



Analysis of polycyclic aromatic hydrocarbons (PAHs) in air using passive sampling calibrated with active measurements[☆]



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ARTICLE INFO

Article history:

Received 5 April 2017

Received in revised form

7 August 2017

Accepted 11 August 2017

Keywords:

Passive air sampling

Air sampling rate

Polycyclic aromatic hydrocarbons

ABSTRACT

There are limited ambient air measurements of extended (beyond EPA Priority 16) lists of polycyclic aromatic hydrocarbons (PAHs). We measured air concentrations of 45 PAHs using passive and active air sampling at 15 sites in a central urban community and one rural site for two years. Passive sampling was conducted with cylindrical XAD-based samplers deployed to capture spatial variability. High volume active samplers with quartz fiber filters for particles and XAD-4 absorbent for gases were deployed at two urban sites and the rural site to calibrate the passive measurements directly. Estimated passive sampling rates (PSRs) were evaluated as functions of meteorological data, seasons, locations, study year, and compared with other studies. Possible particle collection by the passive samplers was evaluated using a variety of particle measurements (TSP, PM₁₀, PM_{2.5} and ultrafines <100 nm). Total PAHs were statistically associated with ultrafine particle concentrations and to a lesser extent PM_{2.5} and PM₁₀, but not TSP. PSRs were more variable when PAH mass loadings were lower and near method detection limits; this occurred more often at the rural site. The PSRs were not statistically associated with meteorological conditions in this study, but wind speed had the highest potential to impact PSR results. The resulting passive PAH measurements are reported with respect to proximity to major roadways and other known air emissions types. PSRs were quantifiable for some PAHs that were found predominantly in the particulate phase in active sampling. This information, together with particle fraction calculations from active sampling, were used to estimate the particulate PAH capture of the passive sampler. Summed PAH (Σ PAH) passive concentrations were measured within the range of 10–265 ng/m³, with the highest concentrations from naphthalene and the lowest detected concentrations from anthracene. These results indicated a stronger seasonal signal within 200 m of a major roadway.

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1. Introduction

Polycyclic aromatic hydrocarbons (PAHs) comprise a category of substances that are emitted to the air mainly from anthropogenic combustion sources (Ravindra et al., 2008). PAH sources do not typically emit a single PAH, but emit complex and somewhat reactive parent PAH and substituted PAH mixtures into the air. PAHs

are semi-volatile substances that can be present in the atmosphere in the gas phase, the particle phase, or both, depending on the environmental conditions and individual PAH vapor pressure. Toxicity and potency varies among individual PAHs, and many of them are known carcinogens, respiratory irritants, and/or are classified as persistent, bioaccumulative toxicants (PBTs).

Ambient air is measured using active or passive sampling techniques, where active samplers pull ambient air through a filter and sorbent material using an air pump. Passive air sampling is the collection of air pollutants by the diffusion of gases, and to a lesser extent particle entrainment, onto and into a sampling medium

[☆] This paper has been recommended for acceptance by Charles Wong.

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(Górecki and Namieśnik, 2002). Passive sampling compliments active air sampling by providing methods that are lower cost, require less security, are noiseless, and do not require power (Gouin et al., 2005). This allows the deployment of more samplers giving higher spatial resolution that is otherwise cost prohibitive. Higher density air sampling in a defined location such as a neighborhood is especially important to public health agencies who aim to answer questions from concerned citizens about potential health effects. However, if passive sampling methods are used either in isolation or in complement to active measurement methods, it is inherent that individual PAH concentrations are determined using PAH specific passive sampling rate (PSR) values in order to evaluate the toxicity or the cancer potency of these complex mixtures in ambient air (MDH, 2016; USEPA, 2010).

Passive air sampling was first used in the late 1920s, but did not become a quantitative methodology until the 1970s (Palmes et al., 1976). Since then, it has grown in use and by the variety of analytes and types of samplers to the present. There are passive air sampling devices for many air toxics and criteria pollutants (Nash and Leith, 2010) including semi-volatiles (Chaemfa et al., 2008; Jaward et al., 2004). Since the effective volume (V_{eff}) of air to the passive sampling media is unknown, the estimation of a PSR is required for the estimation of air concentrations. PSRs may be calculated by a theoretical model (Armitage et al., 2013; Herkert et al., 2016; May et al., 2011), utilized from past studies (Barthel et al., 2012), calculated based on activity of depuration or performance reference compounds (Moeckel et al., 2009), or PSRs may be estimated based on direct calibration from collocated active air measurements (Klanova et al., 2006; Liu et al., 2016; Wania et al., 2003). The use of direct calibration is considered the gold standard for PSR estimation (Herkert et al., 2016), in part because a PSR is estimated for each individual pollutant and site specific characteristics such as ambient temperature and wind speed are reflected in the measurements. The direct calibration of passive samplers to active sampler measurements is well established in the literature (Saini et al., 2015; Armstrong et al., 2014), however, less is known about the particle collection of passive samplers (Markovic et al., 2015).

This manuscript builds upon past investigations of gas and particle sampling rates (Holt et al., 2017), and incorporates the estimation of particulate phase PAH sampling by the passive sampler inferred from associations between ultrafine counts, total PAHs concentrations, particle fraction estimates, and sampling rates for the cylindrical XAD passive sampler. We present active and passive air monitoring results, and the corresponding PSRs for an extended list of PAHs (Table S1) measured over a two-year time period using measurements from co-located passive and active samplers. The study was a collaborative multi-agency effort between the Minnesota Pollution Control Agency (MPCA), the Minnesota Department of Health, and the Mille Lacs Band of Ojibwe Department of Natural Resources and Environment. In this manuscript, (1) we estimate sampling rates from direct calibration of passive samplers, (2) we investigate the potential for uptake of particulate phase PAHs by the XAD passive sampler from in situ particulate measurements (collocated TSP, PM_{10} , $\text{PM}_{2.5}$ and ultrafine measurements), (3) we investigate the seasonal and pollutant variability of sampling rates, and (4) we compare the estimated PSRs from this manuscript to PSRs from the existing literature.

