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Fluid Management Systems

Automated Solid Phase extraction of water samples for EPA8270D

Philip Bassignani

Applications Specialist and Laboratory Manager at Fluid Management Systems

20 years experience in semi-volatile analysis (15 years with contract Labs)

Comparison of LLE vs. SPE Cartridge Methods

LLE	SPE Cartridge
Open to laboratory background	Closed system
Uses >360mls solvent	Uses <60mls solvent
Shaking / Continuous process	Filtration process
Forms emulsions requiring centrifuging	No emulsions formed
Little Selectivity	Wide Selectivity (adsorbent)
Requires water removal	In-line water removal

No Formation of Emulsions

Reduced Solvent Usage

Reduced Exposure to Laboratory Background

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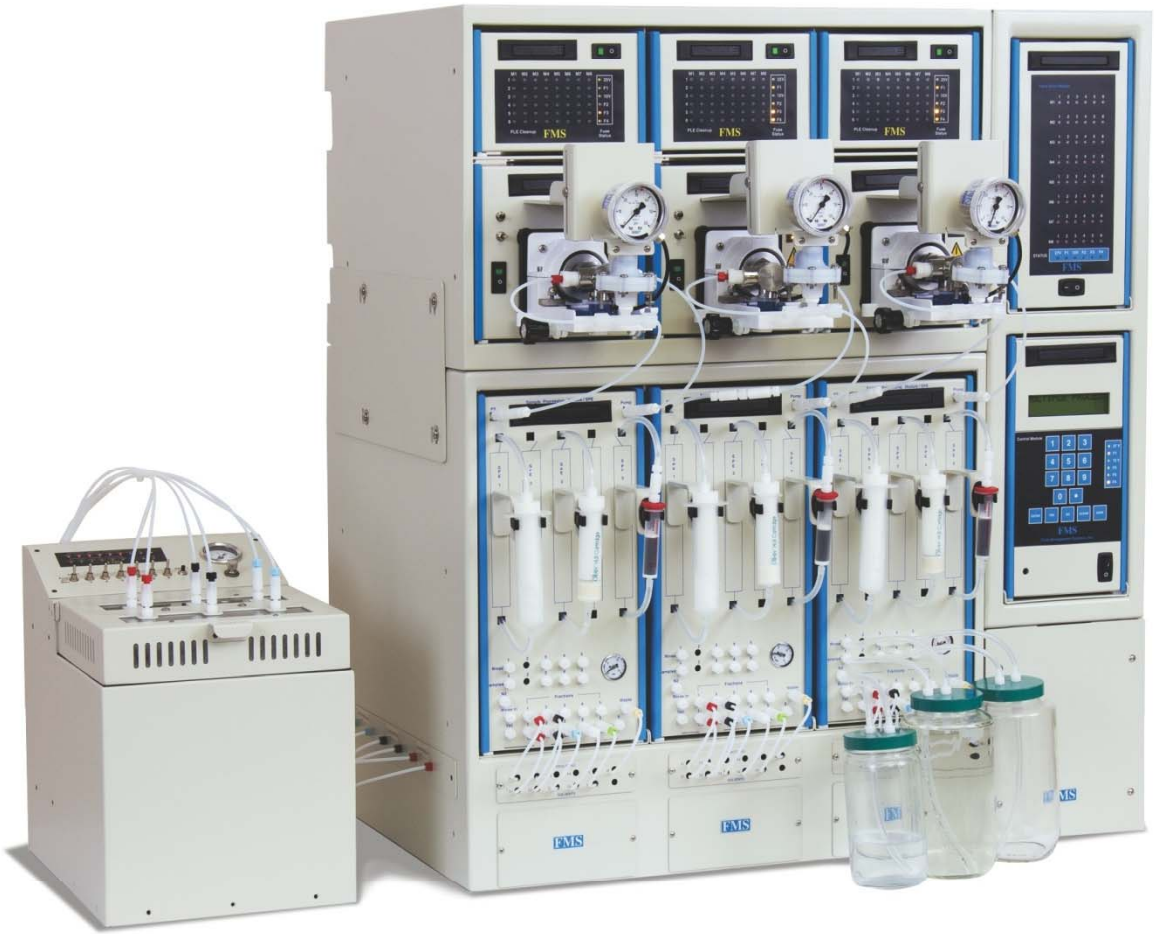
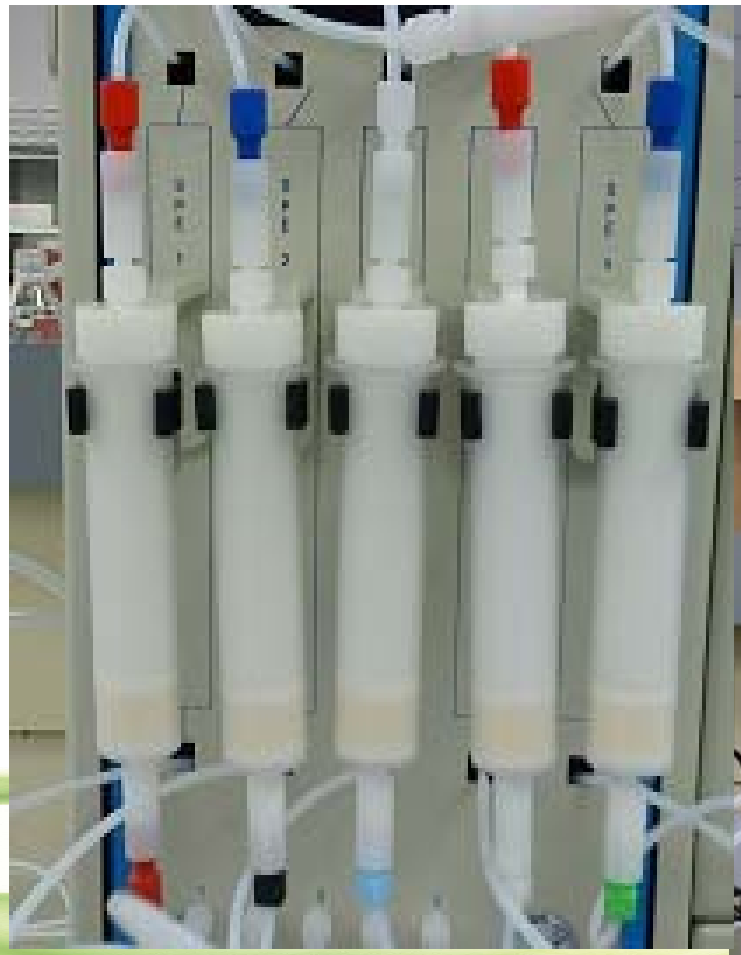


