ONE STEP EXTRACTION & CLEAN-UP SYSTEM FOR
RAPID ANALYSIS OF DIOXINs, PCBs, PAHs & OTHER POPs IN FOOD AND
ENVIRONMENTAL SAMPLES
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Abstract
Since chlorinated and brominated compounds are very toxic at sub-ppt (parts per trillion) and ppq (parts per quadrillion-) levels, the extraction & purification of these compounds becomes a difficult task in sample analysis. For instance performing manual extraction and clean-up for 6 food samples for Dioxins and PCBs analysis could take 3 days or more with much potential for error, human exposure, deadline delays among other things. In addition it is necessary to protect the sample from interfering compounds during the extraction, purification and fractionation processes. Indeed, interfering compounds can be introduced from the air and surrounding environment and the background amount of PCBs in the ambient air and other laboratory surroundings may exceed the detection limits of the sample. An automated one-step extraction and clean-up system has been developed which combines Pressurized Liquid Extraction and multi column purification all in one package. This closed system performs the entire extraction & clean-up for 6 samples in less than two hours producing excellent recoveries while reducing interferences caused by sample handling using traditional methods. Combining one step extraction and clean-up with FMS’s disposable prepacked columns increases the speed of sample prep, reduces human exposure to toxins, and makes the automation of POP analysis affordable.

Introduction
In recent years, advances in analytical techniques have been improved tremendously with the introduction of automated extraction and automated multi column clean-up systems. These new automated systems have improved sample analysis including increased speed and precision during analysis. Scientists are able to perform the entire extraction & Clean-up for detection of Dioxins, PCBs, Pesticides and PAHs in food samples in a very short time.

During the past few years, FMS has developed a one step PLE extraction and clean-up system. The system combines the power of automation and ready to use disposable columns to perform one step extraction and clean-up for Dioxins, PCBs, Pesticides and PAH analysis. This PLE One-step patented system offers features such as automatic documentation, real time plotting of temperature and pressure and a wide range of extraction cells in a modular and expandable design.

Figure 2 diagram describes the plumbing diagram of PLE-Pressurized Liquid Extraction System.

One Step Extraction & Clean-up
Depending on the size of the sample and the extent of the required clean-up, PLE uses one of two techniques to perform purification and clean-up: in-cell clean-up or in-line column clean-up.

PLE Extraction with In-Cell Clean-up
With this technique, the entire extraction and clean-up can be done in one step using In-cell packing material such as silica and carbon. This feature allows rapid extraction and cleanup all in one step.

Figure 1 shows the PLE cartridge with 5 – 250ml capacity, In cell silica, and heating blocks

Table 1 presents typical recoveries of PAH’s using PLE cartridge with In-cell silica.

![Fig 1 - In-Cell Extraction & Clean-up Cartridge and Packing Material](image-url)
Table 1 – In-cell clean-up: 4 g silica 10% deactivated

<table>
<thead>
<tr>
<th>PAHs and Alkil-PAHs</th>
<th>% rec.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Naphthalene</td>
<td>100</td>
</tr>
<tr>
<td>2-Methylnaphthalene</td>
<td>100</td>
</tr>
<tr>
<td>2,6-Dimethylnaphthalene</td>
<td>100</td>
</tr>
<tr>
<td>1,2-Dimethylnaphthalene</td>
<td>105</td>
</tr>
<tr>
<td>2,3,5-Trimethylnaphthalene</td>
<td>94</td>
</tr>
<tr>
<td>Rhodocyclin</td>
<td>0.2</td>
</tr>
</tbody>
</table>

**Fig 1 – PLE Diagram**

**PLE - Extraction with In-line Column Clean-up**

An optional In-line cleanup module allows additional clean-up columns to be added to the output of extraction cartridges for cleaning the sample prior to GC/MS analysis. This powerful feature of PLE saves time and money while producing excellent recoveries and precise results for all analytes. FMS offers a wide variety of disposable Teflon columns.

**Figure 3** shows PLE cartridge coupled with clean-up Column. The In-line technique is the best alternative to performing Extraction and clean-up of fatty samples using two separate procedures (or systems).

**Table 2** presents typical recoveries of PAH’s using PLE cartridge with In-line column.

**Figure 4 and Figure 5** are an example of different one step extraction and clean-up using PLE with optional clean-up modules.

**Fig 3 – PLE Cartridge with In-line Silica Column**
Fig 4 PLE – Extraction System with optional cleanup module
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Figure 5 One Step extraction and cleanup for Dioxin analysis