

# MODIFIED METHOD FOR DETERMINATION OF PAHs IN AMBIENT AIR IN BANGKOK USING GAS CHROMATOGRAPHY-MASS SPECTROMETRY

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**ABSTRACT:** The PAH analysis method was modified using HP-5MS column. Sampling procedure was based on the NIOSH method no.5515. Sorbent tube (XAD-2) and PTFE filter were used to collect PAHs in both gaseous and particulate phases, respectively. The air samples were extracted by n-hexane and analyzed by gas chromatography-mass spectrometry. Results showed that the detection limit of 16 PAHs ranged from 0.6 to 2.4 ng. The percent recoveries of 16 PAHs for sorbent tubes (XAD-2) and PTFE filters were 78.15-90.74% and 78.23-111.55%, respectively. The percent relative standard deviations were both less than 11%. The analysis of 20 air samples; 10 samples from the roadside and the other 10 samples from the local market in Bangkok found that total PAH concentrations were  $520.56 \pm 61.04$  and  $1248.44 \pm 877.93$  ng/m<sup>3</sup>, respectively. The total PAH concentrations from the local market were two times higher than those from the roadside.

**Keywords:** Polycyclic aromatic hydrocarbons (PAHs), Gas chromatography-mass spectrometry (GC-MS), Ambient air, Bangkok

## INTRODUCTION

PAHs are groups of organic compounds with two or more fused aromatic rings formed during incomplete combustion of organic compounds such as coal, oil and gases, garbage, tobacco or charbroiled meat [1]. They are relatively low solubility in water, but are highly lipophilic. PAHs may exist in vapor or particle phases according to their volatility. They are able to combine with suspended particles less than 5  $\mu$ m [2]. The 16 PAHs were chosen for analysis; because they were regarded as priority pollutants from U.S. EPA [3]. Furthermore, some PAHs are recognized as chemical carcinogens to animals and humans [4].

PAHs were determined by high performance liquid chromatography with a UV or fluorescence detector and gas chromatography with a FID or mass spectrometer [5]. Nowadays, Gas chromatography with mass spectrometry (GS-MS) has been widely used due to its sensitivity and selectivity. Moreover, GC-MS was a simple technique [6, 7]. PAHs were extracted using solvent in an ultrasonic bath since it was more rapid and simpler than the other methods, such as Soxhlet extraction [8-10] or Microwave irradiation [3]. This research aimed to modify the analysis method of 16 PAHs both in gaseous and particulate phases in ambient air using GC-MS. The accuracy, precision and detection limits of the method were also tested. Then, the method was used to determine PAHs in ambient air from the roadsides and the local markets in Bangkok area.

## MATERIALS AND METHODS

### Chemical reagents

Two thousand  $\mu$ g/ml mixed standard PAHs in dichloromethane solution (Restek, USA) consisted of acenaphthene, acenaphthylene, anthracene, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene, benzo(k)fluoranthene, chrysene, dibenz(a,h)anthracene, fluoranthene, fluorene, indeno(1,2,3-cd)pyrene, naphthalene, phenanthrene, and pyrene. Methanol, dichloromethane (analytical grade) and n-hexane (GC grade) were purchased from Merck, Germany.

### Instrumentation

Gas chromatography (Agilent 6890N, USA) and a mass selective detector (Agilent 5975, USA) fitted with a HP-5MS capillary column (30m $\times$ 0.25mm, 0.25 $\mu$ m film, J&W Scientific, USA) was used. The operating condition for GC-MS was set up as follows: splitless injection of 2  $\mu$ l, injection temperature of 280 $^{\circ}$ C; the oven temperature program was 80 $^{\circ}$ C, ramped at 15 $^{\circ}$ C/min to 185 $^{\circ}$ C (hold for 5 min), ramped at 10 $^{\circ}$ C/min to 240 $^{\circ}$ C (hold for 10 min), and ramped at 3 $^{\circ}$ C/min to 276 $^{\circ}$ C (hold for 5.5 min). The mass spectrometer used the electron impact mode of 70 eV. The carrier gas was helium at a flow rate of 0.8 ml/min. The mass of primary ions of PAHs were determined using the selected ion monitoring (SIM) mode.

