



Evaluation of two passive samplers for the analysis of organophosphate esters in the ambient air



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ABSTRACT

Both polyurethane foam (PUF) and sorbent-impregnated PUF (SIP) passive air sampling (PAS) methods were deployed and compared separately for the analysis of organophosphate esters (OPEs) in outdoor atmospheric environment. During an continuous period of 84 days, parallel samples were also collected by a high-volume active air sampler (HV-AAS) to assess the contamination levels and to calibrate uptake parameters of PAS. The total concentration of OPEs in both particulate and gaseous phases ranged from 1.50 to 5.64 ng m⁻³ in ambient air. Tris(2-chloroisopropyl) phosphate (TCPP) was the dominating analog, representing 78 ± 9% of total OPE concentration. SIP-PAS showed longer linear-phase sampling period for TCPP, and accumulated more amount of the most volatile triethyl phosphate (TEP) and tributyl phosphate (TBP) homologues, while similar sorption performances of both PAS methods were found for most of the semi-volatile OPEs. Linear sampling rates in PUF-PAS and SIP-PAS disks were calculated for individual OPEs except for TEP and TBP, and the average uptake rates (3.3 ± 1.1 and 3.5 ± 1.7 m³ d⁻¹, respectively) were close to the acknowledged value (4 m⁻³ d⁻¹) for persistent organic pollutants. Besides, isotopic labeled D₁₅-Triphenyl phosphate (TPhP) could be used as a viable deuration compound to calculate site-specific sampling rates of OPEs, with a linear loss of up to ~60% at the end of deployment time.

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1. Introduction

Organophosphate esters (OPEs) are anthropogenic industrial derivatives of phosphoric acid, which are substituted by a combination of alkyl chains, partly chlorinated alkyl chains and aromatic functional groups [1]. Chlorinated OPEs are mainly used as flame retardants (FRs), while the non-halogenated ones have additional uses, such as plasticizers and antifoaming agents, hydraulic fluids, and electronics [2,3]. Environmental measurements of OPEs could date back to the 1970s [4]. Nevertheless, increasing awareness has been focused on the chemicals due to their usage as alternative FRs for polybrominated diphenyl ethers (PBDEs) [5]. The global demands of OPEs are expected to increase and the resulting more production and emissions into the environment may cause re-emerging concern [6,7]. An overview discussed the environmental levels of FRs in several monitoring studies [8], indicating that total OPE concentrations generally exceeded those of PBDEs in indoor air environment, and the human exposure potentials caused by

OPE contamination appeared to be higher.

OPEs are generally recognized as semi-volatile additives [9], which could be released into the environment from commercial products during volatilization, abrasion and dissolution [2]. Adverse health impacts, such as carcinogenic, neurotoxic, reproductive and hemolytic effects [8,10–14], have also been observed in biological tests. Hydrophobic OPE analogues, especially the chlorinated ones, are relatively stable toward biodegradation and might have persistent and long range transport abilities [15]. OPEs have been found globally in various environmental matrices [1,5,15,16], especially the indoor and outdoor environment. Simultaneous air sampling is beneficial to assessing the sources and fates of OPEs through spatial mapping studies. For instance, in order to investigate the occupational exposure to eight organophosphorus compounds, low volume active air samplers were utilized at electronics dismantling facilities and social premises, with total OPE concentrations quantified in the range of 90–3800 ng m⁻³ [16]. High-volume active air samplers (HV-AAS) were also applied to investigate organophosphorus compounds in airborne particles from Indian, Arctic, Pacific and Southern Ocean. The sum of OPEs ranged from 120 pg m⁻³ to 2900 pg m⁻³, and

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the air concentrations decreased sharply from continents toward the open oceans [1].

Active air sampling techniques, such as HV-AAS, could offer information on gas and particle phase distribution in high temporal resolution, however, it is not suitable for massive field monitoring due to the dependence on power supplies as well as instrumental expenses [17]. Nevertheless, passive air samplers (PAS) could provide integrated data on a long sampling period with simplicity and low cost [18], making it feasible to simultaneous sampling at a large geographical scale. Since 2005, a Global Atmospheric Passive Sampling study was performed for detecting persistent organic pollutants (POPs) both spatially and temporally [19,20]. Polyurethane foam (PUF) disk is particularly used as the passive sampling device because of its excellent performance on various kinds of semi-volatile contaminants over periods of several weeks [21]. Shoeib et al. further developed a novel sorbent-impregnated polyurethane foam (SIP) technique, in which XAD adsorbent powder is impregnated onto the macroreticular surface of traditional PUF disk [22]. Compared with PUF, SIP led to higher sorption capacity and longer linear-phase distribution period for more volatile chemicals [17]. Both PUF-PAS and SIP-PAS disks were deployed for measuring polychlorinated biphenyls and neutral polyfluoroalkyl compounds [23], which indicated that SIP-PAS disks had promising sorption capacities for the measurement of both legacy and emerging POPs.

For air sampling of OPEs in indoor air and remote marine atmosphere, active air samplers were generally applied in most previously reported investigations [1,5,24,25]. While, few techniques were tested for passive sampling of OPEs. A passive flux sampler containing an Empore C18FF adsorbent disk and a circular glass plate was developed for the measurement of OPE emissions from building materials in indoor environment [26]. Nevertheless, specific usages of the technique might limit its application to ambient air samples. In this work, both active HV-AAS and passive (PUF-PAS and SIP-PAS) techniques were used for ambient air sampling. The specific objectives include (i) to monitor the contamination levels and compositions of OPEs in outdoor environment, (ii) to establish a robust passive sampling method for OPEs, (iii) to assess the sorption comparability of two PAS disks.

