Determination of Organic Compounds in Drinking Water Using Atlantic® DVB Disks for EPA Method 525.3

***Authors**: Bob Johnson, Biotage, Salem, NH, USA.*
***Revision By:** Matt Harden and Deanna Bissonnette, Biotage, Salem, NH, USA.*
***Keywords:** SVOC, automated solid phase extraction, SPE, drinking water*

Application Note Scope

The purpose of this application note is to outline an automated extraction method utilizing the Atlantic™ DVB SPE Disk and the Biotage automated and manual SPE solutions for the extraction of organic compounds in drinking water. The first section will highlight the use of the Biotage® Horizon 5000 fully automated extraction system and the method used for this application. Additionally, there will be an Application Modification section that will highlight the use of the Biotage® Horizon 4790 (with data and discussion) and Biotage® VacMaster™ Disk for this application.

Introduction

In the June 28, 2012 issue of the Federal Register, the EPA announced the approval of alternate testing methods for use in measuring the levels of contaminates in drinking water and for determining compliance with national primary drinking water regulations. The Safe Drinking Water Act (SDWA) authorizes the EPA to approve the use of alternative testing methods through publication in the Federal Register. The EPA used this streamline authority to make 10 additional methods available for analyzing drinking water samples required by regulation. This expedited approach provides public water systems, laboratories, and primacy agencies with more timely access to new measurement techniques and greater flexibility in the selection of analytical methods. This authority and flexibility helps reduce monitoring costs while maintaining public health protection.

One of the methods approved by this action is Method 525.3 for the determination of semi-volatile organic compounds in finished drinking water. The method analytes are extracted and concentrated from the water using solid phase extraction. Extracts are injected onto a capillary GC column and analyzed using mass spectrometry. Method 525.3 is similar in many ways to its predecessor, method 525.2 (Rev 2.0 – 1995), however there are significant changes which make the newer 525.3 a vastly improved method. Several of the major changes are as follows:

- The sorbent material has been changed from C18 to DVB (divinylbenzene). This yields better recoveries over a wider pH range.
- The preservation/dechlorination scheme has changed from HCl and sodium sulfite, to ascorbic acid, EDTA, and citric acid. This is safer for field sampling crews and allows bottles to be shipped with preservatives pre-added.
- The internal standard is added to the final extract, not prior to the extraction as with 525.2.
- The use of SIM mode is an option for regulated compounds that have difficulty reaching detection limits.
- The surrogate perylene-d12 has been dropped.
- Pentachlorophenol-C13 is now used as an internal standard for pentachlorophenol.

One of the key points to be aware of with method 525.3, is that during the development of the method, the EPA found that several brands of styrene-divinylbenzene (SDVB) and modified SDVB media in cartridge format did not provide satisfactory performance. Therefore, this method specifically identifies those sorbent materials which can be used. Where method modifications are proposed, the analyst must perform the procedures outlined in the initial demonstration of capability (IDC, Sect 9.2), verify that all QC acceptance criteria in this method (Sect. 9) are met, and that method performance in real samples matrices is equivalent to that demonstrated for Laboratory Fortified Sample Matrices (LFSMs) in Sect. 17.

This application note will describe the use of the Biotage® Horizon 5000 automated extractor system, and the Atlantic® DVB SPE disk for the extraction of water samples, as specified in method 525.3.

Instrumentation

- Biotage Instruments:
  - Biotage® Horizon 5000 Automated Extraction System
  - Atlantic® DVB SPE Disk (47 mm)
- Organomation
  - N-Evap Concentrator
- Restek
  - Rxi-5Sil MS 30 m, 0.25 mm ID, 0.25 um df
- Agilent
  - 6890 Gas Chromatograph
  - 5973 Inert MSD
  - 7683B Autosampler

Method Summary

Preservation and Declorination

1. Sample bottles are prepared using 0.10 g/L L-Ascorbic acid, 0.35 g/L trisodium EDTA, and 9.4 g/L potassium dihydrogen citrate (Section 8).
2. A one-liter sample should be collected in this bottle and its pH should be less than or equal to 4.

Extraction

1. Verify that the sample pH is less than or equal to 4.
2. Add surrogate to each sample.
3. Load the sample onto the Biotage® Horizon 5000 extractor and start the extraction process using the method given in Table 1.
4. When complete, remove the collected extract (16–20 mL).
5. Pour the extract through a tube containing 10 g of anhydrous sodium sulfate.
6. Rinse the sodium sulfate using 5 mL of dichloromethane (DCM).
7. Using the N-Evap Concentrator (or equivalent), concentrate the extract to a volume of 0.7 mL using a gentle stream of nitrogen and a water bath temperature of 40 °C.
8. Bring the final volume up to 1 mL, making sure to rinse the concentrator tube with ethyl acetate (EtOAc).
9. Transfer the extract to an autosampler vial and analyze by GC/MS.

