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.

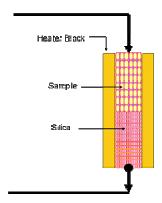
During the past few years, FMS has developed a one step PLE extraction and clean-up system. The system combines the power of automation and ready to use disposable columns to perform one step extraction and clean-up for Dioxins, PCBs, Pesticides and PAH analysis. This PLE One-step patented system offers 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 .

One Step Extraction & Clean-up

Depending on the size of the sample and 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

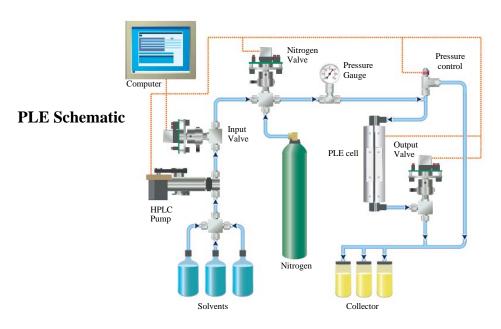
With this technique, the entire extraction and clean-up can be done in one step using In-cell packing material such as silica and carbon. This feature allows rapid extraction and cleanup all in one step.



In-Cell Extraction & Clean-up Cartrige and Packing Material

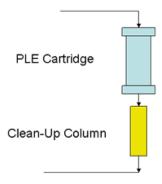
incell clean up: 4 g silica 10 deactivated

	ML07-3:8(1) 1st cycle	ML07-3:8(2) 2nd cycle	
	1g soil+incell clean up	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

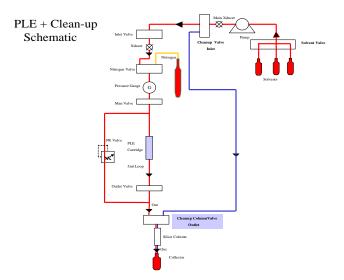


PLE - Extraction with In-line Column Clean-up

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 with capacities from 5 to 250 ml.



In-line Extraction & Clean-up



PAH recoveries using in-line 10 grams silica gel in series with PLE system

PAHs and Alkil-PAHs	% rec
Naphthalene	94
2-Methylnaphtalene	94
1-methylnaphthalen	94
2,6-Dimethylnaphthalene	93
1,2-Dimethylnaphthalene	105
2,3,6-Trimethylnaphthalene	107
2,3,5-Trimethylnaphthalene	94
1,4,6,7-Tetramethylnaphthalene	112
Biphenyl	94
Acenaphthylene	94
Acenaphthene	97
Fluorene	83
Phenanthrene	83
Anthracene	87
Dibenzothiophene	99
2-Mrthylphenanthrene	95
2-Methylanthracene	108
1-Methylphenanthrene	85
Pyrene	114
3,6,-Dimethylphenanthrene	120
1,5/1,7 Dimethylphenanthrene	119
1,2,6-Trimethylphenanthrene	116
Retene	116
Benz(a)anthracene	93
Chrysene	94
Benzo(b)fluoranthenes	96
Benzo(j,k)fluoranthene	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

HPLC pump Gauge Pump 1. Hexane 2. Hexane DCM (I:1) 3. Et OAc-Toluene (I:1) 4. Toluene Sample Vaste Regulator Regulator Regulator Waste Regulator Regulator Regulator Waste

PLE - Extraction with in -line multi column clean-up

 $Fig.~1:~Modified~plumbing~diagram~for~integrated~PLE~and~clean-up~on~the~Power-Prep^{TM}~system.$

Acknowledgements

FMS Thanks the following for providing Data on PLE and Power-Prep systems.

- Prof. Bert van Bavel
- Dr. J.-F. Focant
- Dr. W.E. Turner
- J. Sauló
- Dr.D.G. Patterson Jr.