ANALYTICAL SCIENCES OCTOBER 2017, VOL. 33

A Newly Designed Solid-Phase Extraction-type Collection Device for Precise Determination of Polycyclic Aromatic Hydrocarbons in Air

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A newly designed styrene-divinylbenzene copolymer adsorbent packed solid-phase extraction (SPE)-type collection device for the quantitative determination of airborne polycyclic aromatic hydrocarbons (PAHs) containing two to five rings is reported in this manuscript. This SPE-type collection device offers rapid, easy and quantitative elution of the analytes and easier reuse. A small collection device was initially developed for investigating the basic collection and elution performances of the adsorbent with respect to PAHs. The analytes were quantitatively collected on the adsorbent up to 3 m^3 of air sampling at a sampling temperature of 35°C. The collected analytes were then completely eluted from the adsorbent by passing 3 mL of dichloromethane without carry-over of the analyte. During air collection, because no moisture was trapped on the adsorbent, the subsequent gas chromatography–mass spectrometric analysis was not influenced by moisture. Based on these successful performances, a wide-bore collection device was introduced for collecting larger air samples. After a quantitative investigation of the collection and elution performances of the wide-bore collection device, it device was successfully applied for precise determinations of PAHs in atmospheric air. Further application and employment of the device for the precise determination of semi-volatile organic compounds in environmental air samples is expected due to these excellent results.

Keywords Polycyclic aromatic hydrocarbon, sample preparation, air pollutants, semi-volatile organic compounds

(Received April 13, 2017; Accepted June 27, 2017; Published October 10, 2017)

Introduction

Polycyclic aromatic hydrocarbons (PAHs) are organic compounds that include two or more fused aromatic rings and are considered to be extremely hazardous to human health.[1] Some PAHs are classified as carcinogens by the International Agency for Research on Cancer (IARC).[2] PAHs are formed during the combustion of hydrocarbons at high temperatures, and are widespread environmental contaminants.[3,4] Because PAHs have low vapor pressures, the formations of aerosols and particulate matter (PM) from emitted PAHs has also been reported.[5,6] Actually, a sensitive detection of PAHs by the thermal desorption of PM trapped on a filter was reported.[7]

A wide variety of adsorbents have been used to collect airborne PAHs, such as Tenax, XAD-2, and polyurethane foam (PUF). The US Environmental Protection Agency (EPA) has listed 16 priority PAHs, and has published a standard determination method for PAHs in ambient air using gas chromatography–mass spectrometry (GC-MS).[8] The EPA document describes a sampling procedure that uses a quartz filter and a sorbent cartridge. During air sampling, particulate and aerosol PAHs are trapped on the filter, while some of the more volatile PAHs pass through it and are collected on the adsorbent. To elute the collected PAHs from the adsorbent, Soxhlet extraction with dichloromethane for 18 h is recommended. However, in addition to the long elution time required for this method, its lower recovery of volatile PAHs is a problem.[9–11] Ultrasonic extraction is also a popular method for the elution of PAHs from adsorbents,[10–14] where 20 – 30 min of ultrasonication has typically been used to elute extracted PAHs from XAD resins,[15] although insufficient recovery of PAHs is still a problem here.[12,16] A solid-phase extraction (SPE) type collection device was developed for the collection of air borne PAHs,[17] in which triacontyl (C_{30}) bonded silica was packed into a cartridge. For the precise determination of airborne PAHs, quantitative evaluation using sample preparation steps based on the complete collection and elution of PAHs on a collection device could be necessary. However, the extraction and elution recoveries have not been quantitatively evaluated for commercially available collection devices.

Our research group has been developing an SPE-type collection device for airborne semi-volatile organic compounds (SVOCs). Previously, our research group developed a novel partitioning collection medium.[18,19] Specially prepared

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macroporous silica (MPSi) with a specific surface area of 1.8 $m^2\ g^{-1}$ was used as a support for the collection medium. As the partitioning phase, octadecyl (C_{18}) chains were chemically modified on the surface of the MPSi. The results indicated rapid and complete elution of the analytes (sesquiterpenes and PAHs) from the adsorbent by just passing 10 mL of acetone. However, because the retention power was significantly affected by the sampling temperature, this method was not suitable for the analysis of PAHs in atmospheric air. More recently, poly(dimethylsiloxane) (PDMS)-coated MPSi was developed as an alternative adsorbent for the collection of airborne PAHs.[20] MPSi-PDMS also trapped PAHs based on partitioning, and an excellent elution performance was indicated using 10 mL of acetone with 1 min of ultrasonication. The retention power increased with the PDMS coating, and 250 L of air could be collected without any breakthrough for three-to-six ring PAHs.

