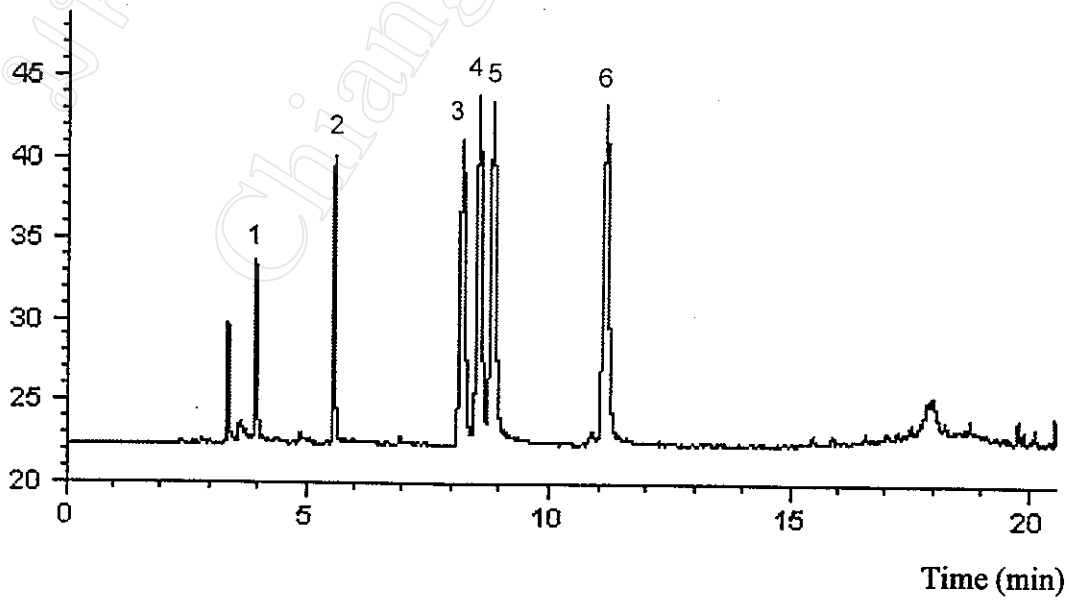

Figure 3.37 GC-FID chromatogram of spiked domestic wastewater at medium level concentration. Peak as in Figure 3.3.

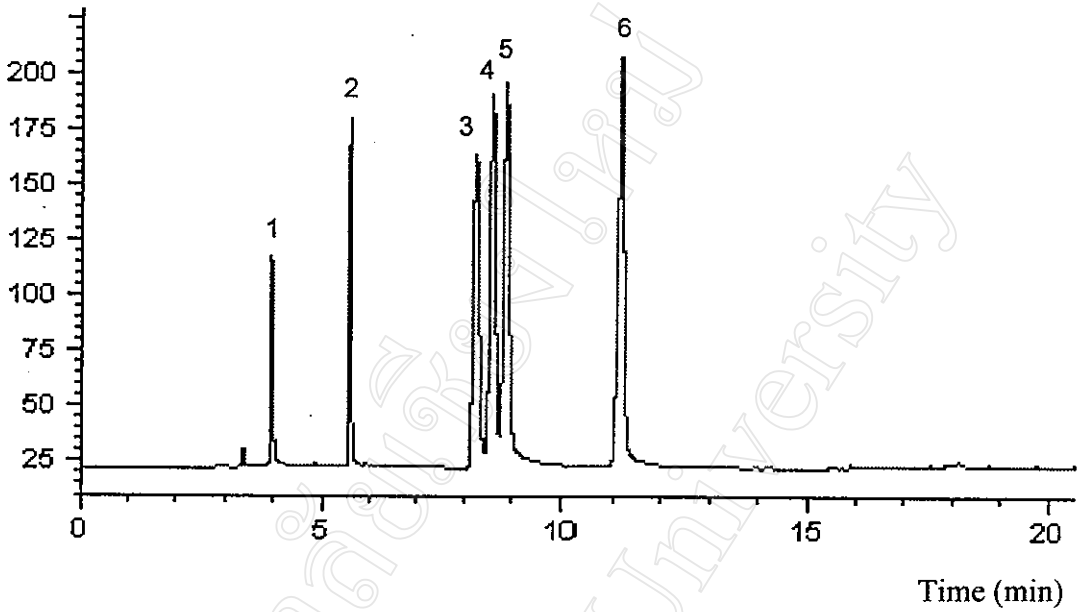

Figure 3.38 GC-FID chromatogram of spiked industrial effluent at high level concentration. Peak as in Figure 3.3.

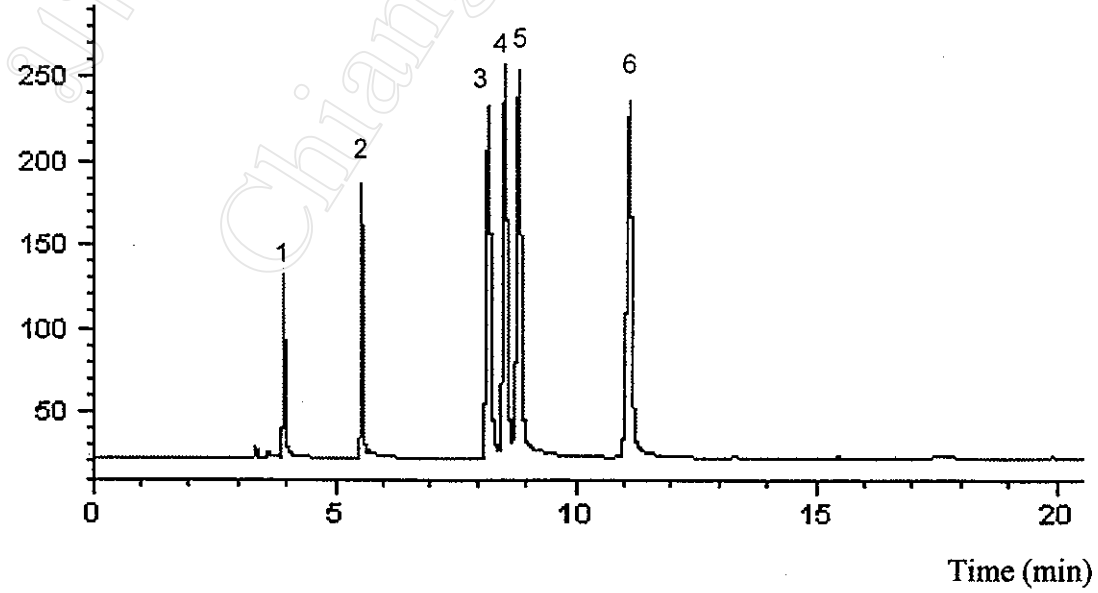

Figure 3.39 GC-FID chromatogram of spiked domestic wastewater at high level concentration. Peak as in Figure 3.3.

