Application Note

The Automated Extraction of Aqueous Samples by Method EPA 8270D Using the TurboTrace™ 8270 SPE System

Introduction

EPA 8270D calls for the extraction for analysis of semi-volatile analytes in various matrices. Target analytes mentioned in the method cover a wide range of compound classes resulting in reporting lists that often approach 100 compounds. In aqueous samples, the combination of compound groups such as phenols, analines, PAHs, phthalates, explosives, pesticides, n-nitrosoamines, and others results in a tedious process of multiple LLE (liquid-liquid extractions) with multiple pH adjustments to extract all the analytes. Large elution volumes (>360 mL of methylene chloride) combined with highly volatile analytes, usually result in low recoveries of target compounds and poor analytical precision.

The use of SPE (Solid Phase Extraction) to automate the extraction process can drastically reduce many of the challenges traditional 8270 extractions pose. By automating the process with the FMS 8270 SPE system, extraction chemists, normally confined to a hood shaking separatory funnels, can be freed up to perform additional tasks.

Instrumentation and Consumables

Instrumentation

- FMS, Inc. TurboTrace™ 8270 SPE system
- FMS, Inc. SuperVap™ concentrator
- FMS, Inc. 200 mL concentrator tubes
- Thermo Trace GC w/DSQ MS

Consumables

- Fisher Pesticide Optima* Methylene Chloride
- Fisher Anhydrous Sodium Sulfate
- Fisher Optima* Methanol
- Fisher HPLC Grade Water
- FMS 1 gram DVB Cartridges
- Fisher Concentrated Sulfuric Acid
- Fisher Sodium Hydroxide

Consumables (*continued*)

- Restek Resprep 2 gram coconut charcoal cartridges (Cat# 26032)
- Restek 8270 matrix spike (Cat# 33073)
- Restek SV Internal Standard Mix (Cat# 31006)
- Restek B/N Surrogate (Cat# 31024)
- Restek Acid Surrogate (Cat# 31025)
- Restek Benzidines mix (Cat# 31834)

Procedure

Prepare 1 liter samples of DI water

Adjust pH of samples to <2 by adding H_2SO_4 drop wise.

Spike samples with 8270 matrix spike, B/N surrogate, and Acid surrogate spiking solutions

Load samples onto FMS TurboTrace 8270 SPE system.

Affix collection bottle to retain post extraction aqueous sample.

Affix coconut charcoal and DVB with pre-filter cartridges to TurboTrace 8270 system.

Figure 1: FMS TurboTrace SPE system with the SuperVap concentrator.

FMS
Fluid Management Systems

SPE

1. Cartridges pre-wet with DCM
2. Cartridges conditioned with MeOH
3. Cartridges conditioned with H_2O
4. Samples passed across DVB cartridges at ~20 mL/min via vacuum
5. Cartridges partially dried with N_2 at 15 PSI
6. Sample bottles sprayed with DCM
7. DCM bottle spray loaded across cartridge and collected through in-line $NaSO_4$
8. Cartridges eluted with additional 10 mL DCM
9. Cartridges purged with N_2 eluting n-Hexane directly to FMS SuperVap concentrator
10. Remove aqueous collection bottle and add NaOH solution to sample mixing thoroughly till pH is >12.
11. Attached sample bottle to system for second pass.
12. DVB Cartridge re-conditioned with MeOH.
13. DVB Cartridge re-conditioned with H_2O
14. Sample passed across both cartridges at ~10 mL/min.
15. Cartridges dried with N_2 for 1 minute each
16. Sample bottle sprayed with DCM
17. DCM bottle spray loaded across DVB cartridge and collected through in-line $NaSO_4$
18. DVB Cartridge eluted with additional 5 mL DCM
19. Coconut charcoal eluted with 20 mL DCM
20. Cartridges independently purged of residual solvent via N_2 stream.

SuperVap Concentrator

1. Preheat temp: 10 minutes at 40 °C
2. Evap mode: 40 °C
3. Nitrogen Pressure: 10 PSI
4. Evaporate extracts 1 mL*

*Evaporator tubes manually rinsed with DCM to ensure no target analytes adhere to evaporator tube walls.

Internal Standard solution added to extract post evaporation for GC/MS analysis.