2. Materials and methods

Site Selection: Air sampling took place in South Minneapolis and on Mille Lacs Band of Ojibwe land in Minnesota USA (Fig. 1, Table S2). The Minneapolis study location is an urban community of high-density residential areas near major highways, whereas the Mille Lacs study location is a forested area on the banks of one of

the largest lakes in Minnesota (Lake Mille Lacs). Active samplers were placed at three existing fixed platform ambient air network sites (MPCA, 2016) with collocated active samplers at the Minnesota network ambient monitoring site Near Roadway (Site ID 962). Passive samplers were co-located with each active sampler, and duplicate passive samplers were sited at Mille Lacs, and Near Roadway. Fourteen passive-only monitors were also sited in South Minneapolis based on modeled results (Pratt et al., 2012), known PAH air emissions sources, demographics, and community member input. The sampling network was dense enough to compare locations for intra-urban variability.

Air Sampling: Active air samples were collected from June 2013 to June 2015 at a frequency of 1 in 12 days for 72 h at stationary fixed platform sites using Tisch Environmental PUF110V high volume samplers set at a flow rate of $120\text{m}^3/\text{day}$. The sampling train included a quartz fiber filter (QFF) (particle), a stainless steel screen, and sieved XAD-4 (gas). Passive sampling was conducted seasonally over the same time period with an XAD-4 resin tube inside a cylindrical passive sampler housing (Schrlau et al., 2011; Zhang et al., 2011). XAD-4 was used instead of the more commonly used XAD-2 due to its higher sorptive capacity from increased pore volume and at least twice the surface area (Lee et al., 2004). Samplers were sited on trees and non-wood utility poles to avoid potential contamination from wood treatment (Kohler et al., 2000).

The MPCA runs an extensive ambient air monitoring network, following USEPA quality assurance plans and standard operating procedures. The fine particle data ($\text{PM}_{2.5}$) were collected using a Met One Instrument BAM-1020 (BAM) continuous mass monitor that collects and report hourly concentrations. The coarse particles (PM_{10}) were collected hourly using a Federal Equivalent Method (FEM), similar to $\text{PM}_{2.5}$. PM_{10} was collected using a monitor Met One Instrument BAM-1020 (BAM), but set up with a PM_{10} cut-off. The hourly concentrations were averaged to the 72 h sample deployment time of the PAH ambient air monitoring. Total suspended particulate is still measured in Minnesota because of an existing state ambient air quality standard for that pollutant. These mass based measurements are 24 h integrated collections using a Tisch Environmental high volume air sampler. The ultrafine particles were collected for the years 2014 and 2015 using a TSI™ Model 3031 ultrafine particle monitor. These 15 min data were reported in 5 channels (20–30 nm, 30–50 nm, 50–70 nm, 70–100 nm, and 100–200 nm), they were summed to <100 nm, and 72 h means were calculated for comparison with the active PAH monitoring data.

Sample Media Preparation and Chemical Analysis: Extraction and chemical analysis of PAHs followed, EPA Method 8270D, Revision 4 (USEPA, 1998a), EPA Method 3545A, Revision 1, Modified (USEPA, 1998b), and a modified EPA Compendium Method Toxics Organics-13A (USEPA, January 1999). Sampler housing and media were cleaned, set up, bagged, and refrigerated at the MDH-PHL (Public Health Laboratory) prior to field deployment. After each use the sampler housings were washed with soap and water and rinsed with deionized water. Before initial use, the raw XAD-4 was washed with deionized water to remove the brine solution and sieved to remove fines (No.30 sieve). After each use, the XAD-4 was cleaned with multiple dichloromethane (DCM) extractions using an Accelerated Solvent Extraction (ASE) apparatus. The cleaned XAD-4 was extracted a final time with acetone to remove DCM for safety reasons and stored in air tight jars in a freezer. The QFF were baked in a muffle furnace at $450\text{ }^{\circ}\text{C}$ for 4 h to remove any organic compounds.

Air samples were held at $\leq 4\text{ }^{\circ}\text{C}$ until the PAH fractions were extracted from QFF or XAD-4 by an ASE method using dichloromethane. The extract was concentrated to 1.0 mL and analyzed by injection of 2.0 μL of the extract into a multimode inlet with a

PAH Monitoring Locations

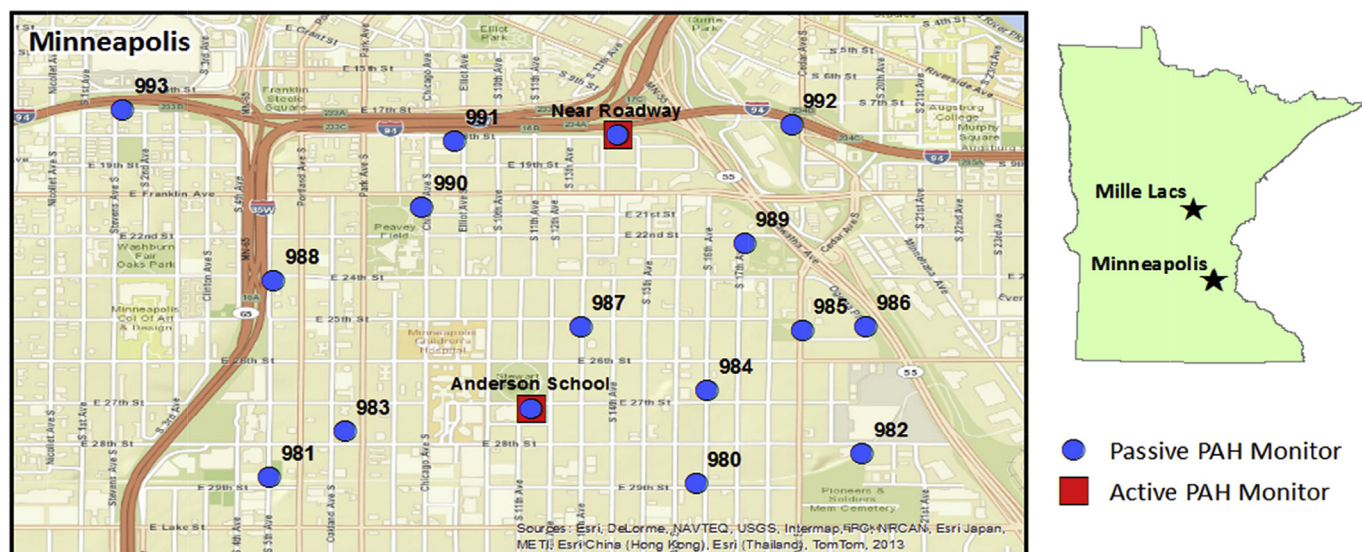


Fig. 1. Map of the sampling site locations. Near Roadway is the name of a Minnesota network ambient monitoring site, with the identification number 962. Anderson School is a Minnesota network ambient monitoring site, with the identification number 963.

splitless single taper liner connected to a fused silica capillary column (J&W Scientific Select PAH Column, 30 m × 0.25 mm I.D. × 0.15 μm film thickness). Quantitation was done using an Agilent model 5977 mass spectrometer using selected ion monitoring/gas chromatography/mass spectrometry (SIM/GC/MS) programmed from 60 °C to 350 °C using 4 °C temperature ramps. Target analytes were quantified by comparing the response in a field sample to the response in a 10 point standard curve (1–1000 ng/mL). A silica gel cleaning procedure (Discovery® Solid Phase Extraction (SPE) DSC-Si Silica Tubes, 3 mL, 500 mg, Supelco, Part # 52654-U) was required for the passive sampling media to remove the mass interferent Dibenzothiophene sulfone for improved Benzo [c]fluorene separation and identification.