Ultrasonic bath (*Ultrasonic steri-cleaner*, Coax group corporation Ltd, Thailand)

SKC personal air sampling pump (Model 224-PCXR8, SKC Inc., USA)

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The 37- mm PTFE membrane, 2- $\mu$ m (Cat.No.225-17-09, SKC Inc., USA) with support pad and spacer ring (Cat. No. 225-23, SKC Inc., USA)

Sorbent tube, XAD-2 resin (Supelco ORBO-43, USA)

#### Calibration curves of PAHs

The 2000  $\mu$ g/ml PAHs in dichloromethane was diluted to 12, 24, 60, 96 and 240 ng/ml with hexane. Each standard mixture of PAHs was analyzed by GC-MS for 3 replications.

#### Sample preparation and analysis

Both filters and sorbent tubes were extracted by 500  $\mu$ l of n-hexane. For sorbent tubes (XAD-2), they were separated into 2 parts, front and back sections. The tubes containing sorbent were extracted with 500  $\mu$ l of n-hexane in an ultrasonic bath at 4°C for 30 min. For PTFE filters, the edges of PTFE filters were removed and the filters were cut into 4 pieces and extracted with 500  $\mu$ l n-hexane in an ice bath (4°C) with occasional agitation for 30 min. After extraction, 2  $\mu$ l of the solution were analyzed by GC-MS. The media blank for both of sorbent tube (XAD-2) and PTFE filter were analyzed in the same manner as the sample.

#### Recovery of PAHs from the sorbent tube and PTFE filter

The back section of sorbent tube and glass wool were removed from the sorbent tube. Then, 5  $\mu$ l of 6  $\mu$ g/ml standard PAHs were injected directly into the front section of sorbent tubes and the PTFE filters in tubes for three replications. Then, they were capped immediately, wrapped in an aluminum foil, left overnight to assure complete adsorption of PAHs and then stored in a freezer before analysis. Blank sorbent tubes and PTFE filters were carried out in the same manner as the sample except that they were not spiked with the standard PAHs. They were analyzed in the same manner as the sample. The recoveries of PAHs from sorbent tubes and PTFE filters were calculated by dividing PAHs recovered with PAHs spiked and multiplied by 100.

#### Reliability of the method for the determination of PAHs

In the development of a sampling and analytical method, the method used must be simple, convenient, accurate and reproducible.

#### Detection limit of the method

The low-level calibration concentrations were selected to determine the detection limit of the method. The known concentrations of PAHs ranged from 1.2 to 12 ng/ml, 1.2, 2.4, 3.6, 4.8, 6.0, 7.2, 8.4, 9.6, 10.8 and 12 ng/ml were prepared and analyzed by GC-MS for three replications. The linear regression equation of each PAH was set up and the standard error of the regression was calculated

following the National Institute for Occupational Safety and Health (NIOSH) method [11]. The limit of detection (LOD) of each PAH was reported as the highest of the calculated LOD, lowest calibration standard or X-intercept if regression has a negative Y-intercept.

#### Accuracy and precision of the method

The two known standard PAH concentrations of 6 and 12  $\mu$ g/ml were prepared. Then, 5  $\mu$ l of known PAH concentrations were injected to both of sorbent tubes and PTFE filters for three replications each day for three days. They were extracted and analyzed in the same manner as the samples. The accuracy and precision were presented as inter-day assays in terms of percent recovery (% recovery) and relative standard deviation (%RSD).

