APPLICATIONS NOTEBOOK

FMS
Fluid Management Systems

Pressurized Liquid Extraction

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PLE Pressurized Liquid Extraction System

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Affordable Laboratory Solutions

PLE™ Pressurized Liquid Extraction

Automated High Speed Extraction System

PLE™ is a high speed Pressurized Liquid Extraction system, designed to perform extraction of multiple samples simultaneously in minutes rather than hours, producing high recoveries and excellent precision for all analytes. Very inexpensive stainless steel extraction cells and end cap filtration keep the operational cost at a minimum.

The optional disposable end cap filtration increases productivity and saves valuable time.

Processes 1 to 6 Samples Simultaneously

PLE processes samples simultaneously saving valuable time.

Reduces Solvent Cost

Uses as little as 15ml of solvent as compared to more than 500ml of of solvent required to perform Soxhlet extractions.

Reduces Solvent Waste

PLE reduces solvent waste through efficient use of solvents.

Increases Productivity

The entire extraction & clean-up may be performed in less than 30 minutes. Traditional methods could take 10 - 16 hours.

Reduces Operating Cost

Rapid extraction & clean-up, along with reduced solvent use and waste, reduces operating costs by as much as 70 percent.

Extraction Cell Size 5 -250 ml

Run small to large sample sizes with the same basic system.

Modular, Expandable & Affordable

With the modular PLE design, one can purchase a 1 to 3 sample system at a very affordable price. The system can be expanded to a 5 or 6 sample system as one's application grows.

One Step Extraction & Clean-up

Optional in-line clean-up module performs the entire extraction and clean-up in one step. Thereby, increasing speed & reducing cost of sample prep.

More efficient and cost effective than traditional processing methods

Fast Automated Extraction

Fast and Efficient Simultaneous Extraction Utilizing High Pressure and Temperature

During the extraction process the solvents inside the PLE™ extraction cell are brought near their supercritical region which has high extraction properties. At high pressure and high temperatures the solvents penetrate the solid samples at a much higher rate permitting a fast and efficient extraction process with minimal solvent usage.

5 to 250ml Extraction Cell Sizes

PLE™ offers 5-250 ml low cost stainless steel extraction cells with end caps Teflon filtration. This wide range of extraction cells allows the use of the same unit for all sample sizes, even in the same run.

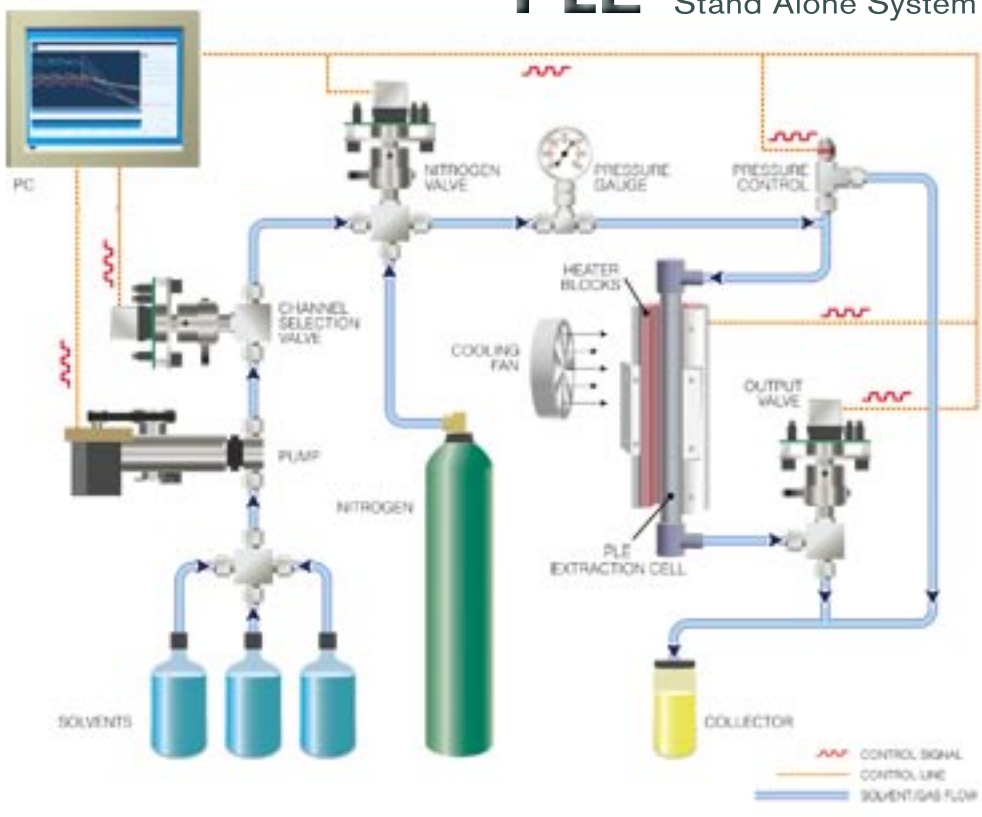
Cross Contamination Free Operation

Optional low cost disposable extraction cells and Teflon end caps filtration ensure trouble free extraction with no cross contamination..

Automatic Operation & Documentation

Real time software allows 6 channels of pressure and 6 channels of temperature data to be plotted simultaneously. This powerful feature allows automatic documentation of the entire extraction data. The temperature and pressure data can be superimposed and printed in graphic or tabular format and stored for future reference.

PLE™ Stand Alone System



Patented One-Step Extraction & Clean-up

The PLE patented one step extraction and clean-up design has the unusual flexibility to perform extraction as well as clean-up in one run. Depending on the sample size and the extent of clean-up, three configurations are available.

PLE/Power-Prep
Dual Extraction & Clean-up System

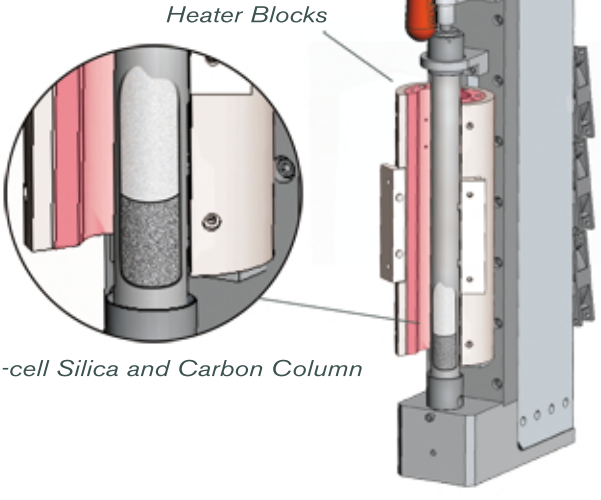
The Dual PLE/Power-Prep consists of two systems in one economical package and is truly the new fontier in rapid sample preparation for POPs analysis. The system can be used to perform extraction, clean-up or both extraction and clean-up. The modular and compact design of the systems enable the user to expand from one to six samples. The user can therefore, start off with a single sample system and expand up to a six sample system as user throughput demand increases.

In-line Column Clean-up

An optional In-line clean-up module allows additional clean-up columns to be added to the output of extraction cells 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 from 0.25 to 50 grams capacity.

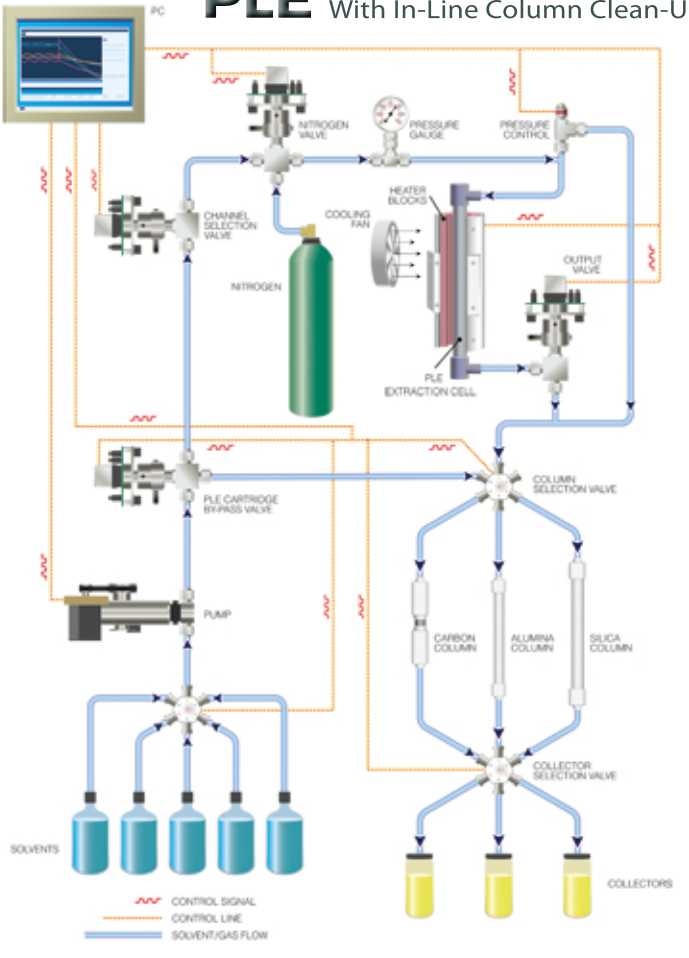
In-cell Column Clean-up

With PLE system, 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 Silica and Carbon Column

PLE™ With In-Line Column Clean-up



Electro Mechanical & Chemical Controls

Complete Control & Monitoring by PC

The entire extraction process is PC controlled allowing the laboratory technician to store and edit extraction protocols, as well as monitor and store extraction data.

