Determination of Semi-Volatile Organic Compounds in Water Using the Oasis HLB SPE Disk for EPA Method 8270D

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Introduction

The purpose of this application note is to demonstrate the use of the Oasis® HLB SPE Disk, with the Horizon Technology SPE-DEX® 4790 Extractor System. The SPE-DEX 4790 Extractor System is a fully-automated, solid-phase extraction system, that provides not only *fast* extraction, but improves the quality and consistency of results, when running U.S. EPA Method 8270D.

EPA Method 8270D measures the semi-volatile organics in solid waste matrices utilizing Method 3535A, and solid-phase extraction (SPE). SPE provides many benefits over conventional liquid-liquid extraction (LLE) techniques, by reducing analysis time and costs, improving accuracy and precision, decreasing solvent usage, and reducing turnaround time from days to hours.

EPA Method 8270D is used to determine most neutral, acidic, and basic semi-volatile organic compounds that are soluble in methylene chloride. Compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols.

The Oasis HLB (Hydrophilic-Lipophilic Balanced) solid-phase extraction disk is able to retain the large list of organic semi-volatile compounds through its unique ratio of hydrophilic N-vinylpyrrolodone and lipophilic divinylbenezene sorbent. The Oasis HLB sorbent has a number of key features that enable it to perform the extraction, including stability at ph extremes and in a wide range of solvents, extraordinary retention of polar compounds, and a hydrophobic retention capacity 3x greater than that of traditional silica-based SPE sorbents.

The Horizon Technology SPE-DEX 4790 provides an automated extraction of liquid samples via SPE methods. The SPE-DEX 4790 can handle samples ranging from 20ml to 4+L. The Horizon Technology Envision Platform Controller provides a user-friendly, web-based controller, capable of interacting with up to eight SPE-DEX 4790 extractors via a standard PC. The Horizon Technology DryVap Concentrator System provides automated sample drying, with a patented membrane technology that automatically concentrates each dried extract by applying heat, vacuum, and sparge flow for up to six samples at once. The environmentally-friendly Horizon Technology Reclaimer Solvent Recovery System is designed to condense and collect solvent vapors which are generated by the DryVap Concentrator System. These automated systems are specifically designed to streamline the sample handling required for the preparation and analysis of environmental samples

The Horizon Technology SPE-DEX 4790® Automated Extraction System, Envision™ Platform Controller, DryVap™ Automated Drying and Concentrating System, and Oasis® HLB Disk.

Instrumentation

- Horizon Technology SPE-DEX 4790 Automated Extractor System.
- Dual pH Kit.
- Horizon Technology Envision Platform Controller.
- Horizon Technology DryVap Concentrator System.
- Horizon Technology DryDisk Separation Membranes.
- Horizon Technology Reclaimer Solvent Recovery System.
- Oasis HLB SPE Disks (47mm).
- Agilent 6890 GC.
- Agilent 5973 Mass Selective Detector.

Method Summary

pH 2 Extraction

1) Adjust 1L aqueous sample to pH 2 with HCl, cap the bottle and mix.
2) Spike 8270 surrogate into samples.
3) Spike 8270 compounds into samples (1.0ml of 50ug/ml spike was used for acid and base compounds).
4) Place a small piece of aluminum foil over the opening of the bottle and screw on the bottle cap adaptor.
5) Remove the original water waste line from the rear panel of the SPE-DEX 4790.
6) Install the sample collection bottle (provided in the dual pH kit). There are two connections. The connection on the side of the cap attaches directly to the water waste fitting on the rear panel of the SPE-DEX 4790 with the Teflon tube provided in the dual pH kit. The connection on top of the sample collection bottle attaches directly into the waste water collection bottle with the tubing provided in the dual pH kit.
7) Load the disk holder with the Oasis HLB 47mm disk.
8) Place a clean VOA vial or equivalent receiver onto the extractor.

9) Load the pH 2 sample onto the SPE-DEX 4790 and turn the sample clockwise 270 degrees.
10) Start EPA Method 8270D extraction method (Table 1), collect extract at high vacuum at 25 in. Hg.
11) Collect extract approximately 20mls.
12) Cap and label extract as acid portion.

pH 12 Extraction

1) Unscrew the cap on the sample collection bottle and adjust the collected water to pH 12 with NaOH and mix.
2) Connect the waste water collection line up to the luer fitting tee (provided in the dual pH kit).
3) Connect the line from the rear of the SPE-DEX 4790 to one of the two remanding openings on the luer fitting tee. Make sure the last connection of the luer fitting tee is capped.
4) Place the sample bottle adaptor (provided in the dual pH kit) on the sample collection bottle.
5) Place a small piece of aluminum foil over the opening of the sample bottle adaptor and screw on the bottle cap adaptor.
6) Load the pH 12 sample onto the SPE-DEX 4790 and turn the sample clockwise 270 degrees.
7) Place a clean VOA vial or equivalent receiver on the extractor.
8) Load EPA Method 8270D (Table 1), and process the sample using the same HLB disk as used in the acidic portion. Collect extract at high vacuum at 25 inHg.
9) Collect extract approximately 20mls.
10) Cap and label extract as base portion.