#### Application in the field

##### Sites of air sample collection

The air sample collection sites were roadsides and local markets located in the central Bangkok. Both types of sample sites are thought to be the common places where PAHs would present in considerably high concentrations due to such surrounding activities as cooking, grilling, and traffics. The air samples were collected during October, 2009 to April, 2010 at which time rainfall is minimal. The 10 air samples were collected from local markets whereas the other 10 air samples were collected from the footpath along streets in several districts of central Bangkok (Figure 1).

##### Air sample collection

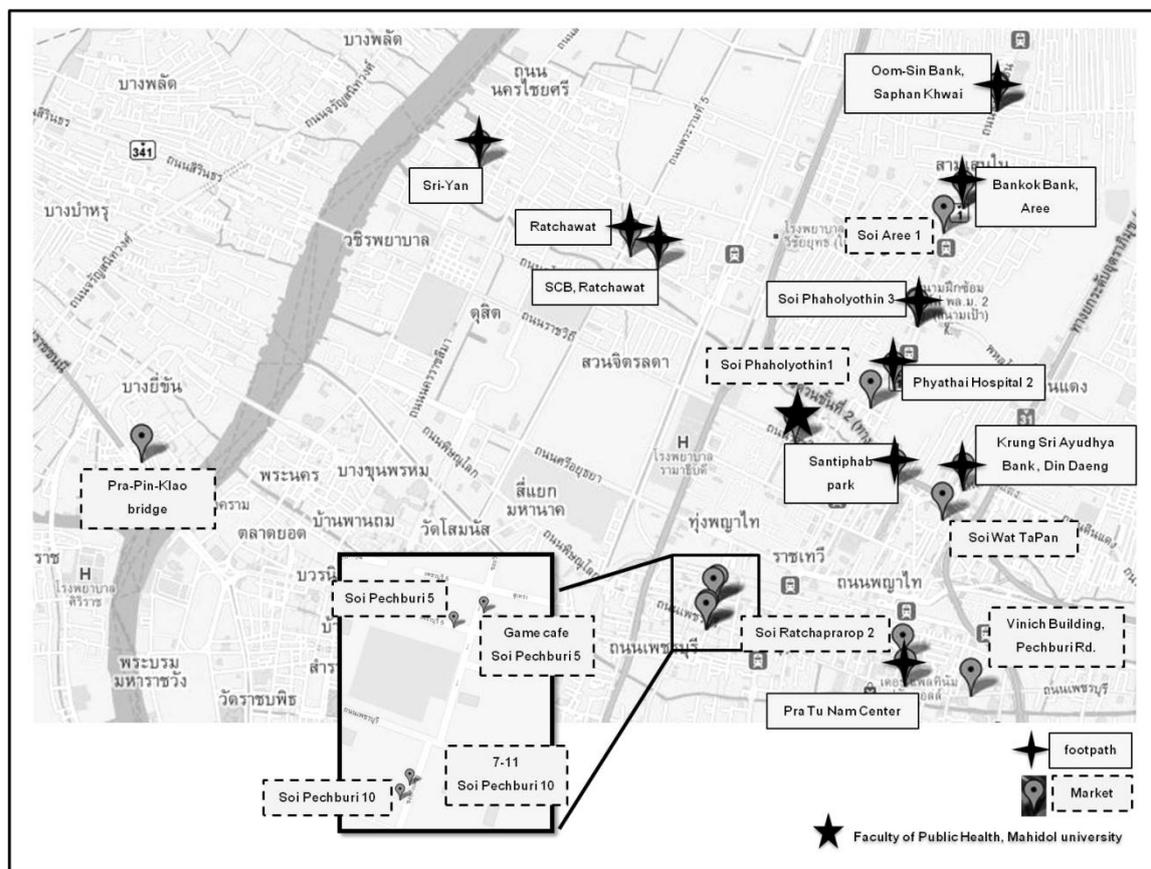
The 20 air samples from ambient air in Bangkok were collected for both particulate and vapor phases using PTFE filters connected to sorbent tubes (XAD-2) according to the NIOSH method 5515 [12]. The PTFE filters were pre-cleaned before sampling by soaking in dichloromethane and methanol solution (1:1, v/v) for 2 min and the filters were dried in the air. The personal sampling pump was calibrated with a cassettes filter holder containing a PTFE filter connected to a sorbent tube (XAD-2) on line by a calibrator meter to calibrate an air flow rate at 2 l/min before and after the sampling. The cassettes filter holder and sorbent tube (XAD-2) were both wrapped in aluminum foil to prevent sample degradation. Ambient air samples were collected at 1.5 m high to collect ambient air of 360-600 l for 3 – 5 hours.