Modular Construction provides for Easy Maintenance

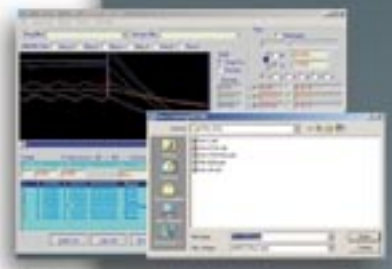
The PLE™ modular units as well as exposed plumbing construction makes for efficient system maintenance. The PLE™ channel is designed to operate independently, should one channel fail the others will continue to perform. This versatility ensures ease of replacement with no down time.

Leak & Clog Free Operation

Simple design along with large bore plumbing enables the PLE to operate virtually leak and clog free.

A Versatile Method Development Tool

The powerful DMS 6000 real time software along with a large variety of extraction cell capacities and the ability to select multiple solvents, different temperature settings, as well as storage of data makes the PLE the perfect development tool.

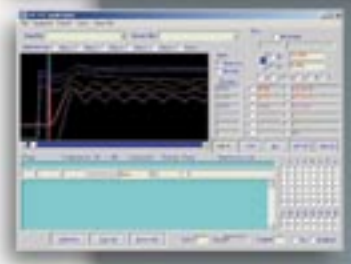


Multiple Extraction

Programming of variable pressure and temperature allows extraction of a variety of different compounds.

Multiple Method Storage

The DMS-6000 Editor allows multiple methods to be stored. In each method parameters such as time, solvent, volume and final temperature can be set for each step. Pressure and temperature as well as dispensed volumes are displayed every second and stored for future reference. Six channels of pressure and six channels of temperature may also be plotted in real time. This powerful feature allows automatic documentation of the entire extraction data.



Applications *Reliable High Speed Analysis*

For the Analysis of:

Dioxins and Furans
PCBs
Pesticides
PAHs
Vitamins & Anibiotics
PBDs and other BFRs
TPH
Herbicides
Explosives

Types of Samples:

Environmental
Soil
Sludge
Air filters & cartridges (XAD and PUFs)
Waste water

Food Samples
Fish
Meat
Fat
Milk
Agricultural products

Biological Samples
Serum
Adipose tissue

Natural Products
Natural products
Herbal products

Pharmaceutical
Vitamins and antibiotics dietary supplements

Industrial Products
Detergents
Paper and pulp
Textiles and fibers

Modular, Expandable & Affordable

The PLE™ is modular and expandable from 1 to 6 samples. allowing the user to expand the system as the demand for higher throughput grows. This feature makes the PLE™ the most affordable on the market.

Recoveries

Dioxins/Furans/PCBs

The following are typical recoveries using fish sample:

2378 - TCDD	75
2378 - TCDF	70
12378 - PeCDD	80
12378 - PeCDF	80
23478 - PeCDF	84
123478 - HXCDD	91
123678 - HXCDD	78
123789 - HXCDD	87
123478 - HXCDF	86
123678 - HXCDF	85
123789 - HXCDF	90
234678 - HXCDF	82
1234678 - HpCDF	82
1234678 - HXCDF	89
OCDD	74
OCDF	74
3344 - PCB (PCB 77)	78
3445 - PCB (PCB81)	82
33445 - PCB (PCB 126)	85
334455 - PCB (PCB169)	77

PAHs

The following are typical recoveries using fish sample:

Naphthalene	98
Acenaphthylene	98
Acenaphthene	95
Fluorene	96
Phenanthrene	103
Anthracene	96
Fluoranthene	102
Pyrene	102
Benz(a)anthracene	99
Chrysene	101
Benzofluoranthenes	99
Benzo(e)pyrene	97
Benzo(a)pyrene	98
Perylene	98
Dibenz(ah)anthracene	100
Indeno(1,2,3,cd)pyrene	99
Benzo(ghi)perylene	99
1-Methylphenanthrene	106
C2-Phen/Anthracene	106
Benzo(b)fluoranthene	96

Pressurized Liquid Extraction Ordering Information

PLE - Pressurized Liquid Extraction System

Part Number	Description
PLE /M1	**PLE modular one sample extraction system** This system Process one sample
PLE/ M2	**PLE modular two sample extraction system** This system Process two samples simultaneously
PLE/ M3	**PLE modular three sample extraction system** This system Process three samples simultaneously
PLE/ M4	**PLE modular four sample extraction system** This system Process four samples simultaneously
PLE/ M5	**PLE modular five sample extraction system** This system Process five samples simultaneously
PLE/ M6	**PLE modular six sample extraction system** This system Process six samples simultaneously

PLE Extraction Cells with Filtration Cartridges

Part number	Description
PLE-CAR250-FLT10	250 ml stainless steel extraction cell with two end cap filtration
PLE-CAR100-FLT10	100 ml stainless steel extraction cell with two end cap filtration
PLE-CAR40-FLT10	40 ml stainless steel extraction cell with two end cap filtration
PLE-CAR20-FLT10	20 ml stainless steel extraction cell with two end cap filtration

PLE Extraction Cells

Part number	Description
PLE-ECEL-SS20	20ml stainless steel extraction cell
PLE-ECEL-SS40	40ml stainless steel extraction cell
PLE-ECEL-SS100	100ml stainless steel extraction cell
PLE-ECEL-SS250	250ml stainless steel extraction cell

PLE Filtration Cartridges

Part number	Description
PLE-FLT-250M-TEF	Teflon, End cap filtration for 250 ml cell
PLE-FLT-100M-TEF	Teflon, End cap filtration for 100, 40, 20 ml cell
PLE-FLT-250M-SS	Stainless steel Reusable End cap filtration for 250 ml cell
PLE-FLT-100M-SS	Stainless steel Reusable End cap filtration for 100, 40,

PLE Accessories

Part number	Description
PLE-HTB-SM	Small Heater blocks for 20 - 40ml extraction cell
PLE-HTB-MD	Medium Heater block for 100ml extraction cell
PLE-HTB-LG	Large Heater block for 250ml extraction cell
PLE-EXT-40	Extender for low volume columns
PLE-EXT-TUB	Extension tubing for low volume column
PLE-INT-SPC-100	Teflon Spacer for 20,40,100 ml cell
PLE-INT-SPC-250	Teflon Spacer for 250ml cell

PLE Modules

Part number	Description
PLE-CNT-MD	PLE Control module
PLE-HPR-MD	High Pressure pump module
PLE-SMP-MD	Sample processing module
PLE-COL-MD	Column module

Expansion Clean-up Modules

Part number	Description
CLUP/1C	Clean-up module with one Column plumbing
CLUP/2C	Clean-up module with two Columns plumbing
CLUP/3C	Clean-up module with one Columns plumbing
PUMP-LP	Pump Module, low pressure
CNTR-LP	Control module, low pressure

Portable Cart & Spil Tray

Part number	Description
SPIL-TRY	Spil Tray
PORT-CRT	Portable Cart

FMS
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Application Note

One Step Extraction, Cleanup and Direct-to-Vial Concentration For PCBs in Soil and Sediment

Introduction

Polychlorinated biphenyls are a class of organic compounds with one to 10 chlorine atoms attached to a pair of bonded benzene rings. PCBs were widely used for many applications, especially as dielectric fluids in transformers, capacitors, and coolants. Due to PCB's toxicity and classification as a persistent organic pollutant, PCB production was banned by the United States Congress in 1979 and by the Stockholm Convention on Persistent Organic Pollutants in 2001.

PCBs have low water solubility and are largely chemically inert, making them extremely resistant to oxidation. These properties allow PCBs (commonly detected using EPA Method 8082) to resist environmental degradation so they tend to accumulate in soil and river sediment. This method determines the concentrations of various PCB congeners and Aroclors in extracts from solid and liquid matrices, including food products using gas chromatography with electron capture detectors (ECD). This new application speeds sample preparation by combining multiple processes into one. The FMS PLE (Pressurized Liquid Extraction) system automatically performs extraction and sample cleanup using the proprietary FMS InCell column and delivers the extract for final concentration directly to a vial for injection into the GC system. This is a first for analysis of PCBs.

Instrumentation

- FMS, Inc. PowerPrep™ PLE system
- FMS, Inc. 5 gm Acidic InCell Silica Column
- FMS, Inc. SuperVap™ Concentrator system
- Thermo Fisher Scientific Polaris Q GCMS

Method summary

PowerPrep PLE system

1. Extraction solvent: Hexane/Methylene Chloride (50/50)
2. Extraction temperature: 120 ºC
3. Extraction pressure: 1500 PSI
4. Extraction time: 15 minutes

SuperVap Concentrator system

1. Pre-heat temp: 55 ºC
2. Pre-heat time: 15 minutes
3. Heat in Sensor mode: 65 ºC
4. Nitrogen Pressure: 15 PSI

Procedure

1. 10 grams of are sample weighed out in 100 mL beaker, repeated for five replicates per matrix.
2. Samples were mixed and dried with Varian Hydro Matrix®.
3. The dried sample is transferred to a FMS extraction cell equipped with an InCell Acid Silica end cap.
4. Samples are spiked with 1 mL (acetone) of a 1 µg/mL PCB congener solution.
5. The void cell volume is filled with Ottawa Sand®, sealed and loaded on the FMS PLE system for extraction.
6. The sample is extracted and automatically transferred to the FMS SuperVap Direct-to-Vial concentrator system.
7. 5 µL ISTD is added to extract (PCB-209) and the extract transferred to GC/MS for analysis.

