

RESEARCH COMMUNICATION

Determination of airborne PAHs using passive sampling with 2,6 diphenyl-p-phenylenoxide as adsorbant

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Abstract: This study focused on initial tests of a small passive air sampler, utilising glass fiber filters and 2,6 diphenyl-p-phenylenoxide as the substrate. The sampler was technically designed for both gaseous and particle-bound polycyclic aromatic hydrocarbons (PAHs). It was further validated in a field sampling campaign in a suburban area and the passive sampling rates were calculated by cross comparison with simultaneous active sampler data. Higher sampling rates were observed for low-molecular-weight PAHs, upon prolonged sampling periods at outdoor ambient air conditions. Although only moderate sampling rates were observed when coupled with a highly sensitive analysis technique based on thermal desorption gas chromatography and mass spectrometry, the passive sampler was shown to be competitive with the other passive samplers in literature. These results are promising and support the future use of this sampler for more quantitative analysis of gaseous and particle-bound PAHs.

Keywords: 2,6 diphenyl-p-phenylenoxide, glass fibre filter, polycyclic aromatic hydrocarbon.

INTRODUCTION

Air pollution caused by polycyclic aromatic hydrocarbons (PAHs) is an issue receiving continuous attention in many urban areas (Jongeneelen, 2001) and significant levels of PAHs have been reported in various occupational environments (Branisteanu & Aiking, 1998; Jongeneelen, 2001). Special attention has been allocated to analysing PAHs and associated compounds from incomplete combustion processes, which are believed to play a role in the formation of reactive oxygen species (ROS) and are also considered as mutagenic and carcinogenic compounds (Bowen, 2003; Molto *et al.*, 2009). Some

selected physico-chemical characteristics of PAHs are denoted in Table I of the Appendix. Considering the negative impact on human health, initiatives for further investigation and better screening methods to monitor air pollution are required. Most of the current PAH sampling methods are labour-intensive and involve pump-driven techniques. Active sampling is accurate and highly quantitative, but complex in design, expensive and requires maintenance. In contrast, passive samplers are cheap, unobtrusive and have a high spatial resolution (Bohlin, 2010). Consequently, passive sampling has emerged as an innovative technique to overcome today's sampling and analytical shortcomings.

The first definition of quantitative passive sampling for gases was introduced by Palmes and Gunnison (1973) in a form of a mathematical model. Later on, many different types of passive air samplers (PASs) were introduced for sampling of different compounds and the theory has been well established for gaseous compounds (Tompkins & Goldsmith, 1977; Huckins *et al.*, 1990).

Most of the existing PASs are designed for gas sampling of semi-volatile organic compounds, and based on high capacity sampling against a linear sampling rate for long durations such as weeks or months. Polyurethane foam (PUF) disks, semi-permeable membrane devices (SPMDs), XAD-resin based samplers and membrane samplers are such examples (Petty *et al.*, 1993; Wania *et al.*, 2003; Harner *et al.*, 2013). In addition, a few low capacity samplers such as polymer coated glass (POG) (Harner *et al.*, 2003) and solid phase micro extraction (SPME) samplers have also been reported (Bohlin, 2010).

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PAHs are known to partition between the gas and particulate phase, with heavier PAHs preferring the particle phase. Eighty four percent of the total PAHs are present in sizes smaller than $0.49\ \mu\text{m}$ (Oliveira *et al.*, 2011). Many existing PASs are designed to sample volatile species by gas diffusion. Hence, the idea of simultaneous sampling of the gas and particle phase has been empirically realised but not yet fully scientifically investigated. Nash and Leith (2010) introduced a small axial PAS for particulate aerosols including a collection substrate underneath a metal mesh cap. They accordingly derived and experimentally verified a deposition model for gaseous and particle-bound PAHs based on both gravity and diffusion.

Since deposition of PAHs is involved in passive sampling, the deposition velocities are equally important. Chamberlain and Chadwick (1953) defined the dry deposition velocity for both particles and gases as the ratio between deposition flux and airborne concentration of the pollutant. For PAHs ranging between naphthalene and perylene, the mean particle deposition velocity for a $2.5\ \mu\text{m}$ particle was estimated to be around $0.02\ \text{cm/s}$ (Horstmann & Mclachlan, 1998).

Thermal desorption (TD) of the semi-volatile organic content from particulate matter and detection by mass spectrometry (MS) was recently introduced as a successful quantitative analytical method (van Drooge *et al.*, 2005; Bates *et al.*, 2008; Molto *et al.*, 2009). It is a faster and more sensitive method compared to solvent based extraction techniques. New collection substrate materials enabling gas adsorption in conjunction with the TD technique is thus of utmost interest, since it can be incorporated into a PAS.