3.5 Analysis of Real Samples

Table 3.19 Results of HSSPME-GC-FID determination of BTEX compounds in real water samples.

Water samples	Concentrations in µg/L (%R.S.D.)					
	Benzene	Toluene	Ethybenzene	p-Xylene	m-Xylene	o-Xylene
Drinking water	ND	ND	ND	ND	ND	ND
Natural surface water	ND	ND	ND	ND	ND	ND
Industrial effluent	ND	ND	ND	ND	ND	ND
Industrial wastewater	ND	116.8 (6.2)	ND	ND	ND	ND
Domestic wastewater	ND	ND	ND	ND	ND	ND
Gasoline station	ND	ND	ND	ND	ND	ND
Motorcycle service garage	ND	7.2 (1.1)	1.4 (3.3)	2.0 (1.1)	2.3 (2.2)	1.7 (2.9)

ND = Not Detected.

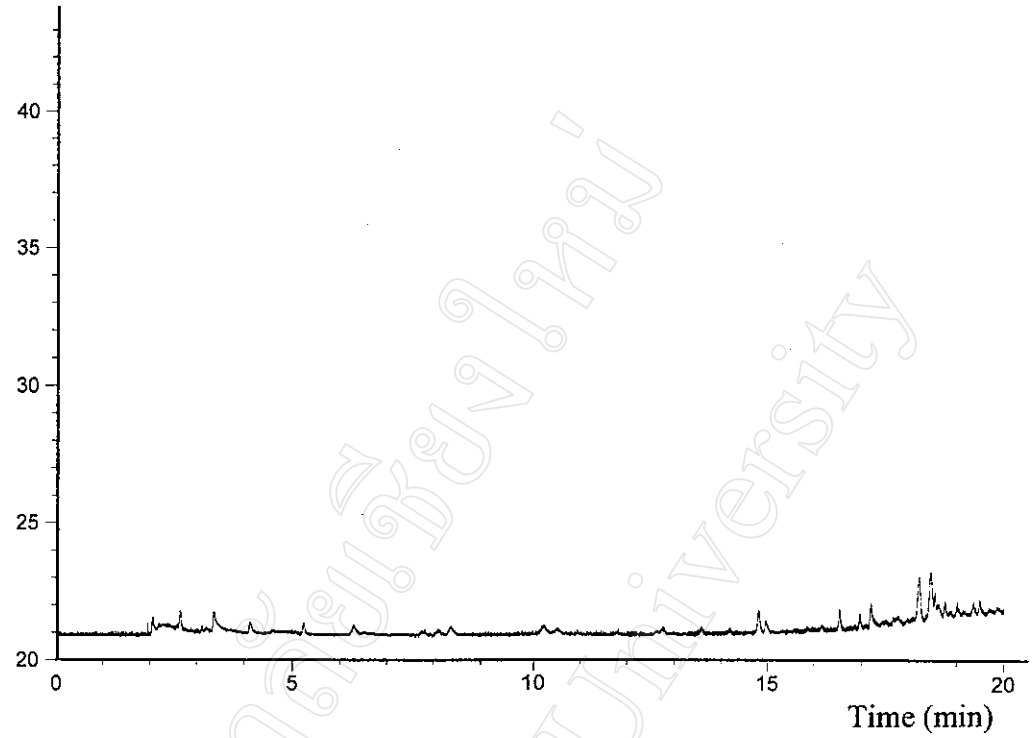

Figure 3.40 GC-FID chromatogram of blank ultrapure water.

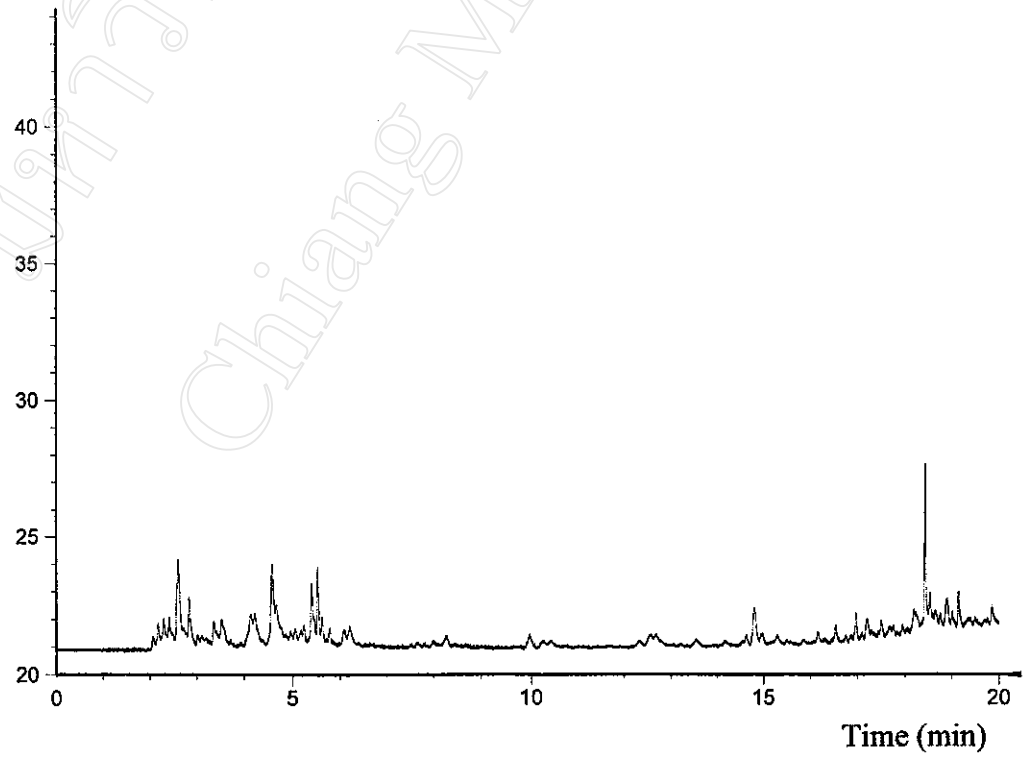

Figure 3.41 GC-FID chromatogram of drinking water sample not detected of BTEX compounds.

