Accelerated Solvent Extraction
Environmental Applications Summary

Pesticides • Chlorinated Compounds • Persistent Organic Pollutants (POPs)
Contents

Introduction
The Accelerated Solvent Extraction System ........................................................................................................... 4

Dioxins and Furans
Extraction of Polychlorinated Dibenzo-p-dioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) from Environmental Samples Using Accelerated Solvent Extraction .......................................................................................................................... 5

Herbicides
Extraction of Chlorinated Herbicides Using Accelerated Solvent Extraction .......................................................................................................................... 6

Organochlorinated Pesticides (OCPs)
Extraction of Chlorinated Pesticides Using Accelerated Solvent Extraction .......................................................................................................................... 7

Organophosphorus Pesticides (OPPs)
Extraction of Organophosphorus Pesticides Using Accelerated Solvent Extraction .............................................................. 8

Persistent Organic Pollutants (POPs)
Rapid Determination of POPs Using Accelerated Solvent Extraction .............................................................................. 9

Polybrominated Diphenyl Ethers (PBDEs)
Analyzing PBDEs in House Dust Samples with the Thermo Scientific TSQ Quantum XLS Ultra GC-MS/MS in El-SRM Mode .......................................................................................... 10
Rapid Determination of PBDEs in Biosolids and Waste Samples Using Accelerated Solvent Extraction ...................................................................................................................................................... 11

Polychlorinated Biphenyls (PCBs)
Accelerated Solvent Extraction of Polychlorinated Biphenyls from Polyurethane Foam Adsorbent Cartridges .................................................. 12
Extraction of Polychlorinated Biphenyls from Environmental Samples Using Accelerated Solvent Extraction .................................................. 13

References
Accelerated Solvent Extraction References .................................................................................................................. 14
Accelerated solvent extraction is an established technique used for fast, accurate sample preparation of air filters, soils, solids, and wastes. Coupled with rapid solvent reduction systems, the often labor-intensive steps of sample preparation can be automated in your laboratory. Accelerated solvent extraction eliminates many of the manual steps involved in preparing samples for analysis, which helps ensure increased reproducibility and accelerates the process significantly.

The Thermo Scientific™ Dionex™ ASE™ Accelerated Solvent Extractor systems use a combination of elevated temperature and pressure with common solvents to increase the efficiency of the extraction process. The result is faster extraction times and a significant reduction in solvent use. Many of the organic solvents used in extractions boil at relatively low temperatures. This is a limitation to techniques such as Soxhlet or automated Soxhlet as the highest temperatures at which extractions take place in these techniques will be the solvent's boiling point. If sufficient pressure is exerted on the solvent during the extraction, temperatures above the boiling point can be used. Therefore all of the advantages of working at elevated temperature can be realized even with solvents with relatively low boiling points. Operating at elevated pressures also accelerates the extraction process. Pumping solvent through a packed bed is easier at elevated pressures; pressurized solvent is forced into the pores of the sample matrix. Hence, the combination of elevated temperatures and pressures allows extractions to occur rapidly and completely.

When extractions are achieved at elevated temperatures, several factors contribute to improved speed, efficiency, and reduced solvent use: 1) solvent strength is higher, 2) diffusion rates are faster, 3) solvent viscosity is decreased, and 4) solute-matrix interactions (dipole attractions, Van der Waals forces, hydrogen bonding, etc.) are more easily disrupted allowing the analytes to be removed from the matrix.

For environmental applications, accelerated solvent extraction is proven to produce equivalent to or better than traditional methods. Accelerated solvent extraction is accepted for use in EPA Method SW-846 3545 for the extraction of pesticides and herbicides, PAHs and semivolatile compounds, PCBs, dioxins and furans, and explosive compounds. Accelerated solvent extraction is also accepted for use in EPA Method SW-846 6860 for the determination of perchlorate and in CLP OLM 04.2A for semivolatile compounds. The Application Briefs compiled here show conditions for extracting pesticides, chlorinated compounds, and POPs from a variety of environmental samples. To view the complete Application Notes, visit our website at www.thermoscientific.com/samplepreparation.
### Introduction

Regulatory agencies are concerned with the high toxicity of PCDDs and PCDFs; great efforts are expended to monitor their presence in environmental samples. The low concentrations at which these compounds are regulated and the strength of their binding to certain matrices pose special challenges to analytical chemists. Accelerated solvent extraction has been applied to the extraction of PCDD and PCDFs from ground chimney brick, urban dust, fly ash and sediment samples. Accelerated solvent extraction complies with U.S. EPA Method 3545 for these compounds.

### Equipment

* Dionex ASE 200 Accelerated Solvent Extractor*

Gas Chromatograph (GC) with Mass Spectrometer (MS) (low resolution or high resolution MS-MS)

Low pressure LC system for sample clean-up

Preparative HPLC for sample clean-up

### Solvents

Toluene, acetic acid, hydrochloric acid. All solvents are pesticide-grade or equivalent and are available from Fisher Scientific.