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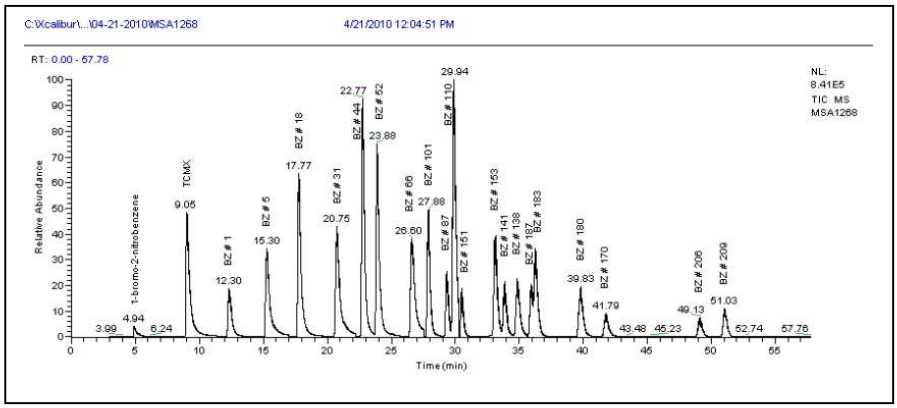
Application Note

Results

Congener	Spiked µg/kg	Sand Mean Rec. µg/kg	%	Sediment Mean Rec. µg/kg	%
BZ #1	100	114.2	114.2%	75.2	75.2%
BZ #5	100	125.2	125.2%	81.3	81.3%
BZ #18	100	119.3	119.3%	93.3	93.3%
BZ #31	100	120.6	120.6%	85.1	85.1%
BZ #44	100	119	119.0%	85.2	85.2%
BZ #52	100	119.2	119.2%	91	91.0%
BZ #66	100	143.6	143.6%	89	89.0%
BZ #87	100	124.7	124.7%	84.7	84.7%
BZ #101	100	122.8	122.8%	89.8	89.8%
BZ #110	100	111.7	111.7%	84.8	84.8%
BZ #138	100	109.4	109.4%	87	87.0%
BZ #141	100	106	106.0%	92.7	92.7%
BZ #151	100	113.1	113.1%	91	91.0%
BZ #153	100	113.3	113.3%	91.4	91.4%
BZ #170	100	120.8	120.8%	86.2	86.2%
BZ #180	100	100.7	100.7%	95	95.0%
BZ #183	100	100.1	100.1%	95	95.0%
BZ #187	100	103.6	103.6%	87.9	87.9%
BZ #206	100	103.2	103.2%	79.3	79.3%
TCMX (IS)	100	102.1	102.1%	96.9	96.9%



Conclusions

The FMS PLE system and the SuperVap™ Direct-to-Vial Concentration system in combination with the FMS 5 gm Acidic Silica gel InCell column automatically performs the extraction, cleanup and concentration of PCB analytes at a high rate of speed producing consistent recoveries and reproducibility for both soil and sediment samples.

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Application Note

One Step Extraction, Cleanup and Concentration of Pesticides from Soil

Introduction

Pesticides are classes of chemicals that are used to treat or control outbreaks of pests, especially insects. Food production facilities as well as safety and health organizations rely heavily on pesticides to increase food yields and/or control diseases spread by insects. Some pesticides have proven hazardous to other animals or the environment and have either been restricted or banned. The need to monitor food products for pesticides is essential as more pesticides are being discovered to have adverse effects. One class of commonly used pesticides is chlorinated pesticides. These compounds are commonly detected using EPA Method 8081. This method determines the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices, including food products, by GC with electron capture detectors (ECD). This new application speeds up and combines multiple sample prep processes into a single, automated method.

The FMS PLE (Pressurized Liquid Extraction) system performs automated extraction and sample cleanup using the FMS proprietary In-cell column and delivers the concentrated extract ready for injection directly into the GC system. This is a first for analyzing chlorinated pesticides by EPA Method 8081.

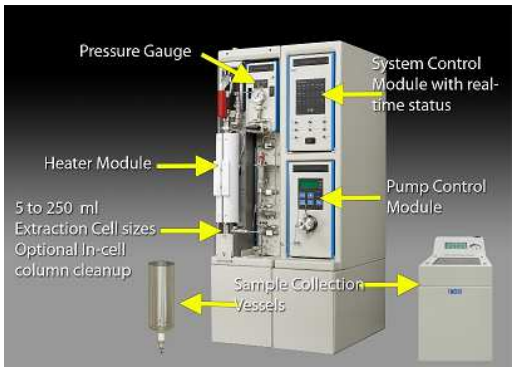
Instrumentation

- FMS, Inc. PowerPrep™ PLE (Pressurized Liquid Extraction) system
- FMS, Inc. 5 gm Acidic InCell Silica Column
- FMS, Inc. SuperVap™ Direct-to-Vial Concentration system
- Thermo Fisher Scientific Polaris Q GCMS

Method Summary

PowerPrep PLE

1. Extraction solvent: Hexane
2. Extraction temperature: 100 ºC
3. Extraction pressure: 1500 PSI
4. Extraction time: 20 minutes



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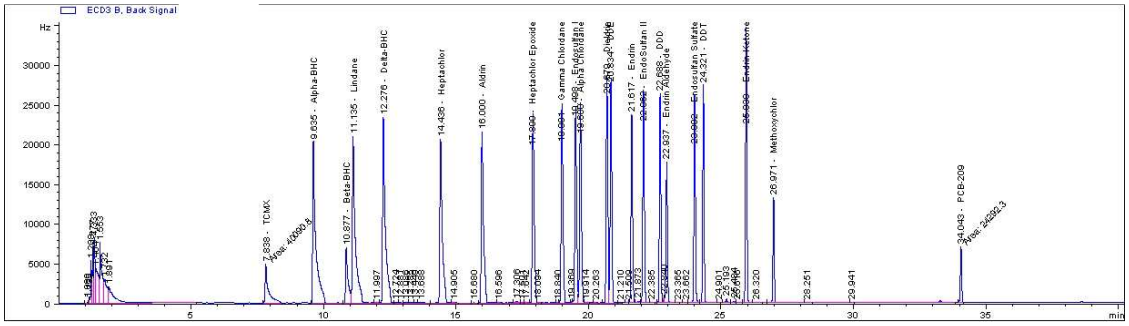
Results

Compound	Recovery (µg/mL)	Recovery
TCMX	4.77	95.4%
Alpha-BHC	4.57	91.4%
Beta-BHC	4.44	88.8%
Gamma-BHC	4.44	88.8%
Delta-BHC	4.46	89.2%
Heptachlor	4.68	93.6%
Aldrin	4.26	85.2%
Heptachlor Epoxide	4.75	95.0%
Gamma-Chlordane	4.23	84.6%
Endosulfan	4.39	87.8%
Alpha-Chlordane	4.22	84.4%
Dieldrin	4.65	93.0%
4,4'-DDE	4.33	86.6%
Endrin	5.2	104.0%
Endosulfan II	5.14	102.8%
Endrin Aldehyde	5	100.0%
4,4'-DDD	4.55	91.0%
Endosulfan Sulfate	4.55	91.0%
4,4'-DDT	4.67	93.4%
Endrin Ketone	4.8	96.0%
Methoxychlor	5.11	102.2%
Deca-PCB	5.37	107.4%

Figure 1: Pesticides in soil



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Conclusions

The FMS PowerPrep™/PLE system in combination with the FMS 2.5 gm Florisil InCell column and the FMS SuperVap™ Direct-to-Vial Concentration system provides fast, automated extraction, cleanup and concentration of samples with excellent recoveries and high reproducibility. The combination of the FMS PowerPrep™ PLE system and the FMS Teflon InCell column demonstrates consistent, reproducible high throughput for automated one-step sample preparation of pesticides extracted from soil samples. The PowerPrep™ PLE system can automatically extract, cleanup and concentrate up to six samples per hour.

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Application Note

Method for the Validation of PAHs in Soil and Sediment Samples Using Pressurized Liquid Extraction and Automated Cleanup

Introduction

PAHs are a group of organic compounds consisting of two or more benzene rings and are often the byproduct of petroleum combustion. Due to their carcinogenic characteristics at relatively low concentrations, they are of particular environmental concern. Seven PAHs have been classified by the US EPA as probable human carcinogens and their toxic characteristics and persistent nature place them among the most extensively monitored organic contaminants.

The following procedure details the use of Pressurized Liquid Extraction (PLE) in conjunction with automated silica gel cleanup (PowerPrep) to deliver a robust, efficient sample prep process for soils and sediments designated for PAH analysis. Outlined, are an Initial Precision and Recovery Study (IPR), a Matrix Detection Level Study (MDL) and a matrix validation of a NIST reference material.

Instrumentation and Consumables

- FMS, Inc. PLE System
- FMS, Inc. PowerPrep System
- FMS, Inc. SuperVap Concentrator System
- FMS, Inc. 50 mL direct-to-vial concentrator tubes
- FMS, Inc. 200 mL concentrator tubes (1 mL termination)
- Thermo Scientific Trace Ultra GC with DSQ MS

Consumables

- FMS, Inc. 6 gram neutral silica columns
- Fisher Pesticide Optima* n-Hexane
- Fisher Pesticide Optima* Methylene Chloride
- Agilent Hydromatrix©
- NIST 1944 RM; NJ River Sediment
- Restek PAH Mixture (Cat# 31841)
- Restek Surrogate Mixture (Cat# 31062)
- Restek SV Internal Standard Mixture (Cat# 31006)

Procedure:

Sample Prep

Samples are weighed out in glass beakers.

For IPR and MDL samples, 20 grams of baked Ottawa sand was used.

Sample portions are spiked with surrogate solutions and/or PAH spiking solution.

Samples are generously mixed with Hydromatrix.

Dried samples are transferred to 40 mL PLE extractions cells.