2,6-diphenyl-p-phenylene oxide (hereon denoted as 2,6 DPP) is a highly porous polymer resin. It is produced by oxidative polymerisation of 2,6-diphenylphenol. 2,6 DPP has been designed for trapping volatile and semi-volatile compounds from air, and can be utilised both as a column packing material and a trapping agent

for organic compounds. Thermal desorption of volatile content is possible due to its high thermal stability and extremely low affinity to water. The surface area of 2,6 DPP is $19 - 35\ \text{m}^2/\text{g}$, pore volume is $2.4\ \text{cm}^3/\text{g}$ and the average pore size is around $200\ \text{nm}$ (Hoshika & Muto, 1978; Clark *et al.*, 1982; Alfeeli *et al.*, 2011).

In this study, we have performed initial tests involving a $12.7\ \text{mm}$ 2,6 DPP based axial sampler combined with the sensitive analysis of TD-GC-MS in an urban roadside area in Umeå, Sweden.

METHODOLOGY

Preparation of samplers

For preparation of the polymer-coated filter substrate, $12.7\ \text{mm}$ diameter glass fibre filters (GFF) (W & R Balston Ltd, UK) were used. 2,6 DPP, commercially known as Tenax[®] TA was purchased from Novakemi Ltd, Holland. After cleaning with n-hexane in acetone (2:1 v/v) for 6 hrs, the GFFs were dried and slightly wetted with dichloromethane. Immediately after that, granular 2,6 DPP ($0.05\ \text{g}$, mesh size $2.0 - 3.5\ \text{mm}$) was poured on top. The sorbent was only partly dissolved resulting in semi-spherical granules on the surface. A metallic mesh ($250\ \mu\text{m}$ mesh size) was mounted approximately $1\ \text{mm}$ above the substrate using a teflon flange. This setup was used so that the sampler could collect gaseous PAHs onto the 2,6 DPP substrate by diffusion, and the heavier particle-bound ones by gravitational settling. Detection of volatile organic compounds (VOCs) in the ppb or ppt level is achievable using thermal desorption techniques such as purging (Alfeeli *et al.*, 2011; Scientific Instrument Services, 2014)

Analytical method

The samplers were folded and placed in TD-tubes and analysed by TD-GC-MS, employing selected ion monitoring for better sensitivity (Wingfors *et al.*, 2013).

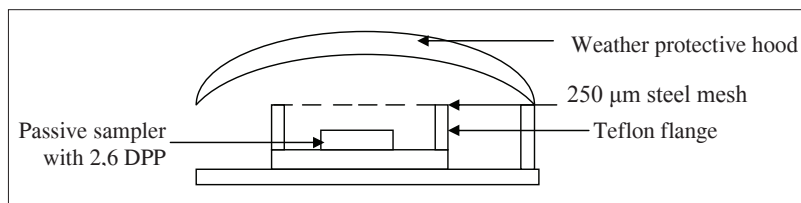


Figure 1: Schematic diagram of the sampling setup

Previously determined retention times from external calibration curves using standard reference material (SRM-1491) were used to identify different PAHs from the sampler, and the peak areas were converted to mass and quantified. All the standard PAH samples were prepared using the above mentioned method and analysed by the same TD-GC-MS based method. For positive identification, a signal to noise ratio of 3:1 and a relative retention time ± 2 seconds, as compared to the standard solution, were used.

Field evaluation of the samplers

The field trial was conducted in Umeå City, Sweden at a location with high automobile traffic, which had, by local dispersion models, been identified as a region of high air pollution (Umeå Kommun, 2007). Five continuous 24 h active samples were obtained during the week by pumping air (200 mL/min) through a 2,6 DPP impregnated GFF enclosed in a rigid plastic cassette. 2,6 DPP containing passive samplers were allowed to continuously sample for seven days at the site. All samplers were placed inside a weather protective hood in order to secure the samplers from rain and strong winds. Due to limitations of pump runtime, the active sampling durations were not identical to those of passive sampling, and average concentrations were used to obtain an integrated value for the week. The sampling height was approximately 10 ft from the ground level.

