



Original Article

Distribution of Phthalate Acid Esters in Indoor and Outdoor Air Samples Collected from Hanoi Metropolitan Areas in Vietnam

Vu Van Tu^{1,2}, Nguyen Viet Anh³, Vu Thi Thanh Nhai¹,
Nguyen Thi Kim Oanh¹, Tu Binh Minh¹, Tran Manh Tri^{1,*}

¹VNU University of Science, 19 Le Thanh Tong, Hoan Kiem, Hanoi, Vietnam

²Vietnam Academy of Science and Technology, 18 Hoang Quoc Viet, Cau Giay, Hanoi, Vietnam

³Vietnam Institute of Industrial Chemistry, 2 Pham Ngu Lao, Hoan Kiem, Hanoi, Vietnam

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Abstract: In this study, ten phthalic acid esters (PAEs) were measured in indoor and outdoor air samples collected from Hanoi metropolitan areas. The target compounds were adsorbed onto a solid adsorbent matrix (C18) with a low-speed pump and then analyzed using gas chromatography-tandem mass spectrometry. The recoveries of surrogate standards ranged from 78.5 to 112% (RSD < 15%). The method detection limits of target compounds were in the range of 0.08-0.3 ng/m³. The total mean concentration of 10 PAEs in indoor and outdoor samples were in ranges of 1400-3710 ng/m³ and 178-487 ng/m³, respectively. Among PAEs, di-(2-ethyl)hexyl phthalate (DEHP) was found at the highest abundance and concentration. The distribution of PAEs in indoor air was significantly higher than those in outdoor air samples. Some pairs of PAEs (BzBP vs. DCHP, DEHP vs. DnHP, DnHP vs. DnOP, and DEHP vs. DnOP) had strong correlations and suggested that they are from the same source. Human exposure doses to PAEs through inhalation were estimated for two age groups (children and adults) based on the measured concentrations of PAEs in air, inhalation rates, and body weights. The estimated daily intake doses ranged from 29.7-750 ng/kg-bw/d and 7.29-181 ng/kg-bw/d for children and adults, respectively.

Keywords: Phthalic acid esters; Air pollution; Indoor air; Outdoor air; Human exposure.

1. Introduction

Phthalates or phthalic acid esters (PAEs) are common plasticizers with featured properties such as soft, flexible, easy to mold,

etc. [1, 2]. Therefore, they are widely used in many consumer products, such as in many different commercial products such as construction materials, household items, medical materials, and personal care products (PCPs) [3, 4]. Global plastic use consumes more than 3 million tons of phthalates per year,

* Corresponding author.

E-mail address: manhtri0908@gmail.com

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and the estimated global market of phthalates in 2020 is expected to reach 10 billion USD [5].

Due to their ubiquity in the environment, human exposure to phthalates leached from waste plastics is virtually unavoidable [5]. Many earlier studies showed that PAEs have been released into different environments, such as soil, sediment, air, dust, water, and food [6-10]. The levels of PAEs in indoor dust samples collected from workplaces and homes in Vietnam were up to 153,000 ng/g and 83,700 ng/g, respectively [8]. Additionally, PAEs were detected in various water samples collected from Hanoi, Vietnam, such as bottled water (mean: 6,400 ng/L), tap water (mean: 11,200 ng/L), lake water (mean: 51,800 ng/L), and wastewater (121,000 ng/L) [11]. PAEs were also quantified with high concentration levels in various micro-environments such as water, soil, dust, air or food, and personal care products collected from China [7, 12]. Furthermore, PAE metabolites were also detected in marine organisms [13] and found in biological or tissue samples such as urine, blood, sweat samples, or semen of infertile men [14-16].