In the present study, we reported a newly designed SPE-type collection device packed with styrene-divinylbenzene (Sty-DVB) adsorbent for the precise collection and elution of two-to-six ring PAHs. Two types of collection devices were developed by packing the particulate adsorbent into glass cartridges with different inner diameters. The fundamental performances were initially investigated with a small collection device. Then, a wide-bore collection device was introduced for collecting larger air samples. After several quantitative evaluations of the collection and elution performances of the collection device, the developed device was successfully applied to the determination of PAHs in ambient air samples.

Experimental

Chemicals

A mixture of 16 standard PAHs (200 mg L^{-1} each) containing naphthalene (Nap), acenaphthylene (Acy), acenaphthene (Ace), fluorene (Flu), phenanthrene (Phe), anthracene (Ant), pyrene (Pyr), fluoranthene (Flt), benz[*a*]anthracene (BaA), chrysene (Chr), benzo[*b*]fluoranthene (BbF), benzo[*k*]fluoranthene (BkF), benzo[*a*]pyrene (BaP), indeno[1,2,3-*cd*]pyrene (Ind), dibenz[*a*,*h*]anthracene (DahA), and benzo[*g*,*h*,*i*]perylene (BghiP) dissolved in methanol/dichloromethane (1:1 v/v) was purchased from Kanto Chemical Co., Inc. (Tokyo, Japan). Acetone (>99.9%) and dichloromethane (>99.9%) were also obtained from Kanto Chemical Co., Inc. A standard stock solution (100 mg L^{-1} each) was prepared by dilution of the mixed solution with acetone. Both 1-methylnaphthalene and 2-methylnaphthalene were obtained from Tokyo Chemical Industry Co., Ltd. (Tokyo, Japan). Sty-DVB polymer particles (Sunpak-H) with 50/80 mesh were made by Shinwa Chemical Industries, Ltd. (Kyoto, Japan). Another Sty-DVB polymer particles of Amberlite XAD-2 with 20/60 mesh (Sigma-Aldrich Japan, Tokyo, Japan) was also used as a sorbent for comparison with the Sunpak-H. The specific surface area (SSA) of XAD-2 is less than 300 $m^2\ g^{-1}$, and its average pore diameter is 9 nm.

Collection devices

The SSA of the Sunpak-H was 100 – 150 $m^2\ g^{-1}$, and its average pore diameter was 30 nm. The small collection device was prepared by packing glass fiber filters, a specially designed polytetrafluoroethylene (PTFE) plate (14.7 mm in diameter with seven holes, HORIBA STEC, Kyoto, Japan), and the Sty-DVB adsorbent (1.0 g) into a glass SPE cartridge (14.9 mm i.d. and 95 mm length, GL Sciences, Tokyo, Japan). The PTFE plate was used to avoid failure of the glass fiber filter during the drying process of the device. The packing length of the sorbent was approximately 20 mm. The packed particles were fixed with another glass fiber filter and a PTFE ring. A PTFE adapter (GL Sciences) was used to connect the cartridge to a gas-sampling pump. A glass cartridge (24.0 mm i.d. and 70 mm length, HORIBA STEC) was employed for the wide-bore collection device. For the wide-bore collection device, a PTFE O-ring, a glass fiber filter, and 3.0 g of the sorbent were packed into the cartridge, which had a packing length of 30 mm. Another glass fiber filter and a specially designed PTFE stopper with seven holes (24.0 mm in diameter, HORIBA STEC) were then packed into the cartridge to fix the sorbent. The stopper has a groove around its girth and fluorine-containing rubber was attached to the groove. To connect the device to a gas-sampling pump, a specially designed PTFE adapter (HORIBA STEC) was used. These collection devices were washed with dichloromethane and acetone before use, and analytes were not detected on the method blank. No swelling of the Sunpak-H particles occurred as a result of contact with either dichloromethane or acetone. An illustration of the prepared collection devices is shown in Fig. 1. A small collection device packed with 1.0 g of XAD-2 was also prepared in the same manner.