SPE Cartridges

Drying Cartridges

Dual Pass Versus Single Pass



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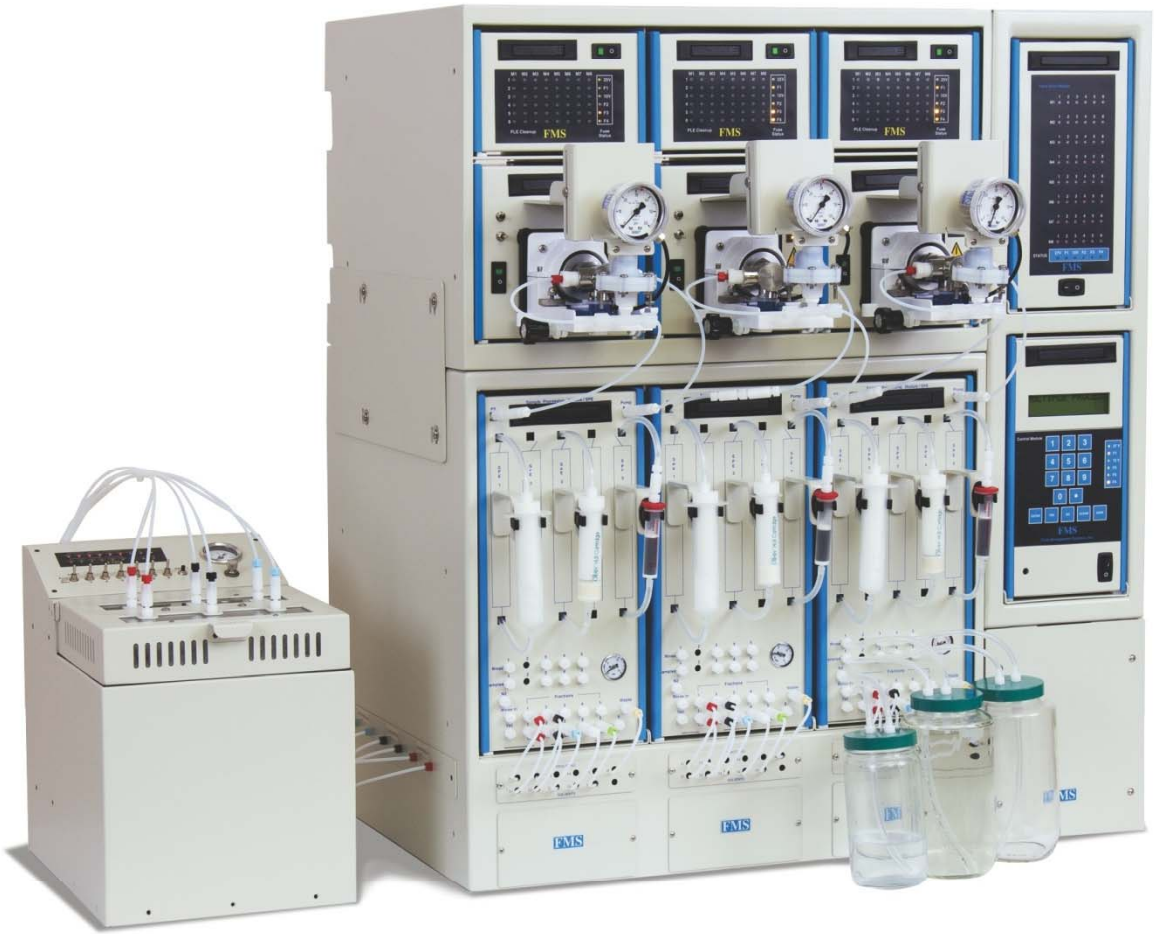

Procedure (Ion Exchange)

- Flush cartridges with DCM
- Nitrogen dry cartridges
- Wet Cartridges with Methanol
- Exchange cartridges with DI Water
- Load Samples (vacuum)
- Dry Cartridges with Nitrogen
- Elute Ion Exchange Cartridge with DCM
- Rinse bottle container with DCM and elute
- Nitrogen Purge Cartridge
- Elute Carbon Cartridge with DCM.
- Nitrogen purge Cartridge
- Rinse Ion exchange Cartridge with Methanol
- Rinse Cartridge with NaOH/'Water solution
- Nitrogen Dry Cartridge
- Elute Cartridge with DCM and Nitrogen purge

Procedure (Dual Pass)

- Flush cartridges with DCM
- Nitrogen dry cartridges
- Flush Cartridges with Methanol
- Flush cartridges with DI Water
- Load Samples (vacuum) (collecting sample in additional bottle with NaOH solution included)
- Dry Cartridges with Nitrogen
- Elute DVB Cartridge with DCM
- Rinse bottle container with DCM and elute
- Nitrogen Purge Cartridge
- Rinse DVB & Carbon Cartridge with Methanol
- Rinse Cartridges with DI water
- Load basic sample across both cartridges.
- Dry Cartridges with nitrogen.
- Elute DVB Cartridge with DCM and Nitrogen purge
- Elute Carbon cartridge with DCM and nitrogen purge