Results

Table 1: Results from 6 1L LCS samples (50 µg/L concentration)

Analyte	Mean
Pyridine	71.63
NDMA	54.44
2-Fluorophenol (Surr)	85.84
Phenol-d5 (Surr)	78.32
Phenol	74.6
Analine	84.06
bis(2-chloroethyl) ether	93.3
2-Chlorophenol	85.99
1,3-Dichlorobenzene	84.86
1,4-Dichlorobenzene	86.29
1,2-Dichlorobenzene	90.67
benzyl_alcohol	90.12
2-methylphenol	91.32
bis(2-chloroisopropyl) ether	94.6
N-nitrosodi-n-propylamine	103.54
4-methylphenol/3-methylphenol	93.3
Hexachloroethane	89.79
Nitrobenzene-d5 (Surr)	96.27
nitrobenzene	111.68
isophorone	94.42
2-Nitrophenol	84.56
2,4-dimethylphenol	99.46
bis(2-chloroethoxy)methane	100.55
2,4-Dichlorophenol	89.01
1,2,4-trichlorobenzene	85.24
Naphthalene	87.54
4-Chloroanaline	80.43
hexachlorobutadiene	81.3
4-Chloro-3-methylphenol	89.26
2-methlynaphthalene	103.61
1-methlynaphthalene	103.95
hexachlorocyclopentadiene	52.78
2,4,6-Trichlorophenol	78.76
2,4,5-Trichlorophenol	79.44
2-Fluorobiphenyl (Surr)	99.03
2-Chloronaphthalene	84.73
2-Nitroanaline	93.23
1,4-dinitrobenzene	93
dimethyl phthalate	97.45
1,3-dinitrobenzene	95.21

Results *(continued)*

Analyte	Mean
Acenaphthylene	89.58
1,2-dinitrobenzene	96.45
3-Nitroanaline	100.57
2,6-dinitrotoluene	101.41
Acenaphthene	95.77
2,4-dinitrophenol	83.84
4-Nitrophenol	99.19
dibenzofuran	94.81
2,4-dinitrotoluene	96.66
2,3,5,6-Tetrachlorophenol	89.25
2,3,4,6-Tetrachlorophenol	87.3
diethyl phthalate	107.46
Fluorene	100.96
4-chlorophenyl phenyl ether	90.49
4-Nitroanaline	112.58
2-Methyl-4,6-dinitrophenol	102.85
NDA-NDPA	89
Azobenzene	101.4
2,4,6-Tribromophenol (Surr)	86.58
4-bromophenyl phenyl ether	90.94
Hexachlorobenzene	92.84
pentachlorophenol	96.05
Phenanthrene	99.97
Anthracene	100.15
Carbazole	108.6
butyl benzyl phthalate	117.76
Pyrene	105.37
Fluoranthene	98.53
Terphenyl-d14 (Surr)	102.05
di-n-butyl phthalate	109.91
benzo[a]anthracene	106.3
Chrysene	102.82
bis(2-ethylhexyl)phthalate	124.46
di-n-octyl_phthalate	118.02
benzo[b]fluoranthene	102.23
benzo[k]fluoranthene	114.84
benzo[a]pyrene	99.25
indeno[1,2,3-cd]pyrene	96.23
dibenzo[a,h]anthracene	94.5
benzo[g,h,i]perylene	95.43
Benzidine	88.21
3,3-dichlorobenzidine	94.6

Conclusions

Evaluation of the performance spikes on the FMS TurboTrace™ 8270 extraction system resulted in average recoveries for nearly all analytes between 70-130%. Combined with the analysis of blank replicates which yielded background concentrations of less than 1 µg/mL for all analytes, the data, when compared to the performance tables within EPA 8270D, shows that automated solid phase extraction is not only equivalent, but in some cases superior to traditional LLE extractions.

Repeatability data gave percent deviations between replicates of less than ±10% for most compounds, and ±20% for all. Fully automated sample loading and eluting of both cartridges independently, and in sequence, eliminates the need to have an extraction chemist separate the cartridges at different stages of the operation. The reduction of human interaction saves time, reduces human error and produces consistent, reproducible results.

When paired with the FMS SuperVap concentrator, the TurboTrace™ 8270 SPE system provides complete, automated sample preparation for aqueous samples by EPA8270D.

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