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Overcoming Sample Prep Challenges in Environmental Analysis

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Benefits of Solid-Phase Extraction in Sample Preparation

Developments in solid phase extraction are alleviating many sample preparation challenges faced by environmental laboratories.

Interview with William Jones

To reduce operating costs and maximize profits, environmental laboratories must become much more efficient in terms of materials, labor, and time. Solid-phase extraction (SPE), which can be automated, produces more consistent results than manual labor techniques and streamlines the process to increase sample throughput. Here, William Jones, Director of Chemical R&D at Biotage, discusses recent SPE developments such as the evolution to multi-modal disks that accommodate complex chemistry in a single pass, the move toward anion exchange resins, and future directions such as the trend toward field sampling.

LCGC: What types of challenges (e.g., in sample preparation) do environmental laboratories face today?

Jones: Today's environmental laboratories face numerous challenges. One, in particular, is high sample-throughput demand. A lot of people need many samples processed, and there is a finite amount of time in which to complete sample analysis. There are often limits on how long a sample can be held before it expires and the laboratory must resample. Thus, laboratories must have speed and efficiency in processing samples.

Sample matrices in environmental laboratories can vary dramatically. These can range from clean drinking water samples, to semi-dirty samples such as groundwater, to extremely dirty samples such as wastewater. Environmental laboratories are constantly trying to reduce operating costs and maximize profits. That means laboratories must be much more efficient in terms of materials, labor, and processing time.

One problem faced by environmental laboratories is high technician turnover rates. New employee training must be conducted frequently. In addition, technicians often have different sample preparation skill sets and that variation must be dealt with. Such variation could cause problems in the consistency and reliabil-

ity of results. Many sample preparation techniques that are still in use today were established years ago and are based on technology that was available at the time. Some of these techniques are dictated by the U.S. Environmental Protection Agency (EPA); other techniques may be carryovers from other regulated environmental test methods. However, sample preparation usually involves a lot of manual effort, which can sometimes be the bottleneck that hinders productivity.

LCGC: How does SPE help alleviate some of these challenges?

Jones: SPE reduces the amount of extraction solvent needed to prepare samples. It also eliminates emulsion formation, which is a problem commonly encountered with liquid–liquid extraction (LLE). When mixing a water sample with a nonpolar solvent, shaking it to perform the extraction, and then extracting analytes from the water into the organic solvent, the sample components can form an emulsion. In that case, the technician must try to break up the emulsion to obtain the organic solvent as part of the sample.

In SPE, the sample passes through a disk and does not come into contact with any solvents; therefore, emulsions are eliminated. In addition, SPE can be automated, which will produce more consistent results than a manual process such as LLE. Through automation, SPE also reduces processing time and solvent usage. SPE can capture target compounds while the sample passes through the disk, whereas LLE requires multiple extraction steps because the process is based on a solution equilibrium.

LCGC: Can you talk about the evolution of these techniques?

Jones: Several forces have driven SPE in certain directions to meet market needs and analytical goals set by regulatory agencies (e.g., International Organization for Standardization [ISO], US EPA). For SPE, there has been an evolution from cartridges to disks because 1 liter is often dictated as the sample size for extraction. Trying to pass a 1-liter sample through a cartridge limits the flow rate (e.g., 10 milliliters per minute) due to the smaller surface area in this media configuration.

With the larger surface area of disks, the sample flow rate increases dramatically from 100 to 200 milliliters per minute. Thus, passing a 1-liter sample through a cartridge may take 100 minutes, whereas using an SPE disk can reduce processing time down to 5 or 10 minutes. Disks have also evolved from a standard size of 47 millimeters to 90 millimeters for selected applications to increase their chemical capacity.

The increase in physical size and chemical capacity can be particularly beneficial for higher-throughput applications with challenging sample matrices, such as oil and grease extractions. In this example, it is possible to extract milligrams (versus micrograms) of material, which is eluted with hexane and evaporated such that a gravimetric measurement can be made. Challenging sample matrices like these have spurred the development of reusable disk holders and custom prefilter

stacks that use benign materials (e.g., microglass fiber). This extraction approach has helped tremendously when working with samples containing high levels of solid materials.

When extracting compounds from environmental samples, particulates and suspended solids can interfere with the media's ability to effectively interact with the target analytes in solution. Filter stacks remove solid materials from the sample matrix, allowing the solution to pass through without interference from the matrix components. Modern filters use relatively inexpensive materials and can often be mixed and matched to accommodate the particulate level for each sample. This improves the accuracy and reproducibility of each extraction without adding a significant amount of cost.

Along with the development of various disk holders, there has been a push to develop a wider range of media to accommodate increasing compound lists and application challenges. These offerings have been expanded to include ion exchange resins and multi-mode disks, just to name a couple of examples. Disks may contain a blend of resins, offering a range of capacities and the ability to capture a wider class of compounds in a given sample run, rather than doing multiple extractions of each sample.

Disks have also been created for the purpose of drying samples. These disks offer a much more convenient means to remove residual water from organic solvent extracts prior to concentration. Before the creation of disk membranes for drying, techniques for drying solvents typically included the use of sodium sulfate, which has several challenges associated with its use, handling and storage.

There has also been an evolution from reusable to disposable disk holders. Disposable disks offer speed and convenience for high-throughput laboratories that need to maximize productivity while reducing overhead costs. Disposable disks eliminate the time that would have been spent cleaning disk holders, while eliminating the risk of sample-to-sample carryover.

LCGC: Can you provide an example of a specific application?

Jones: One example is the development of the Atlantic® One Pass disk, a multi-mode disk that allows hundreds of compounds to be retained with a single pass of a sample solution. The One Pass disk was designed to tackle the challenges of EPA Method 8270, a method for extracting semi-volatile organics from wastewater. Method 8270 contains a list of more than 300 semi-volatile organic compounds. There are approximately 14 different classes of compounds that are categorized as being acidic, basic, neutral, or hydrophilic.

With the One Pass disk, in combination with a carbon cartridge, all of these compounds can be captured in a single run. Comparable speed and efficiency are nearly impossible to achieve with LLE when trying to capture compounds from a list of 300, ranging from extremely hydrophobic to extremely water soluble. It is hard to take compounds that are extremely

water soluble into a hydrophobic solvent. Examples include recoveries for key compounds listed in Method 8270 such as cancer-forming agents (e.g., methyl methanesulfonate) or suspected carcinogens (e.g., N-Nitrosodimethylamine [NDMA]). Recoveries for these compounds are typically poor when using LLE.

The One Pass disk is successful because it contains media with three different retention mechanisms. The mixed-mode disk contains ion exchange resins, as well as an HLB resin to give the disk hydrophilic and lipophilic properties. Under acidic conditions (pH=2), this disk will retain the acidic, neutral and basic analytes outlined in Method 8270. The sample passes through the disk, then to the carbon cartridge to retain the extremely hydrophilic compounds (these are compounds that are typically volatile and have relatively low boiling points). Both the One Pass disk and the carbon cartridge are geared to handle high flow rates, making this setup appropriate for environmental laboratories that are trying to process large sample volumes.

LCGC: Where do you see SPE headed in the future?

Jones: I can see SPE heading in a couple of directions. Right now, there is concern about rapid proliferation of perfluorinated compounds and their intermediates. They come from materials such as Teflon and fire retardants, which persist in the environment. Current EPA methods look for some of these compounds (e.g., perfluorooctanoic acid [PFOA] and perfluorooctane sulfonate [PFOS]) using a reverse-phase resin (styrene divinylbenzene).

Styrene divinylbenzene is ideal for capturing hydrophobic compounds. However, regulators have banned PFOA and PFOS compounds, so manufacturers have been forced to use smaller-chain carbon compounds in their intermediates. These smaller-chain compounds were thought to be healthier in the environment. However, it has been discovered that these "Gen X" compounds are persisting and building up in the environment, just like the longer-chain perfluorinated compounds. They are now being monitored.

The problem with these new compounds is that they are not retained well using traditional methodologies and resins. Therefore, it is necessary to use different SPE modes such as weak anion exchange, WAX-type resins, which capture these anionic compounds more effectively. The carbon footprints of these compounds are too small to be captured effectively by traditional reverse-phase resins.

Another future direction is the use of disks in the field. There has been a lot of interest in using SPE in field sampling. Obtaining samples in the field has led to the discovery that the nature of the disk makes it more amenable to longer-term sample storage. Since compounds are captured directly onto a porous resin, they remain more stable for transport to the laboratory, compared to keeping them in solution for transport. Pore sizes can vary between disks, but if they are smaller

Benefits of Solid-Phase Extraction

than 90 angstroms (which is typical of many resin-based materials), any bacteria present in the sample will be unable to gain access to captured compounds. Thus, sample degradation as a result of being consumed by bacteria is significantly reduced, and the quality of the sample brought to the laboratory is greatly increased.

William Jones
Director of Chemical R&D, Biotage

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Application Note

Improving Horizon Technology Disk Extraction Technology for US EPA Wastewater Method 625.1

Alicia Cannon, Melissa Lever, and Michael Ebitson

Introduction

The US EPA monitors a variety of chemicals in water that may cause harm to humans or wildlife in order to minimize exposure. Method 625 was developed by the Office of Science and Technology in the Clean Water program to monitor a large suite of semivolatile chemicals in wastewater for compliance with the National Pollution Discharge Elimination System (NPDES). NPDES is a system of permitting that regulates the characteristics of water that are released into a waterway, defined by industrial category. The permitting levels are set depending on the waterway's use. If the waterway is used for recreation or is an important wildlife habitat, the limit may be set lower.

The original method was developed in the early 1980s and has been updated several times since then to allow for the use of more modern technology. The latest update has taken place over the last few years and was proposed in a Method Update Rule (MUR) in 2015 (1). The latest version of the method includes a larger suite of analytes (up to 364) and an extensive set of labeled surrogates to better monitor the method performance throughout sample preparation and analysis steps (2).

This application note examines the results from an initial demonstration of capability (DOC) with one liter samples utilizing automated disk solid phase extraction (SPE) for US EPA Method 625.1. Samples were evaluated and measured against the criteria listed in Table 6 of Method 625.1. The analytes chosen for evaluation were from Tables 1, 2 and 3 in Method 625.1. Analytes from Table 3 do not have any acceptance criteria for comparison.