##### Analysis of air samples

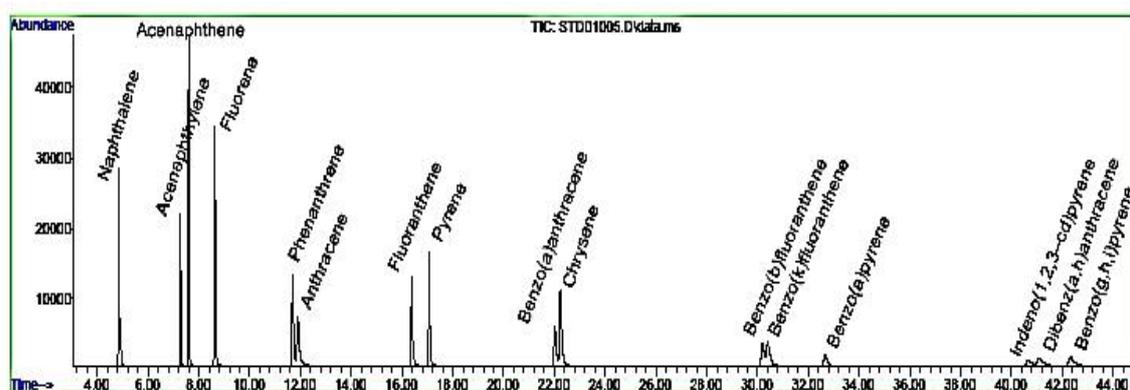
The PTFE filters and sorbent tubes were prepared and analyzed by GC-MS as described above.

##### Statistical analysis

The median was used for descriptive analysis. The mean and standard deviation were also used in this manuscript; the results of this current study can be compared with other studies. The comparison



**Figure 1** Location of 10 air samples collected from local market and the other 10 samples from footpaths along streets in Bangkok



**Figure 2** The chromatograms of 16 priority standard PAHs of 240 ng/ml

between PAH concentrations from the roadside and the local market was illustrated by Mann-Whitney U test.

**RESULT AND DISCUSSION**

**Calibration curves and chromatogram of PAHs analysis**

All 16 standard PAHs were analyzed using GC-MS. The masses of primary ion of each PAH were selected. The chromatograms of 16 PAHs are presented in Figure 2. All PAHs eluted completely

within 45 minutes. Phenanthrene and anthracene peaks, benzo(a)anthracene and chrysene peaks and benzo(b)fluoranthene and benzo(k)fluoranthene peaks are not fully separated.

**Standard curves of PAHs**

The standard curves of each PAH were linear over the concentration range from 12 to 240 ng/ml. The correlation coefficient ( $r^2$ ) of each PAH for three replications ranged from 0.9858 to 0.9996 as presented in Table 1.

