Extraction of PCBs from Environmental Samples Using Accelerated Solvent Extraction

Meets the requirements of U.S. EPA Method 3545

John Ezzell and Bruce Richter
Thermo Fisher Scientific, Salt Lake City, UT, USA

Introduction

Accelerated solvent extraction is a new extraction method that significantly streamlines sample preparation. A commonly used solvent is pumped into an extraction cell containing the sample, which is then brought to an elevated temperature and pressure. Minutes later, the extract is transferred from the heated cell to a standard collection vial for cleanup or analysis. The entire extraction process is fully automated and performed in minutes for fast and easy extraction with low solvent consumption.

Accelerated solvent extraction is used as a direct replacement for solvent-intensive techniques such as Soxhlet and sonication. For the preparation of solid waste samples containing PCBs, accelerated solvent extraction provides more convenient, faster extractions with significantly less solvent usage than these other methods. Accelerated solvent extraction extracts a 10 g sample of a typical solid waste in about 10 min with a total solvent consumption of approximately 15 mL.

PCBs are found in many solid waste materials worldwide. This application note describes the application of accelerated solvent extraction to the extraction of PCBs from sewage sludge, river sediments, marine sediments, and marine tissue (oyster). The procedures described in this application note meet the requirements for sample extraction as determined by U.S. EPA Method 3545 for solid samples.

Equipment

- Thermo Scientific™ Dionex™ ASE™ 200 Accelerated Solvent Extractor,* with 11 mL or larger stainless steel extraction cells
- GC with ECD
- Vials for collection of extracts (40 mL, P/N 49465; 60 mL, P/N 49466)

* Dionex ASE 150 and 350 Accelerated Solvent Extractors can be used for equivalent results.

Solvents

- Hexane
- Acetone

Conditions

System Pressure:	1500 psi*
Oven Temperature:	100 °C
Sample Size:	5 to 10 g
Oven Heatup Time:	5 min
Static Time:	5 min
Flush Volume:	60% of extraction cell volume
Solvent:	Hexane/acetone (1:1), (v/v)
Nitrogen Purge:	1 MPa (150 psi) for 60 s

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

Sample Information

Sewage sludge was obtained from the Fresenius Institute (Taunusstein, Germany). Oyster tissue samples were obtained from the National Oceanographic and Atmospheric Administration (NOAA) Laboratory (Seattle, Washington, USA). The river sediment is a standard reference material, SRM 1939 (National Institute of Science and Technology, Gaithersburg, Maryland, USA). Contaminated soil used in this study was a certified reference material (CRM911-050) purchased from Resource Technology Corporation (Laramie, Wyoming, USA).

Sample Preparation

Samples should be dried and ground. Before filling the cell, a cellulose disk should be placed in the outlet end of the cell. Samples that contain water (greater than 10%) should be mixed in equal proportions with Dionex ASE Prep DE (diatomaceous earth) (P/N 062819).

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Quantification of Sewage Sludge, Oyster Tissue, and River Sediment

Sample extracts from accelerated solvent extraction were prepared for analysis by passing through silver nitrate/sulfuric acid loaded silica gel and alumina columns followed by concentration to 1 mL for GC analysis. PCB analyses were performed by gas chromatography with ECD using a 30 m × 0.25 mm i.d. amine column. Injector and detector were maintained at 300 °C. The GC oven was programmed from 100–300 °C at 10 °C/min following a 5 min hold. External standards were used for calibration.

Quantification of Soil (CRM911-050)

PCB analyses of the soil extracts were performed according to U.S. EPA SW-846 Method 8080. The Dionex ASE 200 Accelerated Solvent Extractor extracts were diluted to 25 mL prior to analysis by GC. Injection was through a split/splitless injector in a GC with dual electron capture detectors. Two 30 m × 0.53 mm i.d. capillary columns provided primary and confirmation data, respectively. Both columns were joined with a fused silica Y connector. The 30 m × 0.53 mm i.d. remaining part of the Y was connected to a 5 m section of deactivated 0.53 mm i.d. fused silica capillary tubing that acted as a guard column. The end of this guard column was inserted into the GC injector. Dual confirmation of the analytes was achieved with a single 5 µL injection. The injector was maintained at 220 °C and both detectors were operated at 320 °C. The oven was programmed from 60–200 °C at 28 °C/min after a 1 min hold, then 265 °C at 10 °C/min holding for 20.5 min. Helium was used as the carrier gas at a linear velocity of approximately 30 cm/s.

Analytical Results

Results from extractions of sewage sludge, oyster tissue, river sediment, and soil are shown in Tables 1 through 4. These tables show the average recoveries and RSDs (%) for PCB congener content of these matrices.[1] Recoveries for all compounds with the exception of one (PCB 153 from the river sediment) are above 77% of the certified or Soxhlet comparison values. Interferences in the river sediment extract also prevented quantification of two low molecular weight PCB congeners (PCB 28 and PCB 52).

The results demonstrate the effectiveness of accelerated solvent extraction as a sample preparation method. Accelerated solvent extraction provides extracts with minimal solvent usage and significant time reduction compared to other extraction methods. Results are comparable to the traditional Soxhlet extraction method.

Accelerated solvent extraction meets the requirements for PCB analysis as described in U.S. EPA SW-846 Method 3545.

Table 1. PCB recoveries from sewage sludge[a].

PCB Congener	Average Recovery, n = 6 (as % of Soxhlet)	RSD (%)
PCB 28	118.1	2.5
PCB 52	114.0	4.7
PCB 101	142.9	7.4
PCB 153	109.5	5.8
PCB 138	109.6	3.9
PCB 180	160.4	7.5

[a] Analyte concentration range: 160–200 µg/kg per component

Table 2. PCB recovery from oyster tissue[a].

PCB Congener	Average Recovery, n = 6 (as % of Soxhlet)	RSD (%)
PCB 28	90.0	7.8
PCB 52	86.9	4.0
PCB 101	83.3	1.5
PCB 153	84.5	3.5
PCB 138	76.9	3.0
PCB 180	87.0	4.3

[a] Analyte concentration range: 50–150 µg/kg per component

Table 3. PCB recovery from river sediment (SRM 1939)[a].

PCB Congener	Average, n = 6 (as % of Soxhlet)	RSD (%)
PCB 101	89.2	3.7
PCB 153	62.3	4.1
PCB 138	122.1	2.3
PCB 180	111.5	5.9

[a] Analyte concentration range: 170–800 µg/kg per component

Table 4. Recovery of arochlor 1254 from soil (CRM911-050).

Run Number	Arochlor Found (µg/kg)
1	1290.0
2	1365.8
3	1283.4
4	1368.6
Average RSD	1327.0 (99.0%) 3.51%

References

1. Richter, B.; Ezzell, J.; Felix, D. *Single Laboratory Method Validation Report: Extraction of Organophosphorous Pesticides, Chlorinated Herbicides, and Polychlorinated Biphenyls Using Accelerated Solvent Extraction (ASE) with Analytical Validation by GC/NPD and GC/ECD*, Document 101124, Dionex Corporation (now part of Thermo Scientific), December 2, 1994.

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