Previous studies on PAEs in the human body have indicated that they are a series of widely used chemicals that are demonstrated to be endocrine disruptors and are detrimental to human health. Despite the short half-lives in tissues, chronic exposure to phthalates will adversely influence the endocrine system and functioning of multiple organs, negatively impacting the success of pregnancy, child growth and development, and reproductive systems in both young children and adolescents [5]. PAEs were approved as one of the organic substances causing endocrine-disrupting chemicals (EDCs) in the human body. The United States Environmental Protection Agency (EPA) classification of di(2-ethylhexyl)phthalate (DEHP) as a B2 "probable human" carcinogen [17, 18].

PAEs are semi-volatile organic compounds (SVOCs). SVOCs in the air are generally collected using active and passive sampling techniques. Currently, active air samplers (AAS) have been used to measure the

abundance of SVOCs in both indoor and outdoor settings [19]. Previous studies used several air sampling methods for PAE analysis with polyurethane foam, XAD-2 resin, Tenax TA tube, and octadecylsilane [6, 10, 20, 21]. In this work, the main purpose is to: i) Monitor the distributions of PAEs in the indoor and outdoor air in various sample locations in Hanoi, Vietnam; ii) Assess the potential sources of PAEs in air by statistical analysis; and iii) Estimate the risk of human exposure to PAEs through inhalation.

2. Materials and Methods

2.1. Chemicals

Ten Target compounds: dimethyl phthalate (DMP), diethyl phthalate (DEP), di-*n*-propyl phthalate (DPP), di-*n*-butyl phthalate (DnBP), diisobutyl phthalate (DiBP), di-*n*-hexylphthalate (DnHP), benzylbutyl phthalate (BzBP), dicyclohexyl phthalate (DCHP), di(2-ethylhexyl)phthalate (DEHP), and di-*n*-octyl phthalate (DnOP) and seven surrogate standards including DMP-*d*4, DEP-*d*4, DPP-*d*4, DBP-*d*4, DiBP-*d*4, BzBP-*d*4, DEHP-*d*4 were purchased from Sigma-Aldrich (USA). *n*-hexane and acetone with analytical purities were acquired from Merck KGaA (Germany), while dichloromethane (DCM) was obtained from Fisher Scientific (UK). The native and surrogate standard solutions were prepared in *n*-hexane. Working standard solutions at seven levels of 1, 5, 10, 50, 100, 200, and 500 ng/mL by diluting the mixed standard solutions.

2.2. Instrumental Analysis

In this study, PAEs were quantified by using a gas chromatograph (GC Trace 1310, Thermo Scientific, USA) interfaced with a mass spectrometer (ISQ7000, Thermo Scientific, USA). A fused silica capillary column (TG-5MS, 30 m × 0.25 mm × 0.25 μm; Thermo Scientific, USA) was used for chromatographic separation of analytes. Detailed instrumental parameters for PAE analysis were reported in previous studies [6, 22].

2.3. Sample Collection

Eleven pairs of indoor and outdoor air samples were collected from private apartments in 2023. In particular, four pairs of them were collected from apartments on the other floors of a building. The sampler was deployed at a 1.0 to 1.5 m height from the ground. The area of a sampled apartment is more significant than 30 m² and usually has more than one person inside. The sampling protocol generally followed those reported in our previous works [6, 22]. With some modifications in this study, a low-volume air sampler (Buck Libra Plus LP-7 230V Pump Kit; AP Buck Inc., USA) was used for collecting air samples from 10–12 h with a flow rate of 4L/min. A glass tube (7 cm length × 0.6 cm ID) with C₁₈ (17% C, 40–63 μm, 60 Å) (SiliCycle, Quebec City, Canada) was used as the main sorbent. Adsorption glass tubes were prepared and preserved in a desiccator at room temperature for no more than two days before collecting samples. Glass tubes were preserved by aluminum foil and then brought to the collecting sample locations. After collecting samples, the glass adsorbent tubes were immediately taken from a pump sampler, and

transported to the laboratory. Then, the samples were stored for a maximum of one week at -4 °C until the analysis

2.4. Sample Preparation

Firstly, the mixed surrogate standard solution's exact amount (200 ng) was spiked into the glass sorbent tubes. Secondly, 15 mL of *n*-hexane/DCM (1:1,...) with GC purity were flowed through the glass sorbent tubes by using a solid phase extraction (SPE) system with a vacuum pump (Rocker 300, Rocker Scientific, Taiwan). Then, the eluted solution was concentrated under a gentle stream of nitrogen to 1 mL. Finally, the sample solutions were transferred into a GC vial for instrumental analysis.

