

ONE STEP EXTRACTION & CLEAN-UP SYSTEM FOR RAPID ANALYSIS OF 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. 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 step. This closed system performs the entire sample-Prep for 6 samples in less than one hour producing excellent recoveries while reducing interferences caused by sample handling using traditional methods.

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.

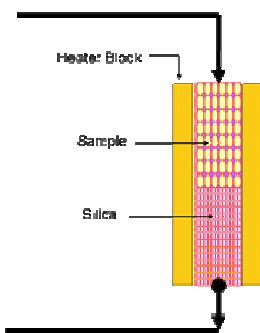
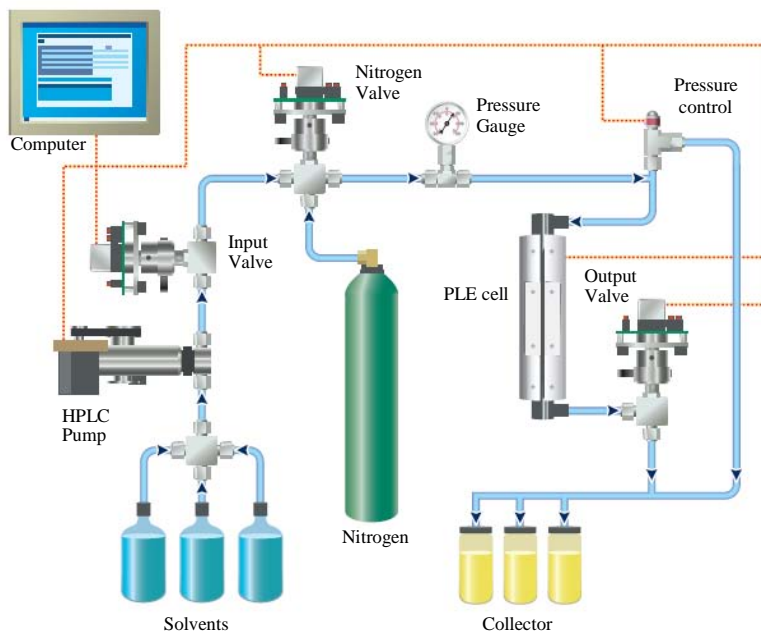


Fig 1 - In-Cell Extraction & Clean-up Cartridge and Packing Material

Table 1 – In-cell clean-up: 4 g silica 10% deactivated

	ML07-3:8(1) 1st cycle 1g soil+incell clean up	ML07-3:8(2) 2nd cycle 1g soil+incell clean up
d8-Naphtalene	84.8	0.1
d10-Acenaphthene	96.2	0.1
d10-Phenanthrene	100	0.2
d12-Chrysene	94.1	0.3
d12-Perylene	90.1	0.3

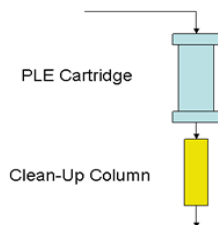
**Fig 2 – PLE Diagram****PLE - Extraction with In-line Column Clean-up**

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**

**Table 2 - PAH recoveries using
In-line 10 grams silica gel in
series with PLE system**

PAHs and Alkil-PAHs	% rec
Naphthalene	94
2-Methylnaphtalene	94
2,6-Dimethylnaphtalene	93
1,2-Dimethylnaphtalene	105
2,3,5-Trimethylnaphtalene	94
Biphenyl	94
Acenaphthylene	94
Acenaphthene	97
Fluorene	83
Phenanthrene	83
Anthracene	87
Dibenzothiophene	99
2-Mrthylphenanthrene	95
1-Methylphenanthrene	85
Pyrene	114
Dimethylphenanthrene	119
Retene	116
Benz(a)anthracene	93
Chrysene	94
Benzo(b)fluorathenes	96
Benzo(j,k)fluorathene	93
Benzo(e)pyrene	91
Benzo(a)pyrene	94
Perylene	95
Dibenz(ah)anthracene	94
Indeno(1,2,3,cd)pyrene	93
Benzo(ghi)perylene	94