Experimental

One liter samples were processed and laboratory control samples (LCS), matrix spike, matrix spike (MS), matrix spike duplicate (MSD) and calibration verifications were evaluated. The samples were extracted using an Atlantic® One Pass SPE disk (Horizon Technology), which is a

Improving Horizon Technology Disk Extraction Technology

Figure 1: SPE-DEX® 5000 Extractor

mixed mode disk containing several functionalities. The process was automated with the SPE-DEX® 5000 (Horizon Technology) extraction system. A carbon cartridge (Max Detect, Horizon Technology) was also used to ensure adequate retention of the light end compounds, such as N-Nitrosodi-n-propylamine.

The Fast Flow Disk Holder (FFDH) was used with the samples because this method is known for varying levels of particulate matter and the sample size was 1L. The FFDH uses a 47mm disk, but allows larger filters to be placed on top to shield the SPE disk from particulates that may cause clogging and keep the flow through the disk fast. The particulate material is retained on the filters and washed with solvent during the elution step, so any material that has been absorbed on the surface will be included in the extraction.

Figure 1 shows a photo of the SPE-DEX® 5000 (Horizon Technology) configured with the One Pass kit, Carbon Cartridge and the Fast Flow Disk Holder.

The resulting extracts were dried using the DryDisk® membrane drying system (Horizon Technology) and evaporated to 1 mL with the DryVap® In-line Drying and Evaporation System (Horizon Technology). The DryVap was chosen for its ability to in-line dry and concentrate extracts at a very low boiling point. Those two features preserve the concentrations of the extracted compounds throughout the drying and concentration process, compared to traditional multistep processes. **Figure 2** shows the DryVap system with DryDisk glassware holding the drying membranes in front. The dried extract is drawn into the concentration tube in the back and the evaporation occurs there. Residual water remains in the front reservoirs separated from the extract.

The extraction method used with the SPE-DEX 5000 is shown in **Table 1**. The conditions for operating the DryVap are shown in **Table 2.**

The samples were measured using a GC/MS (6890GC/5975CMS, Agilent Technologies). The operational conditions are shown in **Table 3.**

All spiking standards used were from (Supelco, Bellefonte, PA). The surrogate mixes were from (Restek Corp, Bellefonte, PA).

Figure 2: DryVap System

Results and Discussion

Table 6 in US EPA Method 625.1 (December 2014) lists criterion by analytes for a variety of characteristics to validate that the method applied with changes will meet the requirements of the original method for a variety of challenging matrices; representative of those that may be encountered in a commercial laboratory. This table is included in Appendix 1 for easy reference.

The first column of Table 6 in Method 625.1 is range in % for recovery of the calibration verification standard. The results for this standard during the testing of the extracts is shown in **Table 4** and meets the criteria listed in Method 625.1 for compliance.

Before running samples, the laboratory must first demonstrate their capability to use a method with an initial DOC, running four spiked reagent water samples through the complete sample preparation and analysis step. The results for recovery accuracy and precision are compared with the range specified for compliance. The range is included in **Table 5** for easy comparison. Analytes with a * indicate that they are from Table 3 and do not have acceptance criteria in Table 6 within Method 625.1. It is up to the laboratory to generate their own acceptance criteria for Table 3 analytes.

The criteria was met in all cases for Table 6 analytes in Method 625.1, indicating the analyte list is under control and further analysis can proceed.

Table 6 demonstrates the results for the MS/MSD. The spike recovery data from the sample is shown in the second column, alongside the range criteria for Table 6 analytes in Method 625.1. The spike and surrogates were added at 100 µg/mL in each liter of sample. The relative percent difference between the spike recovery and spike recovery duplicate is calculated using the equation in Method 625.1. If the data meets the criterion, a pass is indicated in the Pass/Fail column. Analytes with a * indicate that they are from Table 3 and do not have acceptance criteria in Table 6 within Method 625.1. It is up to the laboratory to generate their own acceptance criteria for Table 3 analytes.

As the data indicates, matrix spikes were recovered within the range specified in Table 6 in Method 625.1. The relative percent difference (RPD) between the matrix spike and duplicate sample showed excellent agreement when compared to the limit allowed. The agreement was generally well below the limit, sometimes more than an order of magnitude better.

Conclusion

US EPA Method 625 is an important

Table 1: SPE-DEX 5000 Extraction Method

Step	Operation	Message						Attachment	
1	Pause with Message	Part 1 of 3: Neutrals and Acids Elution. Have the Fast Flow Sediment Disk Holder with OnePass Disk, 1 µm filter, 5 µm filter, top screen over the filters, 250 mL collection flask, and carbon cartridge installed. The down spout of the water in valve must push down on the top screen. Click "Continue" to start Part 1.						None	
Step	**Operation**	**Solvent**	**Solvent Vol. (mL)**	**Purge Time (s)**	**Pump Rate (#)**	**Sat. Time (s)**	**Soak Time (s)**	**Drain Time (s)**	
2	Condition SPE	Acetone	40	60	4	2	60	60	
3	Condition SPE	Reagent Water	20	60	4	2	60	60	

method for evaluation of water pollution or clean-up. It allows a full suite of analytes to be evaluated at one time using GC/MS. Sample preparation is an important part of the process and disk solid phase extraction can provide advantages in using less solvent, eliminating emulsions and providing good extraction across the suite of analytes considered while minimizing exposure.

A number of analytes within Tables 1-3 were extracted simultaneously from a 1-liter sample, which demonstrates the advantage of SPE when facing a challenging extraction and many potential interferences. The results show excellent performance of the One-Pass disk coupled with a Carbon Cartridge for capture of the analytes. Reduced sample volumes can be used for this method to improve performance as long as all Method Detection Limits (MDLs) and reporting limits are met.

Solid phase extraction disks are another tool for the environmental laboratory to consider when evaluating their workflow for increased efficiency and safety. Additional benefits include reduced solvent usage, as well as reduced maintenance and overhead costs. Automating your SPE workflow increases data accuracy and reproducibility. Excellent duplicate agreement was shown here, even for the more difficult samples. Overall, the demonstrated analytical performance meets the criteria required by EPA Method 625.1 and adds to the other benefits of bringingnewer technology into today's modern laboratory.

References

1. Method Update Proposed Rule, *Federal Register,* February 19, 2015, page 8946.

Table 1 (cont'd): SPE-DEX 5000 Extraction Method

Step	Operation		Sample Flow Rate (#)	Done Loading Sample Delay (s)					
4	Load Sample		5	45					
Step	**Operation**	**Solvent**	**Solvent Vol. (mL)**	**Purge Time (s)**	**Pump Rate (#)**	**N2 Blanket**	**Sat. Time (s)**	**Soak Time (s)**	**Drain Time (s)**
5	Wash Sample Container	Reagent Water	20	30	4	Off	2	5	30
Step	**Operation**		**Dry Time (s)**	**Pump Rate (#)**	**N2 Blanket**				
6	Air Dry Disk Timer		360	6	Off				
Step	**Operation**	**Solvent**	**Solvent Vol. (mL)**	**Purge Time (s)**	**Pump Rate (#)**	**N2 Blanket**	**Sat. Time (s)**	**Soak Time (s)**	**Elute Time (s)**
7	Elute Sample Container	Acetone	20	20	4	Off	2	180	180
8	Elute Sample Container	$MeCl_2$	17	15	4	Off	2	180	180
9	Elute Sample Container	$MeCl_2$	17	15	4	Off	2	120	120
10	Elute Sample Container	$MeCl_2$	17	15	4	Off	2	120	120
11	Elute Sample Container	$MeCl_2$	17	15	6	Off	2	120	180
Step	**Operation**	**Message**						**Attachment**	
12	Pause with Message	Part 2 of 3: Ion Exchange Elution. Remove the 250 mL collection flask containing the neutrals and acids elution. Stopper the flask and set aside for part 3. Then install a clean 125 mL flask to collect the ion exchange elution. Click "Continue" to Start Part 2.						None	

2. Method 625.1, December 14 revision, can be found in the MUR, February 20, 2014. Or downloaded here: https://nepis.epa.gov/Exe/ZyPDF.cgi/P100LVHC.PDF?Dockey=P100LVHC.PDF

Table 1 (cont'd): SPE-DEX 5000 Extraction Method

Step	Operation	Solvent	Solvent Vol. (mL)	Purge Time (s)	Pump Rate (#)	N2 Blanket	Sat. Time (s)	Soak Time (s)	Elute Time (s)
13	Elute Sample Container	Acetone	20	20	4	Off	2	0	180
14	Elute Sample Container	1% NH_4OH	20	30	4	Off	2	120	120
15	Elute Sample Container	Acetone	20	20	4	Off	2	180	120
16	Elute Sample Container	$MeCl_2$	17	15	4	Off	2	180	180
17	Elute Sample Container	$MeCl_2$	16	15	4	Off	2	120	180
18	Elute Sample Container	$MeCl_2$	16	15	4	Off	2	120	180
19	Elute Sample Container	$MeCl_2$	16	15	6	Off	2	120	180
Step	Operation	Message						Attachment	
20	Pause with Message	Part 3 of 3: Carbon Cartridge Elution. Remove the carbon cartridge from the tubing lines. Connect the tubing ends together. Using a 20 cc syringe, plunge the carbon cartridge with air through the cap adapter to reseat the carbon bed on the frit. Replace the cap adapter with the funnel cartridge. Replace the disk holder with the cartridge. Replace the 125 mL flask with the 250 mL flask containing the neutrals and acids elution from Part 1. Stopper the 125 mL flask. Click "Continue" to start part 3.						None	
Step	Operation		Dry Time (s)	Pump Rate (#)	N2 Blanket				
21	Air Dry Disk Timer		60	6	Off				
Step	Operation	Solvent	Solvent Vol. (mL)	Purge Time (s)	Pump Rate (#)	N2 Blanket	Sat. Time (s)	Soak Time (s)	Elute Time (s)
22	Elute Sample Container	Acetone	25	20	4	Off	3	60	60
23	Elute Sample Container	$MeCl_2$	17	15	4	Off	3	60	20
24	Elute Sample Container	$MeCl_2$	17	15	4	Off	3	60	20
25	Elute Sample Container	$MeCl_2$	17	15	4	Off	3	60	20
26	Elute Sample Container	$MeCl_2$	17	15	4	Off	3	60	20
27	Elute Sample Container	$MeCl_2$	17	15	6	Off	3	60	60

Table 2: DryVap System Conditions

Parameter	Setting
Dry Volume	200 mL
Heat Power	5
Heat Timer	OFF
Auto Rinse Mode	OFF
Nitrogen Sparge	20 psi
Vacuum	-7 in. Hg

Table 3: GC/MS Conditions

Injection	
Amount	1 µL
Inlet Temperature	280°C
Mode	Splitless
Gas Type	Helium
Column Conditions	**Zebron™ ZB-Semivolatiles (Phenomenex)**
Mode	Consistent Flow
Oven Program	45°C hold for 1 min to 270°C at 15°C/min then to 318 °C at 6 °C/min
MS Ions Monitored	Scan masses 35-550