2.5. QA/QC

Avoiding contamination with PAEs in the working environment was one of the critical quality control steps in this study. Therefore, all glassware was rinsed with double-distilled deionized water, acetone, and *n*-hexane, respectively, and baked at 400 °C for four hours before use. Solvent with purity (GC) > 99.0% was analyzed before sample batches for checking for background contamination.

Table 1. Additional information on QA/QC

PAEs	Blank level (ng)	Recoveries (%)	MDLs (ng/m ³)	MQLs (ng/m ³)
DMP	n.d.	87 ± 6.0	0.10	0.30
DEP	n.d.-3.21	93.0 ± 7.0	0.08	0.25
DPP	n.d.	90.1 ± 8.2	0.08	0.25
DiBP	n.d.-2.68	93.5 ± 8.9	0.08	0.25
DBP	n.d.-3.69	98.5 ± 6.5	0.10	0.30
DnHP	n.d.	94.6 ± 10.9	0.20	0.60
BzBP	n.d.	99.0 ± 7.2	0.30	1.0
DCHP	n.d.	101 ± 5.8	0.25	0.50
DEHP	3.05–24.2	96.8 ± 7.9	0.10	0.30
DnOP	1.56–14.8	97.8 ± 8.7	0.15	0.50
n.d.: Not detection; MDLs: method detection limits; MQLs: method detection limits.				

QC samples (at least 20% total number of injections) were simultaneously analyzed to confirm the contamination and stability of the method. Recoveries of the surrogate standards in blank and actual samples ranged from 78.5% to 112% with relative standard deviation (RSD) < 15%. This study defined method detection limits (MDLs) as shown peaks with signal-to-noise (S/N) ratios > 3. MDLs ranged from 0.08 to 0.30 ng/m³ (Table 1).

2.6. Statistical Analysis and Risk Assessment

Minitab 18® Statistical Software (Minitab Inc.) was used for statistical analysis in this report. Pearson correlation analysis and principal component analysis (PCA) were conducted on the whole dataset to assess correlations and potential sources of PAEs. The level of statistical significance was set at $p < 0.05$. A value of one-half the method detection limits (MDLs) was used in statistical analysis as concentrations below the method detection limits (not detection–n.d.).

Daily intake doses (ID) were calculated based on PAE concentrations quantified in air samples, inhalation rates (IR), and body weights (BW) for adults and children. The ID of selected compounds such as DEP, DBP, and DEHP was compared with respective reference doses (RfD) proposed by the US Environmental Protection Agency [23-25] to estimate hazard quotients (HQ). Equations for ID estimation and exposure parameters were generally adopted from the US EPA [26]. Previous studies [6, 8, 10] were equations for the data sets to conclude.

3. Results and Discussion

3.1. Concentration of PAEs in Air Samples

In this study, PAEs were quantified in twenty-two air samples, including eleven pairs of indoor and outdoor air samples. The total concentrations of 10 PAEs ($\Sigma 10$ PAEs) in indoor and outdoor air samples ranged from 1400 to 3710 ng/m³ and from 178 to 487 ng/m³, respectively (Table 1). There is only DEHP