Concentration

Evaporator Procedure

- Pre-heat: 20 minutes at 55 °C
- Evaporate at 55 °C under 8 PSI nitrogen
- Rinse tubes with DCM at ~ 5mls & 1 ml
- Remove GC vial & add 20 µl ISTD
- Transfer vial to GC/MS

Surrogates

Surrogate	Conc. Spikled	Ion Exchange Mean Rec.	Rev. Phase Mean Rec.
2-Fluorophenol	100 ug/ml	75.6%	79.2%
Phenol-d5	100 ug/ml	76.5%	89.5%
2,4,6-tribromophenol	100 ug/ml	79.6%	91.7%
Nitrobenzene-d8	50 ug/ml	95.2%	85.1%
2-fluorobipenyl	50 ug/ml	93.3%	95.6%
p-terphenyl-d14	50 ug/ml	96.3%	100.0%

PAHs

Compound	Conc. Spiked	Ion Exchange Mean Rec.	Rev. Phase Mean Rec.	Compound	Conc. Spiked	Ion Exchange Mean Rec.	Rev. Phase Mean Rec.
Naphthalene	50 µg/ml	92.7%	86.0%	Fluoranthene	50 µg/ml	100.9%	97.9%
2-Methylnaphthalene	50 µg/ml	91.2%	87.3%	Pyrene	50 µg/ml	86.2%	84.7%
2-Chloronaphthalene	50 µg/ml	86.3%	93.6%	benzo[a]anthracene	50 µg/ml	101.5%	99.0%
Acenaphthylene	50 µg/ml	78.4%	91.0%	Chrysene	50 µg/ml	101.4%	85.9%
Acenaphthene	50 µg/ml	83.9%	92.9%	benzo[b]fluoranthene	50 µg/ml	84.3%	93.1%
Dibenzofuran	50 µg/ml	84.4%	93.1%	benzo[k]fluoranthene	50 µg/ml	83.6%	94.1%
Fluorene	50 µg/ml	89.5%	99.4%	benzo[a]pyrene	50 µg/ml	84.5%	88.6%
Phenanthrene	50 µg/ml	88.6%	93.9%	indeno[1,2,3-cd]pyrene	50 µg/ml	87.3%	100.1%
Anthracene	50 µg/ml	78.6%	81.4%	dibenzo[a,h]anthracene	50 µg/ml	90.1%	81.8%
Carbazole	50 µg/ml	82.4%	106.1%	benzo[g,h,i]perylene	50 µg/ml	85.3%	89.9%

Phthalates

Compound	Conc. Spiked	Ion Exchange Mean Rec.	Rev. Phase Mean Rec.
Dimethylphthalate	50 µg/ml	99.8%	99.5%
Diethylphthalate	50 µg/ml	95.6%	99.4%
Butylbenzyphthalate	50 µg/ml	83.2%	92.2%
di-n-butylphthalate	50 µg/ml	82.6%	97.0%
bis[2-ethylhexyl]phthalate	50 µg/ml	92.1%	92.2%
di-n-octylphthalate	50 µg/ml	77.9%	75.9%

Phenols

Compound	Conc. Spiked	Ion Exchange Mean Rec.	Rev. Phase Mean Rec.	Compound	Conc. Spiked	Ion Exchange Mean Rec.	Rev. Phase Mean Rec.
Phenol	100 µg/ml	76.6%	79.1%	2,4,6-trichlorophenol	100 µg/ml	69.5%	87.6%
2-chlorophenol	100 µg/ml	76.6%	83.3%	2,5,6-trichlorophenol	100 µg/ml	75.5%	55.5%
2-methylphenol	100 µg/ml	80.3%	85.1%	4-nitrophenol	100 µg/ml	85.5%	36.0%
3/4-methylphenol	100 µg/ml	78.3%	86.4%	2,3,4,6-tetrachlorophenol	100 µg/ml	79.4%	71.2%
2-nitrophenol	100 µg/ml	96.0%	86.7%	2-methyl-4,6-dinitrophenol	100 µg/ml	85.0%	101.0%
2,4-dimethylphenol	100 µg/ml	90.4%	84.9%	2,4-dinitrophenol	100 µg/ml	97.2%	98.7%
2,4-dichlorophenol	100 µg/ml	67.8%	75.1%	Pentachlorophenol	100 µg/ml	92.5%	91.2%
2,6-dichlorophenol	100 µg/ml	60.1%	72.0%	Dinoseb	100 µg/ml	65.7%	77.4%
4-chloro-3-methylphenol	100 µg/ml	62.6%	70.9%	benzoic Acid	100 µg/ml	74.7%	38.2%