Table 4: Calibration Verification over the Course of Operation

Analyte	Recovery (%)	Recovery (%)	Recovery (%)	Recovery (%)	Recovery (%)	Recovery (%)	Range for Q %	Pass/ Fail
NDMA	102.9	94.4	96.9	97.8	97.6	94.1	60-140	Pass
1,2,4,5-Tetrachlorobenzene	98.0	97.5	95.0	101.9	100.8	97.2	60-140	Pass
1,2,4-Trichlorobenzene	97.7	98.4	95.0	101.2	100.9	97.2	61-130	Pass
1,3,5,-Trinitrobenzene	95.5	94.7	92.1	97.1	94.6	92.8	60-140	Pass
1,4-Naphthoquinone	94.8	94.7	92.6	97.9	95.4	92.0	60-140	Pass
1-Naphthylamine	85.5	82.6	82.8	87.8	82.6	82.4	60-140	Pass
2,3,4,6-Tetrachlorophenol	93.9	93.5	93.5	98.1	96.3	93.6	60-140	Pass
2,4,5-Trichlorophenol	95.1	94.8	93.9	99.2	95.9	93.0	60-140	Pass
2,4-Dichlorophenol	96.7	96.2	94.4	99.5	96.3	93.1	64-130	Pass
2,4-Dimethylphenol	98.0	97.9	95.2	99.9	97.2	93.8	58-130	Pass
2,4-Dinitrophenol	80.2	81.2	85.7	87.9	85.6	82.1	39-173	Pass
2,4-Dinitrotoluene	93.5	94.2	94.0	98.6	96.9	93.9	53-130	Pass
2,6-Dichlorophenol	95.8	96.5	94.9	99.2	96.3	93.7	60-140	Pass
2,6-Dinitrotoluene	94.5	94.3	92.6	98.9	96.3	93.5	68-137	Pass
2-Chloronaphthalene	97.8	97.4	94.7	100.2	99.6	96.0	70-130	Pass
2-Chlorophenol	97.4	96.6	94.6	98.9	99.2	94.7	55-130	Pass
2-Fluorobiphenyl	99.0	97.9	97.3	100.0	92.5	91.0	60-140	Pass
2-Fluorophenol	99.9	95.8	97.8	95.4	96.3	94.3	60-140	Pass
2-Methylnaphthalene	96.7	98.4	95.3	100.5	98.8	95.3	60-140	Pass
2-Naphthylamine	68.6	70.4	66.2	71.8	65.5	66.7	60-140	Pass
2-Nitroaniline	95.1	94.3	93.4	98.1	94.9	92.5	60-140	Pass
2-Nitrophenol	95.2	96.5	93.7	98.2	96.1	93.7	61-163	Pass
3,3'-Dichlorobenzidine	107.2	106.0	100.4	108.2	102.7	102.2	18-213	Pass
3,3'-Dimethylbenzidine	80.2	84.5	80.1	92.8	86.1	88.0	60-140	Pass
3-Methylcholanthrene	95.0	96.1	92.4	99.4	95.9	94.4	60-140	Pass
3-Nitroaniline	99.2	96.0	96.2	100.6	97.8	96.1	60-140	Pass
4 Aminobiphenyl	87.9	88.2	79.6	86.2	79.9	80.4	60-140	Pass
4,6-Dinitro-2-methylphenol	89.0	90.5	90.9	96.2	94.0	90.7	56-130	Pass
4-Bromophenyl phenyl ether	96.6	99.8	96.0	103.5	100.8	98.1	70-130	Pass
4-Chloro-3-methylphenol	94.8	96.5	95.2	97.7	96.0	91.9	68-130	Pass
4-Chloroaniline	74.7	86.9	75.3	87.5	85.3	82.5	60-140	Pass
4-Chlorophenyl phenyl ether	95.3	97.9	94.9	101.0	98.8	97.5	57-145	Pass
4-Nitroaniline	96.3	92.4	94.9	94.6	94.6	89.9	60-140	Pass
4-Nitrophenol	96.4	89.3	91.5	91.7	90.0	86.8	35-130	Pass
4-Nitroquinoline-1-oxide	89.7	95.9	87.2	104.1	94.4	101.0	60-140	Pass
5-nitro-o-toluidine	94.3	92.9	92.7	98.6	96.3	93.5	60-140	Pass

Table 4 (continued): Calibration Verification over the Course of Operation

	Recovery (%)	Recovery (%)	Recovery (%)	Recovery (%)	Recovery (%)	Recovery (%)	Range for Q	Pass/ Fail
7,12-Dimethylbenz(a)-anthracene	94.6	98.2	95.0	100.2	98.8	95.1	60-140	Pass
Acenaphthene	97.1	96.7	94.6	100.4	97.7	95.0	70-130	Pass
Acenaphthylene	96.9	96.5	94.6	100.5	97.2	94.6	60-130	Pass
Acetophenone	96.7	96.7	94.2	100.0	99.0	95.2	60-140	Pass
Acetylaminofluorene	97.0	95.8	92.1	98.5	94.4	93.7	60-140	Pass
Aniline	90.6	90.6	91.0	96.9	93.3	93.1	60-140	Pass
Anthracene	98.1	98.0	94.5	100.1	98.6	95.0	58-130	Pass
Benz(a)anthracene	96.6	95.1	94.1	98.2	97.2	94.3	42-133	Pass
Benzidine	81.9	84.7	76.3	87.4	82.0	85.3	60-140	Pass
Benzo(a)pyrene	96.5	96.0	93.7	99.3	97.1	94.7	32-148	Pass
Benzo(b)fluoranthene	93.7	94.6	92.2	98.2	96.2	93.2	42-140	Pass
Benzo(ghi)perylene	96.4	93.8	91.3	97.1	92.8	91.1	13-195	Pass
Benzo(k)fluoranthene	97.5	99.3	97.7	101.1	100.2	96.6	25-146	Pass
Benzoic acid	93.6	89.8	92.1	92.9	87.5	86.7	60-140	Pass
Benzyl alcohol	97.1	96.5	94.3	99.1	97.4	94.6	60-140	Pass
Bis(2-chlorethoxy)methane	96.7	98.9	94.3	100.6	98.4	95.5	52-164	Pass
Bis(2-chloroethyl)ether	99.6	99.8	94.7	100.1	99.1	96.5	60-140	Pass
Bis(2chloroisopropyl)ether	95.7	98.8	94.0	102.1	99.7	98.4	63-139	Pass
Bis(2-ethylhexyl) phthalate	85.3	98.6	91.0	102.3	99.9	98.4	43-137	Pass
Carbazole	100.0	96.6	95.1	98.2	97.6	93.5	60-140	Pass
Chrysene	97.4	96.1	94.8	98.1	97.6	93.9	44-140	Pass
Dibenz(ah)anthracene	91.2	93.5	91.1	98.2	94.2	92.5	13-200	Pass
Dibenzofuran	97.0	96.4	94.8	100.7	98.4	95.1	60-140	Pass
Diethyl phthalate	92.5	96.3	93.3	101.4	98.5	97.0	47-130	Pass
Dimethyl phthalate	93.7	95.7	93.3	99.4	97.4	94.4	50-130	Pass
Dimethylaminoazoben-zene	90.2	97.7	93.3	99.5	97.9	95.5	60-140	Pass
Di-n-butyl phthalate	90.9	100.1	91.1	101.8	100.8	98.7	52-130	Pass
Di-n-octyl phthalate	89.3	99.2	92.2	102.3	99.4	98.4	21-132	Pass
Dinoseb	89.8	94.1	90.0	98.5	97.1	94.2	60-140	Pass
Diphenylamine	96.2	96.3	95.3	99.4	97.9	95.3	60-140	Pass
Ethylmethane Sul-fonate	96.8	95.2	94.8	98.6	97.9	95.9	60-140	Pass
Fluoranthene	98.4	96.9	94.8	97.4	97.7	95.1	47-130	Pass
Fluorene	95.6	96.5	93.9	99.4	98.5	94.8	70-130	Pass
Hexachlorobenzene	96.1	99.6	94.4	101.3	99.4	96.2	38-142	Pass
Hexachlorobutadiene	97.9	101.3	95.5	104.7	103.5	100.3	68-130	Pass
Hexachlorocyclopenta-diene	89.9	92.4	88.2	99.4	96.7	94.2	60-140	Pass

Table 4 (continued): Calibration Verification over the Course of Operation

	Recovery (%)	*Recovery (%)*	*Recovery (%)*	*Recovery (%)*	*Recovery (%)*	*Recovery (%)*	*Range for Q*	*Pass/ Fail*
Hexachloroethane	*97.9*	*100.3*	*93.8*	*102.9*	*102.9*	*100.7*	55-130	Pass
Hexachloropropene	*97.5*	*98.9*	*93.8*	*101.4*	*100.6*	*97.1*	60-140	Pass
Indeno(1,2,3-cd)pyrene	*94.3*	*93.3*	*89.6*	*95.2*	*90.8*	*88.6*	13-151	Pass
Isophorone	*95.6*	*98.8*	*93.8*	*100.1*	*98.0*	*95.1*	52-180	Pass
Methapyrilene	*90.2*	*94.0*	*89.5*	*97.1*	*94.8*	*92.9*	60-140	Pass
Naphthalene	*98.5*	*97.9*	*95.0*	*100.2*	*98.8*	*95.6*	70-130	Pass

Table 5: Initial Demonstration of Capability

Analyte	Average DOC	DOC Range	Pass/Fail	SD	Limit for s (%)	Pass/Fail
1,2,4,5-Tetrachlorobenzene*	61.89			5.24		
1,2,4-Trichlorobenzene	57.69	*57-130*	*Pass*	5.08	30	*Pass*
1,2-Dichlorobenzene*	51.60			4.49		
1,3,5,-Trinitrobenzene*	83.54			6.65		
1,3-Dichlorobenzene*	46.37			4.58		
1,3-Dinitrobenzene*	88.29			5.80		
1,4-Dichlorobenzene*	48.39			4.84		
1,4-Naphthoquinone*	77.21			2.15		
1-Naphthylamine*	89.88			1.58		
2,3,4,6-Tetrachlorophenol*	87.10			5.02		
2,4,5-Trichlorophenol*	84.55			4.56		
2,4-Dichlorophenol	86.09	*53-122*	*Pass*	4.45	30	*Pass*
2,4-Dimethylphenol	85.77	*42-120*	*Pass*	4.31	35	*Pass*
2,4-Dinitrophenol	83.62	*D-173*	*Pass*	5.97	79	*Pass*
2,4-Dinitrotoluene	88.97	*48-127*	*Pass*	4.91	25	*Pass*
2,6-Dichlorophenol*	85.58			4.19		
2,6-Dinitrotoluene	89.83	*68-137*	*Pass*	5.49	29	*Pass*
2-Chloronaphthalene	70.88	*65-120*	*Pass*	5.97	15	*Pass*
2-Chlorophenol	77.07	*36-120*	*Pass*	5.10	37	*Pass*
2-Fluorobiphenyl*	83.45			2.46		
2-Fluorophenol*	55.75			6.09		
2-Methyl phenol*	80.66			5.19		
2-Methylnaphthalene*	68.80			5.49		
2-Naphthylamine*	109.87			6.08		