quantified in all of the air samples, followed by DnOP (90.9%) > DEP (86.4%). DMP, DPP, and DCHP in air samples have lower frequencies, with 72.7 %, 68.2%, and 63.6%, respectively. Meanwhile, DiBP, DBP, BzBP, and DnHP have the same frequency with 81.8%. Our study's detection frequency (DF) of PAEs is significantly lower than in a previous study in *Canada* [27]. In addition, DEHP makes the largest contribution to the distribution of PAEs in both indoor and outdoor air samples, ranging from 28.4 to 62.3% and from 9.82 to 52.2%. Additionally, some PAEs contribute less than 10% on average to $\Sigma 10$ PAEs in indoor air samples (DMP, DiBP, DCHP) and outdoor air samples (DCHP). DEHP was extensively used as a plasticizer in many products, especially medical devices, furniture materials, household plastic products, cosmetics, and personal care products [28]. Using large quantities of commercial products containing DEHP may cause it to enter and pollute the environment through release (Table 1).

$\Sigma 10$ PAEs in indoor air samples were higher than outdoor air samples statistically significantly at a 95% confidence level with $p < 0.05$. In detail, $\Sigma 10$ PAEs in indoor air samples ranged from 1400–2970 ng/m³ (mean: 2260 ng/m³; median: 2320 ng/m³). Meanwhile, $\Sigma 10$ PAEs in outdoor air samples ranged from 178–487 ng/m³ (mean: 298 ng/m³; median: 268 ng/m³). DEHP was found at the highest concentration in air samples: indoor air (mean: 1160 ng/m³; median: 1010 ng/m³; DF – *Detection Frequency*: 100%) and outdoor air (mean: 84.7 ng/m³; median: 56.5 ng/m³; R_f : 100%), followed by DEP: indoor air (mean: 361 ng/m³; median: 366 ng/m³; R_f : 100%) and outdoor air (mean: 48.3 ng/m³; median: 38.5 ng/m³; R_f : 72.7%). In contrast, DMP and DCHP had the lowest concentration levels in indoor air samples (mean: 44.9 ng/m³; median: 32.5 ng/m³; R_f : 81.8%) and outdoor air samples (mean: 6.69 ng/m³; median: 4.46 ng/m³; R_f : 81.8%). The existence and fate of PAEs in an indoor environment are attributed to leachability, indoor air volume, rate of air

exchange between the indoor and outdoor environments, humidity, building features, and indoor temperature [29, 30]. With outdoor air, wind direction was also found to impact the air quality inside the building significantly. Wind direction was usually affected by real-time and weather [31].

PAEs quantified results in this study had the same levels as the concentration of PAEs in ordinary offices (mean: 2860 ng/m³; median: 478 ng/m³), classrooms (mean: 3730 ng/m³; median: 2100 ng/m³) [18]. Another study also quantified similar results in indoor air samples in Poland with mean = 2860 ng/m³ and, median = 3030 ng/m³ [20]. Besides, ΣPAEs are lower than Σ6PAEs in the laboratories with 40

samples in Guangzhou, southern China (mean: 6390 ng/m³; median: 1700 ng/m³) [32]. In contrast, previous studies showed lower concentration levels than this study, such as ΣPAEs in bedrooms (mean: 670 ng/m³; median: 670 ng/m³; DF: 100%) and (mean: 643 ng/m³; median: 530 ng/m³; DF: 100%) most used homes in Canada [27]. Levels of PAEs quantified concentration in our outdoor air samples were higher than concentrations detected in Hangzhou, China (mean: 38.0 ng/m³; SD: 16.3 ng/m³) [29], in France (Σ7PAEs: 24.1 ng/m³; SD: 20.4 ng/m³; [33] or in Seoul, Korea [34] but significantly lower than those detected in China [7] and the same levels with PAEs concentration in the outdoor air samples in Shanghai, China [35].