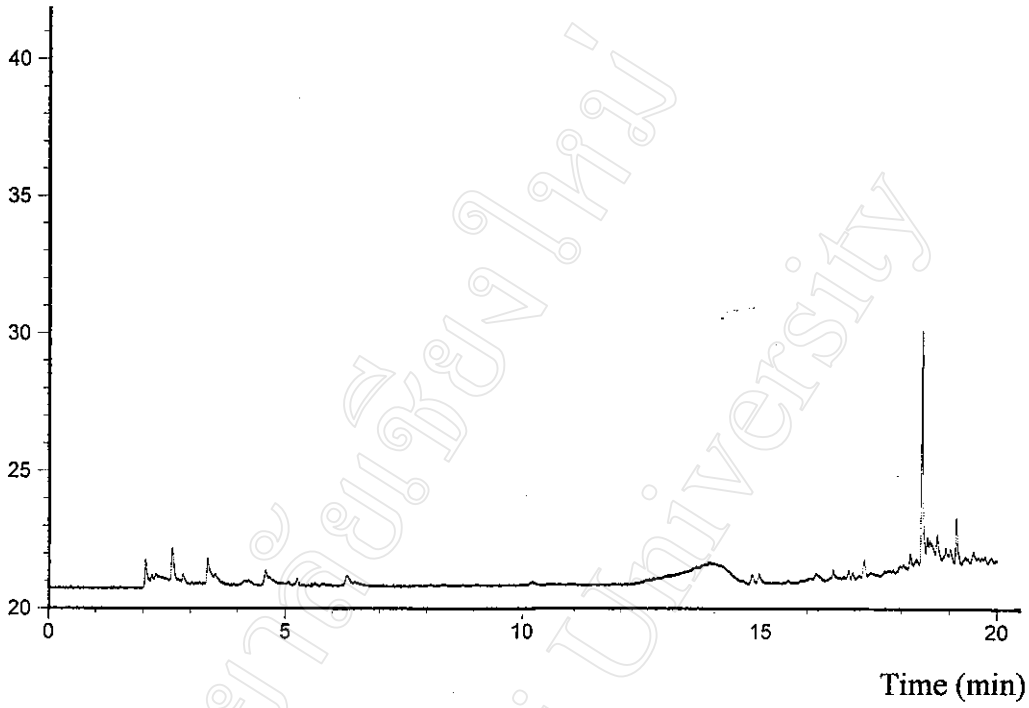

Figure 3.42 GC-FID chromatogram of natural surface water sample not detected of BTEX compounds.

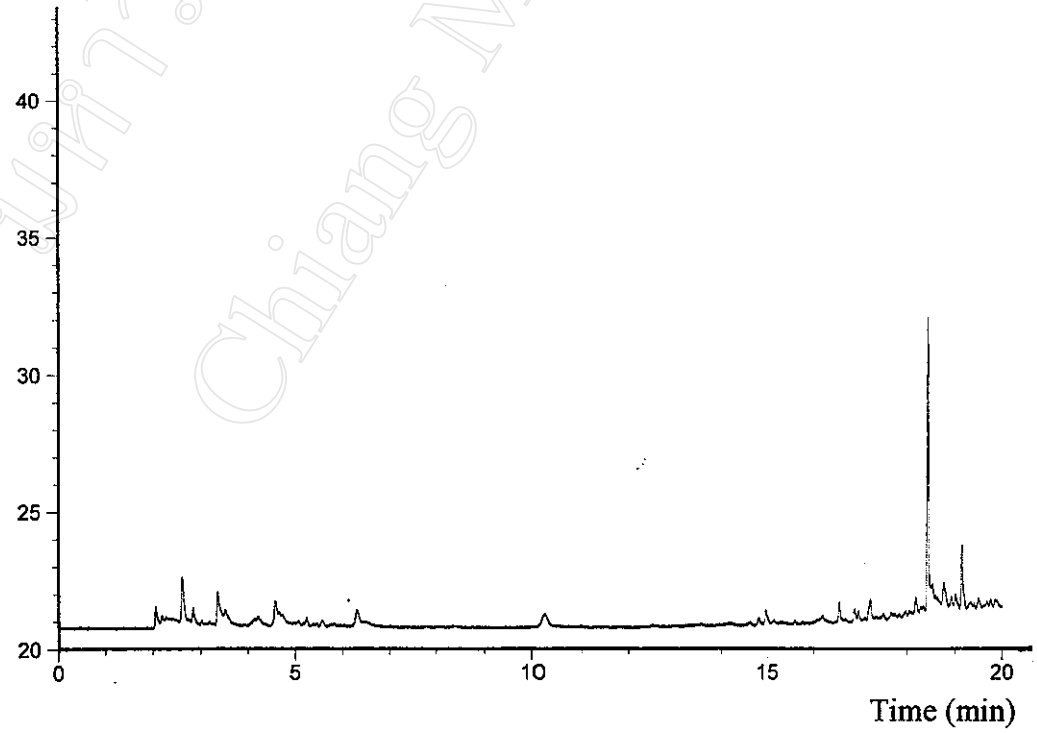

Figure 3.43 GC-FID chromatogram of industrial effluent water sample not detected of BTEX compounds.