Sampling rate (R_s)

R_s is the volume of air, which contacts the sampler over a unit period of time. For an active sampler this is analogous to the volume of air pumped through the active sampler in a unit of time. This parameter is important in quantification of PAHs against active sampler data, which is the current standard. Palms and Gunnison (1973) derived an equation for the R_s in PUF passive samplers. Considering the analogous nature of the sampler used in this study to a PUF sampler, the equation was adapted to estimate the R_s for PAHs. R_s (m^3/day) was established for every compound by a comparison between passive and active sampling according to equation 1 (Palms & Gunnison, 1973).

$$R_s = \frac{M_s}{C_{air}t} \quad \dots(1)$$

M_s is the average mass of analyte in the samplers (ng), C_{air} is the air concentration (ng/m^3) of the PAH determined from active sampling and t is the sampling time (days).

RESULTS AND DISCUSSION

Surface areas of the PUF sampler developed by Shoeib and Harner (2002) and the SPMD sampler designed by Liua *et al.* (2006), were 242.5 cm^2 and 370 cm^2 , respectively. The latter had incorporated a limit of detection (LOD) of 1 ng. A POG sampler with a surface area of 300 cm^2 was developed by Harner *et al.* (2013), where the LOD was 0.1 ng. In comparison, the PAS of this study has a smaller surface area of 1.27 cm^2 and an associated LOD of 0.1 – 0.05 ng. In our case, majority of PAHs met the GC-MS based quantification criteria. The mean concentrations of PAHs (ng/m^3) from active samplers are found in Table 1. The most volatile species (2-ring PAHs) revealed a range of 6.1 – 32 ng/m^3 at the site. Detection criteria were also met for 2 and 3-ring PAHs for all active samplers, whereas 4 and 5-ring PAHs were below the LOD. Although demonstrating low levels, the profile of PAHs followed a realistic pattern that would be expected in an urban location in Europe. Average concentrations of benzo[a]pyrene (BaP) in air for different European countries were estimated by Van Jaarsveld *et al.* (1997), where higher concentrations of BaP were found in the former Czechoslovakia (2.9 ng/m^3), Hungary (1.9 ng/m^3), Germany (1.5 ng/m^3), Poland (1.4 ng/m^3) and Austria (1.3 ng/m^3). Lower concentrations of BaP were reported from Finland (0.034 ng/m^3) and Sweden (0.040 ng/m^3) (Van Jaarsveld *et al.*, 1997). This comparison demonstrates that the BaP concentration of 0.068 ng/m^3 found in this study location was low, but was in the same order of the magnitude to Scandinavian countries.

Passive R_s ranged between 0.09 – 0.15 m^3/day for more volatile PAHs [relative standard deviation (RSD) was ~20 % from naphthalene to 1-methylphenanthrene], indicating successful uptake by diffusion. Also for heavier PAHs useful R_s were obtained, although demonstrating a larger range of 0.02 – 0.29 m^3/day and scatter (mean RSD of 64 % from fluoranthene to perylene). Categorisation of PAH compounds in the sampler as gaseous and particle-bound would be impossible in practice. Therefore, it was assumed that PAHs behave according to their physico-chemical characteristics. PAHs can be released in gas phase as well as supported onto particulate matter (PM) due to their high volatility (Mastral & Calleán, 2000). Most volatile PAH compounds with two or three aromatic rings have been found to be released mainly in gaseous form and the ones with three or more aromatic rings, associated with the PM (Lee *et al.*, 1993). For instance, naphthalene is reported to be present entirely in the gas

Table 1: Concentrations of PAHs obtained from the ambient air sampling trial. LODs and R_s for other passive samplers are given for comparison.