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

### Extraction Conditions

**Solvent:** Toluene or toluene/acetic acid (5%, v/v) if HCl pretreatment currently used

**Temperature:** 175–200 ºC

**Pressure:** 1500 psi**

**Static Time:** 5–15 min

**Static Cycles:** 2 or 3

**Flush Volume:** 60–70%

**Purge Time:** 60–100 s

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

### Analysis

**GC-MS**

### Results

Left table: Average values (ng/kg) from ground chimney brick—comparison of Soxhlet vs. ASE.

Right table: Average values (ng/kg) from urban dust—comparison of Soxhlet vs. ASE.

### Table 1: Average values (ng/kg) from ground chimney brick—comparison of Soxhlet vs. ASE

<table>
<thead>
<tr>
<th>Congeners</th>
<th>Soxhlet (n=1)</th>
<th>ASE (n=2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2,3,7,8-T-PCDD</td>
<td>3.3</td>
<td>3.2</td>
</tr>
<tr>
<td>1,2,3,7,8-P-PCDD</td>
<td>11.8</td>
<td>13.1</td>
</tr>
<tr>
<td>1,2,3,4,7,8-H-PCDD</td>
<td>9.8</td>
<td>8.0</td>
</tr>
<tr>
<td>1,2,3,6,7,8- H-PCDD</td>
<td>11.5</td>
<td>9.5</td>
</tr>
<tr>
<td>1,2,3,7,8,9-HPCDD</td>
<td>n.d. (8)</td>
<td>n.d. (8)</td>
</tr>
<tr>
<td>1,2,3,4,6,7- H-PCDD</td>
<td>113</td>
<td>107</td>
</tr>
<tr>
<td>2,3,7,8-T-PCDF</td>
<td>12.5</td>
<td>18.6</td>
</tr>
<tr>
<td>1,2,3,7,8,12,13,4,6-P-PCDF</td>
<td>9.9</td>
<td>12.0</td>
</tr>
<tr>
<td>2,3,4,7,8-P-PCDF</td>
<td>13.9</td>
<td>18.1</td>
</tr>
<tr>
<td>1,2,3,4,7,8,9-H-PCDF</td>
<td>18.7</td>
<td>23.7</td>
</tr>
<tr>
<td>1,2,3,6,7,8- H-PCDF</td>
<td>10.7</td>
<td>15.8</td>
</tr>
<tr>
<td>2,3,4,6,7,8-HPCDD</td>
<td>3.3</td>
<td>8.7</td>
</tr>
<tr>
<td>1,2,3,7,8-9-HPCDD</td>
<td>n.d. (2)</td>
<td>n.d. (2)</td>
</tr>
<tr>
<td>1,2,3,4,6,7-9-HPCDD</td>
<td>13.2</td>
<td>29.4</td>
</tr>
<tr>
<td>1,2,3,4,7,8-9-HPCDD</td>
<td>n.d. (2)</td>
<td>n.d. (2)</td>
</tr>
<tr>
<td>Toxicity equivalent (NATO)</td>
<td>25.0</td>
<td>28.7</td>
</tr>
<tr>
<td>Toxicity equivalent (EqV)</td>
<td>24.2</td>
<td>29.2</td>
</tr>
</tbody>
</table>

Values are corrected for recovery of 13C-labeled surrogates.

n.d. = not detected. Detection limit, in pg, given in parentheses.

*Sum of two extractions of each sample.

### Table 2: Average values (ng/kg) from urban dust—comparison of Soxhlet vs. ASE

<table>
<thead>
<tr>
<th>Congeners</th>
<th>Soxhlet (n=1)</th>
<th>ASE (n=2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2,3,7,8-T-PCDD</td>
<td>6.0</td>
<td>6.0</td>
</tr>
<tr>
<td>1,2,3,7,8-P-PCDD</td>
<td>52</td>
<td>57</td>
</tr>
<tr>
<td>1,2,3,4,7,8-H-PCDD</td>
<td>46</td>
<td>52</td>
</tr>
<tr>
<td>1,2,3,6,7,8- H-PCDD</td>
<td>120</td>
<td>130</td>
</tr>
<tr>
<td>1,2,3,7,8,9-HPCDD</td>
<td>97</td>
<td>100</td>
</tr>
<tr>
<td>1,2,3,4,6,7- H-PCDD</td>
<td>1000</td>
<td>820</td>
</tr>
<tr>
<td>2,3,7,8-T-PCDF</td>
<td>160</td>
<td>180</td>
</tr>
<tr>
<td>1,2,3,7,8,12,13,4,6-P-PCDF</td>
<td>430</td>
<td>470</td>
</tr>
<tr>
<td>2,3,4,7,8-P-PCDF</td>
<td>380</td>
<td>390</td>
</tr>
<tr>
<td>1,2,3,4,7,8,9-HPCDF</td>
<td>1100</td>
<td>1100</td>
</tr>
<tr>
<td>1,2,3,6,7,8- HPCDF</td>
<td>540</td>
<td>570</td>
</tr>
<tr>
<td>2,3,4,6,7,8-HPCDF</td>
<td>400</td>
<td>360</td>
</tr>
<tr>
<td>1,2,3,7,8-9-HPCDF</td>
<td>42</td>
<td>42</td>
</tr>
<tr>
<td>1,2,3,4,6,7-9-HPCDF</td>
<td>2100</td>
<td>2000</td>
</tr>
<tr>
<td>Toxicity equivalent (NATO)</td>
<td>540</td>
<td>540</td>
</tr>
<tr>
<td>Toxicity equivalent (EqV)</td>
<td>490</td>
<td>510</td>
</tr>
</tbody>
</table>

