

Streamline Your Pesticide Sample Preparation and Analysis in Drinking Water





Agenda

- FMS, Inc. Fluid Management Systems
- Sample Prep vs Analytical Runs in time
- Sample Analysis Workflow
- Sample Prep Workflow
- Total Solution Sample Analysis Strategy





FMS - Fluid Management Systems

- Founded in 1986
- Manufactures Total Solution Sample Preparation and Consumables for GC, GC/MS, LC and LC/MS



Made in the USA





Made in the USA





Made in the USA





Class 1000 Cleanroom for Consumables Manufacturing





Laboratory Workflow Breakdown







FMS Sample Preparation Workflow

Sample Preparation consist of three main instruments: 1- Extraction 2- Sample Clean-Up 3- Concentration = Total Prep





Sample Analysis Work Flow Hours vs Days

Automated Sample Prep Time 1 up to 8 samples = 2hrs





Solid Phase Extraction 30 Min



Sample Cleanup 30 Min



Concentration 30 Min



GC/MS 45Min

Manual Sample Prep Time = 2 to 4 days

Extraction	Concentration	Sample Cleanup	Concentration	GC/MS
24 hours	60 Min	24 to 48 hours	60 Min	45Min /sample



Solid Phase Extraction



EconoTrace®





TurboTrace® ABN





NanoTrace®

TurboTrace®





EconoTrace®



TurboTrace®





TurboTrace® PFC

NanoTrace®









The PLE[®] front end for GC/Ms and LC/MS







PLE





The PLE® Pressurized Liquid Extraction



PLE – Pressurized Liquid Extraction

- Modular and expandable from 1 to 8
- Process 1 to 8 samples in 30 min
- Extraction cell size 5 to 100 ml
- Real time plot of temperature and pressure







- Low Cost POPs analysis
- Runs 2 samples per Module
 In parallel
- Expandable up to 4 Modules
- Run up to 8 samples in Parallel
- Run up to 8 samples in 30 to 40 minutes
- Reduced Solvent
 Consumption





Direct to Vial Concentration

Direct to Vial Concentration

SuperVAP – Concentration System

- 6 Position 25ml Vessel
- 12 Position 50ml Vessel
- 12 Position 20,40,60 ml vial
- 24 Position 2 and 4 ml vial







Solid Phase Extraction



EconoTrace®





TurboTrace® ABN





NanoTrace®

TurboTrace®



Objective

- Develop as many SPE procedures for the testing lab using a single extraction platform.
- Minimize manual steps to lessen error and maximize limited man hours





Reasons for SPE

- Reduced solvent
- Reduced glassware
- Simplified procedures
- Automation versus manual protocols = Reproducibility





Determining Factors

- Ability to load samples by both positive pressure and vacuum.
- Ability to dry cartridges by both vacuum and positive gas pressure (N2 or CO2).
- Easily handle a wide variety of cartridge designs and sizes without cumbersome modifications.





Comparison of LLE/CLE vs. Automated SPE Methods

LLE/CLE

Open to laboratory background

Uses >360mls solvent

Shaking / Continuous process

Forms emulsions requiring centrifuging

Little Selectivity

Requires water removal

Automated SPE

Closed system

Uses <60mls solvent

Filtration process

No emulsions formed

Wide Selectivity (adsorbent)

In-line water removal

Fast and Unattended



Comparison of LLE/CLE vs. Automated SPE Methods

LLE/CLE

No Separation of waste

More volume to evaporate

Massive solvent emission

CLE high electrical costs

Requires lots of solvent for cleaning

Automated SPE

Separates Aqueous and Organic Waste

<60mls solvent to evaporate

6 times less solvent emission

Easily Capture Solvent

Lower solvent costs

Lower Disposal Costs



Reduced Solvent Usage







Methods

- EPA 507
- EPA 508.1
- EPA 513
- EPA 515.2
- EPA 521
- EPA 522

- EPA 525
- EPA 527
- EPA 529
- EPA 548.1
- EPA 550.1
- EPA 552.1





EconoTrace SPE





EconoTrace SPE





EconoTrace SPE









Modular and expandable from 1 to 6 Modules

High Throughput Runs Sample Extraction in Parallel

Uses Positive Pressure Pumping for Precise delivery of Elution and Wash Solvent

Uses Vacuum or Positive Pressure Pumping to Load Samples













508 data OCPs





5515.2 Chlorinated acids

76
72
99
78
106
122
102
90
92
101
96
112
99





521 Nitrosamines

Compound name	Average (%)
NDMA d6	120
NDMA	116
NMEA	125
NDEA	117
NPYR	110
NMOR	107
NDPA	104
NPIP	109
NDBA	103
NDFA	92





522 1,4-dioxane

1,4-dioxane-d8 recoveries

run # 1	87%
run # 2	84%
run # 3	81%













EPA 525.3





Fluid Management Systems 527 Pesticides and Flame Retardants

Compound name	Average (%)
Atrazine	102
BDE-100	84
BDE-153	93
BDE-47	76
BDE-99	78
Bifenthrin	91
Bromacil	113
Chlorpyrifos	91
Dimethoate	109
Esbiol	111
Malathion	102
Nitrophen	107
Oxychlordane	89
Prometryn	97
Propazine	88
Terbufos-sulfone	102
Thiobencarb	90
Vinclozolin	110