Estimation of air concentrations from passive sampler mass loadings: Air concentrations (ng/m³) were estimated from passive mass loadings (ng) by direct calibration to the active air measurement levels. This approach assumes that the passive and active samplers are both sampling the same air. The mass collected by each method is known, and the sampling rate is known for the active sampler. The PSR can be estimated by the ratio since the mass collected is proportional to the sampling rate. We calculated the passive sampling rate separately for each PAH for each passive sampling event because passive diffusion and PAH absorption may vary with ambient conditions and for different compounds. Active samples were collected for 72 h every 12 days constituting 23% of the passive sample duration, so our calibration assumes non sampled times are equivalent to sampled times. The calibration factor (PSR m³/day) is an estimate of the effective volume of air of the XAD-4 passive samplers (Jaward et al., 2004; Shen et al., 2004; Armitage et al., 2013; Harner et al., 2006; Chaemfa et al., 2008). The terms of the PSR equation are as follows:

$$\text{Sampling Rate (m}^3\text{day}^{-1}) = \frac{m_{\text{PAS}}}{C_{\text{AIR}} * \text{Sampling Duration (days)}}$$

where m_{PAS} = mass of specific PAH on passive sampler media (ng), and C_{AIR} = concentration of specific PAH in air from active air sampling (ng m⁻³). The mean active air concentrations were bulk air concentrations (gas + particulate) (Bohlin et al., 2010, 2014;

Klanova et al., 2008; Bohlin et al., 2010; Melymuk et al., 2011) averaged over the duration of the sampling deployment period of three months (7 samples per mean). PSRs were calculated for each set of collocated active and passive samples with results tabulated by site, pollutant, season, and study year. PSRs were then averaged by study location (Minneapolis or Mille Lacs, Table 2). PSRs were calculated seasonally to test for associations with meteorological data, and later calculated as individual gas sampling rates (R_s) and particulate sampling rates (R_p) to test for particle collection by the XAD sampler.

PSRs for triphenylene (analyzed to inform chrysene results) and acenaphthylene (quality assurance issues) were eliminated from reporting and/or the statistical analyses, but were included for informational purposes in Table 2. PSRs with more than 50% data below method detection limits (MDLs) and PSRs equal to zero, where a zero PSR is equivalent to a zero mass loading, were eliminated from statistical analysis. Finally, we confirmed that there were at least five total values within each site and PAH group prior to statistical analysis.

Quality Assurance and Quality Control: The project followed an EPA approved Quality Assurance Project. Data were checked by calibration standards, internal standards, laboratory control standards and duplicates, surrogate spikes; and field and method blanks. Data were accepted without rejection or qualification if laboratory control standard recoveries were between 30 and 150%. Laboratory control standard duplicate relative potential difference was acceptable without rejection or qualification if within ± 50%. Surrogates were run for Benzo [a]pyrene-d₁₂, Fluoranthene-d₁₀, Fluorene-d₁₀, Pyrene-d₁₀, Benzo [e]pyrene-d₁₂, and Benzo [g,h,i] perylene-d₁₂. Data were identified as censored for summary statistics if reported below the MDL, NULL, or reported as zero. The study wide mean MDLs are reported in Table 2, and season specific MDLs are reported in Table S4 in supplemental information.

Passive mass loading at each of the collocated samplers at Mille Lacs (3051) and Near Roadway (962) were tested for agreement by employing linear regression (Table 1). Linear regression was completed for collocated sample pairs with more than 5 of the possible 7 passive mass loading values above zero. Collocated results were in good agreement with the exception of Naphthalene

Table 1
Collocated Passive Sampler Measurement Agreement. Linear regression results for collocated passive sampler results (mass loadings). The parenthetical results exclude the outlying value. *Dibenzothiophene sulfone and triphenylene were included in Table 1 for informational purposes but were eliminated from all other analyses.

	Near Roadway (962)		Mille Lacs (3051)	
Analyte	Adjusted R Squared	P Value	Adjusted R Squared	P Value
2-Methylnaphthalene	–0.12 (0.95)	0.62 (<0.01)	0.92	<0.01
Acenaphthene	0.94	<0.01	0.98	<0.01
Anthracene	0.92	<0.01		
Benz [a]anthracene	0.53	0.04		
Chrysene	0.91	<0.01		
Dibenzothiophene	0.97	<0.01	0.96	<0.01
Dibenzothiophene sulfone*	0.99	<0.01	0.99	<0.01
Fluoranthene	0.94	<0.01	0.80	<0.01
Fluorene	0.84	<0.01	0.85	<0.01
Naphthalene	0.03 (0.95)	0.31 (<0.01)	0.92	<0.01
Phenanthrene	0.88	<0.01	0.87	<0.01
Pyrene	0.79	<0.01	0.74	<0.01
Retene	0.86	<0.01	0.92	<0.01
Triphenylene*	0.84	<0.01		

Table 2
Active air concentrations, passive mass loadings, MDLs, and PSRs are reported for the full study duration and represented as a mean \pm a standard deviation. In some cases the mass loading means are low value estimates, and are reported in italics and bracketed by 0 and the MDL. The resulting PSRs are calculated from the estimated mean and active air concentration or from the passive mass loading MDL and bracketed similar to the passive mass loading results.