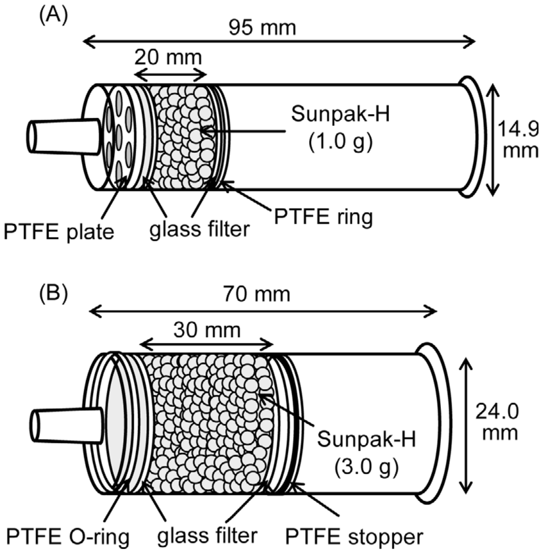

Fig. 1 Illustration of the collection devices packed with Sunpak-H.

Analytical method

The collection and elution performances of the developed devices were evaluated by using the standard stock solution (100 mg L^{-1}). First, 50 μL of the standard solution was dropped on the tip side of the collection device. The device was then connected to a filter holder (EMO-47, GL Sciences) with a 47-mm quartz fiber filter (QR-100, Advantec Toyo Kaisha, Ltd., Tokyo, Japan) using a PTFE tube to trap PM and soot, and to protect the collection device from them. Next, the device was immersed in a constant-temperature water bath (typically kept at 35°C) for 5 min; then, clean air was collected with a prototype gas-sampling unit (HORIBA STEC) or a low volume air sampler (LV-40 BW, Sibata Scientific Technology Ltd., Soka, Japan). The sampling speed was fixed at 5 and 15 L min^{-1} for the small and wide-bore devices, respectively. The analytes collected in the collection device were eluted by passing acetone or dichloromethane as the elution solvent. The volume of the organic solvents was optimized in following experiments for the

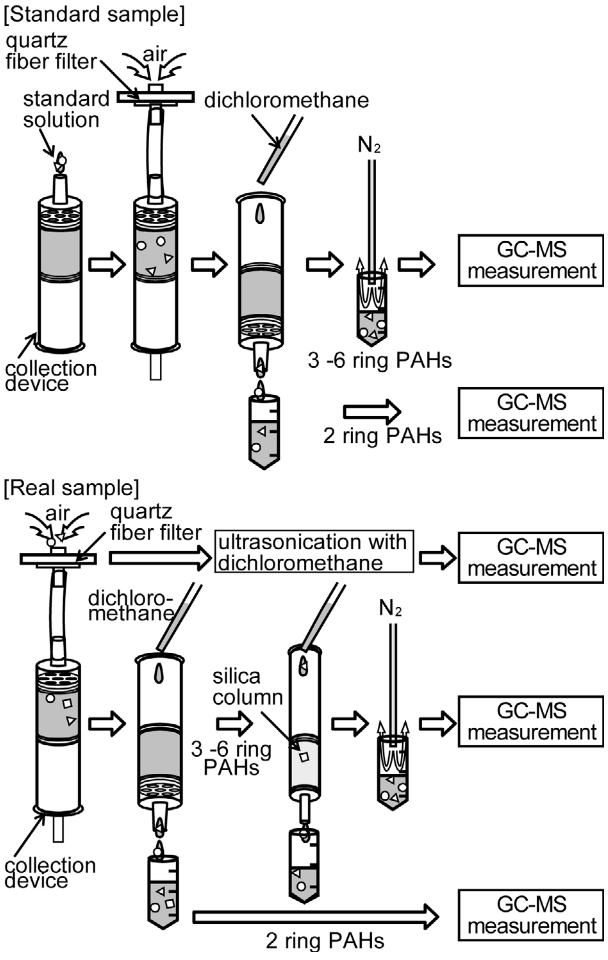

Fig. 2 Illustration of the analytical method.

