

Accelerated Solvent Extraction (ASE) of Polychlorinated Biphenyls from Polyurethane Foam Adsorbent Cartridges

INTRODUCTION

U.S. EPA Method TO-4¹ has been used for a number of years for the determination of polychlorinated biphenyls (PCBs) in air. Sampling is performed by pumping a volume of air through a polyurethane foam (PUF) adsorbent cartridge for 8–12 hours. Once adsorbed, the PCBs are extracted with an organic solvent prior to analysis by gas chromatography with electron capture detection (GC/ECD), according to U.S. EPA Method 608.² The traditional extraction method for PCBs has been Soxhlet, as typified by U.S. EPA Method 3540.³ This method requires 12–14 hours of extraction time and 350 mL of solvent per sample to perform. The size and volume requirements of the PUF cartridges have made alternative extraction methods difficult to employ. Accelerated Solvent Extraction (ASE[®]), however, has been found to be a successful alternative method with several advantages over traditional methods.

ASE is now included in the SW-846 Methods as U.S. EPA Method 3545 (SW-846 Update III).³ This method is approved for the extraction of pesticides (OCP, OPP), herbicides, semivolatiles (BNAs), and PCBs from soils, sediments, and sludges. ASE uses a combination of increased temperature and pressure to increase and optimize the kinetics of the extraction process. Using ASE, sample extractions can be performed in less time and with much less solvent than traditional methods. For example, a 10–30 g soil sample can be extracted in 12 minutes, using 15–45 mL of solvent. ASE is also automated, allowing up to 24 samples to be extracted unattended.

The work reported here discusses the use of ASE for the extraction of PCBs from PUF cartridges. Extraction of PCB congeners and Aroclor[®] mixtures were performed from two different cartridge sizes. In addition to extraction of the cartridges after sampling, the ASE method described herein can be used to clean the PUFs prior to use.

EQUIPMENT

Dionex ASE 200 Accelerated Solvent Extractor*
equipped with 11 or 22 mL cells

Dionex vials for collection of extracts (40 mL, P/N 49465;
60 mL, P/N 49466)

TurboVap[®] II Concentrator (Zymark Corporation)
Gas Chromatograph

Kimwipes[®] (Kimberly-Clark Corporation)

ORBO[™]-1000 PUF cartridge (Supelco, Inc.)

2.5 inch diam. × 3.0 inch PUF cartridge (Graseby GMW)

**ASE 150 and 350 can be used for equivalent results.*

REAGENTS AND STANDARDS

Hexane (Optima grade, Fisher Scientific)

PCB congener standard (Ultra Scientific)

PCB Aroclor standard (Chem Service)

EXTRACTION CONDITIONS

Extraction Solvent:	Hexane
Temperature:	100 °C
Pressure:	1500 psi*
Heat-up Time:	5 min
Static Time:	5 min
Flush Volume:	60%
Purge Time:	100 s
Static Cycles:	1
Total Extraction Time:	12 min per sample
Total Solvent Use:	30 mL per sample

**Pressure studies show that 1500 psi is the optimum extraction pressure for all ASE applications.*

SAMPLE PREPARATION

Orbo-1000 PUFs were loaded directly into 11 mL ASE extraction cells and capped.

3" PUFs were loaded as follows:

1. Place the PUF lengthwise on a Kimwipe.
2. Using a clean weighing spatula, depress the PUF in the middle and roll it up in the Kimwipe, as shown in Figure 1.
3. Slide out the spatula and twist closed the open ends of the Kimwipe, as shown in Figure 2.
4. Twist the rolled PUF into a 22 mL ASE cell and cap, as shown in Figure 3.
5. Following extraction, remove the PUF from the cell, unwrap from the Kimwipe, and allow any residual solvent to evaporate. The PUF will regain its full, original shape after standing at room temperature.