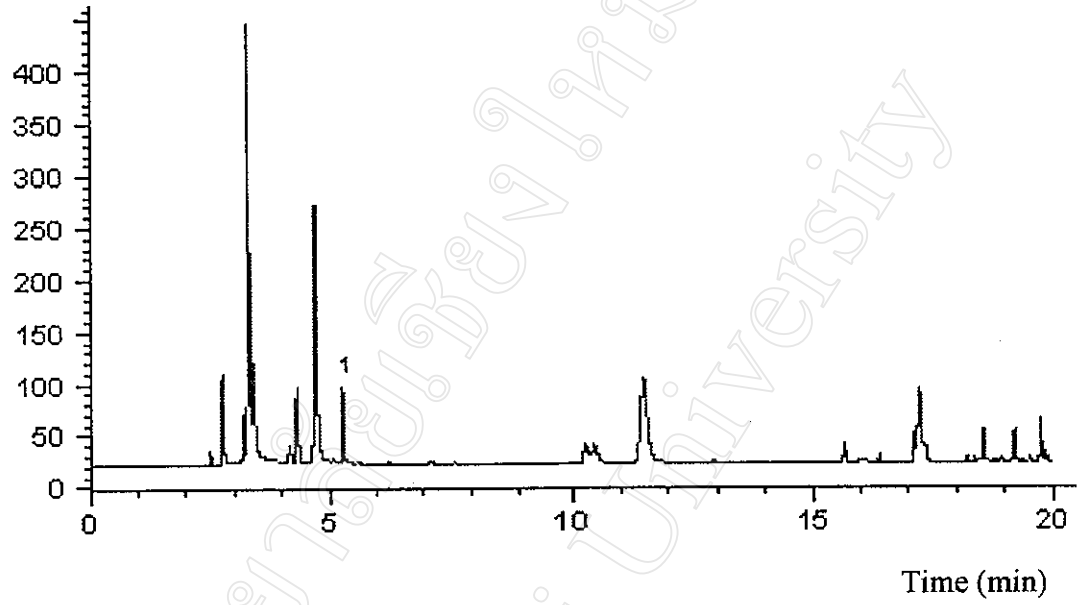

Figure 3.44 GC-FID chromatogram of industrial wastewater sample not detected of BTEX compounds.

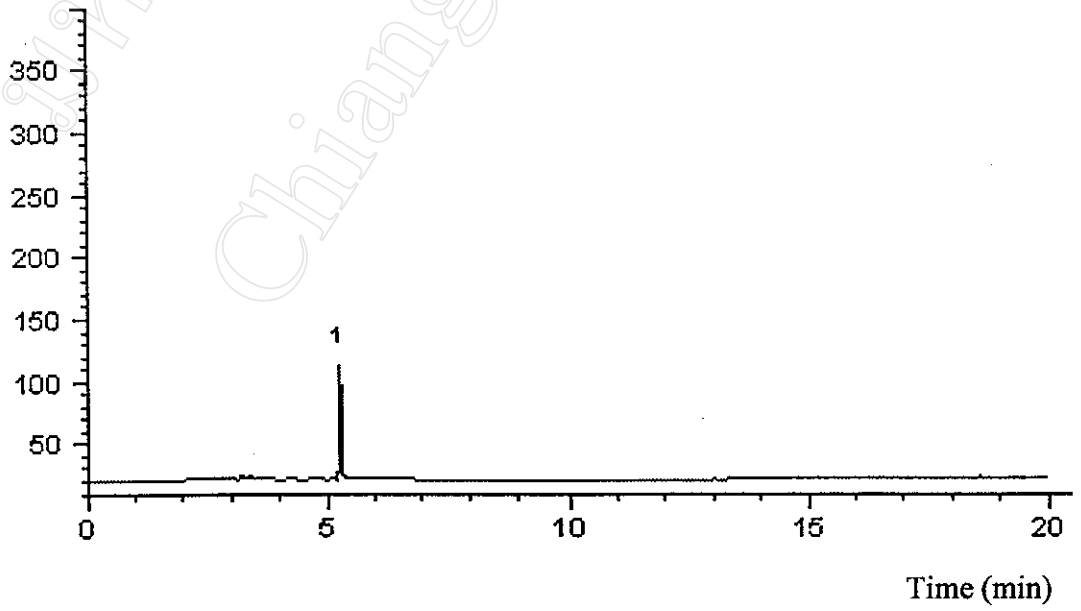

Figure 3.45 GC-FID chromatogram of 200 µg/L of toluene in ultrapure water.
Peak 1= toluene

