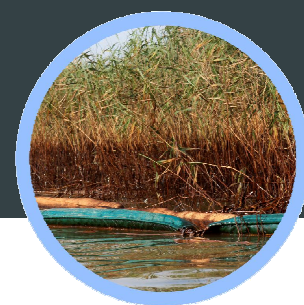


# An MDL study using Automated Solid Phase Extraction of Semi-Volatile Organic Compounds (AB 8270 SIM) in Water



## Introduction

EPA Method 8270 is used to determine the concentration of semi-volatile organic compounds in extracts prepared from solid waste matrices such as soils, air sampling media and water samples. Extractions from water samples can be particularly difficult and time consuming due to the problematic analytes that need to be extracted, concentrated and analyzed as well as the large quantities of solvent the manual sample process requires. Laboratories now have to report analytes at lower reporting limits than previously required, so they often need to employ SIM scan variations of the method.

Given the recovery variability of 8270 compounds and the time it takes to conduct manual extractions, complete automation of the sample prep process is often viewed as the Holy Grail of sample prep. The following application outlines the complete automation of a modified 8270 extraction, delivering a complete, out-of-the box MDL study that demonstrates both precision and accuracy for mixed classes of low-level analytes.

## Instrumentation and Consumables

- FMS, Inc. PowerPrep™ SPE system
- FMS, Inc. SuperVap™ Concentrator system
- FMS, Direct-to-Vial concentrator tubes
- Thermo Certified GC/MS auto sampler vials
- Waters 1 gram Oasis HLB® Cartridge
- Restek 2 gram Sodium sulfate Cartridge
- Thermo Trace GC w/DSQ MS and AS3000 Autosampler

## Reagents

- Fisher Optima\* Methanol
- Fisher Optima\* Methylene Chloride
- Fisher HPLC Water
- Fisher Conc. Sulfuric Acid
- Restek Cat# 31622 Cal Mix #5
- Restek Cat# 316031 Phthalate mix
- AccuStandard Cat# M-8140-06 Diazinon mix
- Ultra Scientific Custom OPP Mix

## Procedure:

### Sample Prep

Five, 1 liter samples are measured out in glass sample collection bottles

Each sample is spiked with 1 mL of spiking solution (dilute of Ultra Scientific, Restek and Accustandard solutions)

The sample PH adjusted to <2 with 1:1 sulfuric acid solution

10 mL Methanol added to each sample bottle

### PowerPrep SPE system

1. HLB Cartridges are conditioned with 10 mL of Methanol
2. HLB Cartridges are conditioned with 10 mL of DI H<sub>2</sub>O
3. Samples are loaded across the HLB Cartridges via vacuum (~75mL/min)
4. Sample bottles are auto rinsed with DI water and the rinse loaded onto the HLB
5. The cartridges are dried with Nitrogen for 2 minutes
6. The HLB cartridges are eluted with 10 mLs of methylene chloride
7. The HLB cartridges are eluted with 10 mLs of methylene chloride
8. Cartridges are purged with N<sub>2</sub>
9. Total time: 61.5 minutes

### SuperVap Concentrator system

1. Pre-heat temp: 45 °C
2. Pre-heat time: 20 minutes
3. Heat in Sensor mode: 45 °C
4. Nitrogen Pressure: 10 PSI
5. End point: 1mL



## Results

Table 1: MDL data compiled over seven replicates.

Compound	Amount Spiked	Amount							Mean	STD Dev	MDL
		SPE #1	SPE #2	SPE #3	SPE #4	SPE #5	SPE #6	SPE #7			
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-methylnaphthalene	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



Figure 1: FMS PowerPrep SPE system with the SuperVap Concentrator

### Conclusions

The results in Table 1 show the recoveries of all seven MDL replicates extracted on the FMS PowerPrep™ SPE/ SuperVap Direct-to-Vial Concentration system. The results demonstrate the ability of the FMS Total Sample Prep approach to deliver precise, consistent recoveries at ultra low level concentrations and with extreme precision. The combination of high recoveries and low standard deviations deliver a low-level MDL that is easy to achieve.

The FMS PowerPrep SPE and SuperVap Direct-to-Vial automated turnkey system is an ideal choice for automating a wide range of compound classes. The system is capable of extracting 5-30 samples in one program which allows the system to grow along with your laboratory's throughput. This turnkey system gives laboratories the ability to rapidly extract and concentrate directly to a vial entire analytical batches with just the click of a mouse.

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