Figure 1



Figure 2



Figure 3

ANALYTICAL

Polyurethane foam cartridges were cleaned prior to use by ASE extraction, using the method described. PCBs were spiked onto PUF cartridges in 50- or 100- μ L volumes using Aroclor or congener standards in hexane. Following extraction, sample extracts were concentrated to 10 or 1 mL under nitrogen using a TurboVap II concentrator. Extract analysis was performed by gas chromatography with single or dual ECD detection using an HP-5890 or HP-6890 GC in accordance with US EPA Method 608. Extraction and analysis of a cleaned, unspiked PUF and Kimwipe was performed in order to verify that no interfering peaks were present.

RESULTS AND DISCUSSION

An initial laboratory capability study was performed by spiking PUF cartridges with 5.00 µg of Aroclor 1248 and extracting 4 samples each by ASE and 4 by traditional Soxhlet. Results presented in Table 1 indicate that both extraction methods generate results well within the criteria established in U.S. EPA Method 608. A method detection limit (MDL) study was then performed. Eight replicate analyses were performed with PUF cartridges spiked to contain 1 µg Aroclor 1248. The results of this study are summarized in Table 2. The MDL for Soxhlet extraction was determined to be 0.19 µg/sample and the MDL for ASE was 0.38 µg/sample.

There is an increasing trend in environmental studies to report PCB concentrations in terms of individual congeners rather than Aroclor groups. Table 3 reports results of ASE extractions performed on PUFs spiked with PCB congeners. An average recovery of 99.5% with an average %RSD of 1.9 (n=5) was achieved for the 8 congeners extracted from these samples.

Additional work was performed using Orbo-1000 PUFs. These PUFs are smaller than the traditional 3 inch PUF, and fit easily into the 11 mL ASE extraction cells. A standard solution containing 50 µg Aroclor 1254 was spiked onto these PUFs and 6 replicates were extracted as described. A recovery of 92.9% with a 2.2% RSD was achieved.

Table 1. Initial demonstration of laboratory performance for ASE and Soxhlet extraction of PUF cartridges.*

Sample #	ASE (µg)	Soxhlet (µg)	US EPA Method 608 Acceptance Criteria
LCS 1	4.63	4.87	
LCS 2	4.44	4.93	
LCS 3	4.72	5.16	
LCS 4	4.10	4.26	
Average (µg)	4.47	4.80	3.42 - 8.40 µg
Average recovery	90%	96%	38 - 158%
Standard Deviation	0.274	0.384	< 1.23

* Recoveries are based on a spiked value of 5.00 µg Aroclor 1248.

Table 2. Method detection limit study for ASE and Soxhlet.*

Sample #	ASE (µg)	Soxhlet (µg)
MDL 1	0.92	0.79
MDL 2	0.97	0.90
MDL 3	0.91	0.95
MDL 4	0.95	0.78
MDL 5	0.80	0.79
MDL 6	1.11	0.88
MDL 7	0.95	0.90
MDL 8	1.21	0.82
Average	0.98	0.85
Standard Deviation	0.13	0.06
MDL (3δ)	0.38	0.19

* Recoveries are based on a spiked value of 1.00 µg Aroclor 1248.

Table 3. Recovery of PCB congeners from spiked PUFs using ASE.

Congener	BZ#*	Spike (µg)	% Recovery	%RSD (n=5)
2-chlorobiphenyl	1	5	100.4	2.3
2,3-dichlorobiphenyl	5	5	100.3	2.0
2,4,5-trichlorobiphenyl	29	5	102.2	1.8
2,2',4,6-tetrachlorobiphenyl	50	10	97.0	1.2
2,2',3,4,5'-pentachlorobiphenyl	87	10	96.1	0.82
2,2',4,4',5,6'-hexachlorobiphenyl	154	10	97.9	1.3
2,2',3,4',5,6,6'-heptachlorobiphenyl	188	10	98.9	1.4
decachlorobiphenyl	209	15	103.1	4.0

* BZ# = Ballschmider number used to identify specific congeners.