Table 1. Biotage® Horizon 5000 extraction method.

Step	Select Solvent	Volume (mL)	Purge (s)	Vacuum	Saturate (s)	Soak (s)	Drain/Elute (s)	Sample Delay (s)
Condition SPE Disk	Ethyl Acetate	11	60	2	1	60	30	
Condition SPE Disk	Dichloromethane	11	60	2	1	60	30	
Condition SPE Disk	Methanol	11	60	2	1	60	5	
Condition SPE Disk	Reagent water	11	60	2	1	5	5	
Load Sample				2				45
Wash Sample Container	Reagent water	10	30	2	1	10	30	
Air Dry Disk				6			60	
Elute Sample Container	Ethyl Acetate	8	30	2	1	90	30	
Elute Sample Container	Dichloromethane	13	15	2	1	90	30	
Elute Sample Container	Dichloromethane	13	15	6	1	90	60	

Application Modifications

Biotage® Horizon 4790 Method Summary

1. Verify that the sample pH is less than or equal to 4.
2. Add surrogate to each sample.
3. Load the sample onto the Biotage® Horizon 4790 Extraction System and start the extraction process using the method given in Table 1.
4. When complete, remove the collected extract (16–20 mL).
5. Pour the extract through a tube containing 10 g of anhydrous sodium sulfate.
6. Rinse the sodium sulfate using 5 mL of DCM.
7. Using the N-Evap Concentrator (or equivalent), concentrate the extract to a volume of 0.7 mL using a gentle stream of nitrogen and a water bath temperature of 40 °C.
8. Bring the final volume up to 1 mL, making sure to rinse the concentrator tube with EtOAc.
9. Transfer the extract to an autosampler vial and analyze by GC/MS.

Table 2. Biotage® Horizon 4790 extraction method*.

Step	Solvent	Soak Time (s)	Dry Time (s)
Prewet 1	Ethyl Acetate	60	30
Prewet 2	Dichloromethane	60	30
Prewet 3	Methanol	60	0
Prewet 4	Reagent Water	5	0
Sample Process			
Air Dry			60
Rinse 1	Ethyl Acetate	90	30
Rinse 2	Dichloromethane	90	30
Rinse 3	Dichloromethane	90	30
Rinse 4	Dichloromethane	60	20

*The data shown in this Application Note was generated using this method. However, EPA recommends the addition of two reagent water wash steps each with 10 sec. soak times and 30 sec. dry times.

Results and Discussion

Table 3 shows the precision and accuracy data obtained from method analytes fortified in reagent water at three concentrations and extracted using the Biotage® Horizon 4790 Extraction System and the Atlantic® DVB SPE disk. The concentrations were 0.25, 2.0, and 5.0 ug/L. The mean recovery values and RSD's are shown.

Table 4 shows the precision and accuracy data obtained for method analytes fortified in finished drinking water from ground and surface water sources and extracted using the same setup, as above. The fortified concentration was 2.0 ug/L. The mean recovery values and RSD's are shown for both synthetic hard water and for surface water.

Recoveries and deviations from both sets are excellent, indicating the Biotage® Horizon 4790 and Atlantic® disk are a viable option for those laboratories looking to increase sample throughput, and reduce labor costs.

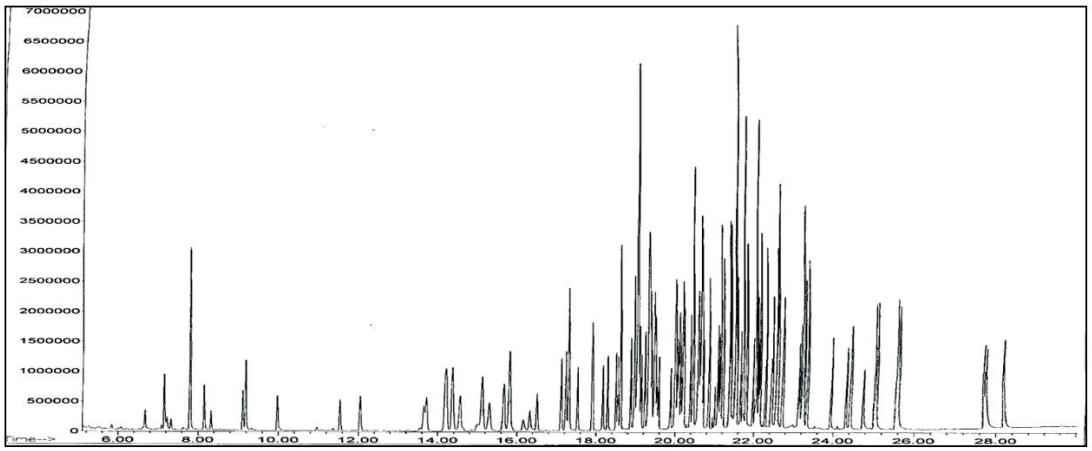

Figure 1: Chromatogram on EPA Method 525.3 Analysis

Table 3. Precision and accuracy of laboratory fortified blanks.