Values are corrected for recovery of 13C-labeled surrogates.

*Sum of two extractions of each sample.

---

**U.S. EPA Method 3545A: Dioxins and furans from ground chimney brick, urban dust, and fly ash**
**U.S. EPA Method 8150A with 3545A: Chlorinated herbicides from soil, sludge, and sediments**

**Equipment**
- Dionex ASE 200 Accelerated Solvent Extractor* equipped with 11 mL or larger stainless steel extraction cells
- Gas Chromatograph (GC) with Electron Capture Detector (ECD)
- Vials for collection of extracts (40 mL, P/N 049465; 60 mL, P/N 049466)

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

**Extraction Conditions**
- Solvent: Dichloromethane/acetone (1:2, v/v), with 4% (v/v) H$_3$PO$_4$/H$_2$O (1:1)
- Temperature: 100 °C
- Pressure: 1500 psi**
- Static Time: 5 min
- Static Cycles: 1
- Flush Volume: 60% of extraction cell volume
- Purge Time: 60 s

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

**Analysis**
- GC-ECD

**Results**
- See tables below.

---

**Table: Average recovery of chlorinated herbicides from three soil types**

<table>
<thead>
<tr>
<th>Chlorinated Herbicide Target Compound</th>
<th>Average Recovery (% of Shaking Method)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2,4-D</td>
<td>116.2</td>
</tr>
<tr>
<td>2,4-DB</td>
<td>112.9</td>
</tr>
<tr>
<td>2,4,5-T</td>
<td>106.6</td>
</tr>
<tr>
<td>2,4,5-TP</td>
<td>117.4</td>
</tr>
<tr>
<td>Dalapon</td>
<td>101.8</td>
</tr>
<tr>
<td>Dicamba</td>
<td>108.1</td>
</tr>
<tr>
<td>Dichlorprop</td>
<td>107.7</td>
</tr>
<tr>
<td>Dinoseb</td>
<td>118.4</td>
</tr>
</tbody>
</table>

*Averages from extraction of sand, loam, and clay soils.

---

**Table: Average recovery and precision for extraction of chlorinated herbicides from three soil types by ASE method.**

<table>
<thead>
<tr>
<th>Matrix</th>
<th>ASE (% of Spike)</th>
<th>ASE* (RSD,%)</th>
<th>Shaking (% of Spike)</th>
<th>Shaking* (RSD,%)</th>
<th>ASE as % of Shaking</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clay (low)</td>
<td>36.1</td>
<td>54.5</td>
<td>42.2</td>
<td>25.2</td>
<td>89.8</td>
</tr>
<tr>
<td>Clay (high)</td>
<td>71.1</td>
<td>15.0</td>
<td>61.6</td>
<td>21.6</td>
<td>112.8</td>
</tr>
<tr>
<td>Loam (low)</td>
<td>56.7</td>
<td>11.2</td>
<td>36.9</td>
<td>78.7</td>
<td>126.6</td>
</tr>
<tr>
<td>Loam (high)</td>
<td>59.9</td>
<td>14.1</td>
<td>43.9</td>
<td>14.7</td>
<td>132.8</td>
</tr>
<tr>
<td>Sand (low)</td>
<td>51.1</td>
<td>12.6</td>
<td>49.7</td>
<td>13.3</td>
<td>111.2</td>
</tr>
<tr>
<td>Sand (high)</td>
<td>69.2</td>
<td>39.5</td>
<td>66.3</td>
<td>35.5</td>
<td>104.4</td>
</tr>
</tbody>
</table>

*Low spiking levels ranged from 50 to 500 µg/kg. High spiking levels ranged from 500 to 5000 µg/kg.

*Each precision (RSD, %) value is the average of seven replicate measurements for each compound, then averaged for all compounds.
Introduction
Accelerated solvent extraction provides a more convenient, faster, and less solvent intensive method than previously available for the extraction of chlorinated pesticides from environmentally important samples. Recoveries of these analytes by accelerated solvent extraction are equivalent to or better than other more solvent intense methods such as Soxhlet. Accelerated solvent extraction also avoids the problem of multiple washing procedures associated with sonication.