**Table 1** The calibration parameters for the 16 PAHs analysis

PAHs	Primary ion (m/z)	Liner regression equation	Correlation coefficient, r <sup>2</sup>
1. Naphthalene	128	y = 161230x + 964.99	0.9994
2. Acenaphthylene	152	y = 105961x - 725.81	0.9995
3. Acenaphthene	154	y = 212616x - 1034.7	0.9998
4. Fluorene	166	y = 176209x - 1459.1	0.9996
5. Phenanthrene	178	y = 114326x - 2700.8	0.9977
6. Anthracene	178	y = 72292x - 2824.1	0.9898
7. Fluoranthene	202	y = 101460x - 1445.5	0.9985
8. Pyrene	202	y = 116009x - 1912.1	0.9980
9. Benzo(a)anthracene	228	y = 56206x - 1879.1	0.9901
10. Chrysene	228	y = 102192x - 3447.1	0.9915
11. Benzo(b)fluoranthene	252	y = 26769x - 852.62	0.9943
12. Benzo(k)fluoranthene	252	y = 32239x - 1285	0.9858
13. Benzo(a)pyrene	252	y = 34530x - 796.01	0.9965
14. Indeno(1,2,3-cd)pyrene	276	y = 15246x - 357.22	0.9968
15. Dibenz(a,h)anthracene	278	y = 20490x - 598.62	0.9938
16. Benzo(g,h,i)perylene	276	y = 32234x - 941.18	0.9954

**Table 2** The %recovery of 16 PAHs from sorbent tube and PTFE filter

PAHs	PAHs added (ng)	PAHs Recovered from sorbent tube (ng)(n = 3)		PAHs Recovered from PTFE filter (ng) (n=3)	
		Mean ± SD	%recovery	Mean ± SD	%recovery
1. Naphthalene	30	30.81 ± 0.25	102.69	25.41 ± 1.51	84.70
2. Acenaphthylene	30	26.51 ± 0.21	88.46	29.25 ± 1.34	97.50
3. Acenaphthene	30	28.60 ± 0.22	95.34	29.65 ± 1.46	98.84
4. Fluorene	30	26.61 ± 0.34	88.70	30.55 ± 1.58	101.83
5. Phenanthrene	30	21.70 ± 0.41	72.34	30.22 ± 1.63	100.74
6. Anthracene	30	21.55 ± 0.28	71.82	28.48 ± 1.43	94.94
7. Fluoranthene	30	19.99 ± 0.43	66.63	31.67 ± 1.53	105.55
8. Pyrene	30	19.79 ± 0.37	65.98	30.92 ± 1.33	103.05
9. Benzo(a)anthracene	30	-	-	29.58 ± 0.66	98.59
10. Chrysene	30	-	-	28.43 ± 0.76	94.77
11. Benzo(b)fluoranthene	30	-	-	28.49 ± 1.26	94.97
12. Benzo(k)fluoranthene	30	-	-	27.44 ± 1.55	91.47
13. Benzo(a)pyrene	30	-	-	28.74 ± 0.50	95.80
14. Indeno(1,2,3-cd)pyrene	30	-	-	27.55 ± 1.81	91.83
15. Dibenz(a,h)anthracene	30	-	-	26.01 ± 3.21	86.69
16. Benzo(g,h,i)perylene	30	-	-	27.19 ± 2.82	90.62

### Recovery of PAHs from sorbent tubes and PTFE filters

The average PAHs recovered from sorbent tubes and PTFE filters ranged from 19.79 to 31.66 ng. The %recovery of PAHs from sorbent tubes ranged from 65.98% to 102.69% (Table 2). Only 8 gaseous phase PAHs having 2-4 aromatic rings recovered from sorbent tubes. The %recovery of PAHs from PTFE filters ranged from 84.70% to 105.55%.

### Reliability of the method for the determination of PAHs

#### Detection limit

The method could detect each PAH ranging from 0.6 to 2.4 ng. The detection limit of phenanthrene was 0.6 ng and the benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, indeno(1,2,3-cd)pyrene, dibenz(a,h)anthracene and benzo(g,h,i)pyrene had the same detection limit of 2.4 ng. The detection limit of anthracene was 0.76 ng and the rest which were acenaphthylene,

acenaphthene, fluorine, fluoranthene, pyrene, benzo(a)anthracene and chrysene, had detection limit of 1.2 ng. The results of this study were compared with previous study of Chen M-R [13] reported LOD from 0.093 to 1.51 ng, which were lower than the LOD of the current study. Analytical techniques for detection of PAHs continue to increase in sensitivity, thus progressively lowering the detection limits for these compounds in ambient air.