Analytes	Fortified Conc. 0.25 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
acenaphthylene	87.3	3.4	86.6	1.1	90.5	1.3
acetochlor	92.3	4.0	93.9	2.6	95.2	1.4
alachlor	95.6	8.0	93.4	2.0	94.6	1.4
aldrin	85.6	1.2	84.7	5.7	90.4	1.2
ametryn	91.4	3.5	93.9	2.9	95.8	1.5
anthracene	87.2	4.6	90.2	2.2	92.9	1.4
atraton	93.2	5.2	94.9	5.3	98.5	2.4
atrazine	94.5	7.4	92.9	2.9	95.0	1.9
benzo[*a*]anthracene	93.6	2.5	90.6	2.1	91.3	1.7
benzo[*a*]pyrene	88.9	3.5	85.8	3.0	88.2	3.9
benzo[*b*]fluoranthene	87.9	3.6	85.1	3.0	89.0	4.0
benzo[*g, h, i*]perylene	88.3	4.2	79.8	3.9	84.5	3.8
benzo[k]fluoranthene	91.1	3.8	85.9	2.7	87.2	3.9
BHT	89.8	5.0	84.9	1.5	87.6	1.2
bromacil	96.9	1.9	94.3	2.8	96.6	3.6
butachlor	94.9	5.0	91.5	2.8	93.0	2.4
butylate	86.9	2.4	88.0	2.4	91.5	1.5
butylbenzylphthalate	104.0	19.0	94.5	3.9	93.6	1.2
chlordane, cis	92.1	6.5	89.2	4.9	89.7	2.5
chlordane, trans	91.2	5.0	89.4	4.2	88.9	2.3
chlorfenvinphos	89.1	7.7	94.0	4.3	95.3	2.4
chlorobenzilate	96.0	4.0	93.8	4.4	92.4	1.9
chloroneb	89.7	4.2	90.9	1.4	93.6	1.3
chlorothalonil	89.0	4.8	94.3	1.6	97.3	2.3
chlorpropham	93.7	7.7	92.5	2.3	95.8	0.8
chlorpyrifos	89.1	2.6	91.3	3.2	94.4	2.1
chrysene	90.7	2.3	92.0	2.6	90.9	1.4
cyanazine	101.0	8.6	108.0	3.3	114.0	3.7
cycloate	90.0	4.9	89.6	1.7	92.4	0.3
dacthal (DCPA)	93.9	3.3	94.3	2.1	96.0	1.2
DDD, 4,4′-	91.0	3.4	91.8	3.4	88.3	1.8
DDE, 4,4′-	83.9	4.3	87.9	6.8	85.6	3.0
DDT, 4,4′-	88.8	6.0	88.3	4.2	86.4	2.4
DEET	91.6	5.5	92.9	2.0	94.5	0.8
di(2-ethylhexyl)adipate	84.1	8.6	76.2	3.3	76.5	3.1
di(2-ethylhexyl)phthalate	ND[e]	11.0	77.0	2.7	72.7	2.8
dibenzo[*a, h*]anthracene	85.3	5.0	76.7	3.7	81.5	4.5
dibutyl phthalate	ND	6.4	111.0	2.2	95.0	1.0
dichlorvos	93.2	4.4	86.7	1.0	89.3	2.3
dieldrin	93.6	9.2	91.3	6.2	91.1	2.8
diethylphthalate	93.6	5.4	91.4	2.2	94.2	0.9
dimethipin	36.0	13.0	32.8	25.0	28.4	9.6
dimethylphthalate	89.4	5.3	90.0	1.0	92.6	1.0