Table 1 Analyte PAHs and their SIM ions

Elution order	Compound	Abbreviation	Primary ion	Secondary ion
1	Naphthalene	Nap	128	126
2	2-Methylnaphthalene	2-Nap	142	141
3	1-Methylnaphthalene	1-Nap	142	141
4	Acenaphthylene	Acy	152	151
5	Acenaphthene	Ace	154	153
6	Fluorene	Flu	166	165
7	Phenanthrene	Phe	178	179
8	Anthracene	Ant	178	179
9	Pyrene	Pyr	202	101
10	Fluoranthene	Flt	202	101
11	Benz[*a*]anthracene	BaA	228	229
12	Chrysene	Chr	228	226
13	Benzo[*b*]fluoranthene	BbF	252	253
14	Benzo[*k*]fluoranthene	BkF	252	253
15	Benzo[*a*]pyrene	BaP	252	253
16	Indeno[1,2,3-*cd*]pyrene	Ind	276	138
17	Dibenz[*a*,*h*]anthracene	DahA	278	139
18	Benzo[*g*,*h*,*i*]perylene	BghiP	276	138

Table 2 Collection recoveries for PAHs by a small device packed with Sunpak-H

Air sampling volume/L	Collection recovery, %			
	Nap	2-Nap	1-Nap	Three-to-six ring PAHs
3000	100	100	100	100
4000	99.5	100	100	100
5000	96.2	100	100	100
6000	93.2	100	100	100

Sampling temperature: 35°C, elution: dichloromethane 3 mL.

respective collection devices. For determining the two ring PAHs, the solution was analyzed by GC-MS without concentration, because two ring PAHs are evaporated by concentration with an N_2 flow. On the other hand, the solution was concentrated to 100 µL with an N_2 flow to determine three-to-six ring PAHs where no losses occurring during the concentration process. The analytical method for the present study is illustrated in Fig. 2. The collection device can be reused after drying the packed particles with an N_2 flow (5 L min^{-1}) for 20 min.

Real sample analysis

Air from a tunnel (Kofu, Japan) and a roadside (Kofu, Japan) were measured as real samples. The air samples were collected at a height of 1.0 m above the ground at ambient temperature in November 2016. During the analysis of real air samples, the eluted solvent was cleaned using a silica-gel column to remove contaminants before GC-MS analysis, which was performed as follows. First, approximately 1 mL of *n*-hexane was added to the concentrated elution solvents (100 µL). The solvent was then transferred to a silica-gel cartridge (Bond Elut SI, silica 2 g, Agilent Technologies, Santa Clara, CA, USA). The vial was rinsed twice with 2 mL of hexane, which was also added to the silica gel cartridge. PAHs were eluted with 6 mL of dichloromethane/hexane (1:9 v/v), and the solution was concentrated to 100 µL with an N_2 flow. During the real sample analysis, the analytes collected on the quartz fiber filter were also determined. After air collection, the quartz fiber filter was placed in a glass vial. Then, PAHs were extracted with 20 mL of dichloromethane with ultrasonication for 20 min. The filter was then removed to another glass vial and ultrasonicated again for 20 min in the same manner. The combined extraction solvent was filtered with filter paper No. 5C (Advantec Toyo Kaisha). The filtered solution was concentrated to 100 µL with an N_2 flow.

GC-MS measurements

A Shimadzu QP5050A GC-MS was used for all GC measurements. Helium (>99.999% purity) was used as the carrier gas at a head pressure of 100 kPa. The injector and interface temperatures were set at 300 and 320°C, respectively. Injection was performed using the split mode at a ratio of 10:1, and 2 µL of the sample solution was injected. A fused-silica HP-5 capillary column (30 m × 0.25 mm, 0.25 µm film thickness, Agilent Technologies) was used for separation of the analytes. The column temperature was initially maintained at 100°C for 4 min, after which it was programmed to increase to 320°C (maintained for 5 min) at a rate of 20°C min^{-1}. The mass spectrometer was operated in the selected-ion-monitoring (SIM) mode. The primary and secondary ions for detecting PAHs are listed in Table 1.

Table 3 Elution recovery of PAHs from a small device packed with Sunpak-H

Solvent volume/mL	Elution recovery, %															
	Nap	Acy	Ace	Flu	Phe	Ant	Pyr	Flt	BaA	Chr	BbF	BkF	BaP	Ind	DahA	BghiP
2	100	100	100	100	100	100	98.3	98.4	100	100	100	100	100	100	100	100
3	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
4	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100

Air sampling volume: 100 L, sampling temperature: 35°C.