Table 1. Summary statistics (mean, median, Q1, Q3, DF) of PAEs in indoor and outdoor air samples from Hanoi, Vietnam

Sampling site	DMP	DEP	DPP	DiBP	DBP	BzBP	DCHP	DnHP	DEHP	DnOP	
Indoor Air	Mean	44.9	361	76.1	49.5	126.2	64.2	61.1	132.6	1159	183
	Q1	5.90	112	n.d.	n.d.	32.5	25.4	4.50	58.5	780	42.5
	Median	32.5	366	71.5	42.5	69.5	52.5	59.8	111	1011	111
	Q3	82.5	428	157	85.4	258	69.5	85.5	225	1452	385
	DF (%)	81.8	100	72.7	72.7	90.9	90.9	81.8	90.9	100	100
Outdoor Air	Mean	27.1	48.3	17.6	27.9	39.8	12.7	6.69	16.16	84.7	17.5
	Q1	n.d.	n.d.	n.d.	12.4	n.d.	n.d.	n.d.	n.d.	39.5	3.87
	Median	21.5	38.5	21.5	21.0	27.8	10.2	4.46	8.82	56.5	8.87
	Q3	58.4	108	28.7	48.5	61.5	26.5	10.2	34.5	124	11.2
	DF (%)	63.6	72.7	63.6	90.9	72.7	72.7	45.4	72.7	100	81.8

3.2. Distribution of PAEs in Air Samples

In this report, four pairs of indoor and outdoor air samples were collected on the ground floor, 8th floor, 27th floor, and 37th floor of a building, respectively (Figure 1). Σ10PAEs in indoor air samples ranged from 6.09 times (ground floor) to 8.34 times (27th floor) than

outdoor air samples. Σ10PAEs in indoor and outdoor air samples ranged from 1480 to 2970 ng/m³ and 190 to 487 ng/m³, respectively. Particularly, Σ10PAEs in indoor and outdoor air samples on the ground floor had the highest concentration levels, with 2970 and 487 ng/m³, respectively. In contrast, the lowest concentration level in indoor air samples was

1480 ng/m³ (37th floor), and in outdoor air samples was 190 ng/m³ (27th floor). Σ 10PAEs in indoor air samples were higher than in outdoor air samples statistically significantly at a 95% confidence level with $p < 0.05$.

In general, the difference in Σ 10PAEs between indoor air samples on different floors is not statistically significant at a 95% confidence level ($p < 0.05$). In contrast, the difference between outdoor air samples is statistically significant, $p > 0.05$. The height above ground (the floor on which the measurements were made) was found to have a significant influence on the concentration of pollutants [36]. A previous study reported that,

according to increasing height, those of biogenic sources, solvent use, and gasoline vehicular emissions decreased, and those of fuel combustion and industrial emissions remained unchanged. Besides, wind direction was also found to significantly impact the air quality inside the building [31].

These may be some of the reasons for the difference between Σ 10PAEs in outdoor air samples on different floors of a building in this study. The other study showed that various factors, such as air ventilation and seasonality, all affected the concentrations of organic compounds [37].

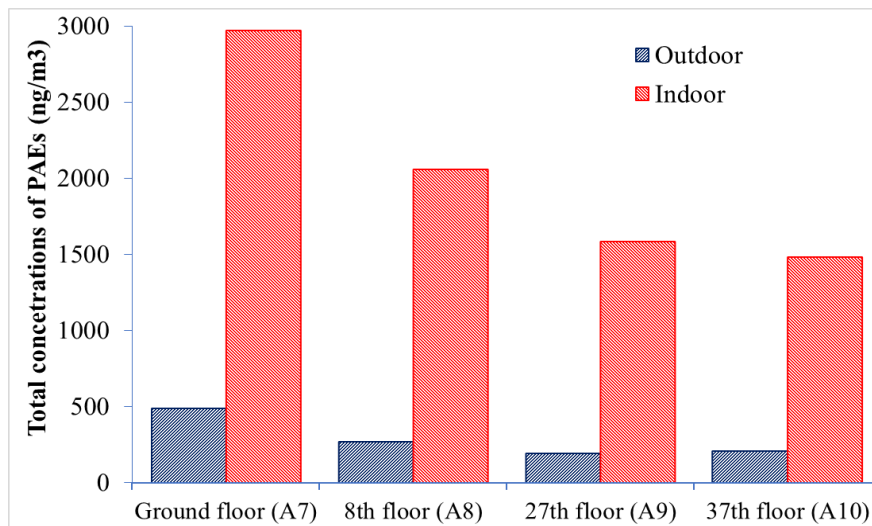


Figure 1. Total concentration (ng/m³) of PAEs in air samples according to altitude.

