

Extraction of Polychlorinated Biphenyls from Polyurethane Foam Filters



Summary

The EDGE® offers a reliable, quick, and efficient sample preparation method for any sample type, including the extraction of polychlorinated biphenyls (PCBs) from polyurethane foam (PUF) filters. It utilizes a combination of pressurized fluid extraction and dispersive solid phase extraction to drastically reduce the sample preparation time, solvent usage, and potential for human error by unprecedented levels. EDGE recovery results are comparable to the traditional extraction method, Soxhlet, for the small and large PUF filters. The extractions performed with the EDGE use less solvent, last seven minutes, and do not require post extraction clean-up or volume transfer. The Soxhlet extraction apparatus took 16 hours, 300 mL of solvent, and multiple vessel transfers, further highlighting the extraction efficiency of the EDGE.

Introduction

In this application note, the EDGE is compared to Soxhlet extraction as a method for the extraction of PCBs from PUF filters. The U.S. EPA TO-4A Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air refers to Soxhlet extraction as the primary extraction method used to accurately quantify PCBs extracted from Polyurethane foam (PUF) filters.¹ PUF is the primary material used to filter and capture persistent organic pollutants (POPs), including PCBs for air monitoring, as described in US EPA TO-4A. PUF filters are used in high-volume air sampling systems, or as passive air samplers (PAS), because of their ability to trap PCBs. As a PAS, PUF filters are economical and do not require electricity, like high-volume air samplers.

The importance of monitoring ambient air for PCBs is related to their negative impact on human health and the environment. PCBs are man-made chlorinated organic compounds containing a biphenyl nucleus on which a combination of one to all ten of the hydrogen atoms are substituted with chlorine. Their use was very popular in the electronics and automotive industries, until the US EPA banned them in 1979. Because of their chemical and physical stability, PCBs do not regularly break down in the environment and are, therefore, still present in soil, air, and water sources. Inhalation of PCBs is connected to a variety of adverse health effects, including cancer. Risk of inhalation exposure is greater in industrial and urban areas, where PCB-contaminated equipment is likely still present. The volatilization of PCBs from spills, landfills, road oils, and other sources, results in measurable atmospheric emissions. Atmospheric transport is the primary mode of the global distribution of PCBs. This further highlights the importance of monitoring the presence of PCBs in ambient air and extracting them for accurate quantification at variable concentrations of contamination.

The efficiencies for the extraction of PCBs from PUF filters obtained with the EDGE are comparable to Soxhlet extraction. A typical Soxhlet extraction takes 16 hours, up to 300 mL of solvent and multiple vessel transfers. In contrast, extraction on the EDGE takes seven minutes, uses less than 50 mL of solvent, and does not require a vessel transfer.

Materials and Methods

Reagents

100 µL of 5 µg/mL congener spike mix was used throughout the study to spike both large and small PUF filters (0.5 µg of PCB mix/PUF filter). Six PCBs, IUPAC Nos. 10, 28, 52, 138, 153, and 180 in a PCB congener mix, were used as the spike mix, which was purchased from Supelco Sigma-Aldrich. The internal standard used was PCB No. 14 (3,5-Dichlorophenyl), purchased from Sigma-Aldrich, to provide quantitative results. ACS Reagent Grade hexane, mixture of isomers, was purchased from Sigma-Aldrich. The small, pre-cleaned PUF filters (22 mm OD x 760 mm L) were obtained from Supelco with certifications of cleanliness. The large, uncleaned PUF filters (6.0 cm OD x 2.5 cm L) were obtained from Supelco and cleaned with the EDGE, using a process developed prior to spiking.

Sample Preparation

Small and large PUF filters were positioned accordingly in the Q-Cup® in order to optimize solvent penetration and extraction efficiency. Prior to sample insertion, the Q-Cups were rinsed with hexane to prevent any cross-contamination and eliminate background noise during analysis. A C9+G1+C9 Q-Disc® sandwich was placed in the bottom cap of the Q-Cup to allow for inline filtration. To insert the large PUF filter into the Q-Cup, the outer circumference was compressed horizontally into the top of the Q-Cup opening. Once inserted from the top, the compressed PUF filter was pushed to the base of the Q-Cup in a way such that it was equally pressed against all sides of the interior wall. See **Image 1**. The small PUF filter was inserted from the base of the Q-Cup, where the cap is removed. The small PUF filter was handled by both ends and folded together in an “S,” so that it supported itself horizontally against the interior wall. See **Image 2**. The base of the Q-Cup containing the Q-Disc was then secured onto the body of the Q-Cup.



Image 1: Insertion of large PUF filter into a Q-Cup



Image 2: Insertion of small PUF filter into a Q-Cup

EDGE Method Small PUF Filters

Q-Disc: C9+G1+C9
Solvent: Hexane
Top Add: 15 mL
Bottom Add: 0 mL
Rinse: 15 mL
Temperature: 100 °C
Hold Time: 10 s
Wash 1: 10 mL Hexane
Wash 2: 10 mL Hexane

EDGE Method Large PUF Filters

Q-Disc: C9+G1+C9
Solvent: Hexane
Top Add: 20 mL
Bottom Add: 5 mL
Rinse: 15 mL
Temperature: 115 °C
Hold Time: 3 min
Wash 1: 10 mL Hexane
Wash 2: 10 mL Hexane

Soxhlet Method

Extractions, using the Soxhlet apparatus were carried out over 16-20 hours, using approximately 150-300 mL hexane per sample. The large and small PUF filters were compressed and pushed to the bottom of the Soxhlet tube, using a solvent-rinsed glass stirring rod. Once in place, the PUF filters were spiked with congener mix and run with 150 mL of hexane for 16 h. Each round-bottom flask was then evaporated down to approximately 30 mL, using a water steam bath. The approximately 30 mL of solvent left were directly transferred into a 40 mL glass collection vial. The round-bottomed flask was rinsed three times with 1-3 mL of clean solvent, that was added to the total volume (~30 mL) in the collection vial.