	Passive Mass Loadings (mean \pm standard deviation)		Mean Passive MDL (ng)	Active Air Concentration (mean \pm standard deviation)		Mean Active MDL (ng/m ³)	Passive Sampling Rate (mean \pm standard deviation)	
	Mille Lacs	South Minneapolis		Mille Lacs	South Minneapolis		Mille Lacs	South Minneapolis
2-Methylnaphthalene	595 \pm 241	4460 \pm 1040	8.3	3.34 \pm 1.65	33.9 \pm 7.84	0.02	2.1 \pm 0.85	1.49 \pm 0.29
Acenaphthene	33.6 \pm 27.4	171 \pm 103	9.58	0.37 \pm 0.38	2.03 \pm 1.26	0.01	1.2 \pm 0.56	0.95 \pm 0.19
Anthracene	3 [0–8]	4 [0–8]	7.92	0.05 \pm 0.08	0.09 \pm 0.05	0.01	0.7 [0–2]	0.5 [0–1]
Benz [a]anthracene	1 [0–4]	2 [0–4]	4.28	0.01 \pm 0.003	0.09 \pm 0.04	0.01	1 [0–4]	0.2 [0–0.5]
Benzo [a]pyrene	ND	1 [0–2]	1.68	ND	0.1 \pm 0.03	0.003	ND	0.1 [0–0.2]
Benzo [b]fluoranthene	detected in only 1 season	3 [0–4]	3.95	detected in only 1 season	0.16 \pm 0.07	0.002	detected in only 1 season	0.2 [0–0.3]
Benzo [b]naphtho [2,1- d]thiophene	detected in only 1 season	1 [0–1]	1.29	detected in only 1 season	0.03 \pm 0.01	0.002	detected in only 1 season	0.4 [0–0.4]
Benzo [c]fluorine	ND	1 [0–3]	3.11	ND	0.02 \pm 0.005	0.006	ND	0.5 [0–2]
Benzo [e]pyrene	ND	1 [0–4]	3.89	ND	0.16 \pm 0.04	0.004	ND	0.07 [0–0.3]
Benzo [g,h,i]perylene	ND	1 [0–6]	6.4	ND	0.2 \pm 0.07	0.006	ND	0.06 [0–0.3]
Benzo [j]fluoranthene	ND	1 [0–8]	8.26	ND	0.1 \pm 0.03	0.01	ND	0.1 [0–0.9]
Benzo [k]fluoranthene	detected in only 1 season	2 [0–4]	3.66	detected in only 1 season	0.07 \pm 0.03	0.007	detected in only 1 season	0.3 [0–0.6]
Chrysene	2 [0–3]	3.72 \pm 1.45	2.77	0.03 \pm 0.01	0.19 \pm 0.1	0.005	0.7 [0–1]	0.28 \pm 0.14
Coronene	ND	detected in only 1 season	10.5	ND	detected in only 1 season	0.005	ND	detected in only 1 season
Cyclopental [c,d]pyrene	ND	1 [0–6]	6.12	ND	0.04 \pm 0.02	0.01	ND	0.3 [0–2]
Chrysene	2 [0–3]	3.72 \pm 1.45	2.77	0.03 \pm 0.01	0.19 \pm 0.1	0.005	0.7 [0–1]	0.28 \pm 0.14
Coronene	ND	detected in only 1 season	10.5	ND	detected in only 1 season	0.005	ND	detected in only 1 season
Cyclopental [c,d]pyrene	ND	1 [0–6]	6.12	ND	0.04 \pm 0.02	0.01	ND	0.3 [0–2]
Dibenz [a,h]anthracene	ND	detected in only 1 season	9.54	ND	detected in only 1 season	0.003	ND	detected in only 1 season
Dibenzothiophene	3 [0–8]	9.96 \pm 3.57	7.95	0.08 \pm 0.07	0.28 \pm 0.18	0.02	0.4 [0–1]	0.51 \pm 0.25
Fluoranthene	26.1 \pm 16.9	137 \pm 54.4	2.5	0.36 \pm 0.19	1.5 \pm 0.57	0.005	0.87 \pm 0.34	1 \pm 0.17
Fluorene	79.2 \pm 33.1	240 \pm 85.3	8.65	0.72 \pm 0.32	2.32 \pm 1.14	0.02	1.3 \pm 0.53	1.2 \pm 0.35
Indeno [1,2,3-cd] pyrene	ND	1 [0–8]	7.92	ND	0.1 \pm 0.03	0.004	ND	0.1 [0–0.9]
Naphthalene	2470 \pm 1200	10600 \pm 2260	21.3	12 \pm 6	75 \pm 16	0.02	2.4 \pm 1	1.6 \pm 0.37
Phenanthrene	151 \pm 72.1	804 \pm 387	8.08	1.54 \pm 0.71	6.38 \pm 2.91	0.005	1.1 \pm 0.31	1.4 \pm 0.21
Pyrene	7.09 \pm 8.43	45 \pm 21	2.29	0.16 \pm 0.1	0.9 \pm 0.3	0.004	0.5 \pm 0.4	0.55 \pm 0.2
Retene	4.93 \pm 3.87	10.9 \pm 5.06	2.48	0.1 \pm 0.05	0.28 \pm 0.16	0.005	0.74 \pm 0.79	0.5 \pm 0.3
Triphenylene	1 [0–12]	3 [0–12]	12.6	0.02 \pm 0.004	0.07 \pm 0.02	0.007	0.5 [0–7]	0.5 [0–2]

and 2-Methylnaphthalene, which were skewed by one outlying result. We did not eliminate these results from PSR calculations since it is unknown which result was erroneous and since these discrepancies did not impact the final PSRs as tested by comparing study year 1 and 2 PSRs.

2.1. Meteorological data

Meteorological data were pulled from nearby National Weather Service ASOS meteorological stations and summarized for comparison with estimated PSRs. The Minneapolis-St. Paul International Airport meteorological station (KMSP) data set was