Results and Discussion

Collection/elution performances of the small collection device

For evaluating the collection recovery of the analyte PAHs, after dropping the standard solution on a small collection device packed with Sunpak-H, another device packed with Sunpak-H was connected in tandem. Then, clean air was collected *via* the two devices, and the collection recovery was calculated from the ratio of PAHs collected in the former and latter devices, while the former collection device was immersed in a constant-temperature water bath (typically kept at 35°C). The collection recoveries for two rings PAHs at a sampling temperature of 35°C are shown in Table 2. A collection recovery of 100% means more than that 99.95% of the analytes remained in the former device after air sampling. With a sampling volume of more than 3000 L, a breakthrough was found for Nap. Methyl naphthalenes and three-to-six ring PAHs were completely collected in the device with sampling volumes of up to 10000 L. The collection recovery by the device packed with XAD-2 was investigated for comparison with Sunpak-H, where the collection recovery of Nap was 96.2 and 89.9% with a sampling volume of 2000 and 3000 L, respectively (Table S1). Therefore, a higher collection power with respect to airborne PAHs by Sunpak-H compared to that by the XAD-2 adsorbent was clearly confirmed. Although the collection power of Sunpak-H packed small collection device could not sufficient for collecting volatile PAHs, it is satisfactory for low volatile compounds, such as BaP.

The elution recovery of the analytes was calculated by sequential elution of the analytes from the collection device with each solvent. First, the optimum desorption solvent was chosen by comparing the elution recoveries with different solvents. The results showed that acetone and dichloromethane provided greater elution recoveries than toluene and hexane. Since a slightly better elution recovery was obtained with dichloromethane than acetone, dichloromethane was employed as the elution solvent for the small collection device. Table 3 gives the elution recoveries of PAHs from the collection device with different volumes of dichloromethane. An elution recovery of 100% means that more than 99.95% of the analytes were eluted during the first elution. All of the investigated PAHs were successfully eluted from the device by passing more than 2 mL of dichloromethane. The elution time for passing 3 mL of dichloromethane was less than 1 min. The elution recovery by the device packed with XAD-2 was also investigated. The device packed with XAD-2 showed an insufficient elution of PAHs, where the elution recoveries ranged from approximately 70 to 90%, even when using 10 mL of acetone or dichloromethane (Table S2). Insufficient elution of PAHs also occurred with a collection device packed with porous silica gel and poly(ethylene glycol) dimethacrylate particles in the preliminary studies. Based on these results, it was believed that the pore size and micropore volume of the sorbent, as well as the sorbent material, contributed to the excellent elution performance for PAHs. The small-size device could be more suitable for the collection of low-volatile SVOC with relatively higher concentration, such as phthalates in indoor air sample.

Table 4 Collection recoveries of PAHs by a wide-bore device packed with Sunpak-H

Air sampling volume/L	Collection recovery, %			
	Nap	2-Nap	1-Nap	Three-to-six ring PAHs
10000	100	100	100	100
15000	99.5	100	100	100
20000	99.4	100	100	100
30000	96.7	100	100	100
40000	96.2	100	100	100

Sampling temperature: 35°C, elution: dichloromethane 10 mL.

In analyze of gaseous environmental samples, moisture in the sample matrix often adversely affects the sample preparation steps and GC-MS measurement. The adsorption of moisture from the air samples was investigated by collecting humid air (relative humidity above 90%, temperature *ca.* 25°C) in the device packed with Sunpak-H. The increase in weight of the device packed with Sunpak-H packed device was 0.01 g after 500 L of humid air had been sampled, while the weight of the device packed with silica gel increased by 0.65 g after the collection of humid air. These results clearly indicated that moisture from the air was not adsorbed on the Sunpak-H, and that environmental air could be collected *via* the Sunpak-H device without desiccant.