Miscellaneous BN compounds

Surrogate	Conc. Spikled	Ion Exchange Mean Rec.	Rev. Phase Mean Rec.	Surrogate	Conc. Spikled	Ion Exchange Mean Rec.	Rev. Phase Mean Rec.
bis[2-chloro]ethylether	50 µg/ml	78.5%	84.0%	3-nitroanaline	50 µg/ml	84.9%	101.0%
bis[2-chloroisopropyl]ether	50 µg/ml	77.0%	83.2%	2-nitroanaline	50 µg/ml	98.1%	98.7%
bis[2-chloroethoxy]methane	50 µg/ml	95.6%	86.3%	4-nitroanaline	50 µg/ml	94.6%	100.2%
hexachloroethane	50 µg/ml	72.9%	84.2%	analine	50 µg/ml	64.0%	67.2%
1,3-dichlorobenzene	50 µg/ml	78.3%	83.3%	2,4-dinitrotoluene	50 µg/ml	105.6%	107.3%
1,2-dichlorobenzene	50 µg/ml	80.4%	85.3%	2,6-dinitrotoluene	50 µg/ml	97.4%	99.9%
1,4-dichlorobenzene	50 µg/ml	79.7%	84.6%	4-chloropnehylphenylether	50 µg/ml	91.1%	86.6%
1,2,4-trichlorobenzene	50 µg/ml	89.4%	84.9%	4-bromophenylether	50 µg/ml	82.3%	83.9%
Nitrobenzene	50 µg/ml	93.3%	84.9%	NDMA	50 µg/ml	35.3%	26.4%
Azobenzene	50 µg/ml	83.3%	98.4%	NDPA	50 µg/ml	67.9%	91.7%
Hexachlorobenzene	50 µg/ml	93.8%	111.0%	Pyridine	50 µg/ml	41.1%	47.6%
Isophorone	50 µg/ml	95.5%	85.0%	3,3-Dichlorobenzidine	50 µg/ml	78.1%	74.1%
Hexachlorobutadiene	50 µg/ml	84.5%	82.1%	Benzidine	50 µg/ml	42.3%	43.3%
Hexachlorocyclopentadiene	50 µg/ml	68.3%	83.6%	Benzyl Alcohol	50 µg/ml	67.5%	45.2%
4-chloroanaline	50 µg/ml	84.7%	83.9%				