Concentration

1) Assemble the DryDisk reservoir with a DryDisk and start the concentration process by adding the acid portion into the DryDisk tube.(Table 2: DryVap Conditions)
2) Once the acidic portion filters through the DryDisk manually rinse the DryDisk reservoir with methylene chloride. Once the methylene chloride has filtered into the concentration tube press stop on the DryVap.
3) Clean the DryDisk reservoir. Empty the water, and then rinse the reservoir with acetone.
4) Load the DryDisk reservoir back onto the DryVap and start the base concentration process using the same conditions.
5) Once the basic portion filters through the dry disk manually rinse the Dry Disk reservoir with methylene chloride.
6) Concentrate the extract to less than 1.0ml (DryVap concentration vessels are graduated to 0.5ml and 1.0ml).
7) Rinse the bottom of the concentrator tube with methylene chloride and bring the extract up to 1.0ml.
8) Transfer the extract to a GC vial.
9) Analyze by GC/MS.

GC/MS Method

Agilent 6890 GC with an Agilent 5973 Mass Selective Detector

Column: DB5MS, 30m x 0.25mm ID, 0.25um

Flow Rate: 9 psi helium which is ramped up with the oven temp to maintain a constant flow.

Temp Ramp:

Temp (°C)	Rate (°C/Min)	Hold (Min)
45	0	1.00
270	15	0.00
320	6.0	0.00

Total Run Time: 24.33 min

Injection Method: Split, Ratio 1:10, 1.0uL injected

Temp (°C)	Rate (°C/Min)	Hold (Min)
280	0	0.00

Table 1 The EPA 8270D Method - programmable into the Envision Platform Controller

Step	Solvent	Soak Time	Dry Time
Prewet #1	Acetone	30 Sec	15 Sec
Prewet #2	Acetone	30 Sec	15 Sec
Prewet #3	Reagent Water	10 Sec	2 Sec
Prewet #4	Reagent Water	10 Sec	2 Sec
Sample Process			
Air Dry			30 Sec
Rinse Step #1	Acetone	3:00 Min	20 Sec
Rinse Step #2	Methylene Chloride	3:00 Min	20 Sec
Rinse Step #3	Methylene Chloride	1:00 Min	20 Sec
Rinse Step #4	Methylene Chloride	1:00 Min	20 Sec
Rinse Step #5	Methylene Chloride	1:00 Min	1:00 Min

Table 2 Conditions used for the DryVap Concentrator System

Parameter	Setting
Dry Volume	20
Heat Power	5
Auto Rinse Mode	OFF
Heat Timer	OFF

Results

The results for the selected EPA Method 8270D compounds are shown in Table 3, listing the compound names and percent recovery. Figure 1 shows the chromatogram of the selected EPA Method 8270D compounds run on the GC/MS.

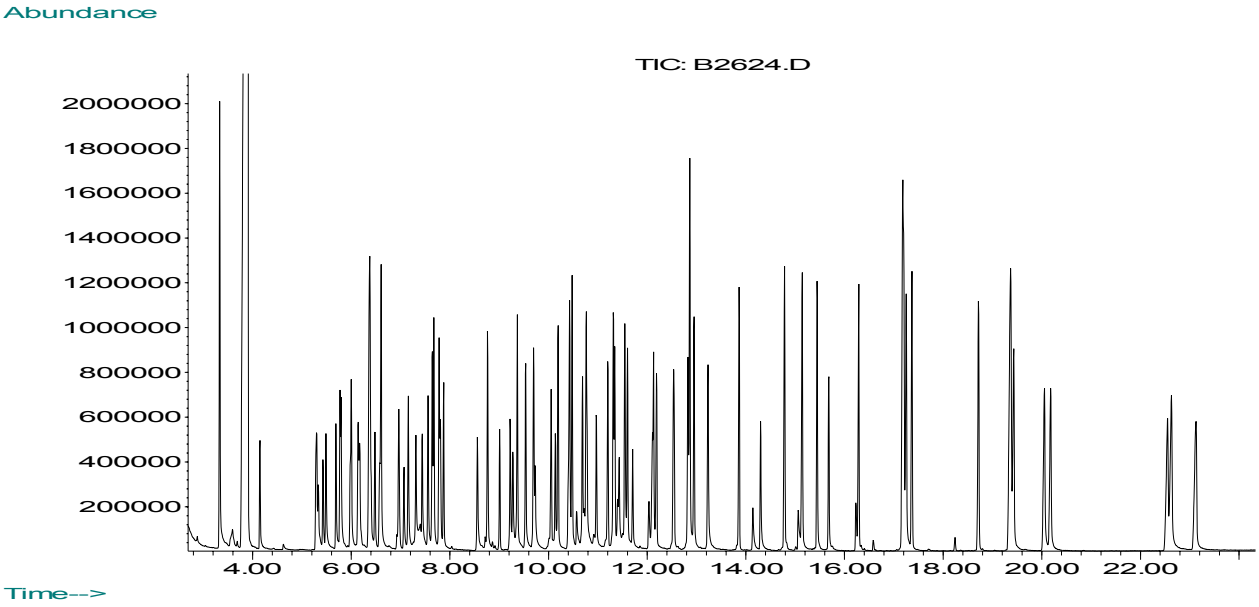
Horizon Technology's fully-automated solid-phase extraction systems using the Oasis HLB SPE disk had a recovery range between 29%-103% for ninety two compounds. 95% of all the compounds were recovered between 60-110%.