Table 5 (continued): Initial Demonstration of Capability

Analyte	Average DOC	DOC Range	Pass/Fail	SD	Limit for s (%)	Pass/Fail
2-Nitroaniline*	87.96			6.80		
2-Nitrophenol	75.83	45-167	Pass	3.66	79	Pass
2-Picoline*	47.44			2.09		
3,3'-Dichlorobenzidine	88.90	8-213	Pass	3.25	65	Pass
3,3'-Dimethylbenzidine*	94.17			2.61		
3+4 Methyl phenol*	82.47			6.41		
3-Methylcholanthrene*	84.77			5.18		
3-Nitroaniline*	91.89			4.77		
4 Aminobiphenyl*	112.58			6.89		
2-methyl-4,6-Dinitrophenol	87.21	53-130	Pass	5.66	122	Pass
4-Bromophenyl phenyl ether	82.74	65-120	Pass	4.84	26	Pass
4-Chloro-3-methylphenol	89.01	41-128	Pass	4.86	44	Pass
4-Chloroaniline*	118.22			8.03		
4-Chlorophenyl phenyl ether	79.56	38-145	Pass	4.55	36	Pass
4-Nitroaniline*	86.93		Pass	4.82		
4-Nitrophenol	92.06	13-129	Pass	4.99	79	Pass
4-Nitroquinoline-1-oxide*	90.69			7.66		
5-nitro-o-toluidine*	93.58			5.44		
7,12-Dimethylbenz(a)-anthracene*	84.98			4.36		
Acenaphthene	76.24	60-132	Pass	5.58	29	Pass
Acenaphthylene	77.33	54-126	Pass	5.81	45	Pass
Acetophenone*	75.52			3.75		
Acetylaminofluorene*	90.49			6.10		
Aniline*	80.22			6.18		
Anthracene*	85.84	43-120	Pass	6.38	40	Pass
Azobenzene*	82.72			4.95		
Benz(a)anthracene	86.41	42-133	Pass	5.20	32	Pass
Benzidine*	99.72			4.45		
Benzo(a)pyrene	86.46	32-148	Pass	5.17	43	Pass
Benzo(b)fluoranthene	87.03	42-140	Pass	4.62	43	Pass
Benzo(ghi)perylene	87.07	D-195	Pass	6.09	61	Pass
Benzo(k)fluoranthene	86.62	25-146	Pass	4.91	38	Pass
Benzoic acid*	57.15			6.57		
Benzyl alcohol*	78.97			3.41		
Bis(2-chlorethoxy)methane	82.26	49-165	Pass	3.42	32	Pass
Bis(2-chloroethyl)ether	68.31	43-126	Pass	2.05	65	Pass
Bis(2chloroisopropyl)ether	68.10	63-139	Pass	3.03	46	Pass
Bis(2-ethylhexyl) phthalate	91.34	29-137	Pass	6.66	50	Pass
Benzyl butyl phthalate	90.69	D-140	Pass	5.14	36	Pass
Carbazole*	89.33			7.42		
Chrysene	87.54	44-140	Pass	5.42	53	Pass
cis-Isosafrole*	75.74			4.98		

Table 5 (continued): Initial Demonstration of Capability

Analyte	Average DOC	DOC Range	Pass/Fail	SD	Limit for s (%)	Pass/Fail
Dibenz(ah)anthracene	87.88	*D-200*	*Pass*	4.85	75	*Pass*
Dibenzofuran*	78.22			5.60		
Diethyl phthalate	90.84	*D-120*	*Pass*	4.45	60	*Pass*
Dimethyl phthalate	89.80	*D-120*	*Pass*	4.36	110	*Pass*
Dimethylaminoazobenzene*	91.85			4.54		
Di-n-butyl phthalate	90.70	*8-120*	*Pass*	6.31	28	*Pass*
Di-n-octyl phthalate	89.40	*19-132*	*Pass*	5.25	42	*Pass*
Dinoseb*	87.13			6.29		
Diphenylamine*	87.58			5.24		
Ethylmethane Sulfonate*	75.62			3.61		
Fluoranthene	87.77	*43-121*	*Pass*	6.82	40	*Pass*
Fluorene	81.07	*70-120*	*Pass*	5.80	23	*Pass*
Hexachlorobenzene	83.49	*8-142*	*Pass*	5.44	33	*Pass*
Hexachlorobutadiene	44.63	*38-120*	*Pass*	4.47	38	*Pass*
Hexachlorocyclopentadiene*	41.53			4.33		
Hexachloropropene*	45.64			4.68		
Indeno(1,2,3-cd)pyrene	85.73	*D-151*	*Pass*	5.28	60	*Pass*
Isophorone	82.02	*47-180*	*Pass*	3.88	56	*Pass*
Methapyrilene*	89.82			4.64		
Methyl Methane Sulfonate*	49.04			2.10		
Naphthalene	66.73	*36-120*	*Pass*	5.59	39	*Pass*
NDMA*	36.34			0.68		
Nitrobenzene	72.65	*54-158*	*Pass*	3.72	37	*Pass*
Nitrobenzene-d5	77.26	*15-314*	*Pass*	4.08		
N-Nitroso-diethylamine*	67.44			2.70		
N-nitroso-di-n-butylamine*	85.87			4.26		
N-nitroso-di-n-propylamine	79.49	*14-198*	*Pass*	4.52	52	*Pass*
N-Nitrosomethyl ethylamine	58.95			1.50		
N-Nitroso-morpholine*	78.99			7.92		
N-Nitroso-piperidine*	82.98			3.96		
N-Nitroso-pyrrolidine*	83.07			4.02		
o-toluidine*	95.47			6.77		
Pentachlorobenzene*	74.93			5.07		
Pentachloroethane*	49.71			4.29		
Pentachloronitrobenzene*	85.84			5.93		
Pentachlorophenol	88.93	*38-152*	*Pass*	7.50	52	*Pass*
Phenacetin*	89.68			6.07		
Phenanthrene	86.11	*65-120*	*Pass*	6.78	24	*Pass*
Phenol	57.80	*17-120*	*Pass*	4.35	39	*Pass*

Table 5 (continued): Initial Demonstration of Capability

Analyte	Average DOC	DOC Range	Pass/Fail	SD	Limit for s (%)	Pass/Fail
Phenol-d5	57.80	*8-424*	*Pass*	4.35		
p-Terphenyl-d14*	91.52			4.98		
Pyrene	87.34	*70-120*	*Pass*	7.55	30	*Pass*
Pyridine*	32.13			2.96		
Safrole*	78.46			5.28		
trans-Isosafrole*	77.99			5.67		

Table 6: MS/MSD

	Average MS/MSD	Range	Pass/Fail	RPD	RPD (%)	Pass/Fail
Analyte	Recovery %	P,Ps(%)		%	Limit	
1,2,4,5-Tetrachlorobenzene*	67.1			9.11		
1,2,4-Trichlorobenzene	62.1	44-142	Pass	4.96	50	Pass
1,2-Dichlorobenzene*	56.5			5.31		
1,3,5,-Trinitrobenzene*	87.7			1.85		
1,3-Dichlorobenzene*	51.1			5.58		
1,3-Dinitrobenzene*	92.9			0.52		
1,4-Dichlorobenzene*	53.1			5.14		
1,4-Naphthoquinone*	82.5			4.89		
1-Naphthylamine*	92.3			5.45		
2,3,4,6-Tetrachlorophenol*	90.5			0.74		
2,4,5-Trichlorophenol*	87.5			1.04		
2,4-Dichlorophenol	89.0	39-135	Pass	0.47	50	Pass
2,4-Dimethylphenol	87.9	32-120	Pass	0.38	58	Pass
2,4-Dinitrophenol	87.5	D-191	Pass	7.00	132	Pass
2,4-Dinitrotoluene	92.9	39-139	Pass	1.44	42	Pass
2,6-Dichlorophenol*	87.8			1.90		
2,6-Dinitrotoluene	93.6	50-158	Pass	3.18	48	Pass
2-Chloronaphthalene	75.9	60-120	Pass	7.87	24	Pass
2-Chlorophenol	81.7	23-134	Pass	3.08	61	Pass
2-Fluorobiphenyl*	86.9			3.43		

Table 6 (continued): MS/MSD

	Average MS/MSD	Range	Pass/Fail	RPD	RPD (%)	Pass/Fail
Analyte	Recovery %	P,Ps(%)		%	Limit	
2-Fluorophenol*	61.8			5.05		
2-Methyl phenol*	84.8			0.68		
2-Methylnaphthalene*	72.7			4.62		
2-Naphthylamine*	113.0			2.93		
2-Nitroaniline*	92.9			1.41		
2-Nitrophenol	80.7	29-182	Pass	4.36	55	Pass
2-Picoline*	49.2			6.96		
3,3'-Dichlorobenzidine	93.3	D-262	Pass	7.92	108	Pass
3,3'-Dimethylbenzidine*	99.3			0.90		
3+4 Methyl phenol*	86.4			1.17		
3-Methylcholanthrene*	87.9			8.34		
3-Nitroaniline*	95.4			4.75		
4 Aminobiphenyl*	114.6			2.36		
2-methyl-4,6-Dinitrophenol	91.9	D-181	Pass	1.83	203	Pass
4-Bromophenyl phenyl ether	85.0	53-127	Pass	3.90	43	Pass
4-Chloro-3-methylphenol	91.1	22-147	Pass	3.06	73	Pass
4-Chloroaniline*	120.7			0.11		
4-Chlorophenyl phenyl ether	82.3	25-158	Pass	4.57	61	Pass
4-Nitroaniline*	90.7			2.92		
4-Nitrophenol	97.3	D-132	Pass	4.31	131	Pass
4-Nitroquinoline-1-oxide*	95.8			1.99		
5-nitro-o-toluidine*	96.8			3.00		
7,12-Dimethylbenz(a)-anthracene*	86.9			5.42		
Acenaphthene	80.1	47-145	Pass	6.28	48	Pass
Acenaphthylene	81.3	33-145	Pass	4.75	74	Pass
Acetophenone*	80.2			5.11		
Acetylaminofluorene*	94.7			6.09		
Aniline*	84.0			7.85		
Anthracene*	89.2	27-133	Pass	7.17	66	Pass
Azobenzene*	86.6			3.64		
Benz(a)anthracene	89.2	33-143	Pass	8.15	53	Pass
Benzidine*	105.5			9.38		
Benzo(a)pyrene	88.8	17-163	Pass	9.10	72	Pass
Benzo(b)fluoranthene	89.2	24-159	Pass	6.83	71	Pass
Benzo(ghi)perylene	90.1	D-219	Pass	11.44	97	Pass
Benzo(k)fluoranthene	89.2	11-162	Pass	7.01	63	Pass
Benzoic acid*	58.7			6.47		
Benzyl alcohol*	81.3			2.07		
Bis(2-chlorethoxy)methane	85.4	33-184	Pass	0.19	54	Pass
Bis(2-chloroethyl)ether	74.6	12-158	Pass	6.50	108	Pass
Bis(2chloroisopropyl)ether	73.7	36-166	Pass	8.23	76	Pass
Bis(2-ethylhexyl) phthalate	92.3	8-158	Pass	0.81	82	Pass