548 Endothall

Endothall	Concentration	% Recovery
Sample #1	48 ug/mL	96
Sample # 2	49 ug/mL	98
Sample # 3	48 ug/mL	96





549.2 Diquat - Paraquat

Compound	%Recovery	Stdev	
Diquat	94	3%	
Paraquat	83	4%	





550.1 PAHs

Compound	%Recovery	Stdev
Naphthalene	71	0.67
2-methylnaphthalene	79	0.70
Acenaphtylene	74	0.78
Acenaphthene	82	0.71
Fluorene	84	0.59
Phenanthrene	94	0.44
Anthracene	96	0.41
Fluoranthene	104	0.33
Pyrene	106	0.31
Benzo(a)anthracene	107	0.35
Chrysene	107	0.35
Benzo(b)fluoranthene	107	0.38
Benzo(k)fluoranthene	107	0.29
Benzo(a)pyrene	107	0.37
Indeno(1,2,3-cd)pyrene	104	0.41
Dibenzo(a,h)anthracene	106	0.42
Benzo(g,h,i)perylene	105	0.40





Automating Solid Phase Extraction and Florisil Clean-up for Organochlorine Pesticides and PCB Aroclors





TurboTrace ABN

Multi Cartridge SPE

Cartridge Chemistry 1

Cartridge Chemistry 2

Real Time Graphical Display of Extraction Steps

Automatic Liquid Level Sensor

Positive Pressure Pump for precise, consistent delivery of Sample and Solvent

Vacuum Pump for High Throughput Sampling



TurboTrace ABN SPE







Fluid Management Systems Clean-up Cartridge(s)







Extraction Procedure

- Condition clean-up Cartridge with elution solvent (10% Acetone in Hexane)
- Condition C18 Cartridge with elution solvent.
- Nitrogen purge off elution solvent
- Condition C18 cartridge with methanol
- Condition C18 cartridge with H₂O
- Load sample across C18 via vacuum
- Nitrogen dry C18 Cartridge





Elution Protocol

- Sample bottle rinsed with elution solvent
- Bottle rinse loaded and eluted across C18 and Clean-up cartridges, collected into FMS SuperVap concentrator
- Cartridges eluted with additional 20mls elution solvents
- Cartridges nitrogen purged into SuperVap

Total elution solvent = 45mls of 90:10 Hexane/Acetone Time = 45minutes to 1 hr



Data: IPR









Data: IPR





Data MDL





Commercial PT Performance





Commercial PT Rank

<u>Rank</u>	Analyte	<u>Rank</u>
11/23	delta-BHC	
13/23	Endosulfan I	1/23
10/23	Endosulfan II	19/23
11/23	Endosulfan sulfate	4/23
1/23	Endrin	13/23
11/23	Endrin aldehyde	21/23
13/23	Endrin ketone	7/22
10/23	gamma-BHC (Lindane, HCH)	16/23
7/26	gamma-Chlordane	
11/23	Methoxychlor	3/23
	Rank11/2313/2310/2311/231/2313/2310/237/2611/23	RankAnalyte11/23delta-BHC13/23Endosulfan I10/23Endosulfan II11/23Endosulfan sulfate1/23Endrin11/23Endrin aldehyde13/23Endrin ketone10/23gamma-BHC (Lindane, HCH)7/26gamma-Chlordane11/23Methoxychlor



Conclusions

- All IPR recoveries within 70%-130%
- Capable of delivering clean blanks and acceptable MDLs
- Excellent performance in live sample matrices and blind PT studies
- Complete automation of the entire process from bottle to GC ready extract.





Expanded Methodology







EPA Methods (examples)

- EPA 8082
- EPA 8081
- EPA 1694
- EPA 1664
- EPA 1613
- EPA 8151

- EPA 1614
- EPA 8310
- EPA 8290
- EPA 8330
- EPA 8141

Any EPA 500 series SPE method



Examples





Automated SPE in Summary

- One Step SPE and Concentration will reduce errors, labor costs, solvent usage and increase your sample throughput
- Automates and Combines the Extraction and Concentration steps in Sample Prep Processing
- In-line evaporation with direct to GC vial tubes
- Provides the Fastest Automated Sample Processing available for SPE Cartridges and Columns



Automated SPE in Summary

- Handles a wide range of Sample sizes and matrix types
- Uses all SPE Cartridge and Column sizes
- Comply with existing methods that require vacuum, positive pressure and precise delivery of sample and solvents
- Program and store an unlimited amount of methods





Conclusions

- Automated SPE extractions and Concentration is a very green technique
 - Reduces Solvent Use
 - Reduces Solvent Disposal Costs
 - Reduces Solvent emissions
- FMS automated SPE systems deliver consistent, reproducible results
- Solid Phase Extraction is a well accepted technology





Conclusions

- New Solid Phase Extraction Chemistries and Sorbents are being developed
- Fully Automated extractions, cleanups and concentrations
- Expandable to meet any SPE method
- Capable of performing in line extract drying and/or Cartridge extract clean-ups





Questions?