Analytes	Fortified Conc. 0.25 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
DIMP	89.9	5.3	83.9	2.7	86.8	1.8
dinitrotoluene, 2,4-	88.6	5.8	92.1	1.4	97.0	1.3
dinitrotoluene, 2,6-	89.3	5.7	89.1	2.1	94.0	0.7
diphenamid	93.8	3.2	94.7	2.1	96.2	1.4
disulfoton	71.0	5.0	77.4	4.9	77.8	4.8
endosulfan I	87.9	9.2	89.6	3.3	91.1	1.4
endosulfan II	94.8	5.8	91.5	6.7	90.6	3.0
endosulfan sulfate	95.1	4.8	93.4	2.7	93.4	1.7
endrin	88.1	2.1	92.5	4.7	93.0	2.3
EPTC	85.8	3.5	86.2	1.0	90.5	1.8
ethion	96.1	4.3	93.5	3.9	92.7	2.8
ethoprop	91.9	4.1	92.1	2.7	95.7	0.7
ethyl parathion	92.9	7.8	88.8	3.1	93.4	2.8
etridiazole	84.6	6.7	88.8	1.4	92.3	1.1
fenamiphos	90.4	13.0	89.1	2.8	92.4	1.7
fenarimol	99.8	5.9	95.9	3.7	100.0	3.4
fluorene	86.6	4.4	88.1	1.2	92.5	0.5
fluridone	95.4	8.2	96.1	5.9	101.0	4.6
HCCPD	72.6	6.9	73.5	2.4	76.5	4.4
HCH, alpha	91.5	5.9	91.1	1.3	93.3	0.7
HCH, beta	96.1	6.1	94.4	2.8	97.0	2.3
HCH, delta	88.5	5.7	90.6	3.4	93.4	1.5
HCH, gamma (lindane)	89.4	4.0	92.1	3.0	92.3	0.9
heptachlor	91.9	9.9	89.8	3.2	92.6	0.3
heptachlor epoxide	86.8	3.3	91.3	5.7	92.8	2.4
hexachlorobenzene	86.8	6.1	89.5	1.9	90.5	1.1
hexazinone	95.0	3.8	97.8	1.8	100.0	1.7
indeno[1,2,3-*c, d*]pyrene	85.3	5.5	80.2	3.7	86.1	4.3
isophorone	88.2	6.0	87.4	1.3	87.1	1.5
methoxychlor	94.1	4.0	93.0	2.6	92.2	1.3
methyl parathion	90.3	3.8	93.6	3.1	95.4	0.4
metolachlor	90.3	5.0	93.9	3.2	96.6	1.6
metribuzin	93.3	4.7	94.8	1.5	95.4	2.0
mevinphos	90.1	4.7	89.4	1.6	93.2	0.9
MGK 264(a)	87.5	4.0	92.3	2.6	94.5	1.5
MGK 264(b)	86.3	7.9	94.4	3.9	96.3	2.5
molinate	90.5	6.9	89.2	1.1	91.6	1.4
napropamide	97.3	7.6	93.3	3.8	92.9	1.2
nitrofen	90.4	5.1	94.4	3.3	94.0	1.3
nonachlor, trans	86.7	4.2	86.8	2.4	86.0	3.3
norflurazon	97.0	3.8	95.1	2.3	98.9	3.3
oxyfluorfen	94.3	5.2	93.2	4.5	92.9	1.9
pebulate	90.5	3.7	88.4	1.6	90.9	2.0
pentachlorophenol	93.0	4.4	97.4	3.9	94.8	3.4