Pressurized Liquid Extraction System

1. Cells filled with hexane: DCM (50:50)
2. Cells pressurized to 1500 PSI
3. Cells heated to 120 ºC for 20 minutes
4. Cells cooled to ambient temperature
5. Cells flushed with 20 mL solvent
6. Cells purged with N_2 and extract discharged to SuperVap Concentrator

SuperVap Concentration System

Preheat temp: 20 minutes at 60 ℃

Evaporation mode w/Sensor temp: 60 ℃

Nitrogen Pressure: 10 PSI

PowerPrep System

1. Condition column(s) w/10 mL DCM
2. Exchange column(s) to Hexane
3. Load sample extract(s)
4. Flush column(s) w/10 mL hexane
5. Elute column(s) w/35 mL DCM
6. Extract eluted to 50 mL SuperVap concentrator tubes with Direct-to-GC vial connection.

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Results

Table 1. Results of four replicate IPR study (spiked at 500 µg/kg)

Compound	Mean Rec.	STD DEV
Naphthalene	85.1%	2.1%
2-Methylnaphthalene	91.5%	2.0%
1-Methylnaphthalene	88.9%	2.1%
Acenaphthylene	101.5%	1.9%
Acenaphthene	96.5%	2.5%
Fluorene	96.9%	3.3%
Phenanthrene	89.1%	4.6%
Anthracene	116.9%	4.5%
Fluoranthene	102.6%	5.9%
Pyrene	101.1%	5.6%
Benzo[a]anthracene	97.4%	4.6%
Chrysene	104.7%	5.1%
Benzo[b]fluoranthene	90.0%	7.1%
Benzo[k]fluoranthene	95.2%	3.7%
Benzo[a]pyrene	89.5%	3.7%
Indeno[1,2,3-cd]pyrene	82.0%	4.7%
Dibenzo[a,h]anthracene	78.7%	4.5%
Benzo[g,h,i]perylene	83.3%	4.4%
Nitrobenzene-D5 (Surr)	93.6%	6.1%
2-Fluorobiphenyl (Surr)	80.2%	3.0%
p-Terphenyl-d14 (surr)	81.7%	5.2%

Table 2. Results of NIST 1944 analysis (reported in mg/kg)

Compound	Cert. Value	Calc. Value
Naphthalene	1.28	.986
2-Methylnaphthalene	.74	.589
1-Methylnaphthalene	.47	.356
Acenaphthylene	NA	.631
Acenaphthene	.39	.363
Fluorene	.48	.371
Phenanthrene	5.27	4.06
Anthracene	1.13	1.51
Fluoranthene	8.92	7.55
Pyrene	9.70	7.58
Benzo[a]anthracene	4.72	3.44
Chrysene	4.86	4.01
Benzo[b]fluoranthene	3.87	3.41
Benzo[k]fluoranthene	2.30	1.83
Benzo[a]pyrene	4.30	3.12
Indeno[1,2,3-cd]pyrene	2.78	2.26
Dibenzo[a,h]anthracene	.42	.445
Benzo[g,h,i]perylene	2.84	2.31

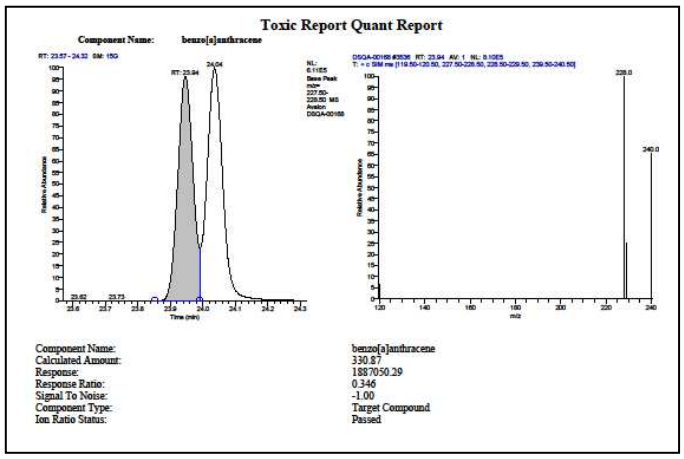
Table 3. Results of 7 replicate MDL study (spiked at 10 µg/kg)

Compound	MDL µg/kg	STD DEV
Naphthalene	2.57	.815
2-Methylnaphthalene	2.82	.905
1-Methylnaphthalene	2.83	.900
Acenaphthylene	2.93	.930
Acenaphthene	3.40	1.08
Fluorene	1.19	.380
Phenanthrene	3.38	1.08
Anthracene	2.83	.900
Fluoranthene	2.68	.850
Pyrene	2.22	.705
Benzo[a]anthracene	3.96	1.26
Chrysene	4.89	1.56
Benzo[b]fluoranthene	1.97	.625
Benzo[k]fluoranthene	3.22	1.03
Benzo[a]pyrene	1.78	.565
Indeno[1,2,3-cd]pyrene	1.68	.535
Dibenzo[a,h]anthracene	3.45	1.10
Benzo[g,h,i]perylene	4.63	1.47

Conclusions

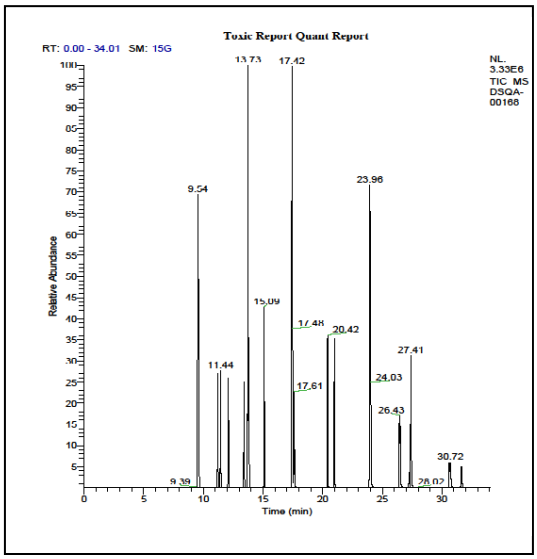
Following the extraction and cleanup with silica gel, the extracts were analyzed on a Thermo Scientific Trace GC with DSQ Mass Spectrometer. All analysis was performed in the selective ionization mode (SIM), with one quantitation ion and one confirmation ion monitored.

Figure 1. Ion spectra for benzo(a)-anthracene from analysis of NIST 1944, showing resolution from chrysene.



Analysis of the extracts showed excellent extraction efficiencies for all compounds analyzed, with minimal deviation between runs. The high level of efficiency enabled the establishment of an MDL below the target of 5 µg/kg for all analytes using the designated sample size. The calculated concentrations for the NIST 1944 sample were between 70-130% for all analytes, thus validating the PLE system extraction for soil and sediment sample. Due to the wide array of other organic contaminants present in the NIST reference sample, the efficiency of the 6 gram silica column was further validated by the clear resolution of each target analyte.

Figure 2. Total ion chromatogram from NIST 1944 analysis.



Using the FMS Pressurized Liquid Extraction system in conjunction with the PowerPrep Sample Cleanup system demonstrates an efficient and robust sample prep method that delivers both high quality results and increased sample throughput. By combining extraction, evaporation and cleanup with direct-to-GC-vial concentration, this automated sample-to-vial process frees laboratory staff to perform other tasks which increases the lab's throughput and quality and consistency of results.

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Application Note

Fast, Automated EPH Fractionation and Cleanup

Introduction

Soil contamination from gasoline, diesel fuel, heating oil, kerosene, or jet fuel leaks or spills is a common occurrence and a global environmental concern. Tanker transfer spills, truck transport spills and leakage from underground storage tanks continue to be sources of petroleum contamination. Recent improvements in transfer techniques, tank designs and materials have significantly reduced the danger of spills and leakage, but problems with installation or lack of operational training and maintenance procedures continue to cause environmental contamination.

In the United States, environmental testing labs identify fuel using the EPA total petroleum hydrocarbon (TPH) method 8015B. The semi-volatile fraction is identified by the distribution pattern displayed when analyzed via GC–FID. Petroleum products are composed of over 250 compounds, making the analysis of all of them difficult. Some states, Massachusetts and Texas among others, have created separate methods for extractable petroleum hydrocarbons (EPH) and volatile petroleum hydrocarbons (VPH). These EPH methods take a more toxicological approach and evaluate the composition of aliphatic and aromatic compounds in an extracted sample. These compounds and compound classes have very different exposure limits. The extracts are fractionated using silica gel and the aliphatic and aromatic groups analyzed separately using GC–FID and give a more accurate assessment of the risk to health. Manual fractionation is very labor intensive and time consuming. The PowerPrep EPH Cleanup and Fractionation System automates EPH fractionation, eliminates errors associated with manual techniques and reduces both glassware and solvent consumption.

Instrumentation

- FMS, Inc. PowerPrep EPH Cleanup and Fractionation System
- FMS, Inc. 6 gm Silica Column
- FMS, Inc. SuperVap Concentrator
- Thermo Fisher Scientific Polaris Q GC/MS

Method summary

PowerPrep PLE system

1. Pre-condition silica column with 30 mL methylene chloride.
2. Rinse column with 30 mL hexane.
3. Dilute sample extract to 9 mL hexane.
4. Spike fraction surrogate compounds (1 mL) into sample extract.
5. Load sample extract onto silica column
6. Elute column with 25 mL hexane, collecting aliphatic fraction.
7. Purge aliphatic fraction line with 5 mL hexane.
8. Elute column with 35 mL methylene chloride, collecting aromatic fraction.
9. Purge aromatic fraction line with 5 mL methylene chloride.
10. Transfer a portion of the extract to a GC vial with insert.
11. Analyze by GC/ECD.