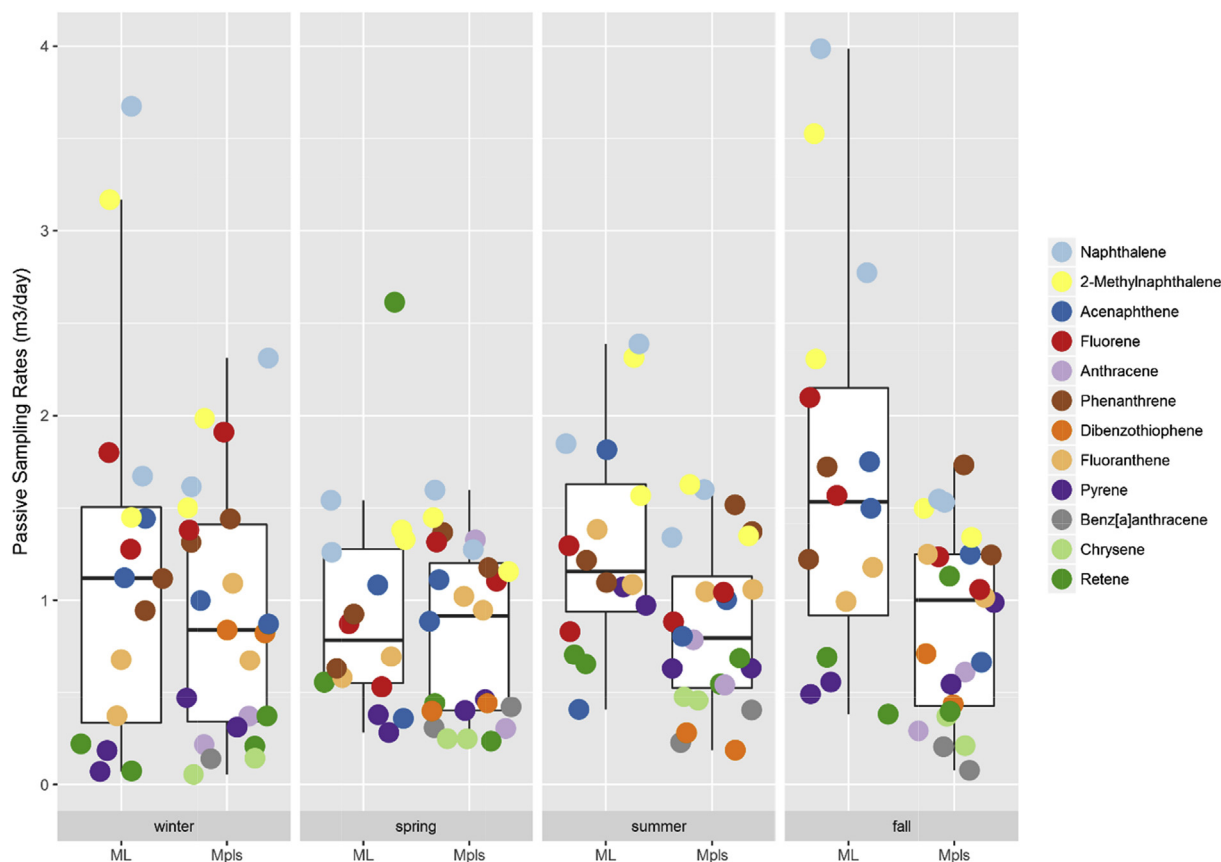


Fig. 2. Comparison of PSRs between south Minneapolis (Mpls) and Mille Lacs (ML) by PAH.

processed to align with Minneapolis PAH data, and data from the Brainerd Airport meteorological station (KBRD) was processed to align with ambient air measurements from Mille Lacs (Table S3, Figs. S1–S4).

3. Results and discussion

Mean active air concentrations, passive mass loadings, and estimated PSRs are reported in Table 2. The active results represent the total (gas plus particle) summarized air concentrations. Concentrations generally decreased as PAH molecular weight increased, and urban concentrations (South Minneapolis) were higher than rural. Passive mass loadings for South Minneapolis reflect means from the Near Roadway site together with the Anderson School site.

3.1. Calculation of PSRs

The PSRs were first estimated in two ways, using gas-phase only (Rs) and using total (gas + particle) loadings (Fig. S5). The results of this study support the use of total (gas plus particle) measurements for estimating PSRs in several ways. Gas-phase only calculations presume that the passive samplers collect only gases and not particles; however, we found non-zero and non-censored passive sampler results for PAHs that were found in the particle phase in active samples in colder months (e.g. Chrysene, Benzo [a]anthracene, Pyrene). A second reason for calculating PSRs using the bulk air concentrations is to account for seasonal gas-particle partitioning by PAHs. Finally, using the bulk air concentration accounts for potential active air sampling artifacts (Bohlin et al., 2014). For

example, gases may react with previously collected particles on the quartz fiber filter and thus be collected on the filter and mistakenly identified as particles (Arp et al., 2007). Finally, airflow across the particle filter may volatilize particle-bound PAHs that are subsequently captured on the absorbent and misidentified as gases (Galarneau and Bidleman, 2006). These issues are alleviated by using total loading for the PSR calculations.

Passive air samplers were deployed for 3 months, and therefore reflect meteorological season averages. We removed two known high-concentration events that were captured by the active samplers since they affected the active air concentrations, but not the seasonal passive mass loadings. The two events captured by the active monitors were an apartment fire on January 1, 2014 and the annual Pow Wow on the Mille Lacs Band's land August 15–17, 2014. Both were uncontrolled combustion events. These events affected the passive monitors for several days over passive deployment. In contrast, the active samplers were affected for a four times larger proportion of their deployment time. For this reason, the inclusion of the extraordinary events resulted in PSR differences of between 3 and 200% for some PAHs. The Pow Wow event produced higher concentrations, and was associated with higher differences in the PSRs. Therefore, the active air results from the event dates were removed from the PSRs calculations.

3.2. Passive sampling rates and meteorological data

The mass loading on the passive sampler is dependent on the diffusion and sorption of the gas phase pollutants onto the sampling media; and for particle phase pollutants likely depend on physical mechanisms of entrainment including diffusion,

impaction, interception, and sedimentation. Since the diffusive layer (Zhang et al., 2013) and particle infiltration into the sampler housing (Markovic et al., 2015) can be modified by ambient meteorological factors, the estimated PSRs were compared to temperature, wind speed, dew point, and estimated wind chill. The PSRs in this study were not statistically different by season (Kruskal Wallis χ^2 -squared 3.5, p-value = 0.32). Wind chill, dew point, and temperature were not significantly correlated with PSRs (PSR ~ Temperature °C Kendall τ = 0.01, p value = 0.89; PSR ~ Dew Point Kendall τ = 0.04, p value = 0.43; PSR ~ wind chill Kendall τ = 0.01, p value = 0.89), but wind speed had a very low but slightly significant negative correlation with PSRs (PSR ~ wind speed Kendall τ = -0.11, p value = 0.04). Approximately 12% of the PSR variability is explained by wind speed in linear regression analysis of these variables. The near significant negative association of wind speed with PSRs suggests it has the potential to influence PSRs across studies and sampling sites, however past studies have found positive relationships between PSRs and wind speed (Zhang et al., 2013). A positive association between wind speed and PSRs is expected since the air-flow around the sampler affects the rate at which turbulent eddies enter the sampler. Past studies have found low impacts of mid-range wind speed (5–15 m/s) on PSRs (Wania et al., 2003), and logarithmic relationships showing increases of PSRs with small increases in wind speeds during calm to low wind speeds (-0 m/s to 1 m/s) (Zhang et al., 2013). Since our PSRs reflect a 3 month deployment, the PSRs were compared to the mean wind speeds observed over the entire sampling period. The variability of these measurements was small, ranging from 2.8 m/s to 4.6 m/s. Therefore, the statistical analysis does not reflect the true variability of wind speeds near the samplers. For this reason, the significant relationship could be a function of the lack of temporal resolution in the data.