2. Experimental

2.1. Materials

Tri(2-chloroethyl) phosphate (TCEP), Tri-n-propylphosphate (TnPP), Tris(2-chloroisopropyl) phosphate (TCPP), Triethyl phosphate (TEP), Tris(2-chloro-1-(chloromethyl)ethyl) phosphate (TDCP), Tributyl phosphate (TBP), Triphenyl phosphate (TPhP), Cresyl Diphenyl Phosphate (CDP), Tri-3-cresyl phosphate (TCrP) were purchased from Dr. Ehrenstorfer GmbH (Wesel, Germany). D₁₅-TEP (98%), D₂₇-TBP (98%) and D₁₅-TPhP (98%) were from Cambridge Isotope Laboratories (Tewksbury, MA). High-performance liquid chromatography (HPLC) grade hexane, dichloromethane, acetone, methanol and acetonitrile were purchased from JT Baker (Phillipsburg, NJ). Ultrapure water was prepared using a Milli-Q system (Millipore, Billerica, MA).

2.2. Sampling

Before sampling, PUF disks (14 cm diameter × 1.35 cm thick, surface area 365 cm², mass 4.40 g, volume 207 cm³, Tisch Environmental, Cleves, OH) were pre-washed by hexane/dichloromethane (1/1, v/v) using accelerated solvent extraction system (ASE 350, Dionex Corporation). SIP disks were prepared by impregnating PUF with finely grounded XAD-4 resin (~0.75 μm,

Supelco, Bellefonte, PA), following the method reported by Shoeib et al. [22]. After that, each of 20.0 ng D₂₇-TBP and D₁₅-TPhP was spiked onto both PAS disks as depuration compounds (DCs). Then, PUF and SIP disks were individually placed inside laboratory-made stainless steel chambers to preserve a low-wind sampling environment [27,28]. The constructions of stainless steel chambers were mentioned elsewhere [22,27]. Both eleven PUF and SIP disks were deployed approximately two meters above the roof on top of the laboratory building (40°00'26.94"N, 116°20'15.10"E) in our research center, with a total height of approximately 14 m. The disks were harvested one at a time on every Friday in the sampling period from July 11 to September 26, 2014.

Parallel samples were also collected by using one HV-AAS sampler, which was located 10 m away from the PAS devices. It operated on every Tuesday and Friday from July 11 to September 30. The sampling rate was 500 L/min, and a total air volume of 600 m³ was collected. In order to trap OPEs from airborne particles, a glass fiber filter (GFF, 10.16 cm diameter, Whatman, Piscataway, NJ) was placed in front of a glass cartridge packed with two PUF (76 mm diameter × 60 mm thick) stacks for gas-phase analytes. All samples were collected, sealed with aluminum foil and stored at -20 °C until analysis.

2.3. Sample pretreatment and instrumental analysis

The GFFs, PUF stacks, PUF-PAS and SIP-PAS disks were pre-treated separately. Prior to extraction, each active sample (GFFs and PUF stacks) was spiked with 20.0 ng D₁₅-TPhP as surrogate standard. While, no additional isotopic-labeled standards were added for passive samples, and D₂₇-TBP and D₁₅-TPhP were tested as depuration compounds. All samples had the same extraction and purification procedures. In brief, hexane/dichloromethane (1/1, v/v) was used as the extraction solvent. Accelerated solvent extraction was performed at 100 °C and 1500 psi in three static cycles of 10 min. The extracts were concentrated to ~2 mL by rotary evaporation and transferred to a gel permeation chromatography column (GPC, 400 × 30 mm, i.d.) packed with Biobeads S-X3 (200–400 mesh, Bio-Rad Laboratories, Hercules, CA). A hexane/dichloromethane mixture (1/1, v/v) was used as eluent and the second fraction of 120 mL eluent was collected after discarding the first 110 mL fraction. The solvents were then concentrated to ~2 mL, and passed through an 8 g neutral alumina column (6–325 mesh, Sigma-Aldrich, St. Louis, MO), which was activated at 600 °C for 6 h and 5% water-deactivated before use. The column was preconditioned using 30 mL of hexane, and all target analytes were eluted with 80 mL of 1:3 hexane/dichloromethane mixture (v/v). Finally, the eluate was concentrated to 1 mL, transferred into LC vials, and solvent exchanged with 980 μL of 6:4 acetonitrile/water mixture (v/v). Twenty microliters of D₁₅-TEP (10.0 ng) was further added into each vial as injection standard.

Analyte quantification was performed on an API 5500 triple-quadrupole mass spectrometer (AB SCIEX Inc. Framingham, MA) interfaced with an Ultimate 3000 ultrahigh performance liquid chromatograph (Thermo Fisher Scientific Inc., Waltham, MA). An Acquity UPLC BEH C8 analyte column (2.1 mm i.d. × 100 mm length, 1.7 μm, Waters) was selected for analyte separation. Column temperature was set as 45 °C. Acetonitrile and water were used as mobile phases, and the flow rate was 0.2 mL/min. Electrospray ionization (ESI) source was employed in positive ion mode. Curtain gas and collision gas were set as 35 and 7 psi, respectively. Ionspray voltage was 5500 v, and ionspray temperature was 450 °C. The flow gradient program as well as confirmation and quantification ions for each analyte in multiple reaction monitoring mode were shown in Tables S1 and S2.

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