Automated Solid-Phase Extraction (SPE) of Endocrine Disruptors in Water

Introduction
Endocrine disrupting compounds (EDCs) include a wide range of compounds that have the potential to interfere with the body’s endocrine system by disrupting the function of natural or endogenous hormones. Examples of potential EDCs include pesticides, such as DDT, dioxins and dioxin-like compounds, polychlorinated biphenyls, flame retardants, and plastic additives. Drinking water supplies need to be tested for the presence of EDCs because of their effect on hormones. The solid-phase extraction (SPE) method described in this Application Brief provides a quick and simple means of concentrating the components using controlled flow rates and volumes that will consistently produce good recoveries.

Instrumentation Used for Sample Preparation
Thermo Scientific™ Dionex™ AutoTrace™ 280 Solid-Phase Extraction instrument.

Sample Preparation
- A 500 mL sample of water is taken for the analysis.
- Screen for the presence of octylphenol, nonylphenol, octachlorostyrene, butylbenzylphthalate, diazinon, BPA, BHA, and BHT.
- Thermo Scientific™ Dionex™ SolEx™ SPE C18, 3 mL Cartridge, with 0.5 g of packing (P/N 074412).
- Dionex SolEx SPE C8, 3 mL Cartridge, with 0.5 g of packing (P/N 074413).
- The solvents methyltertbutylethylene (MTBE) and dichloromethane are used as eluents.
- The eluent can be dried using an in-line anhydrous sodium sulphate cartridge.

Solvents
- Isopropyl alcohol (pesticide quality or equivalent)
- Cyclohexane (pesticide quality or equivalent)
- Extraction solvent: 2.5% (v/v) cyclohexane in isopropyl alcohol (IPA)

Dionex AutoTrace 280 SPE Method and Conclusion
All solvent lines are purged and primed with solvent first. A maximum of five solvents can be used to run a wide range of methods. Independent lines separate the aqueous and organic waste solvents. The instrument sample rack has six positions that can take volumes from 10 mL to 2000 mL and six sample collection positions for the eluent. This method offers an automated rugged and reproducible solution for cleaning up the samples to concentrate and remove interferences.

<table>
<thead>
<tr>
<th>No.</th>
<th>Method: Estimated time 1 h 44 min</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Process six samples using the following method steps:</td>
</tr>
<tr>
<td>2</td>
<td>Condition the column with 5.0 mL of dichloromethane into solvent waste.</td>
</tr>
<tr>
<td>3</td>
<td>Condition column with 5.0 mL of MTBE into solvent waste.</td>
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<tr>
<td>4</td>
<td>Condition column with 5.0 mL of CH₃OH into solvent waste.</td>
</tr>
<tr>
<td>5</td>
<td>Condition column with 5.0 mL of DI water into aqueous waste.</td>
</tr>
<tr>
<td>6</td>
<td>Load 500.0 mL of sample into column.</td>
</tr>
<tr>
<td>7</td>
<td>Rinse column with 5.0 mL of DI water into aqueous waste.</td>
</tr>
<tr>
<td>8</td>
<td>Wash syringe with 5.0 mL of CH₃OH/MTBE 10:90.</td>
</tr>
<tr>
<td>9</td>
<td>Dry column with gas for 15.0 min.</td>
</tr>
<tr>
<td>10</td>
<td>Collect 5.0 mL fraction into sample tube using CH₃OH/MTBE 10:90.</td>
</tr>
<tr>
<td>11</td>
<td>Collect 5.0 mL fraction into sample tube using dichloromethane.</td>
</tr>
<tr>
<td>12</td>
<td>Wash syringe with 5.0 mL of CH₃OH.</td>
</tr>
<tr>
<td>13</td>
<td>End.</td>
</tr>
</tbody>
</table>
Parameters

### Flow Rates
- Cond Flow: 15.0 mL/min
- Load Flow: 10.0 mL/min
- Rinse Flow: 20.0 mL/min
- Elute Flow: 5.0 mL/min
- Cond Air Push: 15.0 mL/min
- Rinse Air Push: 20.0 mL/min
- Elute Air Push: 5.0 mL/min

### SPE Parameters
- Push Delay: 5 sec
- Air Factor: 1.0
- Autowash Vol.: 1.00 mL

### Instrument Parameters
- Max. Elution Vol.: 12.0 mL
- Exhaust Fan On: Yes
- Beeper On: Yes

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