The procedure described in this Application Note meets the requirement for sample extraction as prescribed by EPA Method 3545. This method is applicable to the extraction of water-insoluble or slightly water-soluble volatiles and semivolatiles in preparation for gas chromatographic or GC/MS measurement. The method is applicable to the extraction of chlorinated pesticides from soils, clays, wastes, and sediments containing from 5 to 250 μg/kg of the target compounds.

U.S. EPA Method 3545A: Organochlorinated Pesticides (OCPs) from soil, clay, sludge, and sediments

Equipment
Dionex ASE 200 Accelerated Solvent Extractor* equipped with 11 mL or larger stainless steel extraction cells
Gas Chromatograph (GC) with Mass Spectrometer (MS) or GC Vials for collection of extracts (40 mL, P/N 049465; 60 mL, P/N 049466)

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

Solvents and Reagents
Diatomaceous Earth (DE)
Dispersant for ASE, 1 kg Bottlet (P/N 062819)
Hexane
Acetone

All solvents are pesticide-grade or equivalent and are available from Fisher Scientific.

Extraction Conditions
Solvent: Acetone/hexane (1:1 v/v)
Temperature: 100 °C
Pressure: 1500 psi**
Static Time: 5 min
Static Cycles: 1
Flush Volume: 60%
Purge Time: 60 s

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

Analysis
GC-MS

Results
Average recovery of pesticides from three soil types*—ASE compared to automated Soxhlet.

<table>
<thead>
<tr>
<th>Pesticide</th>
<th>Average Recovery (% of Soxhlet)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alpha BHC</td>
<td>93.3</td>
</tr>
<tr>
<td>Gamma BHC-Lindane</td>
<td>95.6</td>
</tr>
<tr>
<td>Beta BHC</td>
<td>98.6</td>
</tr>
<tr>
<td>Heptachlor</td>
<td>88.0</td>
</tr>
<tr>
<td>Delta BHC</td>
<td>99.5</td>
</tr>
<tr>
<td>Aldrin</td>
<td>94.9</td>
</tr>
<tr>
<td>Heptachlor Epoxide</td>
<td>100.7</td>
</tr>
<tr>
<td>Gamma Chlor dane</td>
<td>99.5</td>
</tr>
<tr>
<td>Alpha Chlor dane</td>
<td>102.0</td>
</tr>
<tr>
<td>Endosulfan 1</td>
<td>100.3</td>
</tr>
<tr>
<td>p,p'-DDE</td>
<td>98.6</td>
</tr>
<tr>
<td>Dieldrin</td>
<td>101.2</td>
</tr>
<tr>
<td>Endrin</td>
<td>97.2</td>
</tr>
<tr>
<td>p,p'-DDD</td>
<td>104.6</td>
</tr>
<tr>
<td>Endosulfan II</td>
<td>105.6</td>
</tr>
<tr>
<td>p,p'-DDT</td>
<td>74.9</td>
</tr>
<tr>
<td>Endrin Aldehyde</td>
<td>104.0</td>
</tr>
<tr>
<td>Endosulfan Sulfate</td>
<td>105.2</td>
</tr>
<tr>
<td>Methocychlor</td>
<td>79.6</td>
</tr>
<tr>
<td>Endrin Ketone</td>
<td>102.9</td>
</tr>
</tbody>
</table>

* Averages from extraction of sand, loam, and clay soils.
Extraction of Organophosphorus Pesticides (OPPs) Using Accelerated Solvent Extraction

**U.S. EPA Method 3545A: OPPs from soil, sludge, and sediments**

**Equipment**
- Dionex ASE 200 Accelerated Solvent Extractor* equipped with 11 mL or larger stainless steel extraction cells
- Gas Chromatograph (GC) with Nitrogen Phosphorous Detector (NPD)
- Vials for collection of extracts (40 mL, P/N 049465, 60 mL, P/N 049466)

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

**Solvents and Reagents**
- Diatomaceous Earth (DE) Dispersant for ASE, 1 kg Bottle (P/N 062819)
- Dichloromethane
- Acetone

All solvents are pesticide-grade or equivalent and are available from Fisher Scientific.

**Extraction Conditions**
- Solvent: Dichloromethane/acetone (1:1 v/v)
- Temperature: 100 °C
- Pressure: 1500 psi**
- Static Time: 5 min
- Static Cycles: 1
- Flush Volume: 60% of extraction cell volume
- Purge Time: 60 s

**Analysis**
- GC-NPD

**Results**

<table>
<thead>
<tr>
<th>Matrix</th>
<th>ASE (% of Spike)</th>
<th>ASE (RSD,%)</th>
<th>Shaking (% of Spike)</th>
<th>Shaking (RSD,%)</th>
<th>ASE as % of Shaking</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clay (low)</td>
<td>55.0</td>
<td>6.2</td>
<td>56.4</td>
<td>7.6</td>
<td>98.8</td>
</tr>
<tr>
<td>Clay (high)</td>
<td>69.2</td>
<td>5.2</td>
<td>72.3</td>
<td>16.3</td>
<td>96.3</td>
</tr>
<tr>
<td>Loam (low)</td>
<td>61.3</td>
<td>11.6</td>
<td>60.4</td>
<td>8.6</td>
<td>103.6</td>
</tr>
<tr>
<td>Loam (high)</td>
<td>61.4</td>
<td>7.8</td>
<td>64.2</td>
<td>6.3</td>
<td>96.5</td>
</tr>
<tr>
<td>Sand (low)</td>
<td>59.0</td>
<td>13.0</td>
<td>63.3</td>
<td>6.7</td>
<td>95.0</td>
</tr>
<tr>
<td>Sand (high)</td>
<td>64.1</td>
<td>11.9</td>
<td>63.2</td>
<td>4.8</td>
<td>101.2</td>
</tr>
</tbody>
</table>