Biotage®

Analytes	Fortified Conc. 0.25 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
	Mean % Recovery	RSD	Mean % Recovery	RSD	Mean % Recovery	RSD
permethrin, cis	87.7	2.0	79.8	1.3	80.9	3.5
permethrin, trans	89.3	3.2	82.3	2.2	83.2	2.0
phenanthrene	88.6	3.9	90.1	2.4	92.6	1.7
phorate	83.7	4.2	88.8	2.3	91.1	1.0
phosphamidon	98.3	6.5	95.2	2.9	95.5	2.0
profenofos	92.0	8.6	93.7	2.8	94.7	4.2
prometon	90.0	3.8	93.9	3.2	96.0	0.8
prometryn	94.0	2.0	98.9	3.9	102.0	2.2
pronamide	92.3	4.0	93.5	1.7	96.1	1.9
propachlor	88.8	5.2	91.5	2.8	94.7	0.9
propazine	95.5	3.3	93.5	2.6	95.7	0.6
pyrene	92.2	4.3	91.8	3.8	92.3	2.1
simazine	93.9	3.8	94.2	1.8	98.7	2.9
simetryn	95.1	2.1	93.8	2.8	95.4	1.0
tebuconazole	93.2	5.3	94.7	2.0	97.0	2.3
tebuthiuron	95.4	11.0	91.3	9.5	99.6	8.9
terbacil	91.4	6.1	94.5	1.9	96.2	2.9
terbutryn	96.5	5.1	93.7	3.0	95.5	1.5
tetrachlorvinphos	95.6	4.9	92.3	3.1	93.0	2.8
triadimefon	94.5	2.4	96.1	3.3	96.7	2.6
tribufos	82.0	5.2	95.8	4.4	93.7	1.6
trifluralin	91.0	5.8	91.2	2.1	95.9	0.8
vernolate	87.3	2.5	87.0	1.1	90.7	1.8
vinclozolin	94.3	8.5	94.7	4.6	94.9	2.2
PCB congeners (by IUPAC#)						
2-chlorobiphenyl (1)	85.3	5.2	87.2	1.8	90.0	0.5
4-chlorobiphenyl (3)	88.2	4.0	88.1	1.3	92.0	1.1
2,4′-dichlorobiphenyl (8)	87.1	5.8	90.8	2.0	92.5	0.4
2,2′,5-trichlorobiphenyl (18)	89.4	2.5	91.0	2.9	92.0	0.5
2,4,4′-trichlorobiphenyl (28)	84.4	6.8	90.6	3.1	90.8	0.4
2,2′,3,5′-tetrachlorobiphenyl (44)	87.2	7.5	89.6	2.8	90.3	1.7
2,2′,5,5′-tetrachlorobiphenyl (52)	89.4	5.4	88.8	2.2	89.4	2.7
2,3′,4′,5-tetrachloroobiphenyl (70)	87.8	8.3	88.3	5.0	89.4	1.7
2,3,3′,4′,6-pentachlorobiphenyl (110)	87.1	3.2	90.5	4.1	88.5	3.4
2,3′,4,4′,5-pentachlorobiphenyl (118)	85.5	3.4	89.0	4.3	84.3	4.1
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	85.9	5.7	88.6	3.9	86.4	2.8
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	84.3	6.6	88.0	4.5	86.7	3.6
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	83.1	4.4	87.7	4.7	85.1	4.6
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	81.9	6.0	87.1	4.5	86.5	2.9
Surrogate Analytes						
1,3-dimethyl-2-nitrobenzene	86.4	4.0	86.2	1.9	88.0	0.9
benzo[a]pyrene-d_{12}	89.6	3.5	93.5	5.1	98.9	4.2
triphenyl phosphate	87.8	2.6	93.3	2.9	99.9	1.8

Biotage®

Table 4. Precision and accuracy of water samples.

Analytes	Fortified Conc.	Synthetic Hard Water[b]		Surface Water[c]	
	µg/L	Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
acenaphthylene	2.0	90.9	2.2	92.1	1.8
acetochlor	2.0	98.7	0.5	97.0	2.6
alachlor	2.0	96.9	1.2	95.0	1.6
aldrin	2.0	93.3	3.4	93.2	2.9
ametryn	2.0	90.8	4.2	90.0	3.2
anthracene	2.0	95.3	2.2	94.8	1.6
atraton	2.0	85.8	5.7	88.2	4.5
atrazine	2.0	92.2	3.2	93.3	0.8
benzo[*a*]anthracene	2.0	92.1	2.4	91.9	1.7
benzo[*a*]pyrene	2.0	87.4	2.3	86.8	2.4
benzo[*b*]fluoranthene	2.0	85.7	3.6	85.8	3.0
benzo[*g, h, i*]perylene	2.0	79.1	1.6	78.4	6.1
benzo[*k*]fluoranthene	2.0	87.9	3.3	85.1	3.5
BHT	2.0	92.6	1.6	93.8	2.4
bromacil	2.0	95.4	4.2	96.1	2.7
butachlor	2.0	93.5	3.6	93.4	2.1
butylate	2.0	95.0	2.0	95.8	3.4
butylbenzylphthalate	2.0	92.8	3.2	92.5	1.9
chlordane, cis-	2.0	89.0	1.7	89.8	2.3
chlordane, trans	2.0	89.7	1.6	90.3	1.3
chlorfenvinphos	2.0	95.2	4.1	95.1	2.3
chlorobenzilate	2.0	94.3	4.1	93.2	1.7
chloroneb	2.0	95.6	1.2	95.6	1.5
chlorothalonil	2.0	97.1	1.9	97.8	1.6
chlorpropham	2.0	97.1	1.4	97.4	2.0
chlorpyrifos	2.0	95.2	2.5	95.0	2.2
chrysene	2.0	94.8	2.7	91.8	2.3
cyanazine	2.0	98.7	7.1	97.4	5.4
cycloate	2.0	94.4	2.3	94.5	2.0
dacthal (DCPA)	2.0	97.5	1.7	98.0	0.6
DDD, 4,4′-	2.0	86.5	3.5	87.5	1.4
DDE, 4,4′-	2.0	83.6	1.7	84.2	2.6
DDT, 4,4′-	2.0	82.4	2.1	82.5	0.6
DEET	2.0	99.3	2.4	99.3	2.9
di(2-ethylhexyl)adipate	2.0	72.1	1.4	73.4	2.0
di(2-ethylhexyl)phthalate	2.0	73.0	3.6	74.7	3.1
dibenzo[a,h]anthracene	2.0	75.8	2.1	75.2	6.3
dibutyl phthalate	2.0	116.0	2.3	114.0	1.0
dichlorvos	2.0	90.0	3.6	91.4	2.2
dieldrin	2.0	90.4	4.9	88.3	1.8
diethylphthalate	2.0	96.7	2.3	96.4	1.1
dimethipin	2.0	45.0	24.0	34.8	17.0
dimethylphthalate	2.0	94.3	1.8	95.2	1.9
DIMP	2.0	84.6	3.5	88.3	5.1