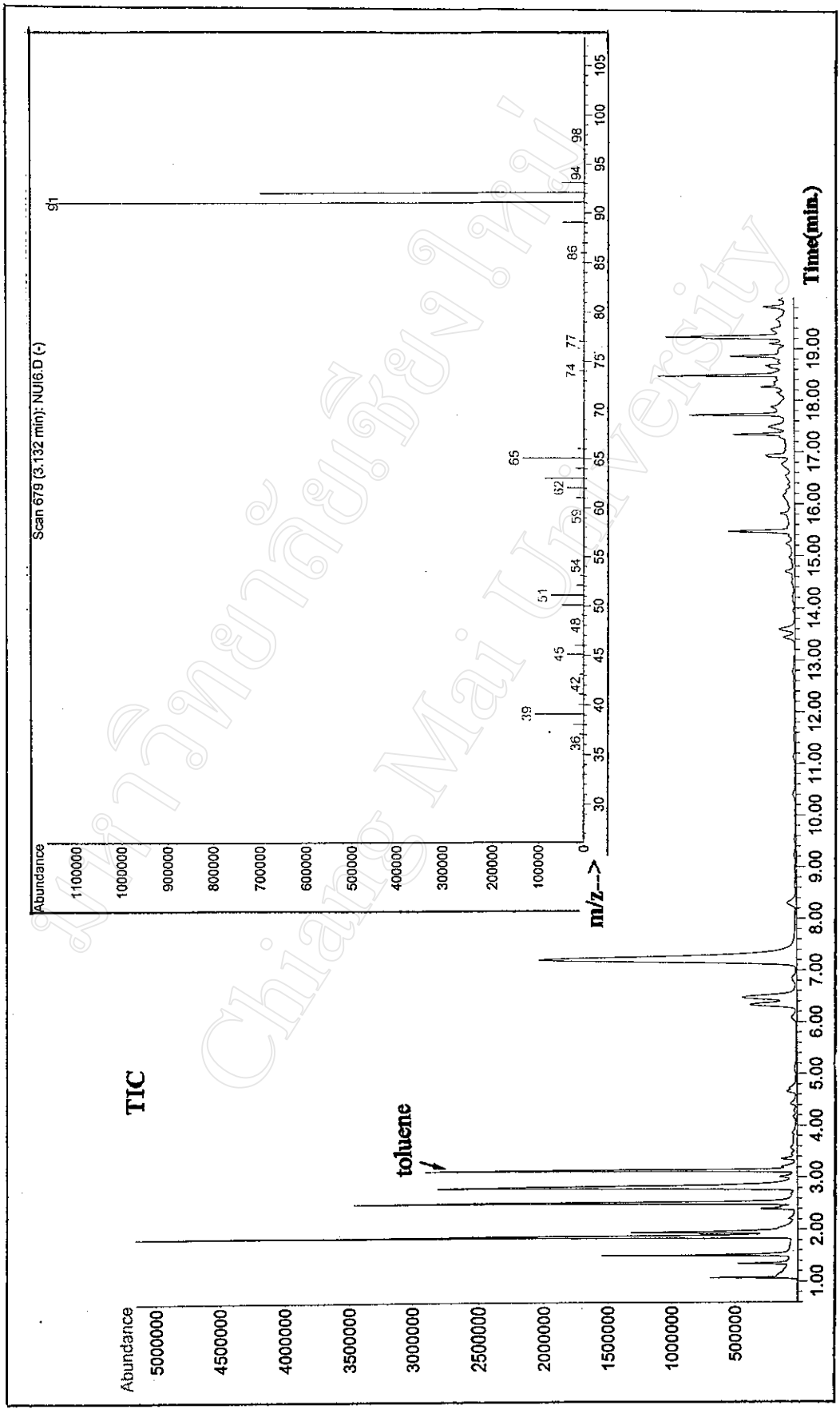

Figure 3.46 Total ion chromatogram of industrial wastewater sample. The inset shows the EI mass spectrum of the peak toluene.

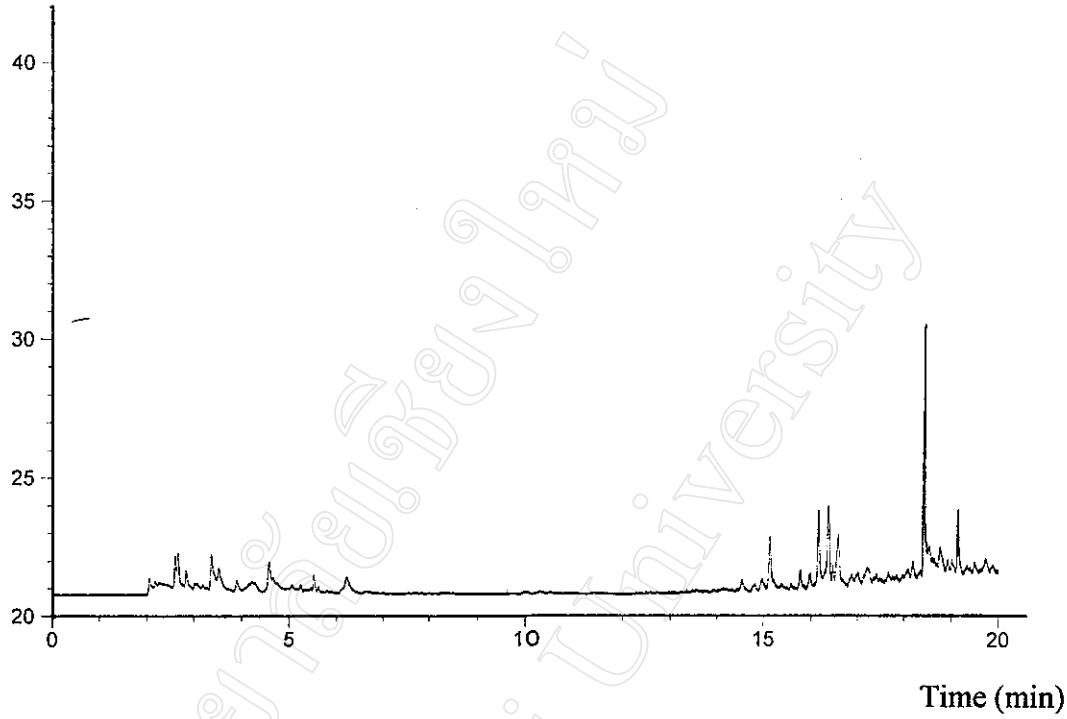

Figure 3.47 GC-FID chromatogram of domestic wastewater sample not detected of BTEX compounds.

