Analysis of Base, Neutral and Acid Semi-volatiles in Municipal and Industrial Waste Water Using EPA Method 8270D with Semi-Automated Solid Phase Extraction (EZSpe®)

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Introduction

Solid Phase Extraction has long been used for the analysis of semi-volatile organics in clean matrices. Methods like EPA 525.3 and EPA 8270D outline performance data for a variety of analytes and products. Due to the unique challenges inherent with waste water matrices laboratories have predominately adhered to LLE (Liquid-Liquid Extraction) protocols. Recent advances in packing materials and semi-automated extraction systems have now made once unheard of extractions of matrices commonplace for SPE.

To meet demands for a low cost method that requires less financial investment than fully automated systems, FMS developed a simple semi - automated system which is fast, inexpensive and yields high quality data.

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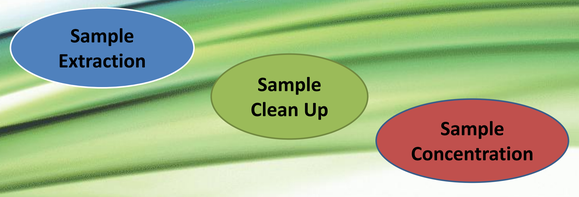
Instrumentation

- FMS EZSpe® System
- FMS SuperVap®
- Vacuum pump
- Thermo Trace GC w/ DSQ MS

Consumables

- FMS, Inc. mixed-bed ABN cartridges
- FMS sodium sulfate cartridge
- FMS 2 gram coconut charcoal cartridges
- Ultra-pure DI water
- Fisher 6 N Hydrochloric Acid; Sodium Hydroxide
- Fisher Pesticide Grade Methanol
- Fisher Pesticide Grade Dichloromethane
- Restek 8270 spiking standards



Procedure

- 6 samples (1L water each) are prepared and acidified with 2 mL HCl till pH ~ 2
- Spike with various standards
- Put sample bottles in place and fill rinse bottles with 35 mL dichloromethane
- Mixed-bed and coconut charcoal cartridges are installed in each of the six positions.

Stage 1a:

- Vacuum is turned on
- Cartridges are conditioned with 10 mL dichloromethane, 20 mL methanol and 35 mL water
- Samples are loaded across cartridges under vacuum
- Cartridges are dried with nitrogen for 10 min
- Sample bottles are automatically rinsed from the rinse bottles with 35 mL dichloromethane

Stage 2a:

- Mixed-bed and coconut charcoal cartridges are separated and dichloromethane from sample bottles is loaded across the mixed-bed cartridges, and the eluent is collected.
- Another 15 mL of dichloromethane is eluted across the cartridges and the total volume is collected as Fraction 1 (acidic fraction).

Stage 1b:

- The mixed-bed cartridges are conditioned with 50 mL 1% sodium hydroxide

Stage 2b:

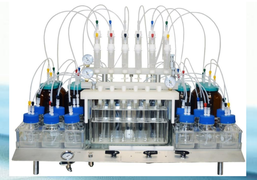
- 45 mL of dichloromethane are passed across the mixed-bed cartridges and the eluent is collected
- Another 25 mL of dichloromethane are passed across the charcoal cartridges, into the same vessel as above, and the total volume is collected as Fraction 2 (basic fraction).
- Extracts are dried over sodium sulfate or in line cartridges can be used downstream from SDVB cartridges

FMS SuperVap®

- Pre-heat temp: 45 ºC
- Pre-heat time: 15 minutes
- Heat in Sensor mode at 45 ºC under nitrogen (7-10 psi)
- Direct to GC Vial Vessel Reduce to 1 mL
- Samples are now ready for analysis

Results: average recoveries (%, 25 ug/L spike)