CONCLUSION

Extraction of polyurethane foam air sampling cartridges is an essential part of current air monitoring regulations. However, currently used extraction methods are both time consuming and solvent intensive. In this Application Note, ASE has been compared to conventional Soxhlet extraction of PCBs from PUF cartridges. Results indicate that ASE provides extraction comparable to conventional methods, but ASE is faster (12 min) and uses much less solvent (30 mL) than is typically associated with these analyses. ASE takes advantage of enhanced solubilization kinetics that occur at temperatures higher than are commonly used to perform solvent extractions. As the efficiency of the extraction process is improved, less solvent and less time are required to complete the process. Because reducing solvent consumption and increasing sample throughput are important concerns to modern laboratories, ASE offers significant advantages for both the production and research lab. In addition, ASE technology is automated, thus increasing the productivity of laboratory personnel. Furthermore, since the ASE 200 Extractor carries out extractions in a sealed system, it protects laboratory personnel from solvent exposure.

REFERENCES

1. U.S. EPA, U.S. Dept. of Commerce National Technical Information Service, Compendum of Methods for the Determination of Trace Organic Compounds in Ambient Air, June 1988.
2. Office of the Federal Register Archives and Records Administration Code of Federal Regulations, Sections 100–140, Protection of the Environment, July 1, 1983.
3. U.S. EPA SW-846, Update III: Test Methods for Evaluating Solid Waste, Method 3545: Fed. Reg. Vol. 62, 114: 32451 U.S. GPO, Washington, DC, June 13, 1997.

ACKNOWLEDGMENTS

The work of Sudhakar Reddy, Kenneth Keeler, and Michael Coyle of the University of Michigan, Department of Occupational Safety and Environmental Health, originally reported as Poster 1846 as the 1998 Pittsburgh Conference, is cited in this application note and is gratefully acknowledged.

LIST OF SUPPLIERS

Zymark Corporation, Zymark Center, Hopkinton, MA 01748, USA. Tel: (508) 435-9500
Supelco, Supelco Park, Bellefonte, PA 16823, USA. Tel: (800) 247-6628
Kimberly-Clark Corporation, 351 Phelps Dr., Irving, TX 75038, USA. Tel: (972) 281-1200
Graseby GMW, 145 S. Miami Ave, Village of Cleves, OH 45002, USA. Tel: (513) 941-2229
Fisher Scientific, 711 Forbes Ave., Pittsburgh, PA 15219, USA. Tel: (412) 562-8300
Ultra Scientific, 250 Smith St., Noth Kingstown, RI 02852, USA. Tel: (401) 294-9400
Chem Service, 660 Tower Ln., West Chester, PA 19381, USA. Tel: (800) 452-9994

ASE is a registered trademark of Dionex Corporation.
Aroclor is a registered trademark of Monsanto.
TurboVap is a registered trademark of Zymark Corporation.
Kimwipes is a trademark of Kimberly-Clark Corporation.
ORBO is a trademark of Supelco, Inc.

Speed • Simplicity • Solutions



Dionex Corporation

1228 Titan Way
P.O. Box 3603
Sunnyvale, CA
94088-3603
(408) 737-0700

North America

U.S./Canada (847) 295-7500

South America

Brazil (55) 11 3731 5140

Europe

Austria (43) 1 616 51 25 Benelux (31) 20 683 9768; (32) 3 353 4294
Denmark (45) 36 36 90 90 France (33) 1 39 30 01 10 Germany (49) 6126 991 0
Ireland (353) 1 644 0064 Italy (39) 02 51 62 1267 Sweden (46) 8 473 3380
Switzerland (41) 62 205 9966 United Kingdom (44) 1276 691722

Asia Pacific

Australia (61) 2 9420 5233 China (852) 2428 3282 India (91) 22 2764 2735
Japan (81) 6 6885 1213 Korea (82) 2 2653 2580 Singapore (65) 6289 1190
Taiwan (886) 2 8751 6655

www.dionex.com



LPN 1083-03 PDF 6/11
©2011 Dionex Corporation