PowerPrep EPH Cleanup and Fractionation System

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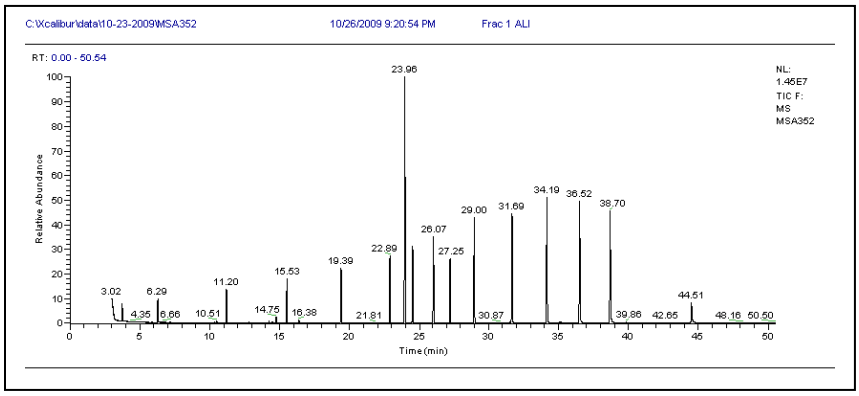
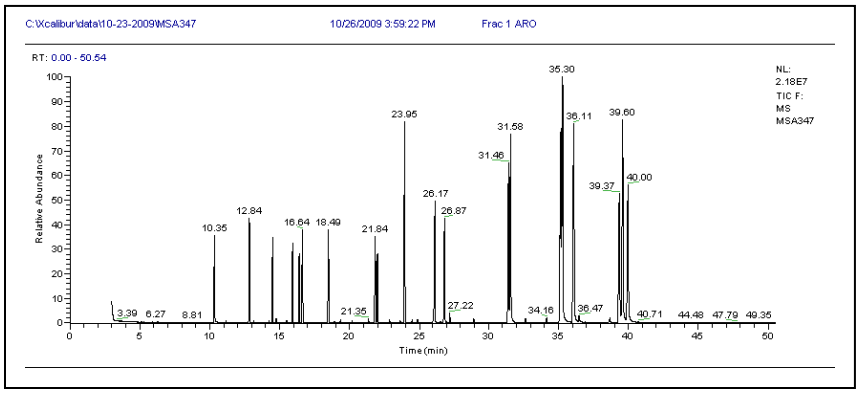
Conclusions

The FMS PowerPrep EPH system in combination with the FMS 6 gm silica gel column and FMS SuperVap concentrator is shown to automatically separate Aliphatic (Alkanes) Hydrocarbon from PAHs (Aromatic) Hydrocarbons at a high rate of speed producing excellent recoveries and reproducibilty. The combination of the FMS PowerPrep EPH system and the FMS Teflon column demonstrates consistent, reproducible, reliable high throughput automated sample fractionation of PAHs. The PowerPrep EPH system can fractionate up to 30 samples per hour.



Aliphatic Fraction 1



Aromatic Fraction 2

Results

Aliphatics

Compound	Conc.	Recovery	Limit
Nonane (C9)	12.547	62.74	30%-130%
Decane (C10)	13.831	69.16	40%-140%
Dodecane (C12)	14.028	70.14	40%-140%
Tetradecane (C14)	13.994	69.97	40%-140%
Hexadecane (C16)	14.944	74.72	40%-140%
Octadecane (C18)	16.131	80.66	40%-140%
Nonadecane (C19)	16.44	82.20	40%-140%
Eicosane (C20)	16.824	84.12	40%-140%
Docosane (C22)	17.374	86.87	40%-140%
Tetracosane (C24)	18.598	92.99	40%-140%
Hexacosane (C26)	19.438	97.19	40%-140%
Octacosane (C28)	19.344	96.72	40%-140%
Triacontane (C30)	18.872	94.36	40%-140%
Hexatriacontane (C36)	16.538	82.69	40%-140%

Aromatics

Compound	Conc.	Recovery	Limit
Naphthalene	13.633	68.17	40%-140%
2-Methylnaphthalene	14.138	70.69	40%-140%
Acenaphthylene	11.62	58.10	40%-140%
Acenaphthene	14.4	72.00	40%-140%
Fluorene	14.951	74.76	40%-140%
Phenanthrene	15.709	78.55	40%-140%
Anthracene	12.216	61.08	40%-140%
Fluoranthene	15.795	78.98	40%-140%
Pyrene	15.809	79.05	40%-140%
Benzo[a]anthracene	18.109	90.55	40%-140%
Chrysene	19.833	99.17	40%-140%
Benzo[b]fluoranthene	20.33	101.65	40%-140%
Benzo[k]fluoranthene	16.151	80.76	40%-140%
Benzo[a]pyrene	14.46	72.30	40%-140%
Indeno[1,2,3-cd]pyrene	18.122	90.61	40%-140%
Dibenzo[a,h]anthracene	19.446	97.23	40%-140%
Benzo[g,h,i]perylene	17.918	89.59	40%-140%

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Application Note

One Step Extraction, Cleanup and Concentration for PBDEs in Soil Samples

Introduction

Polybrominated diphenyl ethers are organo-bromine compounds that are used as flame retardants. Like other brominated flame retardants, PBDEs have been used in a wide array of products, including building materials, electronics, furnishings, motor vehicles, airplanes, plastics, polyurethane foams, and textiles. The hazards of these chemicals have attracted increasing scrutiny, for they have been shown to reduce fertility in humans at household levels and have been linked to liver toxicity, thyroid toxicity, and neurodevelop-mental toxicity in laboratory studies on rats. Studies comparing humans who have higher levels of PBDEs present in their umbilical cord blood at birth have been shown to score lower in tests of mental and physical development. Due to these concerns the European Union has banned the use of PBDEs as have several states in the U.S., including California, Washington and Maine.

Instrumentation and Consumables

- FMS, Inc. PowerPrep™ PLE (Pressurized Liquid Extraction) System
- FMS, Inc. PowerPrep™ sample clean-up system
- FMS, Inc. Classical ABN Column
- FMS, Inc Alumina Column
- FMS, Inc Carbon/Celite column
- FMS, Inc. SuperVap™ Concentrator system
- Thermo Fisher Scientific Polaris Q GCMS

Method Summary

PowerPrep PLE system

1. Extraction solvent: Hexane/Methylene Chloride (50/50)
2. Extraction temperature: 120 ºC
3. Extraction pressure: 1500 PSI
4. Extraction time: 15 minutes

SuperVap Concentrator

1. Pre-heat temp: 55 ºC
2. Pre-heat time: 15 minutes
3. Heat in Sensor mode: 65 ºC
4. Nitrogen Pressure: 15 PSI

Sample preparation and extraction

10 grams of the sample are weighed out in a 100 mL beaker; the process is repeated for eight replicates.

1. Samples are dried with Varian Hydro Matrix®
2. The dried sample is transferred to a FMS extraction cell
3. The samples are spiked with 1 mL (acetone) of 100 ng/mL labeled 1614 spiking solution
4. The cell volume is filled with Ottawa Sand®, sealed and loaded on the FMS PLE system for extraction
5. The sample is extracted and auto-matically transferred to the SuperVap Concentrator.

The sample is concentrated to .5 mL final volume.

The FMS TotalPrep™ system for automated extraction, cleanup and concentration.

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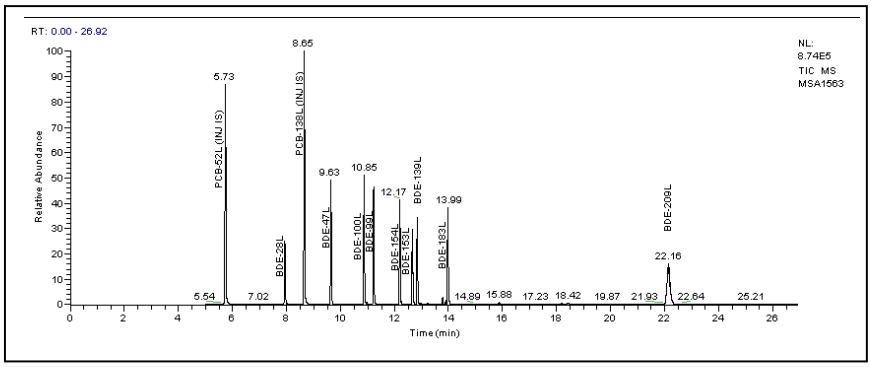
Sample cleanup

1. The extract spiked with Cambridge Isotope 1614 cleanup internal standard (BDE-139L)
2. The columns are preconditioned with solvent.
3. The sample is loaded onto a FMS Classical ABN silica column using the PowerPrep™ solvent pump.
4. The silica column is eluted with 90 mL of hexane and the elution is automatically transferred onto the alumina column.
5. The alumina column is eluted with 180 mL of hexane/methylene chloride (60 mL 2%, 120 mL 50%), the sample is automatically transferred to the carbon column and collected in SuperVap™ Concentrator and brought to dryness.
6. 2 µL of dodecane is added to the extract that has been evaporated to dryness. Sample reconstituted to 10 µL nonane, spiked with 1 µL Cambridge Isotope 1614 Injection Internal Standard and transferred to GC/MS for analysis.

Figure 1: Chromatogram of a soil sample and labelled PBDE recoveries.



Results

Table 1: Mean spike recoveries for labeled compounds over eight replicates.

	Amount Spiked	Mean % Recovery
BDE-28L	100 pg	78.3%
BDE-47L	100 pg	95.5%
BDE-100L	100 pg	107.0%
BDE-99L	100 pg	108.0%
BDE-154L	100 pg	112.6%
BDE-153L	100 pg	111.5%
BDE-183L	100 pg	110.1%
BDE-209L	1000 pg	68.4%

Conclusions

Analysis of eight replicates of soil obtained from the banks of the Charles River in Boston, MA U.S.A. indicates consistent, reproducible recoveries of labelled PBDEs when extracted on the FMS PLE system. Recoveries fell well with in the EPA 1614 recovery window (25-150%), with standard deviations ranging between 2-5% (14% for BDE-209). Native PBDE concentrations detected in samples were at average concentrations of approximately 15.0 ng/kg for BDE 47 and 22.5 ng/kg BDE-99 (dry weight). Other congeners were non-detect at an R.L. of 2 ng/kg (20ng/kg fro BDE-209). Evaluation of the multi column clean-up procedure on the FMS PowerPrep closed system demonstrated efficient sample clean-up allowing low level analysis with excellent reproducibility. Analysis of extracted and cleaned up method blanks verifies FMS columns were free of native PBDEs at instrument detection levels.