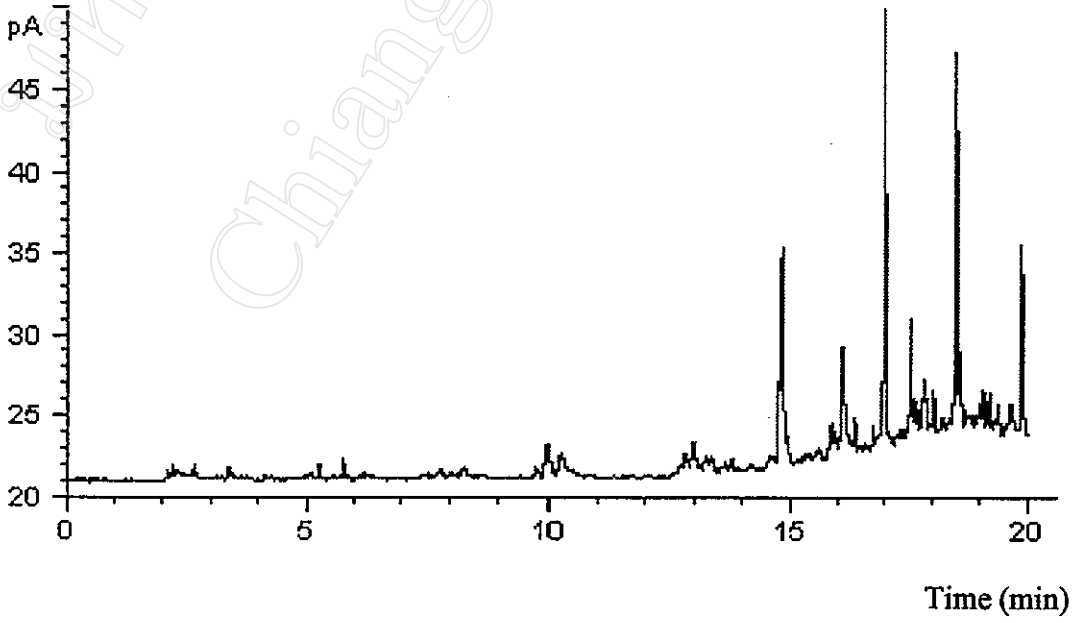

Figure 3.48 GC-FID chromatogram of a real water sample collected from gasoline station not detected of BTEX compounds.

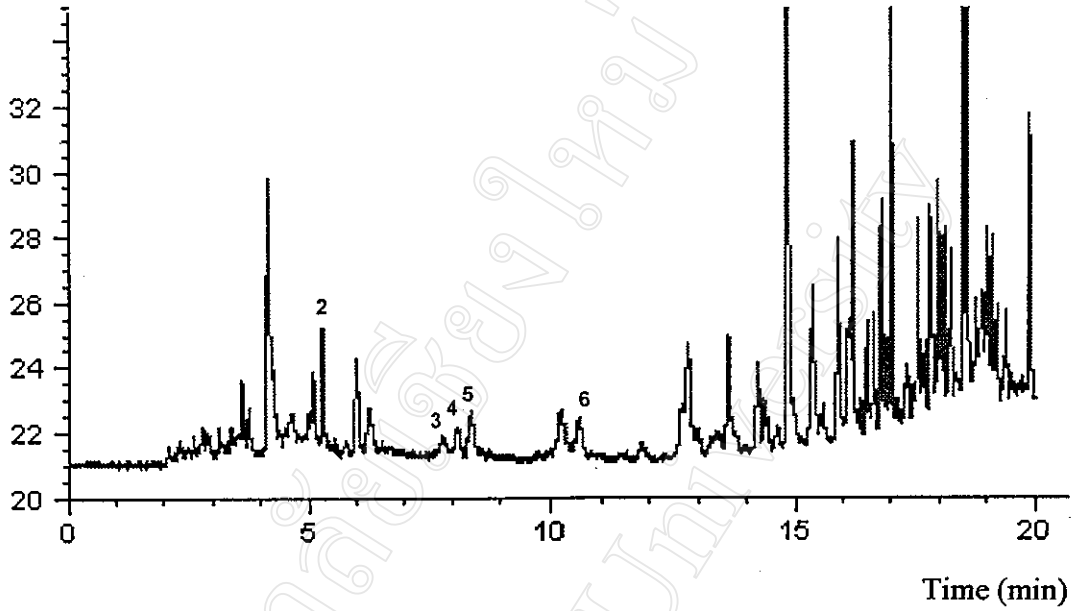

Figure 3.49 GC-FID chromatogram of a real water collected from motorcycle service garage. Peaks : 2 = toluene, 3 = ethylbenzene, 4 = p-xylene, 5 = m-xylene and 6 = o-xylene

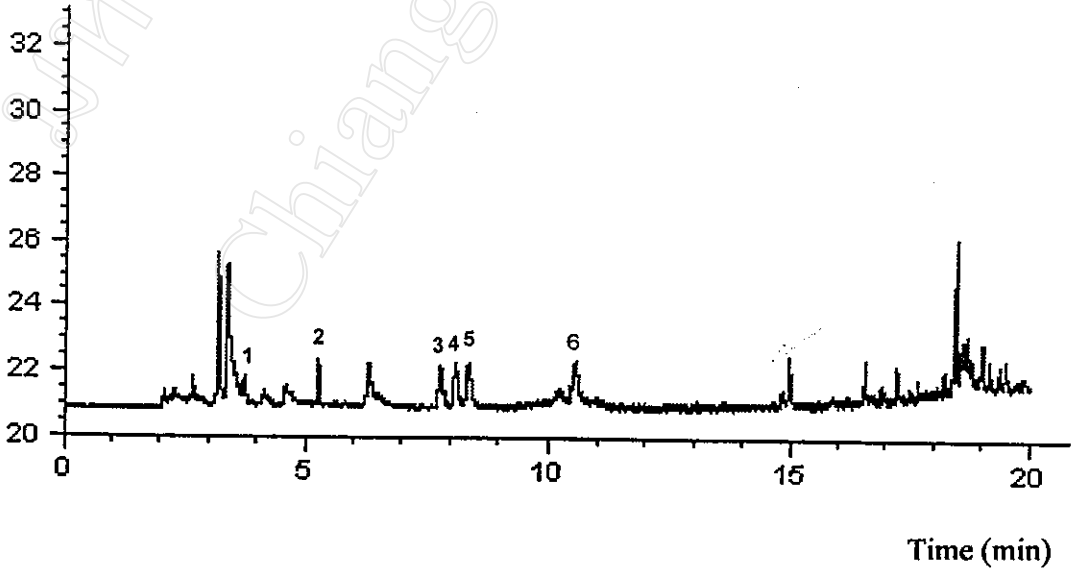

Figure 3.50 GC-FID chromatogram of 4 µg/L of benzene and 2 µg/L of toluene, ethylbenzene, p-xylene, m-xylene and o-xylene in ultrapure water.
Peaks : 1 = benzene and 2-6 correspond to the numbers indicated in Figure 3.49