#### Accuracy and precision

The average recoveries of PAHs from sorbent tubes (XAD-2) ranged from 78.15% to 90.74% at 30 and 60 ng PAH masses and the precision ranged from 2.79% to 10.06%, respectively (Table 3). For PTFE filters, the average recoveries ranged from 78.23% to 111.55% and the precisions ranged from 0.23% to 10.61%. Thus, the accuracy and precision of the PAH analysis method were satisfactory.

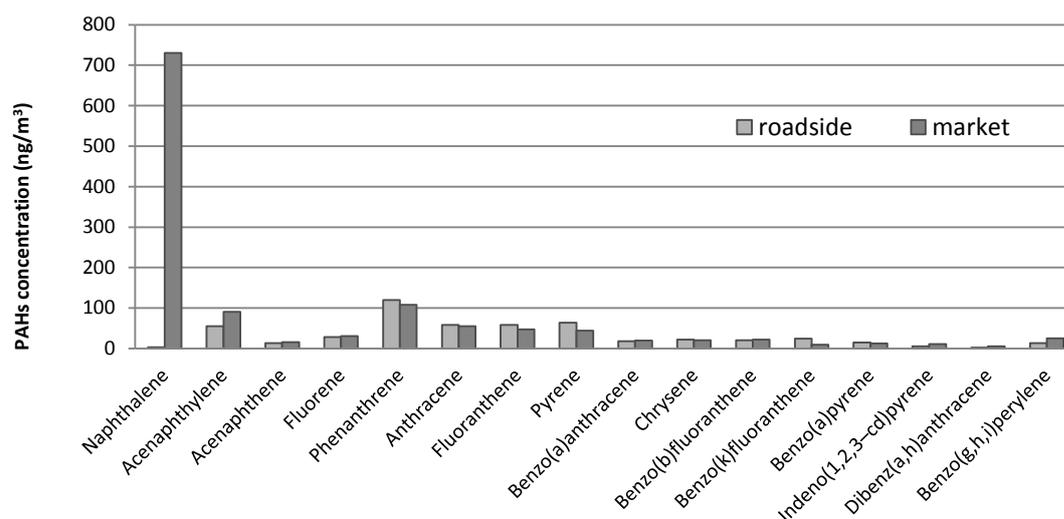
The previous studies reported recovery of PAHs

**Table 3** The accuracy and precision of PAHs for sorbent tube (XAD-2) and PTFE filters

PAHs	Mass of PAHs added (ng)	Inter-day assay (n=3) XAD-2		Inter-day assay (n=3) PTFE filter	
		%RSD	%Recovery	%RSD	%Recovery
1. Naphthalene	30	9.82	78.69	6.27	85.33
	60	5.35	90.74	7.59	89.39
2. Acenaphthylene	30	10.06	79.28	1.93	95.95
	60	2.79	86.78	6.51	104.66
3. Acenaphthene	30	8.12	78.66	3.26	96.80
	60	3.63	89.49	3.75	104.08
4. Fluorene	30	7.78	78.52	3.03	98.94
	60	4.90	87.99	3.17	107.27
5. Phenanthrene	30	7.01	81.13	2.81	97.57
	60	6.17	84.25	2.25	108.57
6. Anthracene	30	6.62	85.11	2.59	92.73
	60	4.02	78.15	2.78	106.24
7. Fluoranthene	30	5.41	82.88	4.68	100.14
	60	5.11	85.03	1.41	111.55
8. Pyrene	30	6.28	82.24	4.79	97.70
	60	5.84	83.33	3.03	108.88
9. Benzo(a)anthracene	30	-	-	4.81	93.80
	60	-	-	0.23	108.06
10. Chrysene	30	-	-	5.35	89.53
	60	-	-	7.33	100.58
11. Benzo(b)fluoranthene	30	-	-	7.14	88.56
	60	-	-	2.22	99.82
12. Benzo(k)fluoranthene	30	-	-	7.72	84.53
	60	-	-	2.80	95.57
13. Benzo(a)pyrene	30	-	-	6.26	89.66
	60	-	-	1.90	100.59
14. Indeno(1,2,3-cd)pyrene	30	-	-	9.06	83.15
	60	-	-	4.01	95.94
15. Dibenzo(a,h)anthracene	30	-	-	9.38	78.23
	60	-	-	10.33	93.13
16. Benzo(g,h,i)perylene	30	-	-	9.40	81.77
	60	-	-	10.61	97.51