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Application Note

Total Extractable Fat Using Pressurized Liquid Extraction (PLE)

Introduction

Regulating fat content is essential to maintaining the quality of food and feed products. The food industry extracts total fat from food samples to analyze for Priority Organic Pollutants (POPs). For several compounds of concern like PCB and PBDE congeners, results are often expressed in amount for gram of lipid.

Traditionally performed by Soxhlet extraction, fat determination is a time consuming process requiring large volumes of solvent and lengthy extraction times. By using pressurized liquid extraction, the entire extraction process can be performed in under an hour resulting in higher throughput.

The following application details the use of the FMS, Inc PLE sytem for the extraction of total fat from a variety of sample matrices.

Instrumentation and Consumables

- FMS, Inc. PLE system
- FMS, Inc. SuperVap™ Concentrator
- FMS, Inc. 200 mL direct-to-vial concentrator tubes
- Mettler Toledo analytical balance
- FMS, Inc PLE extraction cells (10, 20 40 mL)

*Alloy 20 cells required for Hydrolysis

Consumables

- Fisher Pesticide Optima* n-Hexane
- Agilent Hydromatrix
- FMS PLE re-usable end caps

Procedure

Weigh extract collection vials

Sample aliquots are weighed

Samples dried with Hydromatrix

Dried samples transferred are into PLE extraction cells

PLE System

1. Cells filled with n-Hexane
2. Cells pressurized to 1500 PSI
3. Cells heated to 140 ºC for 20 minutes
4. Cells cooled and flushed with n-Hexane (equal to cell volume)
5. Cells purged with N_2 into SuperVap Concentrator

SuperVap Concentrator

1. Preheat temp: 20 minutes at 60 °C
2. Evaporation mode: 60 °C
3. Nitrogen Pressure: 10 PSI
4. Evaporate extracts to total dryness

*Evaporator tubes rinsed with N-Hexane to ensure no collected material sticks to evaporator tube walls.

Post evaporation, weigh collection vials and subtract the pre-weight values.

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Results

Table1: Fat calculations for PLE extracts

Sample	Mean Percent Lipid	RPD	Expected Lipid %	Percent Difference
Animal Feed 1	6.22%	1.0%	6.27%	-0.7%
Animal Feed 2	3.94%	1.3%	4.01%	-1.9%
Peanut Butter Almonds	50.04%	4.8%	50%	0.1%
Non-Dairy Creamer	2.31%	2.7%	2%	14.2%
Parmasean Cheese	24.37%	1.3%	25%	-2.6%
Rolled Oats	5.69%	2.4%	6.25%	-9.4%
Lard	97.7%	0.5%	100%	-2.1%
	Percent Lipid		**Expected Lipid %**	
NIST 1946 Fish Tissue	10.05%		10.17%	-1.2%

Conclusions

Recovered fat calculations yielded values that are close to expected values. RPDs between duplicates demonstrate good reproducibility between the performed extractions. The addition of the NIST 1946 reference material provided a certified fat value for QC of the extraction batch.

When comparing the PLE extraction (< 1 hour total extraction time) to a Soxhlet extraction lasting 16-24 hours, the value of adopting PLE extractions becomes quickly evident. With the demonstrated diversity of matrices capable of being extracted, the PLE is a clearly superior option for high throughput facilities performing fat extractions.

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Application Note

A Quick Screen Method for the Extraction, Cleanup and Concentration of Toxaphene in Fish

Introduction

Prior to its being banned in the U. S. in 1986, toxaphene was widely used throughout the southeastern United States as a pesticide on cotton and soybean crops. Classified as a group 2B carcinogen, it affects the lungs, nervous system and kidneys. Sufficient exposure can be fatal. Despite these dangers, toxaphene is still used today for treating crops outside the United States.

Toxaphene exists as a mixture of roughly 200 organic compounds formed by the chlorination of camphene, which results in a chlorine content of approximately 70%. Because of its persistent nature and the ease with which it enters biological organisms, ingestion of toxaphene through human food sources is a continuing concern since its ban.

Due to a complex and lengthy sample clean-up process, the extraction and analysis of toxaphene can be a daunting process for laboratories. The purpose of this study was to develop a one-step toxaphene extraction and clean-up process from fish tissue that delivers reproducible results.

Instrumentation

- FMS, Inc. PLE™ (Pressurized Liquid Extractor) system
- FMS, Inc. PowerVap™ Concentrator system
- FMS, Direct-to-Vial concentrator tubes
- 2.5 and 5 gram InCell acid (30%) silica gel PLE end caps
- Agilent 7890A GC with µECD

PLE Program

1. Cells are filled with 2:1 Cyclohexane/Pentane
2. Cells are pressurized to 1500 PSI
3. Cells are heated to 120 ºC and held for 15 minutes
4. Cells are cooled and depressurized.
5. Cells are flushed with 80% of cell volume.
6. The remaining solvent purged out of cells with N_2

PowerVap Concentrator

1. Pre-heat temp: 40 ºC
2. Pre-heat time: 15 minutes
3. Heat in Sensor mode: 40 ºC
4. Nitrogen Pressure: 10 PSI

Procedure

Sample Preparation and Extraction

Salmon tissue was finely processed until no visible clumps remained.

Salmon tissue was weighed out in duplicate portions of both 2.5 gram samples and 5 gram samples.

The sample portions were then mixed with Varian Hydro Matrix.

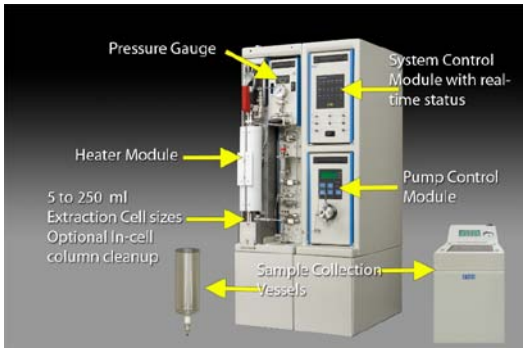
2.5 gram samples were transferred to 40 mL PLE extraction cells equipped with 2.5 gram InCell acidified silica gel end caps. 5 gram samples with transferred to 100 mL Cells equipped with 6 gram acidified silica gel end caps.

The samples are spiked with .1 µg/mL surrogate spiking solution and 4 µg/mL toxaphene spiking solution. Two sample portions were left un-spiked with toxaphene to establish background for sample matrix.

The Cells are capped and loaded onto PLE system. Extraction program initiated.

Extracts automatically transferred to Power Vap system and concentration begun.

Extract removed from PowerVap system (1mL) and transferred to Agilent GC for analysis.



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Results

Table 1: Results of 2.5 gram and 5 gram samples

	2.5 gram	Sample
Compound	Avg. Conc.	% rec.
TCMX	27.8 µg/kg	70%
Decachlorobiphenyl	34.8 µg/kg	87%
Toxaphenne	121.2 µg/kg	76%

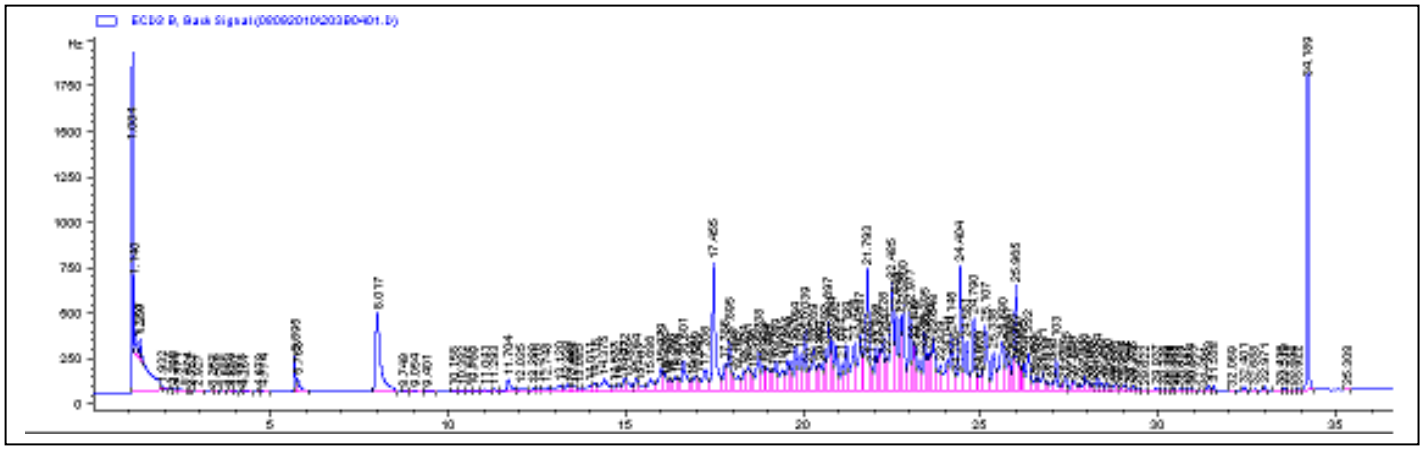
	5 gram	Sample
Compound	Avg. Conc.	% rec.
TCMX	12.8 µg/kg	72%
Decachlorobiphenyl	18 µg/kg	74.5%
Toxaphenne	57.62 µg/kg	98.5%

Conclusions

Analysis of the sample data indicates good recoveries for technical toxaphene using the InCell cleanup for fish tissue samples of both 2.5 and 5 grams. The relative low sample RPDs for the toxaphene and decachlorobiphenyl also indicate excellent reproducibility. A higher RPD was observed for the TCMX recovery in the 5 gram sample due to the higher volatility of the compound and some concluded loss during the longer evaporation time.