3.3. Passive sampling rates and chemical physical parameters

Since there was variation in the PSRs between PAHs, the PSRs were tested for statistical association with several of their chemical-physical parameters (USEPA, 2012; DOE, 2016). The PSRs were statistically associated with chemical-physical parameters that are associated with increased diffusion and volatility in air

(PSR ~ Diffusivity in air cm²/s: Kendall τ = 0.38, p value < 0.01; PSR ~ unitless Henry's Law: Kendall τ = 0.50, p value < 0.01; and PSR ~ Subcooled liquid vapor pressure mmHg: Kendall τ = 0.54, p value < 0.01). These parameters are obvious covariates, but a comparison of linear regression coefficient estimates for each chemical-physical parameter with corresponding PSRs results in subcooled liquid vapor pressure as the highest coefficient (0.86), Henry's Law (0.63) and diffusivity in air with the lowest coefficient (-0.26) and therefore explaining the least variability in PSRs.

3.4. Passive sampling rates, study years and study locations

The air sampling described in this manuscript occurred for 2 years at two distinct study locations. Prior to combining study year one with study year two, statistical differences in the sampling rates for the two study years were tested. Furthermore, since the two study locations were different in land cover, type, and population density the site-based PSRs were also tested for significance. As stated in the previous paragraph, PSRs were neither significantly different between seasons (Fig. 2) nor study years (Kruskal Wallis χ^2 = 0.05, p-value = 0.83). PSRs were significantly different between South Minneapolis and Mille Lacs (Kruskal Wallis χ^2 = 8.38, p-value < 0.01). Mille Lacs had the lowest measured concentrations and greater variability in PSRs. This was true for all PAHs, especially in the fall and winter. The PSR variability (standard deviation/mean) was significantly associated to the percent of censored values in the calculation (adjusted R^2 = 0.16, p-value < 0.01). This suggests that values near or below detection had an effect on the variability of the calculated PSR. PSR variability was highest for high PSRs and when measurements were near or below MDLs and therefore driven by low and less certain measurements.

3.5. Passive sampling rates compared to past measured and modeled studies

There are few published PSRs for the cylindrical XAD-based passive air sampler. Therefore, to confirm that the results from the current study (referred to as "MN Study" in Table 3) were within the range of past studies, a comparison of the PSRs from the South Minneapolis study location and other published results is

Table 3
Comparison of current and past study PSR for XAD cylindrical samplers.

	Measured Passive Sampling Rates (m ³ /day)		
	MN Study (mean and standard deviation of 8 total seasons at two study locations) - Minneapolis Study Location	Calibration Study, Armitage et al., 2013	Wania et al., 2003 and Barthel et al., 2012 applied from Hayward et al. (2010) calibration study
Deployment Time (days)	90	0–122	365
Fluoranthene	1.0 ± 0.17	0.69 ± 0.14	0.76 ± 0.56
Pyrene	0.55 ± 0.2	0.86 ± 0.25	
Chrysene	0.28 ± 0.14	0.93 ± 0.22	
Benzo [b] fluoranthene	0.2 [0–0.3]	<0.008	
Benzo [k] fluoranthene	0.3 [0–0.6]	<0.012	
Benzo [a]pyrene	0.1 [0–0.2]	<0.006	
Indeno [1,2,3-c,d] anthracene	0.1 [0–0.9]	<0.005	
Dibenz [a,h] anthracene	detected only in one season	<0.008	
Benzo [g,h,i] perylene	0.06 [0–0.3]	<0.02	
Naphthalene	1.6 ± 0.37	1.53 ± 0.14	0.95 ± 0.64
Acenaphthene	0.95 ± 0.19	1.49 ± 0.13	
Fluorene	1.2 ± 0.35	0.93 ± 0.31	1.17 ± 0.2
Phenanthrene	1.4 ± 0.21	0.81 ± 0.2	1.11 ± 0.56