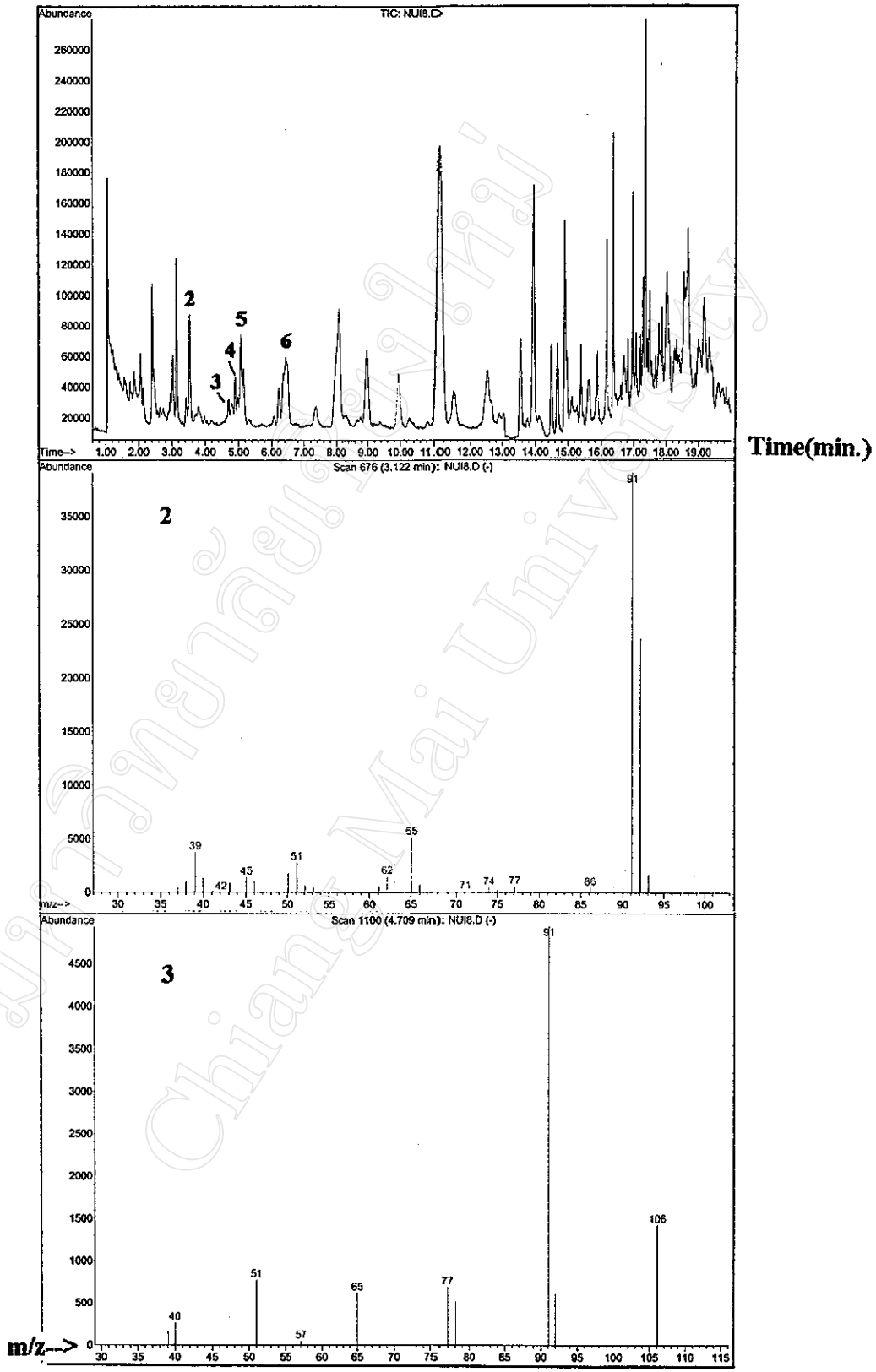

Figure 3.51 Total ion chromatogram of a real water collected from motorcycle service garage. Peak number 2-6 correspond to the numbers indicated in Figure 3.49, 2 and 3 : EI mass spectrum of toluene and ethylbenzene, respectively.