The use of the PLE system with the InCell cleanup and the PowerVap™ Direct-to-Vial concentration system consistently delivered reproducible, one-step extraction, clean-up and concentration for what is traditionally a three-step process. This new method substantially reduced the time of the traditional method. Samples were ready for same-day analysis and used substantially less lab reagents, which saves money.

Figure 1. Shows the chromatogram of a 2.5 gram sample runs on µECD detector.



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Application Note

One Step Extraction, Cleanup and Concentration of Chlorinated Pesticides in Raw Coffee Beans

Introduction

Organochlorine pesticides are man-made chemicals once used widely in agriculture and for mosquito control. Beginning in the 1960's, public awareness of their toxicity began to increase and by the mid 1970's many organochlorine pesticides were banned in the United States. Though some are still registered for use in the United States, today they are used mainly in developing nations.

Classified as persistent organic pollutants, organochlorine pesticides exhibit a wide range of toxic effects including reproductive and neurotoxicity as well as liver and kidney failure. In animals, they have been shown to cause cancer in these organs.

Due to their persistent nature and toxicity, monitoring food sources for the presence of organochlorine pesticides is a growing concern for both food manufacturers and consumers. Foods are typically difficult matrices to extract and analyze. The following procedure describes a rapid and efficient method for the extraction, extract cleanup and concentration of organochlorine pesticides from raw coffee beans.

Instrumentation and Consumables

- FMS, Inc. Pressurized Liquid Extractor (PLE)
- FMS, Inc. SuperVap™ Direct-to-vial Concentration system
- FMS, Inc. direct-to-vial concentrator tubes
- 6 gram InCell Florisil® PLE end cap columns
- Agilent 7890A GC with µECD

PLE Program

1. The Cells are filled with hexane
2. Cells pressurized to 1500 PSI
3. Cells heated to 120 ºC
4. 120 ºC held for 10 minutes
5. Cells cooled and depressurized.
6. The Cells are flushed with 80% of the cell volume hexane.
7. The remaining solvent is purged out of the cells using N_2 directly to concentration tubes

SuperVap Concentrator

1. Pre-heat temp: 40 ºC
2. Pre-heat time: 30 minutes
3. Heat in Sensor mode: 40 ºC
4. Nitrogen Pressure: 15 PSI
5. Sensor End Point Detection

Procedure: Sample Prep and Extraction

The green coffee beans were finely processed until no visible clumps remained.

The coffee bean sample was weighed out in seven replicates of five grams per sample.

The sample portions were then mixed with Varian Hydro Matrix.

10 gram samples were transferred to 100 mL cells equipped with 6 gram Florisil® InCell cleanup end cap columns.

Five samples were spiked, each with 0.1 µg/mL of surrogate spiking solution and pesticide spike. Two sample portions were only spiked with a surrogate solution to establish any background levels in the sample matrix.

The Cells were capped and loaded onto PLE. The extraction is initiated.

The extracts automatically transfer to the SuperVap concentrator and concentration begins.

The Extract is removed from the SuperVap Concentrator with Direct-to-Vial tubes and transferred to Agilent GC for analysis.

Figure 1: FMS Pressurized Liquid Extractor and SuperVap systems.

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Results

Table 1: Mean recoveries and standard deviations from the five extracts after analysis.

Compound	AVG Rec	STD
TCMX	104.8%	4.6
Alpha-BHC	92.0%	1.5
Beta-BHC	89.8%	1.1
Gamma-BHC	89.2%	1.6
Delta-BHC	109.5%	2.7
Heptachlor	87.0%	4.8
Aldrin	95.8%	15.6
Heptachlor Epoxide	81.8%	1.3
Gamma-Chlordane	76.7%	1.5
Endosulfan I	79.9%	1.2
Alpha-Chlordane	78.7%	1.6
Dieldrin	75.4%	1
4,4"-DDE	74.7%	1.5
Endrin	135.0%	3.2
Endosulfan II	81.2%	1.5
4,4'-DDD	92.1%	9.4
Endrin Aldehyde	77.1%	1.8
Endosulfan Sulfate	98.8%	2.5
4,4'-DDT	83.4%	23.9
Endrin Ketone	80.8%	4.6
Methoxychlor	91.2%	17.9
Deca-PCB	93.6%	10.4

Conclusions

Analysis of the PLE data on the GC demonstrates excellent reproducibility, consistent recoveries and from a traditionally difficult sample matrix. Adding to the efficiency of the entire sample prep process was the use of the InCell extraction cleanup end cap column. The InCell column eliminates the need to perform post cleanup and a second concentration step. Eliminating the second concentration step dramatically reduces the risk of lower analyte recovery of volatile compounds. With the addition of the Direct-to-Vial concentration tube, no sample transfer is necessary. This allowed the sample to be extracted and cleaned up by the PLE system and then sent automatically to the SuperVap™ Concentrator system where the final extract is concentrated directly to a vial for final GC analysis.

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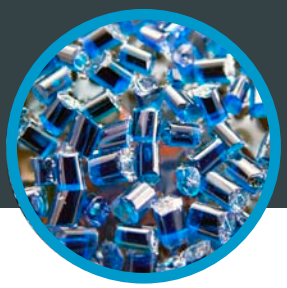
Application Note

Extraction of Antioxidants from High Density Polyethylene (HDPE) By Pressurized Liquid Extraction (PLE)



Introduction

Plastics and other polymers are used in a wide spectrum of applications from industrial usage to packaging, including the packaging of food and pharmaceutical products. To enhance functionality, chemical additives are often added to polymers. These additives enable polymers to be more durable, pliable and make them less subject to degradation. To ensure that the specified amounts of an additive or combination of additives are incorporated into a polymer. After the extrusion process, a rapid and accurate analytical method is required to measure additives and other process by products.

This application note demonstrates the use of pressurized liquid extraction using the FMS PLE system to extract polymer additives for quatitative analysis by HPLC. Four specific additives of known concentrations were extracted from high density polyethylene (HDPE) and the recoveries measured.

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Instrumentation and consumables

- FMS, Inc. Pressurized Liquid extraction System
- Thermo Certified GC/MS auto sampler vials
- FMS 5 mL Stainless Steel PLE Cartridge
- FMS Universal Stainless Steel PLE End Caps

Reagents

- Fisher Optima* 2-Propanol
- Fisher Optima* Cyclohexane

Sample preparation and PLE procedure

Sample Prep

1. A sample of high density polyethylene was milled to a fine particle size to increase surface area.
2. Duplicate samples of 2 grams each were weighed out and transferred to FMS 5 mL extraction cells and sealed with stainless steel re-usable end caps.
3. Samples were loaded on FMS PLE system.

Pressurized Liquid Extraction

Solvents Mix: 2-Propanol/Cyclohexane (95:5)

Solvent pump rate: 35 mL/min

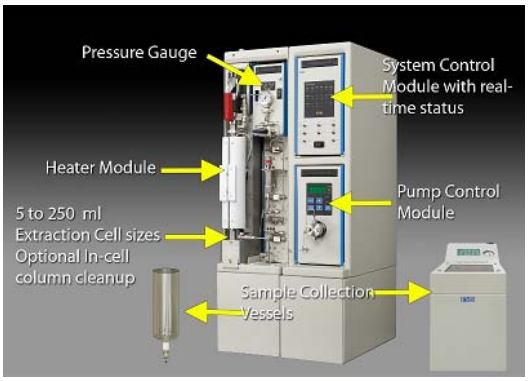
Pre-heat temperature: 100 °C

Extraction temperature: 120 °C

Extraction time: 20 minutes

Cycles: 3

Total time: 1.5 hours



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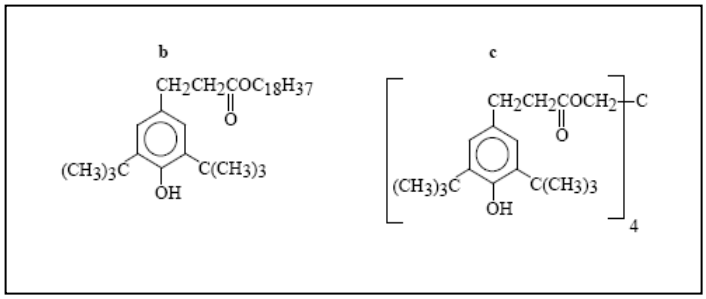
Results

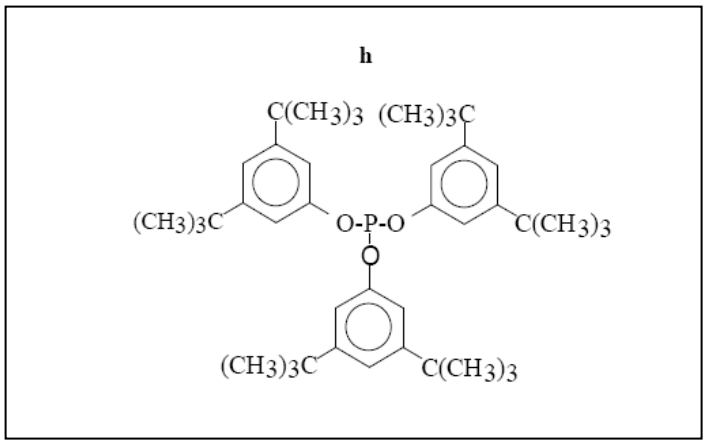
Table 1: Dual extractions of HDPE

	Cell #1	Cell #2	RSD
Erucamide®	70.80%	71.60%	0.712
Irganox® 1010	81.30%	81.00%	0.8115
Irganox® 1076	81.20%	80%	0.806
Irgafos® 168	116.60%	115.60%	1.161

Molecular formulas of two hindered phenols extracted. b) Irganox® 1076 & c) Irganox® 1010



Molecular formula for aromatic phosphite extracted, h) Irgafos® 168



Conclusions

Analysis of the four antioxidant additives in the HDPE samples demonstrates the FMS PLE system's ability to generate excellent recoveries for all four compounds (70-130%). The low relative RPDs also demonstrate the ability to generate consistent, reproducible extraction efficiencies between runs.