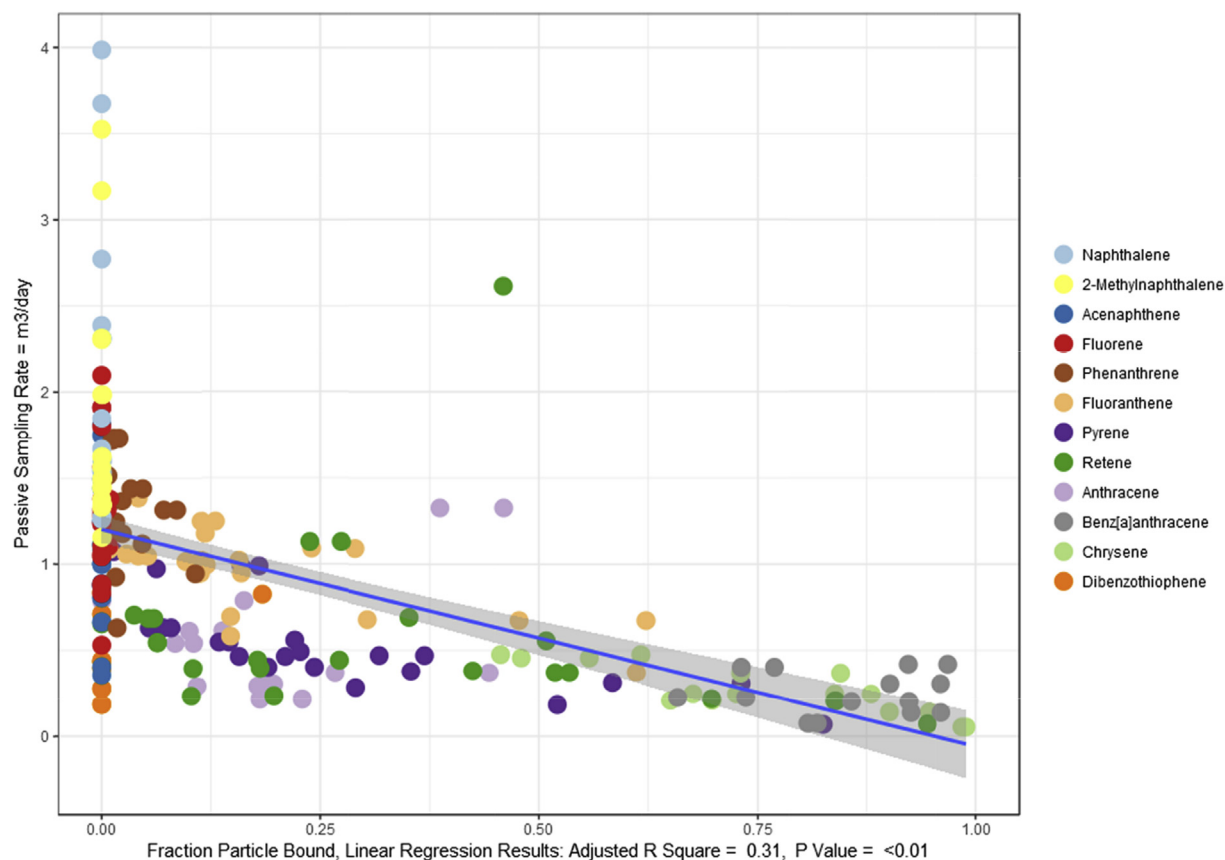


Fig. 3. The fraction of PAH captured on the filter (particulate) compared to the estimated PSR for that time period and for the PAH. $m = -1.26$, Y-Intercept = 1.2.

presented. The South Minneapolis study location was reported because there were higher detections, and therefore more certain values measured there. Overall, the results were similar, and would support PSRs being useful to estimate PAH concentrations to within a factor of two (Armitage et al., 2013). In several past studies, a PSR of 0.52 was applied to estimate concentrations of all measured PAHs (Barthel et al., 2012; Choi et al., 2009). This is appropriate for a screening level analysis of air concentrations, but not as useful for the estimation of the carcinogenic potency in the air mixture. In future studies, since the study location differences were driven mainly by low concentration based uncertainty, we would recommend the use of the study-wide mean PSRs from the South Minneapolis study location as presented in Table 3. Chrysene and fluoranthene were the only PAHs with relative potency factors that were captured by the passive samplers in levels above data completeness and censoring rules. The potency of this air mixture will be investigated in future manuscripts.

3.6. Passive sampling rates and fraction particle bound

Higher molecular weight PAHs were predominantly measured on the active sample filters, and therefore were particle bound, especially in colder temperatures. Since some of these particle-bound PAHs were detected in passive samples (Table S5), there may be a small amount of particle entrainment. However, the particle capture for this sampler type is apparently very low since there were also many high molecular weight PAHs measured below MDLs. The relationship between particle bound fraction and PSR was tested by pairing in time the mean particle bound fraction

measured on active samples with the time and location relevant PSR from the passive samples. This association was found to be significant and negative (Fig. 3), explaining 31% of the variability in the PSRs (adjusted $R^2 = 0.31$). Overall, the most variability in PSRs with respect to fraction particle bound was found for the PAHs measured predominantly to entirely in the gas phase (i.e. fraction particle bound = 0).

3.7. Active air PAH concentration comparison to collocated particulate measurements

The active sample concentrations measured at Near Roadway-962 were also compared to Minneapolis particulate measurements including TSP, PM_{10} , $PM_{2.5}$, and ultrafines (20–30 nm, 30–50 nm, 50–70 nm, and 70–100 nm, <100 nm). All of the particulate measurements were collected at the Near Roadway site, except PM_{10} which is measured at a site in downtown Minneapolis a little over 2.5 km to the northwest. None of the active PAH measurements were statistically associated with TSP, only fluoranthene was statistically associated with PM_{10} (adjusted $R^2 = 0.17$, P value < 0.01), and only naphthalene was associated with $PM_{2.5}$ (adjusted $R^2 = 0.11$, P value = 0.01). However, Pyrene, Phenanthrene, Naphthalene, and Fluoranthene were significantly associated with ultrafine particles under 100 nm (Table S6). Chrysene was statistically associated with all UFP particle sizes (30–50 nm, 50–70 nm, and 70–100 nm), but not with 20–30 nm or summed UFP under 100 nm. Since some of the predominantly particle-bound PAHs were detected in passive samples (Table S5), it is possible that there is a small but detectable particle collection by