Biotage®

Analytes	Fortified Conc.	Synthetic Hard Water[b]		Surface Water[c]	
	µg/L	Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
dinitrotoluene, 2,4-	2.0	93.4	5.6	98.2	3.0
dinitrotoluene, 2,6-	2.0	92.9	3.3	95.5	1.1
diphenamid	2.0	95.5	2.6	95.2	1.8
disulfoton	2.0	80.0	5.1	73.4	10.0
endosulfan I	2.0	68.1	2.7	59.4	7.7
endosulfan II	2.0	90.6	6.2	89.7	4.8
endosulfan sulfate	2.0	92.6	3.2	92.1	2.4
endrin	2.0	90.6	3.4	93.0	2.5
EPTC	2.0	93.0	2.0	94.6	2.0
ethion	2.0	92.8	3.2	89.3	1.1
ethoprop	2.0	99.1	2.1	98.7	2.6
ethyl parathion	2.0	95.4	1.9	96.3	6.8
etridiazole	2.0	96.1	2.6	95.1	2.8
fenamiphos	2.0	90.3	4.4	91.8	2.9
fenarimol	2.0	98.7	4.5	94.4	1.8
fluorene	2.0	93.3	1.7	93.0	2.4
fluridone	2.0	89.1	6.1	87.0	10.0
HCCPD	2.0	83.8	2.7	85.4	3.8
HCH, alpha	2.0	95.2	2.6	96.4	3.6
HCH, beta	2.0	99.6	3.1	98.5	2.9
HCH, delta	2.0	94.8	3.3	94.2	2.4
HCH, gamma (lindane)	2.0	92.8	1.8	94.1	2.8
heptachlor	2.0	90.5	3.7	91.4	3.7
heptachlor epoxide	2.0	95.4	4.5	92.3	3.0
hexachlorobenzene	2.0	93.6	1.3	92.8	1.8
hexazinone	2.0	95.1	1.6	94.9	4.4
indeno[1,2,3-c,d]pyrene	2.0	80.6	2.7	80.6	6.3
isophorone	2.0	90.9	2.3	91.9	1.4
methoxychlor	2.0	93.4	3.6	93.8	1.8
methyl parathion	2.0	95.4	2.6	98.3	4.9
metolachlor	2.0	97.0	1.6	98.0	1.6
metribuzin	2.0	93.7	1.0	93.6	1.5
mevinphos	2.0	95.0	2.5	95.6	1.3
MGK 264(a)	1.6	95.7	2.3	93.9	2.2
MGK 264(b)	0.4	98.4	3.6	98.1	4.0
molinate	2.0	93.9	2.2	94.5	1.7
napropamide	2.0	91.3	4.4	93.1	1.9
nitrofen	2.0	93.2	3.3	95.6	1.8
nonachlor, trans	2.0	86.7	3.7	86.3	2.9
norflurazon	2.0	97.1	3.1	96.0	1.5
oxyfluorfen	2.0	93.5	3.2	95.3	0.8
pebulate	2.0	92.8	2.3	94.3	2.4
pentachlorophenol	8.0	96.8	2.7	96.6	2.2
permethrin, cis	2.0	77.7	3.1	77.3	2.1
permethrin, trans	2.0	80.1	2.9	79.1	1.8