Table 3 Concentration and % recovery of compounds

Compound Name	% Recovery
2-Flourophenol	80.04
Phenol-d6	73.30
Phenol	73.76
Aniline	35.34
Bis(2-chloroethyl)ether	73.56
2-Chlorophenol	79.18
1,3-Dichlorobenzene	65.26
1,4-Dichlorobenzene	66.20
1,2-Dichlorobenzene	69.52
Benzyl alcohol	72.04
2-Methyl phenol	83.64
Bis(2chloroisopropyl)ether	79.38
Acetophenone	81.80
3+4 Methyl phenol	83.24
N-nitroso-di-n-propylamine	80.50
Hexachloroethane	62.78
Nitrobenzene-d5	81.68
Nitrobenzene	77.84
Dimethylaniline	57.36
Isopherone	85.64
2-Nitrophenol	84.82
2,4-Dimethylphenol	84.30
Bis(2-chlorethoxy)methane	84.42
Benzoic acid	90.52
2,4-Dichlorophenol	82.32
1,2,4-Trichlorobenzene	75.48
Napthalene	79.78
2,6-Dichlorophenol	85.62
4-Chloroaniline	68.48
Hexachloropropene	58.16
Hexachlorobutadiene	69.64
4-Chloro-3-methylphenol	86.20
2-Methylnapthalene	82.90
Hexachlorocyclopentadiene	66.20
2,4,6-Trichlorophenol	88.14
2,4,5-Trichlorophenol	78.42
2-Flourobiphenol	87.28
2-Chloronapthalene	84.44
Diphenyl ether	85.60
2-Nitroaniline	93.68
Dimethyl phthalate	91.04
2,6-Dinitrotoluene	92.10
Acenaphthylene	86.10
3-Nitroaniline	81.50
Acenaphthene	87.58

Compound Name (cont.)	% Recovery
2,4-Dinitrophenol	102.88
Pentachlorobenzene	84.08
4-Nitrophenol	83.16
Dibenzofuran	88.26
2,4-Dinitrotoluene	91.02
2,3,4,6-Tetrachlorophenol	89.78
Diethyl phthalate	93.14
Fluorene	90.80
4-Chlorophenyl phenyl ether	90.28
4-Nitroaniline	83.58
4,6-Dinitro-2-methylphenol	95.36
Diphenylamine	90.46
Azobenzene	84.58
2,4,6-Tribromophenol	80.62
1,3,5,-Trinitrobenzene	87.60
Phenacetan	90.42
4-Bromophenyl phenyl ether	87.02
Hexachlorobenzene	88.14
Pentachlorophenol	86.28
Pentachloronitrobenzene	87.74
Dinoseb	88.66
Phenanthrene	90.56
Anthracene	86.98
Carbazole	90.26
Di-n-butyl phthalate	90.22
4-Nitroquinoline-1-oxide	64.12
Methapyrilene	70.60
Flouranthene	88.14
Benzidine	50.00
Pyrene	86.70
p-Terphenyl-d14	86.74
Dimethylaminoazobenzene	89.70
3,3`Dimethylbenzidine	28.90
Butyl benzl phthalate	90.32
3,3'-Dichlorobenzidine	83.16
Benz(a)anthracene	87.10
Chrysene	88.40
Bis(2-ethlyhexyl)phthalate	90.76
Di-n-octyl phthalate	85.64
7,12-Dimethylbenz(a)anthra	82.76
Benzo(b)fluoranthene	85.20
Benzo(k)fluoranthene	88.20
Benzo(a)pyrene	83.84
Indeno(1,2,3-cd)pyrene	84.90
Dibenz(ah)anthracene	80.22
Benzo(ghi)perylene	86.58

Figure 1 Chromatogram of selected EPA Method 8270D compounds analyzed on the GC/MS



Conclusions

The content in this application note demonstrates that the Horizon Technology fully-automated extraction, drying and concentration systems used with the Oasis HLB disk are capable of fully-automating selected EPA Method 8270D compounds, resulting in data that is both accurate and precise. Extraction times were typically 20 min for each pH portion and drying and concentrating times were approximately 40 minutes.

The Horizon Technology SPE-DEX 4790 Automated Extractor System, coupled with the Envision Platform, DryVap Concentrator System and the Reclaimer Solvent Recovery System reduces analyst labor, solvent usage, turnaround time, and greatly improves accuracy and precision.