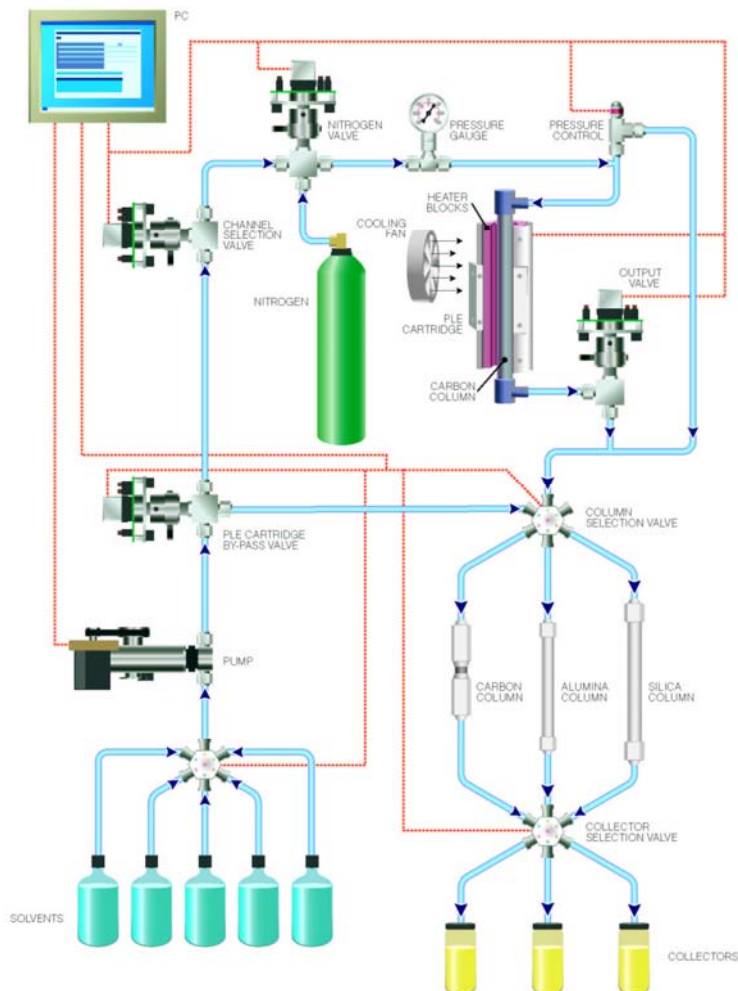


Fig 4 PLE – Extraction System with optional cleanup module

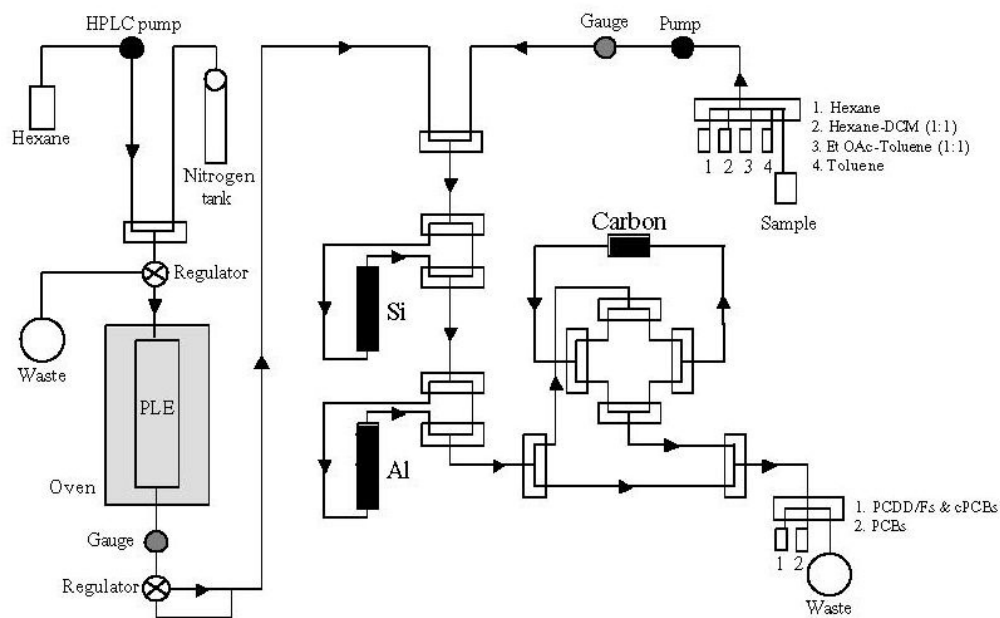


Figure 5 One Step extraction and cleanup for Dioxin analysis

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