Biotage®

Analytes	Fortified Conc.	Synthetic Hard Water[b]		Surface Water[c]	
	µg/L	Mean % Recovery[d]	RSD	Mean % Recovery[d]	RSD
phenanthrene	2.0	93.8	1.2	94.3	2.3
phorate	2.0	92.0	1.5	93.0	4.1
phosphamidon	2.0	92.7	5.3	94.6	1.7
profenofos	2.0	94.2	2.9	95.7	2.0
prometon	2.0	87.4	6.1	86.1	3.6
prometryn	2.0	93.3	4.5	91.5	2.8
pronamide	2.0	95.7	2.3	96.4	1.7
propachlor	2.0	97.1	2.8	96.3	1.3
propazine	2.0	93.8	1.8	93.0	3.0
pyrene	2.0	93.0	2.5	93.7	1.4
simazine	2.0	91.8	2.7	92.9	2.7
simetryn	2.0	88.4	3.8	89.6	3.4
tebuconazole	2.0	97.0	2.3	96.8	2.4
tebuthiuron	2.0	97.7	6.1	98.8	8.0
terbacil	2.0	96.1	2.0	96.5	2.6
terbutryn	2.0	92.4	5.4	89.6	4.2
tetrachlorvinphos	2.0	91.7	3.9	91.8	3.1
triadimefon	2.0	97.6	3.9	93.8	4.2
tribufos	2.0	95.2	3.7	93.5	2.3
trifluralin	2.0	100.0	1.8	99.1	2.0
vernolate	2.0	92.6	2.4	94.0	2.5
vinclozolin	2.0	97.5	1.1	97.6	3.9
PCB congeners (by IUPAC#)					
2-chlorobiphenyl (1)	2.0	90.8	2.0	92.0	2.4
4-chlorobiphenyl (3)	2.0	91.8	2.3	92.4	3.2
2,4′-dichlorobiphenyl (8)	2.0	94.9	2.0	95.0	2.7
2,2′,5-trichlorobiphenyl (18)	2.0	95.2	2.2	94.4	1.8
2,4,4′-trichlorobiphenyl (28)	2.0	93.6	1.9	92.9	3.4
2,2′,3,5′-tetrachlorobiphenyl (44)	2.0	94.4	3.8	92.1	2.1
2,2′,5,5′-tetrachlorobiphenyl (52)	2.0	91.1	3.6	91.4	2.5
2,3′,4′,5-tetrachloroobiphenyl (70)	2.0	87.8	1.2	89.4	1.2
2,3,3′,4′,6-pentachlorobiphenyl (110)	2.0	86.6	1.3	88.0	1.8
2,3′,4,4′,5-pentachlorobiphenyl (118)	2.0	83.3	2.9	86.9	1.7
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	2.0	82.6	1.8	84.7	2.2
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	2.0	82.5	2.0	85.9	2.8
2,2′,4,4′,5,5′- hexachlorobiphenyl (153)	2.0	81.5	1.8	83.4	2.9
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	2.0	79.3	2.6	81.3	1.6
Surrogate Analytes					
1,3-dimethyl-2-nitrobenzene	2.0	88.7	2.9	90.6	3.8
benzo[*a*]pyrene-d_{12}	2.0	94.1	2.4	93.4	3.2
triphenyl phosphate	2.0	92.2	1.8	92.4	1.7

Biotage®

Biotage® VacMaster™ Disk Method Summary

1. Repeat the following steps for each active Biotage® VacMaster™ Disk station.
2. Set up the VacMaster Disk manifolds ensuring all waste lines and vacuum lines are attached. Set the vacuum pump to -24"Hg.
3. Prepare the disk holder assembly (47 mm): ensure the support screen is flat in the center of the disk holder. Place the Atlantic® DVB Disk on top of the support screen with the ripples of the disk on top and add any prefilters on top of the disk. Place the disk holder assembly on the VacMaster Disk manifold ensuring there is a tight seal with the luer fitting.
4. If using the multifunnel, place onto the disk holder assembly. If not using the multifunnel, omit those directions throughout the method.
5. Condition the SPE Disk:
   a. Guide for each conditioning step in table 5 below:
      i. Measure the appropriate volume of solvent into a graduated cylinder and pour into the disk holder assembly.
      ii. Using a Nalgene Wash Bottle (phthalate free), rinse the multifunnel and disk holder in a circle for about 3 seconds using the same SOLVENT (approximately 5 additional mL).
      iii. SATURATE the disk for the time indicated (in SECONDS). (Saturate means: quickly turn the knob to the appropriate waste destination and back to the "OFF" position. This brings the solvent into the disk media bed).
      iv. SOAK the disk for the time indicated (in SECONDS).
      v. DRAIN to the appropriate waste destination for the time indicated (in SECONDS). Switch to the "OFF" position.
6. Load the Sample:
   a. For multifunnel: quickly and efficiently angle the bottle to rest on the multifunnel upside-down.
   b. For no multifunnel: pour a portion of the sample into the disk holder.
   c. Adjust the vacuum between -10"Hg and -15"Hg for sample load (please note, if the sample is flowing too slowly, the vacuum can be increased). Drain the sample to "AQUEOUS" waste. Continue to pour the sample into the disk holder ensuring the disk does not go dry or overflow for the duration of sample load.
7. Wash Sample Container:
   a. Guide for each wash step in table 6 below:
      i. Measure appropriate volume of REAGENT into a graduated cylinder, pour into the sample bottle, and swirl around. Pour the solvent in the sample bottle into the disk holder assembly.
      ii. Using a Nalgene Wash Bottle (phthalate free), rinse the multifunnel and disk holder in a circle for about 3 seconds using the same REAGENT (approximately 5 additional mL).
      iii. SATURATE the disk for the time shown (IN SECONDS).
      iv. SOAK the disk for the time shown (IN SECONDS).
      v. DRAIN to "AQUEOUS" waste for the time shown (in SECONDS). Switch to the "OFF" position.