The advantages of Pressurized Liquid Extraction over traditional Soxhlet extractions are faster analyses (1.5 hours versus 15 hours) and reduced solvent volume (up to 200 mL). This Pressurized Liquid Extraction technique using the FMS PLE extraction system is an ideal method for extracting antioxidant additives from HDPE.

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Application Note

Dioxins, PCBs and PBDEs in Human Serum Using Automated Pressurized Liquid Extraction, Multi-Column Cleanup, and Concentration.

Introduction

Because of its complexity, human serum is one of the most challenging sample matrices encountered. Organic contaminants often exist at low lipid concentrations (~600 mg/dl) that require extremely low detection levels and ultra clean blanks. The manual sample preparation process for human serum consists of multiple, time-consuming steps that are messy, difficult to reproduce and yield inaccurate results.

By combining the analysis of multiple analytes into a single extraction and concentration method using the Pressurized Liquid Extraction (PLE) system, the PowerPrep system to fractionate analyte classes, and the SuperVap Concentrator, the sample preparation process can be streamlined into a rapid, reproducible method.

Instrumentation

- FMS, Inc. PLE System
- FMS, Inc. PowerPrep System
- FMS, Inc. SuperVap Concentrator
- FMS, Inc. 200 mL direct-to-vial concentrator tubes
- FMS, Inc. 200 mL concentrator tubes (1 mL termination)
- Thermo Scientific Trace Ultra GC with Quantum TSQ
- Thermo Scientific Trace Ultra GC with DFS HRMS

Consumables

- FMS, Inc. PBDE free ABN columns
- FMS, Inc. PBDE free Alumina columns
- FMS, Inc. PBDE free Carbon columns
- Fisher Optima* Toluene
- Fisher Optima* ETAC
- Fisher Optima* n-Hexane
- EMD Omni* Benzene
- Fisher Optima* Methylene Chloride
- Agilent Hydromatrix©
- Fisher Formic Acid
- NIST 1958 RM; Fortified Human Serum

Consumables *continued*

- Cambridge Isotopes EDF-4143, Dioxin, Furan, & PCB in Tissue Calibration Standards
- Cambridge Isotopes EDF-4144B, Spiking solution for Dioxin, Furan, and PCB in Tissue
- Cambridge Isotopes EDF-4145, Recovery Standard for Dioxin, Furan, and PCB in Tissue
- Cambridge Isotopes EC-5366, PCB Calibration Standards
- Cambridge Isotopes EC-5367, PCB Spiking Solution
- Cambridge Isotopes EO-5319, BFR calibration Standards
- Cambridge Isotopes EO-5158, BFR Spiking Solution
- Cambridge Isotopes EO-5169, Recovery Standard

Procedure

1. PLE cells are filled with Hydromatrix (baked at 500 ºC)
2. Sample amounts are measured (up to 20 mLs)
3. Serum samples are spiked with appropriate labeled surrogates for analytes of interest
4. Samples are transferred to extraction cells via a large volume pipettor.
5. Formic acid is added to samples on a 1:5 ratio
6. Cells are capped and allowed to equilibrate for 30 minutes before loading onto the PLE system

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Procedure

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Pressurized Liquid Extraction system

1. Cells filled with hexane: DCM (50:50)
2. Cells pressurized to 1500 PSI
3. Cells heated to 120 ºC (2 cycles, 20 and 10 minutes)
4. Cells cooled to ambient temperature
5. Cells flushed with 20 mL solvent
6. Cells purged with N_2 and extract discharged to SuperVap Concentrator.

SuperVap concentration system

1. Preheat temp: 20 minutes at 60 °C
2. Evap mode w/Sensor temp: 60 °C
3. Nitrogen Pressure: 10 PSI

PowerPrep system

1. Columns conditioned
2. Load sample extract(s)
3. Columns eluted with Hexane and collected (F1)
4. Alumina and Carbon columns eluted with DCM: Hexane and collected (F2)
5. Carbon column rinsed with ETAC:Ben (F3)
6. Carbon column back eluted with Toluene (F4)

Fractions F1, F2, F3 collected in a single vessel for the analysis of PCBs and PBDEs Fraction F4 contains PCDDs, PCDFs and Co-Planar PCBs.

Results

Table 1. Mean recoveries and deviations for labeled compounds over 20 samples.

Compound	Mean Rec.	STD DEV
2,3,7,8-TCDD	67.4%	8.5%
1,2,3,7,8-PeCDD	78.1%	9.8%
1,2,3,4,7,8-HxCDD	81.8%	10.0%
1,2,3,6,7,8-HxCDD	67.8%	8.0%
1,2,3,7,8,9-HxCDD	81.1%	9.9%
1,2,3,4,6,7,8-HpCDD	60.9%	7.0%
OCDD	60.6%	6.3%
2,3,7,8-TCDF	71.7%	8.8%
1,2,3,7,8-PeCDF	76.6%	9.2%
2,3,4,7,8-PeCDF	78.2%	10.0%
1,2,3,4,7,8-HxCDF	75.5%	9.0%
1,2,3,6,7,8-HxCDF	75.8%	8.9%
2,3,4,6,7,8-HxCDF	75.1%	9.1%
1,2,3,7,8,9-HxCDF	96.9%	13.0%
1,2,3,4,6,7,8-HpCDF	69.6%	7.1%
1,2,3,4,7,8,9-HpCDF	NA	NA
OCDF	68.7%	10.0%
PBDE-28	65.1%	19.1%
PBDE-47	73.1%	22.3%
PBDE-100	74.6%	25.1%
PBDE-99	79.5%	25.5%
PBDE-154	78.5%	25.6%
PBDE-153	85.8%	26.9%
PBDE-183	98.3%	29.9%
PBDE-209	95.5%	25.7%
PCB-28	81.6%	27.8%
PCB-52	80.0%	25.9%
PCB-101	81.0%	28.7%
PCB-105	75.0%	21.3%
PCB-114	78.0%	20.8%
PCB-118	69.5%	19.8%
PCB-123	74.9%	21.4%
PCB-128	75.2%	21.2%
PCB-138	75.1%	21.4%
PCB-153	77.5%	22.2%
PCB-156	71.0%	20.5%
PCB-157	67.7%	19.9%
PCB-167	65.0%	21.4%
PCB-170	86.4%	19.3%

Table 1 continued

Compound	Mean Rec.	STD DEV
PCB-178	63.7%	22.9%
PCB-180	65.2%	18.6%
PCB-189	71.7%	18.1%
PCB-194	86.4%	21.4%
PCB-206	82.9%	23.0%
PCB-209	100.8%	23.4%
PCB-77	73.4%	9.2%
PCB-81	67.9%	7.5%
PCB-126	78.9%	10.7%
PCB-169	101.3%	14.0%

Table 2. Results of NIST 1958 analysis.

Compound	Calc. Conc.	Cert Value
2,3,7,8-TCDD	80	97.3
1,2,3,7,8-PeCDD	90	114
1,2,3,4,7,8-HxCDD	10	98.5
1,2,3,6,7,8-HxCDD	270	363
1,2,3,7,8,9-HxCDD	90	103
1,2,3,4,6,7,8-HpCDD	410	595
OCDD	2540	2750
2,3,7,8-TCDF	100	107
1,2,3,7,8-PeCDF	80	107
2,3,4,7,8-PeCDF	180	221
1,2,3,4,7,8-HxCDF	90	102
1,2,3,6,7,8-HxCDF	90	110
2,3,4,6,7,8-HxCDF	670	958
1,2,3,7,8,9-HxCDF	90	99.6
1,2,3,4,6,7,8-HpCDF	ND	NA
1,2,3,4,7,8,9-HpCDF	120	86.2
OCDF	130	88.6
PBDE-28	425.74	470
PBDE-47	750.31	661
PBDE-100	417.14	482
PBDE-99	449.02	499
PBDE-154	359.62	450
PBDE-153	376.31	460
PBDE-183	365.71	461
PBDE-209	366.41	415

Table 2. continued

Compound	Calc. Conc.	Cert Value
PCB-105	437.04	415
PCB-114	41.13	47.4
PCB-118	412.13	418
PCB-123	41.02	54.4
PCB-156	381.57	424
PCB-157	343.81	426
PCB-167	341.44	409
PCB-170	339.70	429
PCB-180	358.75	470
PCB-189	380.17	409
PCB-77	NA	NA
PCB-81	NA	NA
PCB-126	7890	8050
PCB-169	8013	8400

Conclusions

The results of the 20 sample study indicate that Pressurized Liquid Extraction (PLE) combined with the PowerPrep multi-column fractionation and SuperVap Concentration systems generated efficient extractions. The relatively low deviation between recoveries demonstrates the robustness of the extraction process as well as the ability to deliver a high level of reproducibility across multiple samples. The calculated concentrations of the NIST 1958 extraction further demonstrate the efficiencies of the extraction and the ability to recover native compounds from the matrix. Also, the need to perform an additional cholesterol removal step, typically required with traditional SPE extractions of human serum, was eliminated.

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