Table 6 (continued): MS/MSD

	Average MS/MSD	Range	Pass/Fail	RPD	RPD (%)	Pass/Fail
Analyte	Recovery %	P,Ps(%)		%	Limit	
Benzyl butyl phthalate	91.7	D-152	Pass	1.99	60	Pass
Carbazole*	93.1			5.94		
Chrysene	89.4	17-168	Pass	10.27	87	Pass
cis-Isosafrole*	79.9			3.64		
Dibenz(ah)anthracene	91.2	D-227	Pass	6.46	126	Pass
Dibenzofuran*	82.7			6.37		
Diethyl phthalate	94.3	D-120	Pass	1.07	100	Pass
Dimethyl phthalate	93.2	D-120	Pass	0.97	183	Pass
Dimethylaminoazobenzene*	93.4			4.06		
Di-n-butyl phthalate	93.9	1-120	Pass	5.12	47	Pass
Di-n-octyl phthalate	90.7	4-146	Pass	2.69	69	Pass
Dinoseb*	91.0			1.83		
Diphenylamine*	91.3			2.03		
Ethylmethane Sulfonate*	79.1			3.34		
Fluoranthene	91.6	26-137	Pass	6.57	66	Pass
Fluorene	85.1	59-121	Pass	5.18	38	Pass
Hexachlorobenzene	86.1	D-152	Pass	6.56	55	Pass
Hexachlorobutadiene	48.8	24-120	Pass	5.56	62	Pass
Hexachlorocyclopentadiene*	46.5			4.15		
Hexachloropropene*	49.9			5.69		
Indeno(1,2,3-cd)pyrene	89.6	D-171	Pass	11.95	99	Pass
Isophorone	85.0	21-196	Pass	0.36	93	Pass
Methapyrilene*	92.8			3.99		
Methyl Methane Sulfonate*	53.4			8.39		
Naphthalene	71.4	21-133	Pass	4.06	65	Pass
NDMA*	38.6			17.34		
Nitrobenzene	77.4	35-180	Pass	5.18	62	Pass
Nitrobenzene-d5	82.0			5.54		
N-Nitroso-diethylamine*	71.4			6.09		
N-nitroso-di-n-butylamine*	88.2			0.45		
N-nitroso-di-n-propylamine	83.6	D-230	Pass	3.64	87	Pass
N-Nitrosomethyl ethylamine	62.8			11.95		
N-Nitroso-morpholine*	82.3			13.96		
N-Nitroso-piperidine*	85.7			2.10		
N-Nitroso-pyrrolidine*	85.7			0.86		
o-toluidine*	100.1			1.58		
Pentachlorobenzene*	78.8			7.32		
Pentachloroethane*	55.3			7.13		
Pentachloronitrobenzene*	89.1			5.28		
Pentachlorophenol	93.5	14-176	Pass	3.03	86	Pass

Table 6 (continued): MS/MSD

	Average MS/MSD	Range	Pass/Fail	RPD	RPD (%)	Pass/Fail
Analyte	Recovery %	P,Ps(%)		%	Limit	
Phenacetin*	92.7			3.73		
Phenanthrene	88.8	54-120	Pass	6.07	39	Pass
Phenol	62.2	5-120	Pass	5.59	64	Pass
Phenol-d5	61.8			6.61		
p-Terphenyl-d14*	89.1			6.72		
Pyrene	90.8	52-120	Pass	7.51	49	Pass
Pyridine*	32.3			4.49		
Safrole*	83.0			3.83		
trans-Isosafrole*	82.8			1.56		

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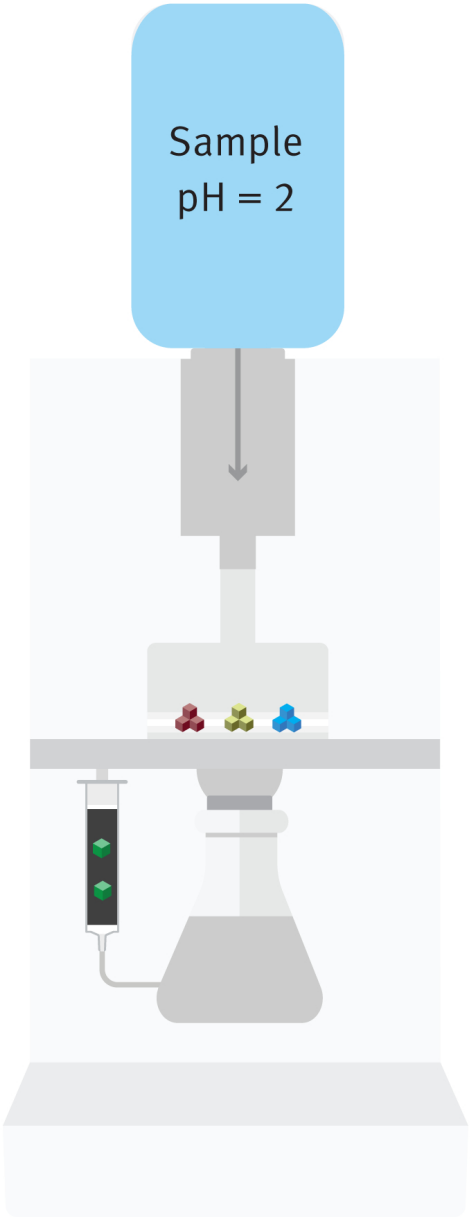

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Application Note

Improving Automated SPE Disk Extraction Technology for Semi-volatile Compounds in Groundwater Using US EPA Method 8270E

Alicia Cannon, Melissa Lever, and Michael Ebitson

Introduction

Semivolatile organic compounds (SVOC) have a variety of chemical properties that have been found to cause harmful effects to both humans and the environment. Accurate measurements are challenging to obtain because SVOCs readily adsorb onto surfaces, and are found in common household items such as cleaning agents, personal care products, electrical components, pesticides, water and food. Affects to health depend on the chemical nature of the compound in conjunction with the degree of exposure; yet have been known to include allergenic symptoms, reproductive and endocrine issues. Laboratories around the world measure these compounds in water, soil, and leachates from waste sites. US EPA Method 8270E can be used to determine the concentration of SVOCs extracted from liquid, solid and leachate samples in effort to limit exposure and the spread of these persistent organic pollutants (1).

While almost all laboratories test for less than the full list of 243 compounds included in the method, typical laboratories will often measure a large suite of 80 to 100 compounds. Compound classes that can be extracted using this method 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.

This application note will demonstrate the results of Initial Demonstration of Proficiency (IDP) evaluations for compliance with US EPA Method 8270E to determine a list of 114 semi-volatile organic compounds that are neutral, acidic, and basic. In addition to the IDP, a typical groundwater sample will be evaluated which

Figure 1. SPE-DEX 5000 Extractor with One Pass Kit

contains a different matrix and particulate content than that in the IDP. The Matrix Spike (MS)/ Matrix Spike Duplicate (MSD) are presented. Solid phase extraction is described as a suitable sample preparation alternative in Method 8270 and companion method US EPA 3535 outlines the general use of SPE. Suitable sorbent material in disk format for this list of analytes and a modern system for automation will be demonstrated (2).

Experimental

Outline of SPE Procedure

The SVOCs in this method were extracted using automated sample preparation solutions for solid phase extraction (SPE), drying and concentration. The samples were extracted using an Atlantic® One Pass Disk (Horizon Technology) which is a mixed-mode disk containing several functionalities. Two different pre-filters, Atlantic Fast Flow 1 µm and 5 µm (Hori-

Figure 2. DryVap System

zon Technology) with a fine mesh screen were used, as well as a carbon cartridge (Horizon Technology) to ensure adequate retention of difficult to obtain light end compounds, such as NDMA and N-Nitrosomethyl ethylamine.

The Fast Flow Disk Holder was used with the samples because groundwater and leachate samples can at times, have varying levels of particulate matter. The Fast Flow Disk Holder uses a 47 mm disk, but allows larger diameter pre-filters to be placed on top to shield the SPE disk from particulates that may cause clogging, which allows the sample to flow through the disk more quickly. The particulates are retained on the filters and washed with solvent during the elution steps, so any compounds that have been adsorbed on the particulates will be included in the extraction.

The extraction was completed using the SPE-DEX® 5000 (Horizon Technology) (**Figure 1**); an automated extractor which allows various types of liquid samples to be processed directly from the original

Table 1: SPE-DEX 5000 Extraction Method

Step	Operation	Message						Attachment	
1	Pause with Message	Part 1 of 3: Neutrals and Acids Elution. Have the Fast Flow Disk Holder with One Pass disk, 1 µm filter, 5 µm filter, top screen over the filters, 250 mL collection flask, and carbon cartridge installed. The down spout of the water in valve must push down on the top screen. Click "Continue" to start Part 1.						None	
Step	**Operation**	**Solvent**	**Solvent Vol. (mL)**	**Purge Time (s)**	**Pump Rate (#)**	**Sat. Time (s)**	**Soak Time (s)**	**Drain Time (s)**	
2	Condition SPE Disk	Acetone	40	60	4	2	60	60	
3	Condition SPE Disk	Reagent Water	20	60	4	2	60	60	

sample container. The system includes intuitive software for easy programming, and automatic sample and solvent delivery.