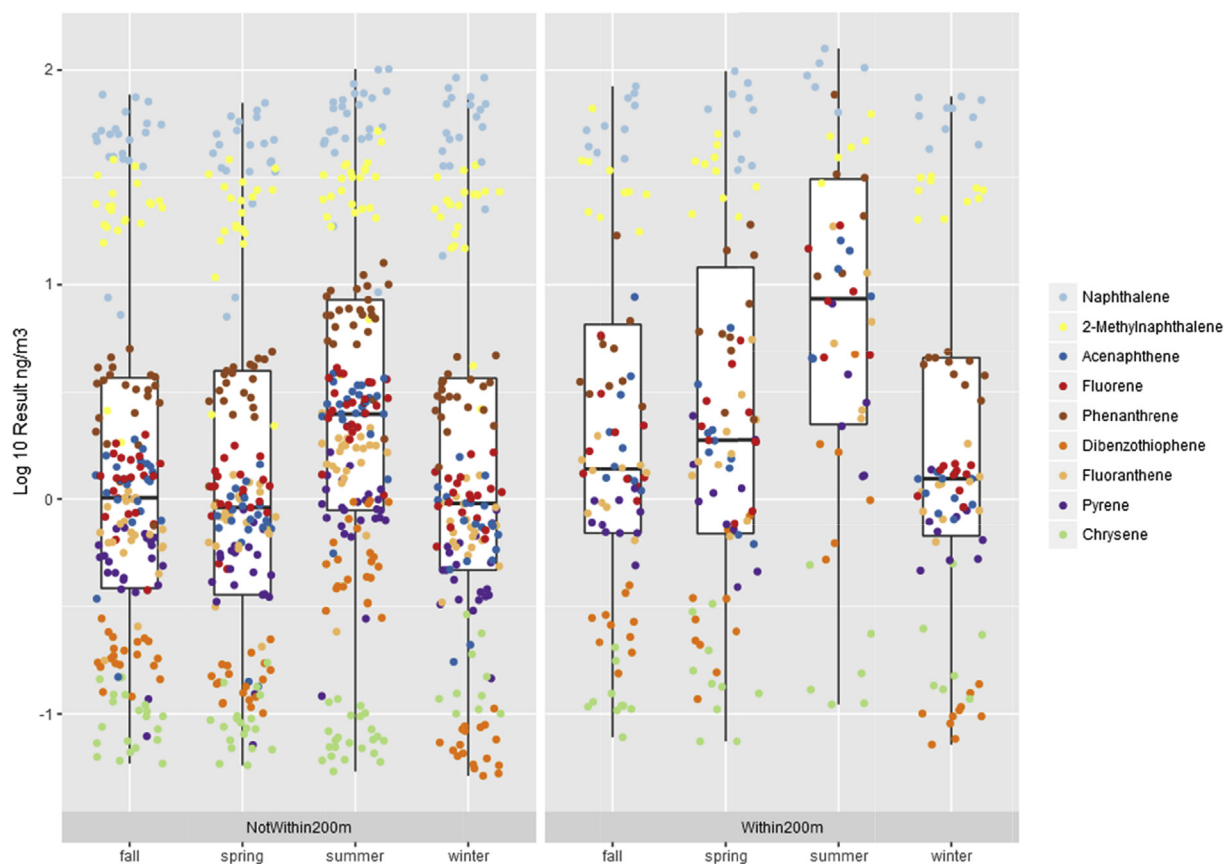


Fig. 4. Passive concentration results by major roadway status, analyte, and season.

these passive samplers, which has been found in past PUF-PAS sampler studies (Chaemfa et al., 2009; Bohlin et al., 2014; Harner et al., 2013). Many of our sampling sites were near traffic sources, and in that near-source environment PAHs may be associated with ultrafine particles that are transported similar to gases and are more likely than larger particles to diffuse into and be retained by the passive samplers.

Chrysene is a PAH with gas particle partitioning behavior that is dependent in part on temperature. Since chrysene was found exclusively on filters in the winter, the winter R_p is expected to be a true particle phase sampling rate. We estimated the gas (R_s) and particle phase (R_p) sampling rates for chrysene and compared them to the gas – particle partitioning from active sampler measurements. The total chrysene mass on the passive samplers divided by the chrysene mass on the active filters gives the proportion particulate bound PAHs that were captured by the passive samplers. The result was that 2% of the particulate bound chrysene in winter was collected on the passive samplers. A similar result (~2.5%) was found for detected levels of Benzo[a]anthracene. We infer from the significant associations between UFPs and PAHs at the Near Roadway site that these pollutants are from the same source, and that PAHs may be bound to the UFP size fraction. Identifying the particle size distribution of PAHs is complicated by several factors, including the possibility that UFPs may pass through the QFF and be collected on XAD-4 and identified as gases. Further work is needed to better understand the particle size distribution of atmospheric PAHs (Pratt et al., 2017), and to determine the particle collection efficiency of passive air samplers. Such analyses have implications for air toxics risk assessments of PAHs since the majority of

carcinogenic PAHs are particle-bound.

3.8. Seasonal and spatial passive air concentrations

In general, the passive concentrations were higher for unbuffered sites (962, 992) nearest to roadways, and lower at the rural Mille Lacs site. The 983 site was a bus stop, and was high for several PAHs including phenanthrene, fluorene, acenaphthene, dibenzothiophene, fluoranthene, chrysene, and pyrene. The 982 site, which was adjacent to a foundry and an asphalt production facility, was higher for naphthalene than other locations. This site was eliminated from the following analysis investigating sites nearest to roadways due to the potential impacts from these point sources.

The 16 passive sites were categorized as follows in order to investigate roadway signals: (1) within 200 m of a heavily trafficked roadway, (2) within 200 m but obstructed or not within 200 m (Table S2). Heavily trafficked roadways were defined as highways. The “obstructed” category was created for sampling sites within 200 m of a major roadway, but obstructed by seasonal tree canopy or a highway noise wall. We observed a stronger seasonal signal at the sites within 200 m of a major roadway, and less change from season to season at obstructed sites and those sites not within 200 m of a major roadway (Fig. 4). All PAHs (including Σ PAH) were statistically (as per Kruskal Wallis ranked sum test) different by major roadway status except: retene and dibenzothiophene. Retene is a marker for woodsmoke and had a higher general signal in the fall. Dibenzothiophene is a diesel emission marker and was very high at a bus stop, was more prevalent in the fall and summer, and

largely below MDLs in the winter.

4. Conclusions

Measurements from collocated active and passive samplers at two urban and one rural location were used to calculate PAH PSRs and estimate air concentrations at a dense urban network of passive samplers. The PSRs were more variable when PAH loadings were lower and near MDLs; this occurred more often at the rural site. Extraordinary events like uncontrolled fires have the potential to bias PSRs if calibrating from direct active air measurements. The PSRs were not strongly statistically associated with meteorological conditions in this study, but wind speed had the highest potential to impact PSR results. PSRs were positively associated with chemical-physical parameters that reflect higher volatility and gas phase diffusion. PSRs were negatively associated with estimates of fraction particle bound from active air sampling results. Passive sampling allowed for measurements with more intensive spatial resolution than regulatory grade monitors, and the results indicated a stronger seasonal signal within 200 m of a major roadway. Future work from the study includes estimation of potency from the passive and active sample.

Main findings

Higher molecular weight PAHs (particulate phase) had low but detectable passive sampling rates. Based on this information, the percent particulate phase PAH capture was estimated for the passive samplers. PAHs had a stronger seasonal signal within 200 m of highways.

Acknowledgements

The study was completed through funding from a USEPA Community Air Toxics Grant entitled *Calibrating concern about PAHs (Polycyclic Aromatic Hydrocarbons) in urban air using monitoring and modeling* (RFP No.: EPA-OAR-OAQPS-11-05). The authors wish to acknowledge the Simonich Laboratory and the Superfund Research Program at Oregon State University for donation of the passive samplers and analytical and technical support throughout the project. Paul Swedenborg made this project possible by his supervisory support at the MDH Public Health Laboratory throughout the proposal, analytical development and air sampling. We would also like to acknowledge Dr. James Armitage and Dr. Ed Swain for their assistance with the direction and writing of the manuscript.

Appendix A. Supplementary data

Supplementary data related to this article can be found at <http://dx.doi.org/10.1016/j.envpol.2017.08.049>.

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