* Low spiking level is approximately 250 µg/kg. High spiking level is approximately 2500 µg/kg.

* Each recovery and precision (%RSD) value is the average of seven replicate measurements for each compound, then averaged for all compounds.

**Introduction**

The use of accelerated solvent extraction in the extraction of OPP compounds from solid wastes is a more convenient, faster, and less solvent-intensive method than previously available. OPP recoveries by accelerated solvent extraction are equivalent to recoveries from Soxhlet and other methods. Accelerated solvent extraction also avoids the problem of multiple washing procedures associated with sonication.

The procedures described in this Application Note meet the requirements for sample extraction as prescribed by U.S. EPA Method 3545A. This method is applicable to the extraction of water-insoluble or slightly water-soluble OPPs in preparation for gas chromatographic measurements. This method is applicable to soils, solid wastes, and sediments containing 250–2500 µg/kg of OPPs.
Rapid Determination of Persistent Organic Pollutants (POPs) Using Accelerated Solvent Extraction

Introduction
The United Nations Environmental Program (UNEP) has been implemented in an effort to combat the release of selected POPs. POPs are found in environmental samples such as soils, sludges, solid and semi-solid waste, and sediments. POPs are also found in biological samples such as human breast milk, and fish tissue. UNEP is interested in eliminating POPs from the environment because these compounds are considered toxic, carcinogenic, and mutagenic, and degrade slowly in the environment, posing a threat to the global environment.

Accelerated solvent extraction is equivalent to U.S. EPA Methods 3540, 3541, 3550, and 8150 for the extraction of organochlorine pesticides (OCPs), organophosphorous pesticides (OPPs), semivolatiles or base neutral acids (BNAs), chlorinated herbicides, polycyclic aromatic hydrocarbons (PAHs), and polychlorinated biphenyls (PCBs). Accelerated solvent extraction complies with U.S. EPA Method 3545A for these compounds.

U.S. EPA Methods 3540, 3541, 3550, and 8150: POPs from various environmental samples

Equipment
Dionex ASE 200 Accelerated Solvent Extractor* with Solvent Controller (P/N 048765)
Use either:
22 mL stainless steel extraction cells (P/N 048764)
11 mL stainless steel extraction cells (P/N 048765)
33 mL stainless steel extraction cells (P/N 048766)

Cellulose filters (P/N 0469458)
Vials for collection of extracts (40 mL, P/N 049465; 60 mL, P/N 049466)
Analytical balance (to read to nearest 0.0001 g or better)

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

Extraction Conditions

Pesticides and PCBs (8081/8082)
Solvent: Hexane/acetone (1:1), (v/v)
Temperature: 100 °C
Pressure: 1500 psi**
Static Time: 5 min
Static Cycles: 1–2
Flush Volume: 60%
Purge Time: 60–120 s

Hexachlorobenzene (8270)
Solvent: Dichloromethane/acetone (1:1), (v/v)
Temperature: 100 °C
Pressure: 1500 psi**
Static Time: 5 min
Static Cycles: 1–2
Flush Volume: 60%
Purge Time: 60–120 s

Dioxins (PCDD and PCDF) (8290)
Solvent: Toluene (100%) or toluene/acetic acid (5%, v/v) if HCl pretreatment currently used
Temperature: 75–200 °C
Pressure: 1500 psi**
Static Time: 5–15 min
Static Cycles: 2–3
Flush Volume: 60–70%
Purge Time: 60–120 s

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

Analysis
GC-MS
GC-MS/MS

Results
Average recovery of pesticides from three soil types—ASE compared to automated Soxhlet.

<table>
<thead>
<tr>
<th>Pesticide</th>
<th>Average Recovery (% of Soxhlet)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heptachlor</td>
<td>88.0</td>
</tr>
<tr>
<td>Aldrin</td>
<td>94.9</td>
</tr>
<tr>
<td>Gamma Chlordane</td>
<td>99.5</td>
</tr>
<tr>
<td>Alpha Chlordane</td>
<td>102.0</td>
</tr>
<tr>
<td>Dieldrin</td>
<td>101.2</td>
</tr>
<tr>
<td>Endrin</td>
<td>97.2</td>
</tr>
<tr>
<td>p,p’-DDT</td>
<td>74.9</td>
</tr>
</tbody>
</table>

* Averages from extraction of sand, loam, and clay soils.
Analyzing PBDEs in House Dust Samples with the Thermo Scientific TSQ Quantum XLS Ultra GC-MS/MS in El-SRM Mode

U.S. EPA Method 1614: PBDEs from House Dust

**Equipment**
- Dionex ASE 350 Accelerated Solvent Extractor equipped with 22 mL or larger stainless steel extraction cells
- Gas Chromatograph (GC) with Mass Spectrometer (MS)

**Solvents**
- Methylene chloride
- Silica gel

All solvents are pesticide-grade or equivalent and are available from Fisher Scientific.