Analyte	Average (%)	EPA Window	Analyte	Average (%)	EPA Window
N-nitrosodimethylamine-d6_(S)	35		dimethyl_phthalate	77	D-112
N-nitrosodimethylamine	76		3-Nitroaniline	84	
Pyridine	78		2,6-dinitrotoluene	84	50-158
bis(2-chloroethyl)_ether-d8_(S)	113		Acenaphthylene-d10_(S)	89	
Phenol-d5_(S)	107		Acenaphthylene	94	33-145
Phenol	107	5-112	1,2-Dinitrobenzene	80	
Aniline	101		2,4-dinitrophenol	80	
2-Chlorophenol-d4_(S)	101		Acenaphthene	79	47-145
bis(2-chloroethyl)_ether	95	12-158	Dibenzofuran	84	
2-Chlorophenol	96	23-134	4-Nitrophenol	58	D-132
1,3-Dichlorobenzene	76	20-124	2,4-dinitrotoluene	60	39-139
1,4-Dichlorobenzene	84	D-172	4-Nitrophenol-d4_(S)	95	
1,2-Dichlorobenzene	82	32-129	2,3,5,6-Tetrachlorophenol	98	
Benzyl_Alcohol	107		2,3,4,6-Tetrachlorophenol	86	
2-Methylphenol	104		diethyl_phthalate	84	D-114
2,2'-oxybis(1-chloropropane)	102		Fluorene-d10_(S)	71	
N-nitrosodi-n-propylamine	112	D-230	Fluorene	98	59-121
4-Methylphenol-d8_(S)	109		4-chlorophenyl_phenyl_ether	97	25-158
3/4-Methylphenol	104		4-Nitroaniline	79	
Hexachloroethane	74	40-113	2-Methyl-4,6-dinitrophenol-d2_(S)	62	
Nitrobenzene-d5_(S)	100		2-Methyl-4,6-dinitrophenol	45	
nitrobenzene	101	35-180	DPA/NDPA	18	
isophorone	96	21-196	Azobenzene	87	
2-Nitrophenol-d4_(S)	82		Hexachlorobenzene	103	
2-Nitrophenol	82	29-182	pentachlorophenol	82	14-176
2,4-dimethylphenol	88	32-119	Anthracene-d10_(S)	81	
bis(2-chloroethoxy)methane	96	33-184	Phenanthrene	96	27-133
2,4-Dichlorophenol-D3_(S)	82		Anthracene	96	54-120
2,4-Dichlorophenol	79	39-135	Carbazole	98	
1,2,4-trichlorobenzene	62	44-142	di-n-butyl_phthalate	120	1-118
Naphthalene	83	21-133	Fluoranthene	102	26-137
4-Chloroaniline	91		Pyrene-d10_(S)	97	
4-Chloroaniline-d4_(S)	80		Benzidine	104	
hexachlorobutadiene	85	24-116	Pyrene	98	52-115
4-Chloro-3-methylphenol	99	22-147	Chrysene	94	
1-Methylnaphthalene	82		benzo[a]anthracene	94	17-168
2-Methylnaphthalene	80		3,3'-Dichlorobenzidine	77	33-143
hexachlorocyclopentadiene	116		bis(2-ethylhexyl)phthalate	123	8-158
2,4,5-Trichlorophenol	69		benzo[b]fluoranthene	103	24-159
2,4,6-Trichlorophenol	68	37-144	benzo[k]fluoranthene	103	11-162
2-Chloronaphthalene	73	60-118	benzo[a]pyrene-d12_(S)	91	
2-Nitroaniline	86		benzo[a]pyrene	116	17-163
1,4-Dinitrobenzene	91		indeno[1,2,3-cd]pyrene	85	D-171
dimethyl_phthalate-d6_(S)	70		dibenzo[a,h]anthracene	83	D-227
1,3-Dinitrobenzene	89		benzo[g,h,i]perylene	104	D-219



FMS EZSpe system

Conclusions

Reviewing the sample data shows high recoveries for multiple 8270 analytes, demonstrating excellent efficiency for all classes of compounds. Samples can be taken from collection bottle to GC vial in one quick, consistent, reproducible process that will save laboratories both time and money.

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