Wide-bore collection device

Based on the above excellent collection and elution performances with respect to PAHs of the small device packed with Sunpak-H, a wide-bore collection device was developed for the sampling of larger volumes. By introducing a wide-bore glass cartridge, the gas-sampling speed was significantly increased compared to that of the small device. The pressure loss of the wide-bore collection device was 21.4 kPa at a gas-sampling speed of 15 L min^{-1}. Owing to the increase in the diameter of the collection device, the linear velocity of the sample air was decreased; thus, an increase in the analyte collection recovery was expected. In addition, increasing the packing length of the sorbent was also advantageous with respect to the collection of analytes. The collection recoveries of two ring PAHs by the wide-bore collection device are given in Table 4. Sampling volumes of up to 10000 L of Nap were completely trapped in the collection device. These results clearly indicated that the retention power with respect to a volatile analyte was significantly increased by increasing the

Table 5 Elution recovery of PAHs from a wide-bore device packed with Sunpak-H

Solvent volume/ mL	Elution recovery, %															
	Nap	Acy	Ace	Flu	Phe	Ant	Pyr	Flt	BaA	Chr	BbF	BkF	BaP	Ind	DahA	BghiP
5	100	100	100	100	100	100	99.7	99.6	99.5	100	100	100	100	100	100	100
8	100	100	100	100	100	100	99.7	99.6	100	100	100	100	100	100	100	100
9	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
10	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100

Air sampling volume: 5000 L, sampling temperature: 35°C.

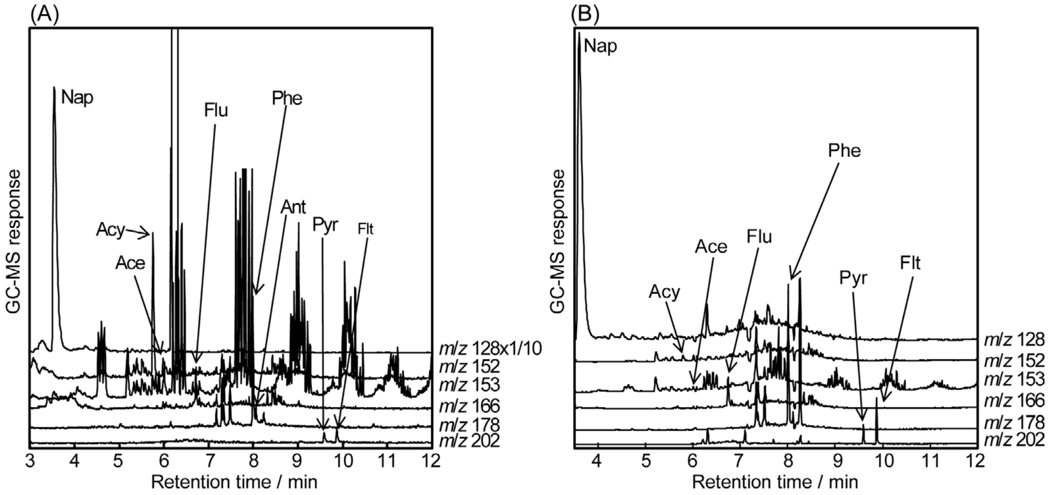

Fig. 3 Typical chromatogram for the determination of PAHs collected in the wide-bore collection device packed with Sunpak-H; (A) tunnel air and (B) roadside air.

inner diameter of the collection device. No breakthrough was observed for methyl naphthalenes and three-to-six ring PAHs with sampling volumes of up to 50000 L. Also, the wide-bore device did not adsorbed moisture from atmospheric air; the weight of the device increased by less than 0.01 g after the collection of 5000 L of moist air (relative humidity above 89%, temperature of 22.5°C).

The elution recovery of PAHs by the wide-bore collection device eluted with dichloromethane is shown in Table 5. Compared to the small collection device, a slightly larger amount of elution solvent was required for complete elution of the collected PAHs, although the analytes were completely eluted by passing 9 mL of dichloromethane, while the desorption time was within 3 min. The volume of elution solvent required for the wide-bore collection device was determined to be 10 mL.