The one liter samples were spiked and acidified to pH 2 with hydrochloric acid (HCl) and placed on the SPE-DEX 5000. Each of the three positions on the SPE-DEX 5000 was set up with the One Pass Kit (Horizon Technology). The resulting extracts were dried using DryDisk® Membranes (Horizon Technology) and concentrated with a DryVap® (Horizon Technology) (**Figure 2**) for automatic in-line drying and concentration.

The extraction method used with the SPE-DEX 5000 is shown in **Table 1.** The conditions for operating the DryVap are shown in **Table 2.** Most of the steps using the SPE-DEX 5000 were completely automated and require no operator intervention. For the few steps where a new flask is required for an elution step, a clear message is shown in the software to make the operation of the system simple to follow.

The samples were measured using GC/MS (6890GC/5975CMS, Agilent Technology). Operational conditions are shown in **Table 3.** All spiking standard solutions used were from Supelco, Bellefonte, PA. The surrogate mixes were from Restek Corp., Bellefonte, PA. Samples were spiked with 50 ug/L of analytes.

Results and Discussion

Prior to implementation of the sample preparation method, EPA Method 8270 requires each laboratory to complete an Initial Demonstration of Proficiency (IDP) by performing at least four replicate reference samples, taken through the entire sample preparation and analysis steps. Section 1.1 of Method 8270 suggests that method operation is considered appropriate if average recovery falls between 50 – 150 %. This

Table 1 (cont'd): SPE-DEX 5000 Extraction Method

Step	Operation		Sample Flow Rate (#)	Done Loading Sample Delay (s)					
4	Load Sample		5	45					
Step	**Operation**	**Solvent**	**Solvent Vol. (mL)**	**Purge Time (s)**	**Pump Rate (#)**	**N2 Blanket**	**Sat. Time (s)**	**Soak Time (s)**	**Drain Time (s)**
5	Wash Sample Container	Reagent Water	20	30	4	Off	2	5	30
Step	**Operation**		**Dry Time (s)**	**Pump Rate (#)**	**N2 Blanket**				
6	Air Dry Disk Timer		360	6	Off				
Step	**Operation**	**Solvent**	**Solvent Vol. (mL)**	**Purge Time (s)**	**Pump Rate (#)**	**N2 Blanket**	**Sat. Time (s)**	**Soak Time (s)**	**Elute Time (s)**
7	Elute Sample Container	Acetone	20	20	4	Off	2	180	180
8	Elute Sample Container	$MeCl_2$	17	15	4	Off	2	180	180
9	Elute Sample Container	$MeCl_2$	17	15	4	Off	2	120	120
10	Elute Sample Container	$MeCl_2$	17	15	4	Off	2	120	120
11	Elute Sample Container	$MeCl_2$	17	15	6	Off	2	120	180
Step	**Operation**	**Message**						**Attachment**	
12	Pause with Message	Part 2 of 3: Ion Exchange Elution. Remove the 250 mL collection flask containing the neutrals and acids elution. Stopper the flask and set aside for part 3. Then install a clean 125 mL flask to collect the ion exchange elution. Click "Continue" to Start Part 2.						None	

note explains that actual recoveries may vary depending on the sample matrix, number of constituents being analyzed concurrently, analytical instrumentation, and the preparation method used. The note also includes a list of compounds that have historically been considered problematic (Section 1.4). Section 13.1 suggests that performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC

Table 1 (cont'd): SPE-DEX 5000 Extraction Method

Step	Operation	Solvent	Solvent Vol. (mL)	Purge Time (s)	Pump Rate (#)	N2 Blanket	Sat. Time (s)	Soak Time (s)	Elute Time (s)
13	Elute Sample Container	Acetone	20	20	4	Off	2	0	180
14	Elute Sample Container	1% NH_4OH	20	30	4	Off	2	120	120
15	Elute Sample Container	Acetone	20	20	4	Off	2	180	120
16	Elute Sample Container	$MeCl_2$	17	15	4	Off	2	180	180
17	Elute Sample Container	$MeCl_2$	16	15	4	Off	2	120	180
18	Elute Sample Container	$MeCl_2$	16	15	4	Off	2	120	180
19	Elute Sample Container	$MeCl_2$	16	15	6	Off	2	120	180
Step	Operation	Message						Attachment	
20	Pause with Message	Part 3 of 3: Carbon Cartridge Elution. Remove the carbon cartridge from the tubing lines. Connect the tubing ends together. Using a 20 cc syringe, plunge the carbon cartridge with air through the cap adapter to reseat the carbon bed on the frit. Replace the cap adapter with the funnel on the cartridge. Replace the disk holder with the cartridge. Replace the 125 mL flask with the 250 mL flask containing the neutrals and acids elution from Part 1. Stopper the 125 mL flask. Click "Continue" to start part 3.						None	
Step	Operation		Dry Time (s)	Pump Rate	N2				
21	Air Dry Disk Timer		60	6	Off				
Step	Operation	Solvent	Solvent Vol. (mL)	Purge Time (s)	Pump Rate (#)	N2 Blanket	Sat. Time (s)	Soak Time (s)	Elute Time (s)
22	Elute Sample Container	Acetone	25	20	4	Off	3	60	60
23	Elute Sample Container	$MeCl_2$	17	15	4	Off	3	60	20
24	Elute Sample Container	$MeCl_2$	17	15	4	Off	3	60	20
25	Elute Sample Container	$MeCl_2$	17	15	4	Off	3	60	20
26	Elute Sample Container	$MeCl_2$	17	15	4	Off	3	60	20
27	Elute Sample Container	$MeCl_2$	17	15	6	Off	3	60	60

Table 2: DryVap System Conditions

Parameter	Setting
Dry Volume	200 mL
Heat Power	5
Heat Timer	OFF
Auto Rinse Mode	OFF
Nitrogen Sparge	20 psi
Vacuum	-7 in. Hg

performance criteria to meet the needs of the application. The method criteria is for guidance purposes only.

Table 4 presents data obtained for 4 consecutive runs completed as an IDP study per Section 9.4 with the use of Horizon Technology's suit of automated sample equipment. For all compounds except for those of which EPA Method 8270E considers to be problematic (e.g., pyridine, NDMA, hexachlorocyclopentadiene and 2-picoline), average recovery fell in the 50 - 150% recovery range, and most were better, showing this preparation technique to be acceptable. Standard Deviation for this set of data was found to be 0.77-13 demonstrating reproducibility of results. Blank results are also shown (surrogate recoveries not shown) to indicate the low level of contamination in the process.

Matrix effects on method performance is documented by the analysis of 2 replicates of unspiked samples or a matrix spike (MS) and matrix spike duplicate (MSD). Since the groundwater samples extracted were not expected to contain target analytes, a matrix spike and matrix spike duplicate pair was performed as indicated in the method. Results for the MS/MSD are shown in **Table 5** and meet the criteria listed in Method 8270E for compliance having average percent recoveries fall within 50-150% for all compounds analyzed; except those of which EPA Method 8270E considers to be problematic. Relative Percent Difference (RPD) between the data sets was observed to be well below the generally accepted RPD value of 20%, except for one pair of hexachlorocyclopentadiene (light sensitive) MS/MSD pairs, where the RPD was 25%.

The data presented is normalized against internal standards to demonstrate the extraction efficiency of compounds in EPA Method 8270E and to eliminate any external sources of error from the analytical instrumentation.

Conclusion

The precision and accuracy shown in the Initial Demonstration of Proficiency demonstrate that extraction with automated SPE, combine with, extract drying and evaporation is an effective solution for a wide range of semivolatile compounds extracted from ground water samples. The results are compliant with the method requirements and show a solid base for laboratory use.

Solid phase extraction is an effective extraction technique that reduces the

Table 3: Agilent 6890/5975MS

Injection	
Amount	1 µL
Inlet Temperature	280°C
Mode	Splitless
Gas Type	Helium
Column Conditions	**Zebron™ ZB-Semivolatiles (Phenomenex)**
Mode	Consistent Flow
Oven Program	45°C hold for 1 min to 270°C at 15°C/min then to 318 °C at 6 °C/min
MS Ions Monitored	Scan masses 35-550

solvent that must be evaporated before GC/MS introduction, reducing costs and time required for sample preparation. SPE disks, especially when used with the Fast Flow Disk holder can handle large amounts of particulate and will drastically reduce the possibility of emulsion formation, making extractions more consistent.

This system can be extended to leachate preparation from soil samples or water matrices with high levels of particulate, such as wastewater. With today's modern analytical instrumentation the requirement for one liter samples may not be necessary for all projects and smaller volume sample (such as 100 mL) preparation has been demonstrated for semivolatile compounds (3).

References

1. US EPA Method 8270E, https://www.epa.gov/sites/production/files/2017-04/documents/method_8260d_update_vi_final_03-13-2017_0.pdf .

2. US EPA Method 3535A, https://www.epa.gov/sites/production/files/2015-12/documents/3535a.pdf.

3. AN1211710_01, Solid Phase Extraction (SPE) in US EPA Method 625.1, the Performance of Smaller

Table 4: IDP Initial Demonstration of Proficiency (IDP)