**Extraction Conditions**
- Solvent: Methylene chloride (100%)
- Temperature: 100 °C
- Pressure: 1500 psi*
- Static Time: 5 min
- Static Cycles: 3
- Flush Volume: 60%
- Purge Time: 120 s

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

**Analysis**
- GC-MS

**Results**

House dust sample in NCI (top) and El-SRM (bottom) showing BDE47 at a level of 0.07 ppb.

House dust sample in NCI (top) and El-SRM (bottom) showing BDE 183 at a level of 0.17 ppb.

**Introduction**

Polybrominated diphenyl ethers (PBDEs) were introduced in the 1960s as flame retardants and are used today in a wide variety of household apparatuses, consumer electronics, furniture and more. Environmental levels of PBDEs have been continuously increasing due to their special persistence. Certain congeners have been banned completely and are currently in the list of the Stockholm convention’s POPs. Sources of human intake are typically via ingestion (nutritional) and the inhalation of indoor and house dust.

Due to growing concerns over the health risks from constant exposure to this class of compounds and the accumulation effects in the food chain, suitable analytical methods are required to quantify flame retardant compounds at low levels in a variety of matrices.
Introduction
Polybrominated diphenyl ethers (PBDEs) were developed in the early 1970s and are used today as flame retardants for various consumer products, including clothing, furniture, and plastics. Many of these consumer products are disposed in municipal landfills where the PBDEs then leach into groundwater and accumulate in certain biosystems. Recent studies indicate that PBDE concentrations in these biosystems are on the rise. In the 1980s, PBDEs were discovered in European waterways, which led the European Union to ban their production and use. Although the toxicity of PBDEs is still under investigation, evidence suggests that PBDEs may compromise endocrine or hepatic functions.

In this Application Note, PBDEs were extracted from human breast milk (freeze-dried), sediments, fish tissues, and polymers.

U.S. EPA Method 1614: PBDEs from sediments

**Equipment**
Dionex ASE 200 Accelerated Solvent Extractor* with Solvent Controller (P/N 048765)*
22 mL stainless steel extraction cells (P/N 048764)
Gas Chromatograph (GC) with Mass Spectrometer (MS)
Cellulose filters (P/N 0469458)
Vials for collection of extracts, 60 mL (P/N 049466)

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

**Solvents and Reagents**
Diatomaceous Earth (DE)
Dispersant for ASE, 1 kg Bottle (P/N 062819)
Methylene chloride
All solvents are pesticide-grade or equivalent and are available from Fisher Scientific.

**Extraction Conditions**

**Sediments**
Solvent: Methylene chloride (100%)
Temperature: 100 °C
Pressure: 1500 psi**
Static Time: 5 min
Static Cycles: 2
Flush Volume: 60%
Purge Time: 120 s

**Analysis**
GC-MS

**Results**
With a detection limit of 0.5 μg/kg, the GC/MS analysis found BDE-47 in 22% of the 332 sediment sample extracts. BDE-47 is one of two major constituents of Penta, which is a commercial product used to flame retard polyurethane foam.
Accelerated Solvent Extraction of Polychlorinated Biphenyls from Polyurethane Foam Absorbent 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. The traditional extraction method for PCBs has been Soxhlet, as typified by U.S. EPA Method 3540. 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 has been found to be a successful alternative method with several advantages over traditional methods.

U.S. EPA Method TO-41: PCBs from air sampling PUF cartridges

Equipment
Dionex ASE 200 Accelerated Solvent Extractor* equipped with either:
- 22 mL stainless steel extraction cells (P/N 048764)
- 11 mL stainless steel extraction cells (P/N 048765)
Vials for collection of extracts (40 mL, P/N 049465; 60 mL, P/N 049466)
Gas Chromatograph (GC)
Kimwipes® (Kimberly-Clark Corporation)
2.5 inch diam. x 3.0 inch PUF cartridge

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

Solvents
Hexane
All solvents are pesticide-grade or equivalent and are available from Fisher Scientific.

Extraction Conditions
Solvent: Hexane
Temperature: 100 °C
Pressure: 1500 psi**
Static Time: 5 min
Static Cycles: 1
Flush Volume: 60%
Purge Time: 100 s

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

Analysis
GC

Results
See tables below.