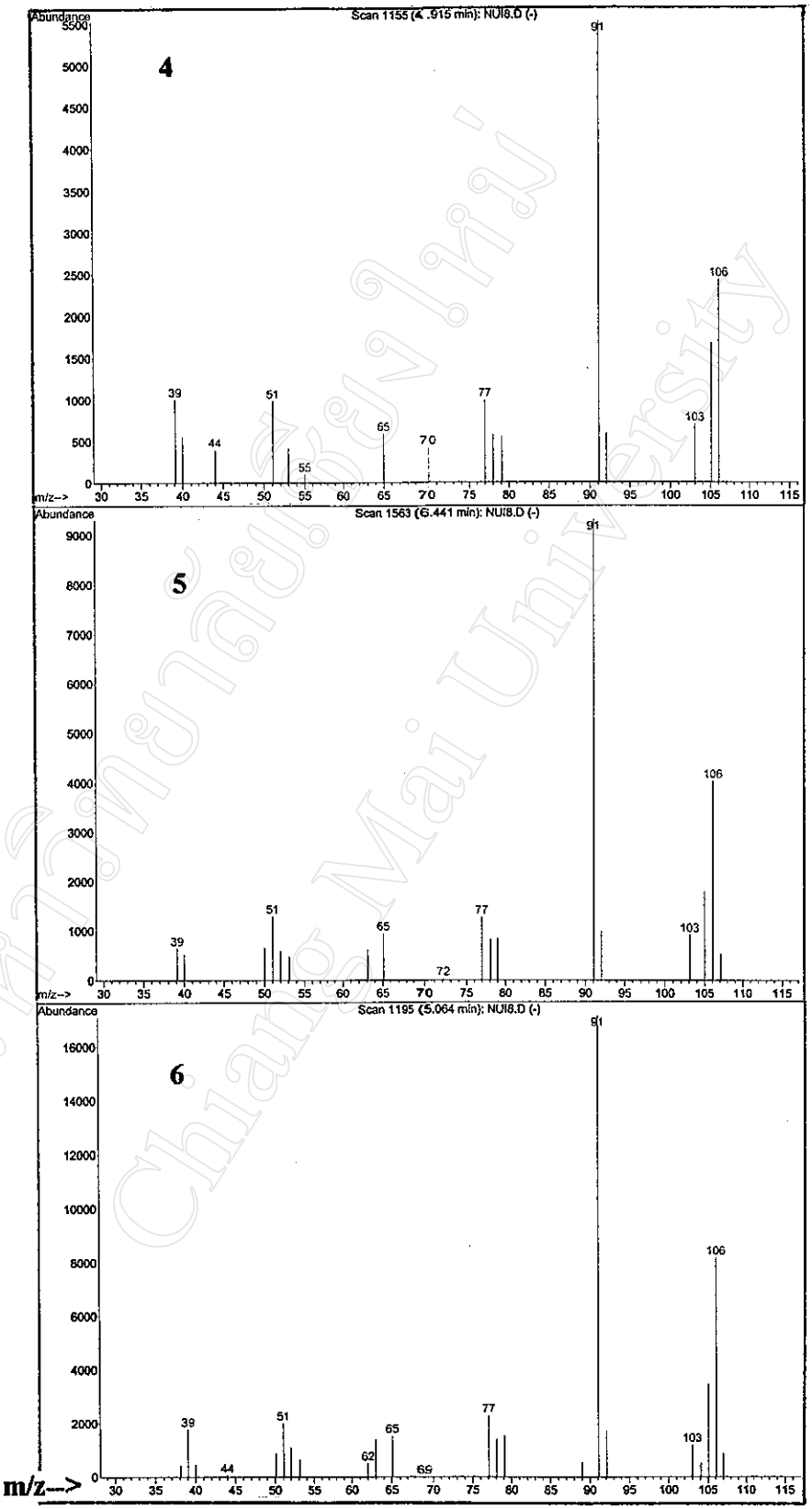

Figure 3.52 (continued) EI mass spectrum of 4 : p-xylene, 5 : m-xylene and 6 : o-xylene.

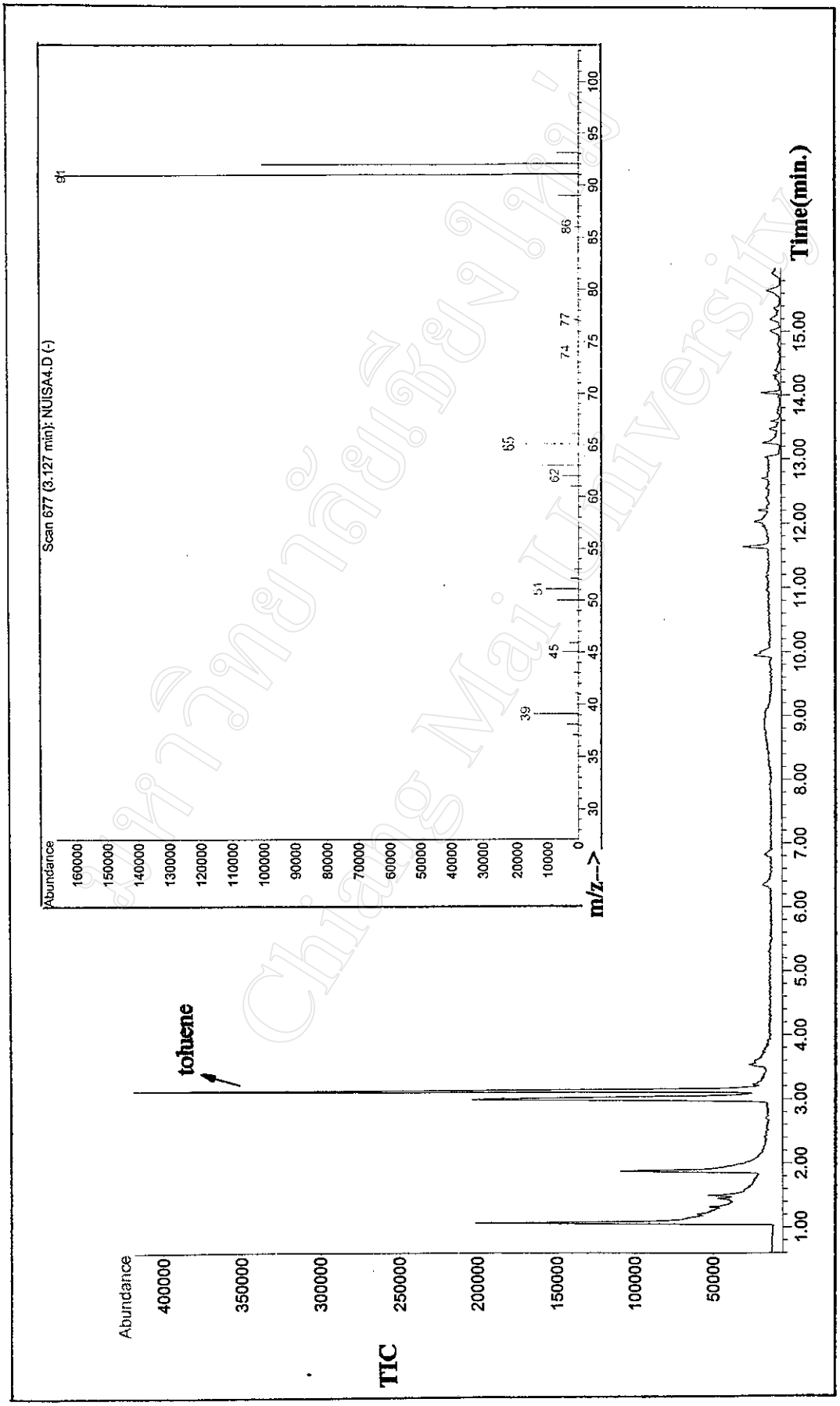

Figure 3.53 Total ion chromatogram of a human blood sample by HSSPME-GC-MS under optimal conditions extraction. The inset shows the EI mass spectrum of the peak toluene.