Name	Passive mean (ng/sample)	Active mean (ng/m ³)	This study		Other studies	
			R_s (m ³ /day)	LOD (ng/PAS)	R_s (m ³ /day)	LOD (ng/PAS)
Naphthalene	26 ± 2.9	32 ± 17	0.11	0.02	3.5 ± 0.2 ^{##}	0.2 [#]
2-methylnaphthalene	12 ± 1.5	15 ± 12	0.12	0.02	3.5 ± 0.2 ^{##}	0.2 [#]
1-methylnaphthalene	8.6 ± 1.5	10 ± 8.0	0.12	0.02	3.5 ± 0.2 ^{##}	0.2 [#]
Biphenyl	4.7 ± 0.63	7.5 ± 4.9	0.089	0.03	3.5 ± 0.2 ^{##}	0.2 [#]
2,6-dimethylnaphthalene	6.3 ± 0.97	6.1 ± 5.4	0.15	0.04	3.5 ± 0.2 ^{##}	0.2 [#]
Acenaphthylene	0.39 ± 0.19	3.4 ± 2.8	0.016	0.05	2.4 ± 0.3 [*]	0.1 – 2.0 [*]
Acenaphthene	1.4 ± 0.25	2.8 ± 2.6	0.069	0.05	2.4 ± 0.3 [*]	0.1 – 2.0 [*]
2,3,5-trimethylnaphthalene	1.5 ± 0.20	1.9 ± 1.1	0.11	0.05	2.4 ± 0.3 [*]	0.1 – 2.0 [*]
Fluorene	2.0 ± 0.13	4.3 ± 2.8	0.068	0.05	2.4 ± 0.3 [*]	0.1 – 2.0 [*]
Phenanthrene	1.4 ± 0.28	6.0 ± 3.0	0.032	0.01	2.4 ± 0.3 [*]	0.1 – 2.0 [*]
Anthracene	1.5 ± 0.68	4.8 ± 4.5	0.046	0.04	2.4 ± 0.3 [*]	0.1 – 2.0 [*]
1-methylphenanthrene	0.067 ± 0.01	0.57 ± 0.38	0.017	0.03	2.4 ± 0.3 [*]	0.1 – 2.0 [*]
Fluoranthene	0.12 ± 0.02	1.5 ± 1.6	0.012	0.03	2.4 ± 0.3 [*]	0.1 – 2.0 [*]
Pyrene	0.058 ± 0.02	0.4 ± 0.15	0.0056	0.03	1.2 ± 0.2 [*]	0.1 – 2.0 [*]
Benz[a]anthracene	0.004 ± 0.001	0.25 ± 0.50	0.002	0.05	1.2 ± 0.2 [*]	0.1 – 2.0 [*]
Chrysene	0.057 ± 0.05	0.14 ± 0.80	0.059	0.05	1.2 ± 0.2 [*]	0.1 – 2.0 [*]
Benzo[b]fluoranthene	0.12 ± 0.07	0.058 ± 0.05	0.29	0.05	1.2 ± 0.2 [*]	0.1 – 2.0 [*]
Benzo[k]fluoranthene	0.029 ± 0.02	0.058 ± 0.04	0.072	0.05	1.2 ± 0.2 [*]	0.1 – 2.0 [*]
Benzo[e]pyrene	0.062 ± 0.05	0.065 ± 0.07	0.14	0.05	1.2 ± 0.2 [*]	0.1 – 2.0 [*]
Benzo[a]pyrene	0.035 ± 0.01	0.068 ± 0.07	0.074	0.1	1.2 ± 0.2 [*]	0.1 – 2.0 [*]
Perylene	0.037 ± 0.02	0.047 ± 0.02	0.11	0.1	1.2 ± 0.2 [*]	0.1 – 2.0 [*]

Comparative data as per Shoeib and Harner, 2002⁺, Bohlin, 2010^{*}, and Gouin *et al.*, 2010[#]

phase, while BaP and other compounds with 5 or 6 rings are known to be adsorbed to PM (Mastral & Calleán, 2000).

Previously published R_s are in the order of one magnitude higher and they are also shown in Table 1 for comparison. However, those samplers are based on analytical methods demonstrating a higher LOD by one order of magnitude, thus resulting in a similar overall performance (Shoeib & Harner, 2002; Bohlin, 2010; Gouin *et al.*, 2010).

CONCLUSION

The 12.7 mm GFF sampler with solid 2,6 DPP showed promising sampling rates to measure the full suite of ambient air PAHs in a short-term urban air sampling campaign. The lower sampling rates exhibited by the sampler in comparison to larger, higher-capacity PUF passive samplers is compensated by the superior mass transfer by the thermal desorption method used in the analytical measurement. The use of GFF-2,6 DPP based

collection substrate indicated simultaneous sampling of both particulate and gaseous phases, which is considered a great advantage due to the particle association of heavier PAHs.

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Appendix

Table I: Physico-chemical properties of common PAHs (Bohlin, 2010)

Name of the PAH	Number of aromatic rings	Log K _{OA}	Distribution in gas phase in ambient air (% at 25° C)	Classification
Naphthalene	2	6.8 – 7.9	> 95	Volatile
Acenaphthylene	3	6.8 – 7.9	> 95	Volatile
Acenaphthene				
Fluorene				
Phenanthrene				
Anthracene				
Fluoranthene	4	8.8 – 8.9	50 – 90	Semi-volatile
Pyrene				
Benzo[a]anthracene				
Chrysene				
Benzo[b]fluoranthene	5	10	< 30	Less volatile/ particle bound
Benzo[k]fluoranthene				
Benzo[a]pyrene				
Indeno[1,2,3 – c,d]pyrene	6	12	<10	Less volatile/ particle bound
Dibenz[a,h]anthracene				
Benzo[g,h,i]perylene				