Compounds:	% Rec	% Rec	% Rec	% Rec	Average % Recovery	SD
1,2,4,5-Tetrachlorobenzene	72.0	72.1	68.1	73.2	71.4	2.2
1,2,4-Trichlorobenzene	72.0	71.2	68.9	70.4	70.6	1.3
1,2-Dichlorobenzene	59.6	61.7	58.7	61.4	60.4	1.4
1,3,5,-Trinitrobenzene	87.0	85.6	86.9	89.7	87.3	1.7
1,3-Dichlorobenzene	53.9	57.8	55.2	57.1	56.0	1.8
1,3-Dinitrobenzene	91.2	88.6	91.9	93.2	91.2	1.9
1,4-Dichlorobenzene	56.4	60.0	56.6	59.5	58.1	1.9
1,4-Naphthoquinone	86.0	76.2	74.2	71.0	76.9	6.5
1-Naphthylamine	99.5	84.4	96.0	91.9	93.0	6.5
2,3,4,6-Tetrachlorophenol	93.2	90.8	91.3	94.3	92.4	1.6
2,4,5-Trichlorophenol	89.6	87.5	88.9	91.1	89.3	1.5
2,4,6-Tribromophenol	93.3	92.6	94.3	95.2	93.8	1.2
2,4,6-Trichlorophenol	91.0	88.9	89.5	91.8	90.3	1.3
2,4-Dichlorophenol	91.0	89.0	90.2	90.5	90.2	0.83
2,4-Dimethylphenol	87.5	83.6	87.7	87.7	86.6	2.0
2,4-Dinitrophenol	80.0	79.7	79.5	84.4	80.9	2.3
2,4-Dinitrotoluene	93.4	91.4	93.2	94.4	93.1	1.3
2,6-Dichlorophenol	89.5	87.2	87.7	90.0	88.6	1.4
2,6-Dinitrotoluene	95.4	91.4	95.0	94.9	94.2	1.9
2-Chloronaphthalene	82.9	81.4	81.4	82.5	82.1	0.77
2-Chlorophenol	79.9	78.4	77.3	78.4	78.5	1.1
2-Fluorobiphenyl	83.4	80.1	81.7	83.3	82.1	1.6
2-Fluorophenol	55.0	61.1	56.1	57.6	57.5	2.6
2-Methyl phenol	81.9	83.5	82.4	83.3	82.8	0.77
2-Methylnaphthalene	81.2	80.4	79.8	80.9	80.6	0.61
2-Naphthylamine	124	111	118	121	118	5.6
2-Nitroaniline	93.5	93.1	95.5	96.4	94.6	1.6
2-Nitrophenol	79.5	79.4	77.3	80.6	79.2	1.4
2-Picoline	44.6	48.5	45.0	47.9	46.5	2.0
3,3'-Dichlorobenzidine	107	97.3	102	106	103	4.2
3,3'-Dimethylbenzidine	129	109	114	126	119	9.6
3+4 Methyl phenol	83.7	85.4	85.1	86.9	85.3	1.3
3-Methylcholanthrene	88.4	82.5	89.1	91.1	87.8	3.7
3-Nitroaniline	110	105	107	110	108	2.2
4 Aminobiphenyl	129	128	119	129	126	5.0
4,6-Dinitro-2-methylphenol	89.6	86.9	88.6	90.3	88.9	1.4
4-Bromophenyl phenyl ether	88.6	89.6	90.3	91.7	90.0	1.3
4-Chloro-3-methylphenol	93.0	92.0	94.2	95.9	93.8	1.7
4-Chloroaniline	124	119	120	124	122	2.9
4-Chlorophenyl phenyl ether	86.7	87.1	88.5	88.6	87.7	0.96
4-Nitroaniline	90.7	89.9	89.2	90.0	90.0	0.64

Table 4 (cont'd): IDP Initial Demonstration of Proficiency (IDP)

4-Nitroquinoline-1-oxide	93.0	91.7	91.6	96.0	93.1	2.0
5-nitro-o-toluidine	103	99.3	102	104	102	2.1
7,12-Dimethylbenz(a)-anthracene	87.8	82.1	87.4	90.2	86.9	3.4
Acenaphthene	86.2	84.4	86.5	86.0	85.8	0.94
Acenaphthylene	87.5	85.4	86.7	87.1	86.7	0.89
Acetophenone	80.2	78.4	76.0	77.8	78.1	1.7
Acetylaminofluorene	102	100	100	102	101	0.97
Aniline	89.8	85.1	81.2	84.4	85.1	3.5
Anthracene	89.8	89.2	90.6	93.0	90.6	1.7
Azobenzene	91.8	89.7	91.4	92.0	91.2	1.0
Benz(a)anthracene	93.6	92.2	92.2	94.4	93.1	1.1
Benzidine	109.5	76.9	89.3	90.9	91.7	13
Benzo(a)pyrene	90.1	88.7	88.8	92.1	89.9	1.6
Benzo(b)fluoranthene	91.2	89.5	89.5	93.8	91.0	2.0
Benzo(ghi)perylene	94.2	91.5	91.9	95.3	93.2	1.8
Benzo(k)fluoranthene	94.0	92.1	91.4	92.5	92.5	1.1
Benzoic acid	61.2	68.7	62.9	68.5	65.3	3.8
Benzyl alcohol	84.8	82.9	83.4	82.6	83.4	0.97
Bis(2-chlorethoxy)methane	88.5	84.3	84.8	84.9	85.6	1.9
Bis(2-chloroethyl)ether	72.2	71.6	68.7	71.0	70.9	1.5
Bis(2chloroisopropyl)ether	75.2	74.1	71.0	73.2	73.4	1.8
Bis(2-ethylhexyl) phthalate	103	103	103	106	104	1.3
Butyl benzyl phthalate	98.7	97.4	98.5	99.3	98.5	0.77
Carbazole	95.4	93.6	95.4	95.0	94.8	0.86
Chrysene	92.6	93.3	91.0	94.6	92.9	1.5
cis-Isosafrole	85.9	84.0	83.8	84.4	84.5	0.96
Dibenz(ah)anthracene	92.9	88.0	86.7	90.6	89.6	2.7
Dibenzofuran	87.8	86.7	87.7	87.9	87.5	0.54
Diethyl phthalate	96.8	94.3	97.5	97.2	96.4	1.4
Dimethyl phthalate	95.0	92.6	94.8	94.8	94.3	1.1
Dimethylaminoazobenzene	97.3	93.4	97.3	95.9	96.0	1.8
Di-n-butyl phthalate	101	99.0	102	102	101	1.4
Di-n-octyl phthalate	101	101	102	103	102	0.98
Dinoseb	91.1	91.8	93.1	94.1	92.5	1.4
Diphenylamine	94.2	90.6	94.6	93.9	93.3	1.8
Ethylmethane Sulfonate	81.5	75.2	75.9	75.9	77.1	2.9
Fluoranthene	93.3	92.8	93.4	94.9	93.6	0.93
Fluorene	89.2	87.3	88.1	89.3	88.5	0.96
Hexachlorobenzene	88.3	90.0	89.3	92.6	90.0	1.8
Hexachlorobutadiene	54.3	54.4	50.6	54.4	53.4	1.9
Hexachlorocyclopentadiene	49.2	45.9	39.2	40.6	43.7	4.6
Hexachloroethane	53.9	58.4	53.9	57.2	55.8	2.3
Hexachloropropene	54.7	55.1	52.3	54.7	54.2	1.3

Table 4 (cont'd): IDP Initial Demonstration of Proficiency (IDP)

Isophorone	86.2	83.2	82.6	83.6	83.9	1.6
Methapyrilene	89.5	87.0	89.0	88.8	88.6	1.1
Methyl Methane Sulfonate	54.5	49.9	58.2	60.5	55.8	4.6
Naphthalene	77.0	75.4	73.6	75.3	75.3	1.4
NDMA	38.0	39.6	47.7	50.0	43.8	5.9
Nitrobenzene	78.0	76.9	73.9	76.4	76.3	1.7
Nitrobenzene-d5	79.0	77.0	74.5	77.2	76.9	1.8
N-Nitroso-diethylamine	69.8	69.0	67.2	69.1	68.8	1.1
N-nitroso-di-n-butylamine	89.9	89.0	90.6	91.2	90.2	0.94
N-nitroso-di-n-propylamine	80.9	77.7	77.0	79.5	78.8	1.8
N-Nitrosomethyl ethylamine	61.0	62.2	62.2	65.3	62.7	1.8
N-Nitroso-morpholine	88.2	84.9	86.0	88.0	86.8	1.6
N-Nitroso-piperidine	84.6	81.9	81.4	83.2	82.8	1.4
N-Nitroso-pyrrolidine	83.4	79.8	81.8	84.5	82.4	2.0
o-toluidine	102	95.1	91.3	97.1	96.3	4.3
Pentachlorobenzene	79.1	79.6	78.8	82.1	79.9	1.5
Pentachloroethane	55.5	60.2	56.9	59.8	58.1	2.3
Pentachloronitrobenzene	92.6	91.3	89.9	93.2	91.8	1.5
Pentachlorophenol	88.2	88.8	88.2	92.8	89.5	2.2
Phenacetin	93.6	94.8	95.0	96.6	95.0	1.2
Phenanthrene	90.1	90.2	91.3	92.6	91.0	1.2
Phenol	56.1	57.5	58.4	57.0	57.2	0.95
Phenol-d5	56.8	57.3	58.1	57.7	57.5	0.56
p-Terphenyl-d14	93.9	91.9	89.3	93.9	92.3	2.2
Pyrene	93.1	91.6	92.7	94.2	92.9	1.1
Pyridine	31.4	36.1	32.5	35.6	33.9	2.3
Safrole	88.1	84.3	87.5	86.3	86.5	1.7
trans-Isosafrole	85.8	87.2	85.3	87.2	86.4	1.0