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

<table>
<thead>
<tr>
<th>Sample #</th>
<th>ASE (µg)</th>
<th>Soxhlet (µg)</th>
<th>US EPA Method 608 Acceptance Criteria</th>
</tr>
</thead>
<tbody>
<tr>
<td>LCS 1</td>
<td>4.63</td>
<td>4.87</td>
<td></td>
</tr>
<tr>
<td>LCS 2</td>
<td>4.44</td>
<td>4.93</td>
<td></td>
</tr>
<tr>
<td>LCS 3</td>
<td>4.72</td>
<td>5.16</td>
<td></td>
</tr>
<tr>
<td>LCS 4</td>
<td>4.10</td>
<td>4.26</td>
<td></td>
</tr>
<tr>
<td>Average</td>
<td>4.47</td>
<td>4.80</td>
<td>3.42 - 8.40 µg</td>
</tr>
<tr>
<td>Average Recovery</td>
<td>90%</td>
<td>96%</td>
<td>38 - 158%</td>
</tr>
<tr>
<td>Standard Deviation</td>
<td>0.274</td>
<td>0.384</td>
<td>&lt;1.23</td>
</tr>
</tbody>
</table>

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

Method detection limit study for ASE and Soxhlet.

<table>
<thead>
<tr>
<th>Sample #</th>
<th>ASE (µg)</th>
<th>Soxhlet (µg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MOL 1</td>
<td>0.92</td>
<td>0.79</td>
</tr>
<tr>
<td>MOL 2</td>
<td>0.97</td>
<td>0.90</td>
</tr>
<tr>
<td>MOL 3</td>
<td>0.91</td>
<td>0.95</td>
</tr>
<tr>
<td>MOL 4</td>
<td>0.95</td>
<td>0.78</td>
</tr>
<tr>
<td>MOL 5</td>
<td>0.80</td>
<td>0.79</td>
</tr>
<tr>
<td>MOL 6</td>
<td>1.11</td>
<td>0.88</td>
</tr>
<tr>
<td>MOL 7</td>
<td>0.95</td>
<td>0.90</td>
</tr>
<tr>
<td>MOL 8</td>
<td>1.21</td>
<td>0.82</td>
</tr>
<tr>
<td>Average</td>
<td>0.98</td>
<td>0.85</td>
</tr>
<tr>
<td>Standard Deviation</td>
<td>0.13</td>
<td>0.06</td>
</tr>
<tr>
<td>MDL (3σ)</td>
<td>0.38</td>
<td>0.19</td>
</tr>
</tbody>
</table>

*Recoveries are based on a spiked value of 1.00 µg Aroclor 1248.
Introduction
Polychlorinated biphenyls (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.

Accelerated solvent extraction is 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.

Equipment
Dionex ASE 200 Accelerated Solvent Extractor* equipped with 11 mL or larger stainless steel extraction cells
Gas Chromatograph (GC) with Electron Capture Detector (ECD)
Vials for collection of extracts (40 mL, P/N 049465; 60 mL, P/N 049466)
* Dionex ASE 150 and 350 can be used for equivalent results.

Solvents and Reagents
Diatomaceous Earth (DE) Dispersant for ASE, 1 kg Bottle (P/N 062819)
Acetone
Hexane
All solvents are pesticide-grade or equivalent and are available from Fisher Scientific.

U.S. EPA Method 3545A: PCBs from soil, sludge, and sediments

Extraction Conditions
Solvent: Hexane/acetone (1:1), (v/v)
Temperature: 100 °C
Pressure: 1500** psi
Static Time: 5 min
Static Cycles: 1
Flush Volume: 60% of extraction cell volume
Purge Time: 60 s

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

Analysis
GC-ECD

Results
PCB recoveries from sewage sludge*.

<table>
<thead>
<tr>
<th>PCB Congener</th>
<th>Average Recovery, n = 6 (as % of Soxhlet)</th>
<th>RSD (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PCB 28</td>
<td>118.1</td>
<td>2.5</td>
</tr>
<tr>
<td>PCB 52</td>
<td>114.0</td>
<td>4.7</td>
</tr>
<tr>
<td>PCB 101</td>
<td>142.9</td>
<td>7.4</td>
</tr>
<tr>
<td>PCB 153</td>
<td>109.5</td>
<td>5.8</td>
</tr>
<tr>
<td>PCB 138</td>
<td>109.6</td>
<td>3.9</td>
</tr>
<tr>
<td>PCB 180</td>
<td>160.4</td>
<td>7.5</td>
</tr>
</tbody>
</table>

*Analyte concentration range: 160–200 µg/kg per component

PCB recovery from river sediment (SRM 1939)*.

<table>
<thead>
<tr>
<th>PCB Congener</th>
<th>Average, n = 6 (as % of Soxhlet)</th>
<th>RSD (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PCB 101</td>
<td>89.2</td>
<td>3.7</td>
</tr>
<tr>
<td>PCB 153</td>
<td>62.3</td>
<td>4.1</td>
</tr>
<tr>
<td>PCB 138</td>
<td>122.1</td>
<td>2.3</td>
</tr>
<tr>
<td>PCB 180</td>
<td>111.5</td>
<td>5.9</td>
</tr>
</tbody>
</table>

*Analyte concentration range: 170–800 µg/kg per component

Recovery of Aroclor 1254 from soil (CRM911-050).