**Table 4** Total PAH concentrations in ambient air in Bangkok

PAHs	PAHs concentrations (ng/m <sup>3</sup> )		
	Roadside (n = 10)	Market (n = 10)	Ambient air (n = 20)
1. Naphthalene	3.26 ± 5.89	730.01 ± 882.61	366.64 ± 712.75
2. Acenaphthylene	54.97 ± 15.99	90.51 ± 48.12	72.74 ± 39.38
3. Acenaphthene	13.16 ± 5.88	16.08 ± 7.66	14.62 ± 6.81
4. Fluorene	28.62 ± 14.97	30.45 ± 11.63	29.53 ± 13.08
5. Phenanthrene	120.25 ± 31.26	107.88 ± 41.41	114.06 ± 36.27
6. Anthracene	58.73 ± 17.04	55.43 ± 34.08	57.08 ± 26.28
7. Fluoranthene	58.29 ± 12.36	47.03 ± 16.04	52.66 ± 15.08
8. Pyrene	63.49 ± 10.60	44.49 ± 18.74	53.99 ± 17.74
9. Benzo(a)anthracene	17.90 ± 10.24	20.09 ± 9.55	19.00 ± 9.70
10. Chrysene	21.83 ± 9.48	20.50 ± 8.94	21.16 ± 8.99
11. Benzo(b)fluoranthene	20.20 ± 12.00	22.08 ± 16.41	21.14 ± 14.03
12. Benzo(k)fluoranthene	24.63 ± 15.34	9.67 ± 16.08	17.15 ± 17.11
13. Benzo(a)pyrene	14.75 ± 8.48	12.57 ± 11.12	13.66 ± 9.69
14. Indeno(1,2,3-cd)pyrene	5.19 ± 11.96	10.75 ± 18.42	7.97 ± 15.38
15. Dibenzo(a,h)anthracene	2.14 ± 6.70	5.41 ± 12.00	3.78 ± 9.60
16. Benzo(g,h,i)perylene	13.16 ± 17.11	25.48 ± 33.18	19.32 ± 26.46
<b>Total PAHs</b>	<b>520.56 ± 61.04</b>	<b>1248.44 ± 877.93</b>	<b>884.50 ± 711.53</b>
<b>Total BaP<sub>eq</sub></b>	<b>24.96 ± 12.61</b>	<b>26.32 ± 25.23</b>	<b>25.64 ± 19.43</b>



**Figure 3** The comparison between PAH concentrations from the roadside and the local market

ranging from  $85.3 \pm 6.8\%$  to  $131.7 \pm 9.3\%$  [3] and from  $78.6\%$  to  $93.5\%$  [13] and the precisions ranging from  $1.28\%$  to  $8.89\%$  [13]. The recovery of the current study was similar to the previous studies. Therefore, the developed GC-MS method can be used for assessment of PAH concentrations in ambient air.