Analysis

The sample extracts were collected in individual glass collection vials that were evaporated down to slightly below 5 mL, using a Genevac evaporator. Each sample was spiked with internal standard and brought to exactly 5 mL total volume, to achieve a final concentration of 0.1 µg/mL. The samples were analyzed via GC-MS. The analysis of the PCB congener mix was conducted on an Agilent 7890A GC with a 5975C MSD. A Phenomenex ZB-5MSplus 30 m, 0.25 mm column was used. The inlet parameters were set as follows: 250 °C and 1 µL splitless injection with 0.8 mL/min flow rate, using Helium as the carrier gas. The GC conditions were as follows: initial column temperature started at 60 °C and increased to 200 °C at 30 °C/min, held for 2 min, then ramped at 10 °C/min to 295 °C and held for 6 min. Quantitation was based on a 5-point multi-level calibration curve, in the concentration interval 60 pg/L -150 pg/L for the individual PCB congeners, and PCB14 was used as the internal standard at 80 pg/L. Selective Ion Monitoring mode was used to identify each specific PCB. From that, the total area was used for quantifying each peak.

Results and Discussion

The results presented here show the EDGE is an effective method for the extraction of PCBs from large and small PUF filters, compared to Soxhlet extraction. The US EPA method TO-4A indicates that sample recoveries for individual compounds generally fall within the range of 90 to 110%, but recoveries ranging from 65 to 125% are considered acceptable.¹ The EDGE PCB recovery data fall within the 90-110% recovery range for both small and large PUF filters. **Table 1** and **Table 2** display the data for the extraction of small PUF filters, using both the EDGE and Soxhlet extractions, respectively. **Table 3** and **Table 4** display data for the extraction of large PUF filters, using the EDGE and Soxhlet extraction, respectively. The EDGE and Soxhlet extraction recoveries for all six PCBs spiked in the small and large PUF filters are in the acceptable range, determined by US EPA TO-4A.

The US EPA method TO-4A also provides the acceptable criteria for the percent relative standard deviation (RSD) and states that most compounds range from ± 5 to $\pm 30\%$. The RSD values for the EDGE in **Table 1** are noteworthy, for the small PUF filters had RSD values ranging from 0.5-3.0% but do not exceed 3%, demonstrating the excellent precision for identical sample extractions, using the EDGE. The %RSD values for the EDGE in **Table 3** for the large PUF filters are also notable. They range from 2.2-7.1%, which is below and within the acceptable range of ± 5 to $\pm 30\%$ for each PCB.

Overall, the EDGE recovery data for both small and large PUF filters demonstrate adequate recoveries, well within the acceptable ranges for the US EPA TO-4A method. The EDGE recovery data for the small PUF filters were slightly better than Soxhlet extraction, and the EDGE large PUF filter extraction data compares favorably to Soxhlet extraction. It is important to note that the average EDGE extractions for all six PCBs, extracted from both PUF sizes, were performed in seven min, with 40 mL of solvent; Soxhlet extraction is performed in 16 hours and requires up to 300 mL of solvent.

Table 1. EDGE Small PUF Filter Extraction (n=7)

PCB	% Soxhlet*	Mean	RSD
10	100	91	0.9
28	101	92	1.4
52	100	92	0.5
138	105	102	2.2
153	103	100	1.8
180	108	106	3.0

* % Soxhlet is the quotient of the mean EDGE recoveries divided by the mean Soxhlet extraction recoveries for individual PCBs.

Table 2. Soxhlet Small PUF Filter Extraction (n=9)

PCB	Mean	RSD
10	91	1.6
28	91	1.6
52	92	1.2
138	97	1.3
153	97	1.5
180	98	1.3

Table 3. EDGE Large PUF Filter Extraction (n=7)

PCB	% Soxhlet*	Mean	RSD
10	101	92	2.2
28	98	90	6.0
52	98	93	7.0
138	93	95	7.1
153	93	96	6.7
180	91	95	4.4

* % Soxhlet is the quotient of the mean EDGE recoveries divided by the mean Soxhlet extraction recoveries for individual PCBs.

Table 4. Soxhlet Large PUF Filter Extraction (n=8)

PCB	Mean	RSD
10	90	3.0
28	92	4.6
52	95	4.2
138	102	4.6
153	103	2.9
180	105	4.0

References

1. Winberry, W. T. Jr., et al., *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, U.S. Environmental Protection Agency, Center for Environmental Research Information, Office of Research and Development, Cincinnati, OH, EPA TO-4A, January 1999.*

Conclusion

After the comparison of extraction techniques, the EDGE showed a significant advantage over Soxhlet extraction. Soxhlet extraction is a substantial bottleneck in sample preparation, since it is time-consuming and requires extensive extractions. Soxhlet extraction is also inefficient, using large volumes of solvent, further highlighting Soxhlet extraction has a clear disadvantage, when compared to the EDGE. The combined PFE and dSPE dynamic extraction allows EDGE to extract PCBs from PUF filters in a fraction of the time compared to Soxhlet extraction. Both the EDGE and Soxhlet extractions provided acceptable recoveries and RSD values for all six PCBs observed, relative to the acceptance criteria provided in US EPA method TO-4A.

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