Table 5: Matrix Spike (MS) and Matrix Spike Duplicate (MSD) Results-Ground Water

Compounds:	MS-1 Rec	MSD-1 % Rec	Avg (%)	RPD	MS-2 % Rec	MSD-2 % Rec	Avg (%)	RPD
1,2,4,5-Tetrachlorobenzene	78.8	65.1	72.0	9.5	72.1	72.7	72.4	0.43
1,2,4-Trichlorobenzene	73.5	65.0	69.2	6.1	70.9	72.9	71.9	1.4
1,2-Dichlorobenzene	61.8	54.9	58.4	5.9	61.6	66.8	64.2	4.1
1,3,5,-Trinitrobenzene	91.1	85.4	88.2	3.2	84.0	84.0	84.0	0.04
1,3-Dichlorobenzene	56.8	50.2	53.5	6.2	57.4	62.7	60.0	4.4
1,3-Dinitrobenzene	96.6	88.7	92.7	4.3	89.1	86.1	87.6	1.7
1,4-Dichlorobenzene	59.0	51.4	55.2	6.9	59.1	63.9	61.5	3.9
1,4-Naphthoquinone	86.7	72.0	79.4	9.3	76.8	75.1	76.0	1.1
1-Naphthylamine	103	89.5	96.2	7.0	85.3	81.5	83.4	2.3
2,3,4,6-Tetrachlorophenol	94.1	87.0	90.6	3.9	89.0	88.3	88.7	0.41
2,4,5-Trichlorophenol	89.8	85.9	87.9	2.3	87.4	86.1	86.8	0.70
2,4,6-Tribromophenol	95.6	90.8	93.2	2.6	90.2	91.9	91.1	0.94
2,4,6-Trichlorophenol	91.9	86.2	89.1	3.2	88.6	86.9	87.8	0.98
2,4-Dichlorophenol	92.6	86.2	89.4	3.6	87.7	87.9	87.8	0.13
2,4-Dimethylphenol	90.0	83.9	86.9	3.5	86.6	84.8	85.7	1.1
2,4-Dinitrophenol	82.5	76.5	79.5	3.8	77.4	76.9	77.1	0.35
2,4-Dinitrotoluene	96.1	88.1	92.1	4.3	88.6	87.4	88.0	0.69
2,6-Dichlorophenol	90.6	85.1	87.8	3.1	87.8	87.6	87.7	0.14
2,6-Dinitrotoluene	95.5	89.8	92.7	3.1	90.5	88.6	89.6	1.1
2-Chloronaphthalene	85.9	76.7	81.3	5.6	80.4	80.6	80.5	0.12
2-Chlorophenol	80.8	75.0	77.9	3.7	78.4	82.2	80.3	2.4
2-Fluorobiphenyl	87.6	79.0	83.3	5.2	83.6	83.1	83.3	0.29
2-Fluorophenol	71.5	58.6	65.1	9.9	66.7	66.3	66.5	0.36
2-Methyl phenol	87.4	81.9	84.7	3.3	84.1	84.7	84.4	0.34
2-Methylnaphthalene	83.4	76.8	80.1	4.1	79.3	80.7	80.0	0.85
2-Naphthylamine	127	112	120	6.5	109	108	108	0.06
2-Nitroaniline	97.5	90.5	94.0	3.7	92.3	89.6	91.0	1.5
2-Nitrophenol	83.5	75.3	79.4	5.1	81.5	81.5	81.5	0.00
2-Picoline	47.0	42.0	44.5	5.6	47.6	49.6	48.6	2.0
3,3'-Dichlorobenzidine	109	102	105	3.3	99.9	98.2	99.1	0.88
3,3'-Dimethylbenzidine	129	117	123	5.0	110	102	106	3.7
3+4 Methyl phenol	89.8	83.7	86.8	3.5	84.5	86.3	85.4	1.0
3-Methylcholanthrene	90.4	84.7	87.5	3.3	80.0	82.6	81.3	1.6
3-Nitroaniline	111	99	105	5.6	104	102	103	0.99
4 Aminobiphenyl	127	123	125	1.6	123	116	120	2.8
4,6-Dinitro-2-methylphenol	91.6	86.5	89.1	2.9	83.0	83.7	83.3	0.42
4-Bromophenyl phenyl ether	91.5	84.9	88.2	3.7	86.1	86.4	86.3	0.13
4-Chloro-3-methylphenol	96.7	90.1	93.4	3.5	89.5	90.2	89.9	0.42
4-Chloroaniline	129	117	123	5.0	118	117	117	0.51
4-Chlorophenyl phenyl ether	89.4	82.2	85.8	4.2	84.3	83.9	84.1	0.25

Table 5 (cont'd): Matrix Spike (MS) and Matrix Spike Duplicate (MSD) Results- Ground Water

4-Nitroaniline	95.4	85.7	90.6	5.3	87.1	85.3	86.2	1.0
4-Nitrophenol	90.7	90.1	90.4	0.3	86.2	88.3	87.2	1.2
4-Nitroquinoline-1-oxide	97.9	89.2	93.6	4.6	86.7	87.6	87.2	0.49
5-nitro-o-toluidine	105	96.2	101	4.6	95.7	93.1	94.4	1.3
7,12-Dimethylbenz(a)-anthracene	88.8	83.9	86.4	2.8	79.4	84.3	81.8	3.0
Acenaphthene	89.0	81.3	85.2	4.5	82.5	83.1	82.8	0.33
Acenaphthylene	88.8	82.3	85.5	3.8	83.9	83.9	83.9	0.01
Acetophenone	80.7	72.8	76.7	5.1	77.4	80.9	79.2	2.2
Acetylaminofluorene	102	96.5	99.3	2.8	96.8	96.8	96.8	0.00
Aniline	91.9	79.2	85.5	7.4	84.7	86.0	85.3	0.77
Anthracene	92.9	84.7	88.8	4.6	86.2	87.1	86.6	0.52
Azobenzene	93.1	86.0	89.6	4.0	87.7	86.9	87.3	0.47
Benz(a)anthracene	92.8	88.4	90.6	2.5	89.0	90.3	89.6	0.71
Benzidine	103	88.1	95.5	7.7	70.4	80.2	75.3	6.5
Benzo(a)pyrene	90.3	85.4	87.9	2.8	85.6	86.9	86.3	0.77
Benzo(b)fluoranthene	92.3	86.4	89.4	3.3	87.4	88.7	88.0	0.74
Benzo(ghi)perylene	93.6	87.6	90.6	3.3	90.8	90.7	90.8	0.07
Benzo(k)fluoranthene	94.0	88.6	91.3	3.0	88.4	89.8	89.1	0.81
Benzoic acid	71.2	63.9	67.6	5.4	70.4	64.7	67.6	4.2
Benzyl alcohol	85.4	74.9	80.1	6.5	82.2	84.0	83.1	1.1
Bis(2-chlorethoxy)methane	88.6	82.1	85.3	3.8	83.7	85.8	84.8	1.2
Bis(2-chloroethyl)ether	72.0	64.4	68.2	5.6	70.1	76.4	73.3	4.3
Bis(2chloroisopropyl)ether	75.3	67.2	71.3	5.7	72.6	78.1	75.4	3.7
Bis(2-ethylhexyl) phthalate	101	98.0	99.5	1.5	95.7	98.2	97.0	1.3
Butyl benzyl phthalate	97.5	93.3	95.4	2.2	92.3	94.9	93.6	1.4
Carbazole	98.0	90.8	94.4	3.8	90.8	91.1	91.0	0.21
Chrysene	92.9	86.8	89.9	3.4	88.3	90.1	89.2	1.0
cis-Isosafrole	88.1	80.7	84.4	4.4	82.2	84.3	83.3	1.2
Dibenz(ah)anthracene	87.4	83.2	85.3	2.5	85.6	85.9	85.7	0.20
Dibenzofuran	90.2	83.3	86.7	3.9	84.1	84.5	84.3	0.19
Diethyl phthalate	98.9	92.2	95.6	3.5	91.4	91.2	91.3	0.088
Dimethyl phthalate	96.6	89.6	93.1	3.7	90.5	89.3	89.9	0.66
Dimethylaminoazobenzene	96.7	93.9	95.3	1.5	89.8	92.8	91.3	1.7
Di-n-butyl phthalate	102	97.5	99.7	2.2	95.6	96.1	95.9	0.26
Di-n-octyl phthalate	99.5	95.3	97.4	2.2	93.7	96.8	95.2	1.6
Dinoseb	94.4	91.7	93.1	1.5	85.8	89.4	87.6	2.1
Diphenylamine	95.8	88.9	92.3	3.7	89.4	88.7	89.1	0.38
Ethylmethane Sulfonate	78.7	72.0	75.3	4.5	75.0	81.6	78.3	4.2
Fluoranthene	95.9	88.1	92.0	4.2	89.8	90.1	89.9	0.17
Fluorene	89.4	83.5	86.5	3.4	85.0	84.5	84.8	0.27
Hexachlorobenzene	91.0	84.6	87.8	3.6	87.0	87.0	87.0	0.023
Hexachlorobutadiene	63.1	47.6	55.4	14	55.1	58.7	56.9	3.1

Table 5 (cont'd): Matrix Spike (MS) and Matrix Spike Duplicate (MSD) Results- Ground Water

Hexachlorocyclopentadiene	64.3	38.5	51.4	25	46.8	46.8	46.8	0.021
Hexachloroethane	56.0	49.2	52.6	6.5	55.9	61.9	58.9	5.1
Hexachloropropene	60.5	50.8	55.6	8.7	56.2	59.6	57.9	2.9
Indeno(1,2,3-cd)pyrene	91.6	86.4	89.0	2.9	88.4	89.4	88.9	0.54
Isophorone	86.5	79.7	83.1	4.1	82.8	83.3	83.0	0.31
Methapyrilene	90.0	84.9	87.5	2.9	84.2	84.7	84.5	0.28
Methyl Methane Sulfonate	55.6	53.2	54.4	2.2	48.9	64.2	56.6	14
Naphthalene	79.1	70.4	74.8	5.8	76.3	78.0	77.2	1.2
NDMA	41.1	39.6	40.4	1.9	37.4	52.4	44.9	17
Nitrobenzene	78.2	72.2	75.2	4.0	75.8	79.6	77.7	2.4
Nitrobenzene-d5	80.2	73.3	76.7	4.5	78.8	81.5	80.1	1.7
N-Nitroso-diethylamine	71.1	62.6	66.9	6.3	68.4	75.1	71.8	4.7
N-nitroso-di-n-butylamine	91.5	86.5	89.0	2.8	86.7	86.4	86.5	0.16
N-nitroso-di-n-propylamine	80.1	79.2	79.6	0.6	75.7	80.6	78.2	3.1
N-Nitrosomethyl ethylamine	62.1	54.9	58.5	6.2	61.7	70.1	65.9	6.3
N-Nitroso-morpholine	88.2	80.1	84.2	4.9	82.8	88.4	85.6	3.3
N-Nitroso-piperidine	84.4	78.3	81.3	3.8	79.6	82.8	81.2	2.0
N-Nitroso-pyrrolidine	83.7	79.9	81.8	2.3	78.7	82.3	80.5	2.3
o-toluidine	101	91.4	96.3	5.1	91.6	91.7	91.7	0.0
Pentachlorobenzene	83.3	73.7	78.5	6.1	77.2	78.8	78.0	1.0
Pentachloroethane	58.4	51.5	54.9	6.3	59.2	64.5	61.8	4.3
Pentachloronitrobenzene	94.1	87.2	90.7	3.8	87.5	87.7	87.6	0.13
Pentachlorophenol	90.7	86.0	88.4	2.7	85.9	86.6	86.3	0.39
Phenacetin	97.9	91.4	94.7	3.4	91.8	90.1	90.9	0.92
Phenanthrene	93.8	86.4	90.1	4.1	87.7	88.2	88.0	0.28
Phenol	68.7	56.2	62.5	10	59.8	62.0	60.9	1.8
Phenol-d5	69.3	57.0	63.2	9.8	62.2	63.4	62.8	1.0
p-Terphenyl-d14	92.1	86.9	89.5	2.9	89.5	90.4	89.9	0.47
Pyrene	94.8	86.7	90.7	4.4	89.2	89.3	89.2	0.045
Pyridine	32.2	29.9	31.1	3.7	34.6	35.2	34.9	0.86
Safrole	88.9	83.0	86.0	3.5	84.1	84.4	84.2	0.14
trans-Isosafrole	89.7	82.4	86.0	4.2	87.7	85.3	86.5	1.4

Samples, available at www.horizontechinc.com.