### Application in the field

#### PAH concentrations in ambient air

The PAH analysis method was modified from other methods [13, 14] to be a simple and reliable method for analysis of PAHs in air. Twenty air samples were collected; 10 samples from the roadsides and the other 10 samples from the local markets. The naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene and pyrene were found in gas phase at high concentration because of their intrinsic highly volatile nature. The medians of total PAH concentrations from the roadside and the local market were  $511.98$  and  $976.26$   $\text{ng/m}^3$ , respectively. In addition, the mean and standard deviation of total PAH concentrations from the roadside and the local market were  $520.56 \pm 61.04$  and  $1248.44 \pm 877.93$   $\text{ng/m}^3$ , respectively (Table 4). The total PAH concentration from the local market was two times higher than those from the roadside ( $p$ -value  $< 0.05$ ) probably because naphthalene was found in high concentration in two samples in the local market. The result of this study was similar to the study of Chen Y et al. reported that naphthalene (67-89%) was the most abundant gaseous PAHs found in all samples from 6 commercial restaurants in Hong Kong [15].

The comparison between PAHs found from the roadside and the local market is presented in Figure 3. The major PAHs on the roadside were phenanthrene, pyrene, fluoranthene, anthracene and

acenaphthylene, while the major PAHs from the local market were naphthalene, phenanthrene, acenaphthylene, anthracene, fluoranthene and pyrene. With regards to health effects of these PAHs, anthracene, benzo(a)pyrene and naphthalene are direct skin irritants while anthracene and benzo(a)pyrene are also reported to be skin sensitizers. Naphthalene can cause breakdown of red blood cells if inhaled or ingested in large amounts [16].

The average total PAHs concentration from 20 air samples was  $884.50 \pm 711.53$   $\text{ng/m}^3$ . Converting PAHs into  $\text{BaP}_{\text{eq}}$  according to Nisbet and LaGoy [17], the carcinogen potency of PAH concentrations was estimated by calculation of the  $\text{BaP}$  equivalent concentration based on toxic equivalent factors (TEFs). The average total  $\text{BaP}_{\text{eq}}$  concentration was  $25.64 \pm 19.43$   $\text{ng/m}^3$ . The  $\text{BaP}$  contribution to the dose of carcinogenic PAHs in this study was not very high.

When compared PAH concentrations found in this study with previous studies, total PAH concentrations in ambient air in Bangkok ranged from  $7.10$  to  $83.04$   $\text{ng/m}^3$  [18] and from  $76$  to  $189$   $\text{ng/m}^3$  [19]. Furthermore, the average concentration of 8 PAH exposure of traffic polices in Bangkok was  $72.79$   $\text{ng/m}^3$ ; the major PAHs found was benzo(g,h,i)perylene and indeno(1,2,3-cd)pyrene<sup>2</sup>. The average 15 PAHs concentrations in ambient air of Brazil ranged from  $8.94$  to  $62.5$   $\text{ng/m}^3$  [20] and from  $0.01$  to  $0.9$   $\text{ng/m}^3$  [21]. The PAH concentrations in the former studies were considerably lower than those found in the current study because the current study analyzed 16 PAHs and most of PAHs detected were in the gaseous phase in air samples but the former studies did not collect the gaseous phase of PAHs. In addition, some of the former studies collected only  $\text{PM}_{2.5}$  and analyzed for PAHs. The total PAH concentrations

in industry, urban and rural areas in Taiwan were  $1650 \pm 1240$ ,  $1220 \pm 520$ , and  $831 \pm 427$  ng/m<sup>3</sup>, respectively [22]. The PAH concentrations in urban area in Taiwan were considerably higher than those found in the current study. The varying in PAH concentrations in urban area can be explained by different sampling and analysis method, volume and duration of air sample collection. Regarding to traffic sources in Taiwan, the average total PAH concentration was  $8,110$  ng/m<sup>3</sup> and the major PAHs found was naphthalene followed by acenaphthene and fluorine [23].

## CONCLUSION

The modified method can be used for analysis of PAH concentrations in ambient air. The method used sampling equipment and sampling protocol following National Institute of Occupational Safety and Health, method 5515 and preparation of sample using n-hexane as extraction solvent and analysis with the GC-MS. The method is simple with satisfactory accuracy, precision and detection limit. The GC/MS can also be used to confirm the identification of each PAHs.

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