<table>
<thead>
<tr>
<th>Run Number</th>
<th>Aroclor Found (µg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1290.0</td>
</tr>
<tr>
<td>2</td>
<td>1365.8</td>
</tr>
<tr>
<td>3</td>
<td>1283.4</td>
</tr>
<tr>
<td>4</td>
<td>1368.6</td>
</tr>
<tr>
<td>Average</td>
<td>1327.0 (99.0%)</td>
</tr>
<tr>
<td>RSD</td>
<td>3.51%</td>
</tr>
</tbody>
</table>


A. Rubel, R. Bierl. Routine analysis of vinicultural relevant fungicides, 

H. Miyamoto, K. Ohtsuka, Y. Fukuda, Y. Ishibashi. Rapid extraction of 


M. D. David, S. Campell, Q. X. Li. Pressurized fluid extraction of nonpolar pesticides and polar herbicides using situ derivatization. Analytical Chemistry. 2000: 72,15, 3665–3670.


S. Campbell, Q.X. Li. NA+ EDTA assisted insitu derivitization pressurized fluid extraction of polar herbicides in soil. Analytica Chimica Acta. 2001: 434, 283–289.


V. Camel. Recent extraction techniques for solid matrixes-supercritical fluid extraction, pressurized fluid extraction and microwave-assisted extraction: their potential and pitfalls. *The Analyst.* 2001: 126, 1182–1193.


S. Sporring, S. Bowadt, B. Svensmark, E. Bjorklund. Comprehensive comparison of classical Soxhlet extraction with Soxtec extraction, ultrasonication extraction, supercritical fluid extraction, microwave assisted extraction and accelerated solvent extraction for the determination of polychlorinated biphenyls. *Journal of Chromatography A*. 2005: 1090, 1–9.


Pu Wang, Qinghua Zhang, Yawei Wang, Thanh Wang, Xiaomin Li, Lei Ding, Guibin Jiang. Evaluation of Soxhlet extraction, accelerated solvent extraction and microwave-assisted extraction for the determination of polychlorinated biphenyls and polybrominated diphenyl ethers in soil and fish samples. *Analytica Chimica Acta*. 2010: 663 (1) 43–48.


<table>
<thead>
<tr>
<th>Country</th>
<th>Phone</th>
</tr>
</thead>
<tbody>
<tr>
<td>Australia</td>
<td>+61 3 9757 4486</td>
</tr>
<tr>
<td>Austria</td>
<td>+43 1 333 50 34 0</td>
</tr>
<tr>
<td>Belgium</td>
<td>+32 53 73 42 41</td>
</tr>
<tr>
<td>Brazil</td>
<td>+55 11 3731 5140</td>
</tr>
<tr>
<td>China</td>
<td>+852 2428 3282</td>
</tr>
<tr>
<td>Denmark</td>
<td>+45 70 23 62 60</td>
</tr>
<tr>
<td>France</td>
<td>+33 1 60 92 48 00</td>
</tr>
<tr>
<td>Germany</td>
<td>+49 6126 991 0</td>
</tr>
<tr>
<td>India</td>
<td>+91 22 2764 2735</td>
</tr>
<tr>
<td>Italy</td>
<td>+39 02 51 62 1267</td>
</tr>
<tr>
<td>Japan</td>
<td>+81 6 8885 1213</td>
</tr>
<tr>
<td>Korea</td>
<td>+82 2 3420 8600</td>
</tr>
<tr>
<td>Netherlands</td>
<td>+31 76 579 55 55</td>
</tr>
<tr>
<td>Singapore</td>
<td>+65 6289 1190</td>
</tr>
<tr>
<td>Sweden</td>
<td>+46 8 473 3380</td>
</tr>
<tr>
<td>Switzerland</td>
<td>+41 62 205 9966</td>
</tr>
<tr>
<td>Taiwan</td>
<td>+886 2 8751 6655</td>
</tr>
<tr>
<td>UK/Ireland</td>
<td>+44 1442 233555</td>
</tr>
<tr>
<td>USA and Canada</td>
<td>+847 295 7500</td>
</tr>
</tbody>
</table>

www.thermoscientific.com/samplepreparation

©2012 Thermo Fisher Scientific Inc. All rights reserved. ISO is a trademark of the International Standards Organization. All other trademarks are the property of Thermo Fisher Scientific Inc. and its subsidiaries. This information is presented as an example of the capabilities of Thermo Fisher Scientific Inc. products. It is not intended to encourage use of these products in any manners that might infringe the intellectual property rights of others. Specifications, terms and pricing are subject to change. Not all products are available in all countries. Please consult your local sales representative for details.

Thermo Scientific Dionex products are designed, developed, and manufactured under an ISO 9001 Quality System.

4/11/18, E 10/12