3.3. Potential Sources of PAEs in Air

The Pearson correlation matrix was used for this report's data set to find the strong correlation between pairs of PAEs and assess the origin of each pair of individual PAEs (Table 2). Some pairs of PAEs had strong correlations, such as BzBP versus DCHP ($r = 0.643$), DEHP versus DnHP ($r = 0.660$), DnHP versus DnOP ($r = 0.720$), and DEHP versus DnOP ($r = 0.797$). Besides, DEP versus DEHP was the pair of PAEs with the highest correlation with $r = 0.825$ in this data set. They were two of the most used PAEs

in commercial products and were listed as hazardous pollutants by the US EPA. In general, high-molecular-weight PAEs such as BzBP, DEHP, DCHP, DnHP, and DnOP had strong correlations with each other. In reality, they were used a lot as a plasticizer in common indoor or outdoor products such as vinyl tiles, vinyl gloves, adhesives, caulks, food conveyor belts, carpet tiles, artificial leather, tarps, automotive trim, and traffic cones [4, 38]. These results proposed that high-molecular-weight PAEs in air samples collected in Hanoi urban areas could have the exact origin because of their strong correlations.

In addition, the principle component analysis (PCA) was used to initially find assessments of the potential sources of PAEs in the air samples (Figure 2). From the results of the rigenanalysis of the correlation matrix, PC1 and PC2 accounted for the highest percentage of the cumulative variance, with 37.4% and 23.0%, respectively. After analyzing eigenvectors, the result indicated that PC1 and PC2 had high correlations with some high molecular weight (HMW) PAEs. Particularly, PC1 positively correlated with DCHP, DnHP,

and DEHP. PC2 had a strong correlation with DnOP. DEHP and DnOP are mainly used as plasticizers, and they are applied in household plastic products and cosmetics or personal care products (PCPs) [1, 39]. Meanwhile, DnHP and DCHP are widely used as a binder in ink or coating products [40, 41]. PCA results indicated that some high-molecular-weight PAEs accounted for the distribution and the main pollutant factors. Besides they had a high impact on the accumulation of PAEs in the air samples.

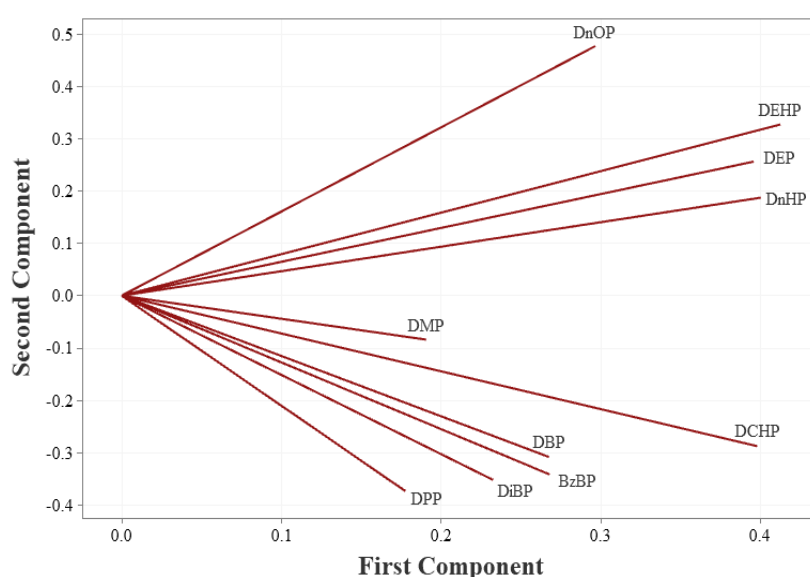


Figure 2. Principal component analysis of PAEs in the air samples.