The relative standard deviations (RSDs) of the peak areas of the standard analytes ($n = 5$, repetitive sample dropping, air collection, and elution) were less than 12%. These RSDs were the same as those obtained when the GC-MS measurements were repeated using manual injections. This excellent repeatability could be due to the complete collection and elution of the analyte PAHs by the collection device packed with Sunpak-H. The limit of detection (LOD) for two ring PAHs (in standard solution) was 0.02 mg L^{-1}. For analysis of two ring PAHs, 10000 L air was collected and the elution solvent (10 mL) was directly analyzed by GC-MS. Thus, the LOD for two ring PAHs in air was calculated to be 20 ng m^{-3}. The LODs of the three to six ring PAHs for standard solutions were in the range from 0.01 to 0.05 mg L^{-1}. For the analysis of three-to-six ring PAHs, the air sampling volume was assumed to be 50000 L and the elution solvent was concentrated to 100 μL. The LODs for three-to-six ring PAHs in air were calculated to be in the range from 0.02 to 0.1 ng m^{-3}.

Finally, the wide-bore collection device was applied to the determination of PAHs in atmospheric air. A typical chromatogram for the determination of PAHs in tunnel air collected with the wide-bore collection device packed with Sunpak-H is shown in Fig. 3A; 600 L tunnel air was collected at 10 L/min for 60 min, and the temperature and relative humidity in the tunnel were 21°C and *ca.* 65%, respectively. Figure 3B shows the PAHs in roadside air. This is from a road that goes through the above tunnel; this sample was collected at approximately 800 m from the tunnel. A volume of 21600 L roadside air (relative humidity *ca.* 50 - 65%, temperature *ca.* 25 - 35°C) was collected (15 L min^{-1} for 24 h). The quantification results for the PAHs in the tunnel and roadside air collected both on the quartz fiber filter and in the collection device packed with Sunpak-H are listed in Table 6. For the real sample analysis, PAHs collected in the collection device were also completely eluted with 10 mL of dichloromethane, and any analytes were detected in a sequential second elution. The quantitative result for Nap in roadside air is not accurate because a breakthrough occurred for Nap with sampling volumes of more than 15000 L. Although the sampling points are on the same road, significantly higher concentrations of PAHs were detected in the tunnel. As was found in previous work, PAHs with low vapor pressure are retained on quartz fiber filters as PM, and they remain on the filter during air sampling.[20,21]

Table 6 Quantitative results for PAHs in real sample air

		Concentration/ng m^{-3}									
		Nap	Acy	Ace	Flu	Phe	Ant	Pyr	Flt	BaA	Chr
Tunnel	Sunpak-H	9.9×10^3	92	91	61	94	54	38	36	N.D.	N.D.
	Filter	2.2×10^3	31	72	35	N.D.	N.D.	52	43	50	29
	Total	12.1×10^3	123	163	96	94	54	90	79	50	29
Roadside	Sunpak-H	*ca.* 300	1.5	3.6	4.3	12	N.D.	3.0	4.6	N.D.	N.D.
	Filter	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	1.1	N.D.	N.D.
	Total	*ca.* 300	1.5	3.6	4.3	12	N.D.	3.0	5.7	N.D.	N.D.

N.D.: not detected.

In the present study, BaA and Chr were only detected on the quartz fiber filter, and they did not move to the collection device. This result is in good agreement with those in a previous study.[20] Separated quantification of PAHs trapped on quartz fiber filter and adsorbent could be have meaning for understanding the component of PM and gas-particle distribution of PAHs.

As shown in Figs. 3A and 3B, SVOC peaks that were considered to originate from diesel exhaust were also collected and determined (*m*/*z* 153). Since the developed device could be used to collect VOCs having a higher carbon number, further application of the proposed method is expected, such as in investigations of photochemical ozone creation.

Conclusion

A newly designed collection device for the precise determination of airborne PAHs was developed with a Sty-DVB adsorbent, known as Sunpak-H. Moisture from the air was not trapped on the adsorbent during air collection. The developed device indicated a rapid and quantitative elution of the collected analytes by the simple passing of an elution solvent, and also easier reuse. The usefulness for the quantitative determination of airborne PAHs with the developed collection device, compared to a conventional adsorbent, was also quantitatively indicated. The wide bore device was successfully applied for the determination of airborne PAHs in air samples from a tunnel and a roadside. The proposed method could be further applied for precise determinations of several SVOCs, such as sesquiterpenes, phthalates, and those related to photochemical ozone and aerosol creation.

Acknowledgements

This work was supported by JSPS KAKENHI (Grant Number 15K17875). One of the authors (I. U.) acknowledges support from Prof. Y. Saito, Toyohashi University of Technology and from S. Kawakubo of University of Yamanashi.

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