Table 5. Disk conditioning.

Solvent	Volume (mL)	Saturate (sec.)	Soak (sec.)	Waste Destination	Drain (sec.)
Ethyl Acetate	11	1	60	Organic	30
Dichloromethane	11	1	60	Organic	30
Methanol	11	1	60	Organic	5
Reagent Water	11	1	5	Organic	5

Table 6. Sample container wash.

Reagent	Volume (mL)	Saturate (sec.)	Soak (sec.)	Waste Destination	Drain (sec.)
Reagent Water	10	1	10	Aqueous	30

Biotage®

8. Air Dry the SPE Disk:
    a. Return the vacuum to -24"Hg and continue to air dry the SPE disk to "AQUEOUS" waste for an additional 60 SECONDS. Switch to the "OFF" position.
    b. Remove the sample bottle from the multifunnel.
9. Elute the SPE Disk: (Please note: the elutions will go into the collection flask inside the chamber, not to waste containers)
    a. Place a clean 125 mL 24/40 tapered Erlenmeyer flask or 40 mL VOA vial using the VOA vial insert into the VacMaster Disk collection chamber. Place the cover on the chamber. Remove the disk holder assembly and place the disk holder assembly into the luer fitting on top of the collection chamber. Attach the luer fitting of the collection chamber assembly onto the manifold.
    b. Guide for each elution step in table 7 below:
        i. Measure appropriate volume of solvent into a graduated cylinder, pour into the sample bottle, and swirl around. Pour the solvent in the sample bottle into the disk holder assembly.
        ii. Using a Nalgene Wash Bottle (phthalate free), rinse the multifunnel and disk holder in a circle for about 3 seconds using the same SOLVENT (approximately 5 additional mL).
        iii. SATURATE the disk for the time indicated (in SECONDS) to "ORGANIC".
        iv. SOAK the disk for the time indicated (in SECONDS).
        v. DRAIN to "ORGANIC" for SECONDS. Switch to the "OFF" position.
        vi. Remove the chamber lid to release the vacuum from inside the chamber.

Table 7. Disk elution.

Solvent	Volume (mL)	Saturate (sec.)	Soak (sec.)	Waste Destination	Elute (sec.)
Ethyl Acetate	8	1	90	Organic	30
Dichloromethane	13	1	90	Organic	30
Dichloromethane	13	1	90	Organic	60

EUROPE
Main Office: +46 18 565900
Toll Free: +800 18 565710
Fax: +46 18 591922
Order Tel: +46 18 565710
Order Fax: +46 18 565705
order@biotage.com
Support Tel: +46 18 56 59 11
Support Fax: + 46 18 56 57 11
eu-1-pointsupport@biotage.com

NORTH & LATIN AMERICA
Main Office: +1 704 654 4900
Toll Free: +1 800 446 4752
Fax: +1 704 654 4917
Order Tel: +1 704 654 4900
Order Fax: +1 434 296 8217
ordermailbox@biotage.com
Support Tel: +1 800 446 4752
Outside US: +1 704 654 4900
us-1-pointsupport@biotage.com

JAPAN
Tel: +81 3 5627 3123
Fax: +81 3 5627 3121
jp_order@biotage.com
jp-1-pointsupport@biotage.com

CHINA
Tel: +86 21 68162810
Fax: +86 21 68162829
cn_order@biotage.com
cn-1-pointsupport@biotage.com

KOREA
Tel: +82 31 706 8500
Fax: +82 31 706 8510
korea_info@biotage.com
kr-1-pointsupport@biotage.com

INDIA
Tel: +91 11 45653772
india@biotage.com

Distributors in other regions are listed on www.biotage.com

Literature Number: AN081-HOR-V.1

Biotage®