3.4. Risk Assessment of Human Exposure to PAEs through Inhalation

There have been various previous studies evaluating the risk assessment of human exposure to PAEs from several microenvironments such as dust, air, water, food, and personal care products [8-11, 42, 43]. In this study, our data set was used to calculate daily intake doses (ID) of individual PAEs through inhalation (US EPA, 2011) and hazard quotients (HQ) for non-cancer endpoints of DEP, DBP, and DEHP (Table 3). Daily intake dose results of adults and children were in ranges of 7.29-181 ng/kg-bw/d and 29.7-750 ng/kg-bw/d, respectively. Generally, daily intake dose

values were much lower than the reference dose of some PAEs such as DEP (800,000 ng/kg-bw/d; [23], DnBP (100,000 ng/kg-bw/d; US EPA, [24], and DEHP (20,000 ng/kg-bw/d; US EPA, [25].

In comparison, daily intake doses of children exposed to PAEs through inhalation had higher levels than previous studies [8, 10]. The ID values through air inhalation were also lower than the risk assessment of PAEs in outdoor dust samples on the Tibetan Plateau, China (adults: 24.7 ng/kg-bw/d; children: 15.0 ng/kg-bw/d), [44] or than risk assessment of PAEs through the consumption of bottled drinking water [11] or coffee [43].

Table 2. The estimated human exposure doses to PAEs through inhalation for adults and children

Compounds	Adults	Children
<i>Intake doses (ng/kg-bw/d, median and range)</i>		
DMP	7.29 (NA–17.7)	29.7 (NA–72.1)
DEP	56.7 (4.99–140)	234 (20.7–577)
DPP	12.0 (NA–33.3)	49.6 (NA–73.8)
DiBP	8.01 (NA–18.4)	32.7 (NA–73.8)
DBP	20.1 (NA–56.3)	82.6 (NA–232)
DnHP	10.1 (NA–29.1)	47.8 (NA–120)
BzBP	9.58 (NA–33.1)	39.6 (NA–137)
DCHP	20.8 (NA–43.3)	86.0 (NA–179)
DEHP	181 (71.5–320)	750 (296–1330)
DnOP	28.6 (3.50–70.6)	118 (14.5–291)
<i>Hazard quotients (median and range)</i>		
DEP	7.09×10^{-5} (6.24×10^{-6} – 1.74×10^{-4})	2.93×10^{-4} (2.59×10^{-5} – 7.21×10^{-4})
DBP	3.98×10^{-4} (NA– 1.12×10^{-3})	8.26×10^{-4} (NA– 2.32×10^{-3})
DEHP	9.06×10^{-3} (3.58×10^{-3} – 1.60×10^{-2})	3.75×10^{-2} (1.48×10^{-2} – 6.63×10^{-2})
NA: Not available due to non-detected compounds		

Hazard Quotient values (including DEP, DBP, and DEHP) for adults and children in this data set ranged from 10^{-5} to 10^{-3} and from 10^{-4} to 10^{-2} , respectively. This result showed similarities in level compared to some previous studies [6, 8, 10].

4. Conclusions

This study has provided new insights into the distribution of PAEs in indoor and outdoor air from Hanoi urban areas. The level of PAE pollution in the air according to space, height and dispersion origin was also investigated and evaluated in this report. To our knowledge, this is the first study to report the distribution of PAEs in air according to altitude. Based on measured concentrations, the risk of exposure to PAEs through air inhalation was estimated for adults and children at doses of 7.29–181 ng/kg-bw/d ng/g and 29.7–750 ng/kg-bw/d, respectively. Overall, the research has provided

basic scientific data that may help environmental management agencies issuing of regulations to control these pollutants in the future.

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