Efficiency for SIM

Compound	Amount Spiked	SPE #1	SPE #2	SPE #3	SPE #4	SPE #5	SPE #6	SPE #7	Mean	STD Dev	MDL
Anthracene	0.05	0.0467	0.0510	0.0510	0.0500	0.0499	0.0515	0.0500	0.050	0.0016	0.0051
bis(2-ethylhexyl)phthalate	2	1.7920	1.9400	2.4600	1.8760	2.1200	1.9180	1.9040	2.001	0.2252	0.7076
dibenzo[a,h]anthracene	0.05	0.0401	0.0407	0.0438	0.0355	0.0394	0.0404	0.0407	0.040	0.0025	0.0077
Chloropyrifos	0.125	0.1450	0.1500	0.1638	0.1363	0.1638	0.1488	0.1475	0.151	0.0100	0.0313
Pyrene	0.05	0.0575	0.0580	0.0580	0.0555	0.0565	0.0565	0.0585	0.057	0.0011	0.0034
Dichlorobenil	0.125	0.0964	0.0986	0.0998	0.0961	0.0960	0.0976	0.1013	0.098	0.0020	0.0063
Dimethylphthalate	2	1.0760	0.9840	0.9300	0.8640	0.9580	0.9760	1.0200	0.973	0.0670	0.2104
Di-n-butylphthalate	2	1.9360	1.8960	2.0200	1.9420	1.9500	1.9200	2.0000	1.952	0.0437	0.1373
benzo[a]anthracene	0.05	0.0449	0.0471	0.0515	0.0463	0.0479	0.0464	0.0447	0.047	0.0023	0.0073
Chrysene	0.05	0.0415	0.0428	0.0458	0.0419	0.0425	0.0434	0.0424	0.043	0.0014	0.0044
indeno[1,2,3-cd]pyrene	0.05	0.0386	0.0398	0.0420	0.0357	0.0396	0.0396	0.0395	0.039	0.0019	0.0059
Phenanthrene	0.05	0.0560	0.0545	0.0540	0.0500	0.0525	0.0550	0.0560	0.054	0.0021	0.0067
benzo[b]fluoranthene	0.05	0.0469	0.0489	0.0525	0.0446	0.0462	0.0446	0.0471	0.047	0.0028	0.0087
2-methlynaphthalene	0.05	0.0476	0.0465	0.0425	0.0465	0.0424	0.0461	0.0474	0.046	0.0022	0.0069
benzo[a]pyrene	0.05	0.0434	0.0459	0.0500	0.0422	0.0429	0.0432	0.0442	0.045	0.0027	0.0084
Acenaphthylene	0.05	0.0378	0.0359	0.0317	0.0353	0.0355	0.0379	0.0407	0.036	0.0028	0.0088
Malathion	0.125	0.1288	0.1500	0.1650	0.1425	0.1600	0.1500	0.1450	0.149	0.0119	0.0373
Di-n-octylphthalate	2	1.6420	1.8580	2.3200	1.7600	1.9840	1.8860	1.8360	1.898	0.2143	0.6736
Acenaphthene	0.05	0.0431	0.0376	0.0349	0.0369	0.0390	0.0405	0.0468	0.040	0.0040	0.0127
Fluorene	0.05	0.0410	0.0384	0.0339	0.0369	0.0375	0.0392	0.0415	0.038	0.0026	0.0081
benzo[k]fluoranthene	0.05	0.0458	0.0479	0.0570	0.0447	0.0475	0.0483	0.0458	0.048	0.0041	0.0130
Diazanon	0.05	0.0555	0.0530	0.0565	0.0575	0.0615	0.0560	0.0595	0.057	0.0028	0.0087
Fluoranthene	0.05	0.0595	0.0585	0.0585	0.0545	0.0585	0.0565	0.0590	0.058	0.0017	0.0055
Butylbenzylphthalate	2	1.8800	2.0800	2.4600	2.0200	2.0800	2.0600	1.9660	2.078	0.1832	0.5757
Diethylphthalate	0.05	1.8340	1.5380	1.4020	1.4920	1.5720	1.5860	1.8800	1.615	0.1767	0.5553
Naphthalene	0.05	0.0430	0.0424	0.0408	0.0411	0.0400	0.0416	0.0455	0.042	0.0018	0.0057
benzo[g,h,i]perylene	0.05	0.0363	0.0355	0.0387	0.0334	0.0375	0.0364	0.0372	0.036	0.0017	0.0053
Prometon	0.125	0.1375	0.1198	0.1133	0.0984	0.1288	0.1209	0.1350	0.122	0.0135	0.0425

Direct to GC Vial Evaporator Tubes

Conclusions

Solid Phase Extraction is a viable alternative to liquid-liquid extraction for EPA-8270D

Questions?