Empore™ SDB-XC SPE Disks are Combined with the Empore™ EZ-Trace Extraction System to Extract SVOCs in Water using EPA Method 525.3

Application Note
Environmental

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Abstract

CDS Empore™ (formerly 3M™ Empore™) EZ-Trace is an extraction system allowing for simultaneous extractions and significantly reducing preparation time of multiple samples. This application note demonstrates the performance of the EZ-Trace using the Empore™ SDB-XC disk under EPA Method 525.3 for reagent grade and ground water. The recovery is determined for 125 analytes for this method and demonstrates that the Empore™ EZ-Trace system provides a clean and efficient extraction with the Empore™ SDB-XC disk.

Introduction

EPA Method 525.3 is a targeted list of 125 semi-volatile organic compounds in drinking water, 15 more than the previous version 525.2. Target compounds of interest in this method include organochlorine, -nitrogen and -phosphorous pesticides, polycyclic aromatic hydrocarbons (PAHs), and polychlorinated biphenyl (PCBs).[1] In addition to an expanded list of target compounds, Method 525.3 also was developed to provide an update on methods in sample preservation and sample preparation by exploring new solid phase extraction (SPE) technologies. Method 525.2 to 525.3 saw the transition from C18 silica-based sorbents to newer polymeric based sorbents. The attractiveness of polymeric based sorbents is that they provide greater stability at lower pH, are readily wettable with water, and remove the complications of silanol interactions that are present with silica-based sorbents.[2]

In this application note, multiple extractions are performed simultaneously by the Empore™ EZ-Trace to extract semi-volatile organic compounds from water samples, either reagent grade or ground, using 47 mm Empore™ SDB-XC, polymeric-based SPE disks. Extraction is performed on the EZ-Trace under the negative pressure of a vacuum pump. The extract is evaporated, diluted, and then analyzed via GC-MS. The recovery and concentrations of 125 analytical standards are determined from the five-point calibration curve of each standard. The results are assessed by the efficiency, reproducibility, and cleanliness of the extraction in combination with the accuracy and precision of the SDB-XC disk.

The validation data presented herein was determined on four replicate measurements for each sample from the same lot of SDB-XC disks. MDLs were not determined as part of this validation.

Experiment Setup

SPE disk:
Solid phase extraction (SPE) was done with Empore™ SDB-XC 47mm disks (catalog # 2240; Fisher Scientific part # 13110020; VWR part # 76333-136).

Extraction System:
Four extractions were performed simultaneously with the Empore™ EZ-Trace SPE vacuum system (catalog # 8000; VWR part # 76449-580).

Table 1: List of analytical calibration standards, surrogate, and internal standards and their concentrations.

Analytical Calibration Standards	Concentration (μg L^{-1})
organochlorine pesticide (OCB)	500
organonitrogen pesticide (ONP)	500
organophosphorous pesticide (OPP)	500
polycyclic aromatic hydrocarbon (PAH)	500
polychlorinated biphenyl (PCB)	500
surrogate	500
Internal Standards (IS)	
polycyclic aromatic hydrocarbon (PAH)	500
pentachlorophenol- $^{13}C_6$ (PCP)	1000

Chemicals:
EPA Method 525.3 calibration standards, internal standards, and the surrogate were purchased as pre-mixed calibrated solutions from Restek (Bellefonte, PA). Calibration standards were purchased as five separate solutions and are shown in Table 1 along with their concentrations. Also shown in Table 1 is the surrogate concentration as well as the internal standards (IS) and their concentrations. The GC-MS calibration standard decafluorotriphenylphosphine (DTFPP) was also purchased from Restek. Reagents potassium dihydrogen citrate, L-ascorbic acid, and ethylenediaminetetraacetic acid (EDTA) trisodium were all purchased from Sigma Aldrich (St. Louis, MO). Methanol and dichloromethane were purchased from Sigma Aldrich (St. Louis, MO) and ethyl acetate from EMD Millipore (Darmstadt, Germany). Water was treated in house using a Milli-Q Water Treatment System.

Preparartion of Standards:
The primary dilution standard (PDS) was prepared at a concentration of 50 µg L^{-1} for each of the 5 analytical calibration standards. This solution was refrigerated until ready for use.

Water samples, either reagent grade or ground, were first treated with approximately 0.1 g L-ascorbic acid, 0.35 g EDTA, and 9.4 g potassium dihydrogen citrate. Each water sample was buffered to pH 3.8 to prevent microbial growth and analyte degradation.

Methods:
1. Spike the first reagent water sample with 4 µL of PDS and 10 µL of surrogate.

2. Assemble all 4 disk adapter and glass filtration assemblies on the Empore™ EZ-Trace using a 47 mm Empore™ SDB-XC Solid Phase Extraction (SPE) disk.

3. Wash each disk with 5 mL ethyl acetate. Adjust the waste valve for organic waste. Pull a small amount of solvent through the disk by vacuum by turning the primary flow path valve to the waste position; shut off the vacuum valve and allow the disk to soak for about one minute.

4. Then wash each extraction apparatus and disk by adding 10 mL of methanol to the reservoir. Pull a small amount through the disk; shut off the vacuum valve and allow the disk to soak for about one minute. Do not let the disk dry.

5. Condition each disk by adding approximately 10 mL of water to the reservoir. Adjust the waste valve for aqueous waste and pull a small amount through the disk. Do not let the disk dry.

6. Add each 1 L water sample to a filtration reservoir. Under vacuum, filter as quickly as the vacuum will allow.

7. Before all the water samples have filtered through their SPE disks, wash each bottle with 10 mL of water and add to the filtration reservoirs.

8. Before disk drying, wash each SPE disk with another 5-10 mL of water to wash any salts off the disks.

9. Dry the disks under -20 psi vacuum for 10 minutes. At 10 minutes, shut the primary flow path valve off.

10. Rinse the sample bottle with 5 mL of ethyl acetate and add to the reservoir. Adjust the primary flow path valve to elute and pull a small amount through the disk. Let the disk soak for one minute.

11. Repeat step 9 using 5 mL dichloromethane.

12. Rinse the filtration reservoir with 5 mL of 1:1 ethyl acetate : dichloromethane.

13. Dry the combined eluant with 10 g of granular anhydrous sodium sulfate and collect in an evaporation vial. Rinse the collection tube and sodium sulfate each with a 5 mL portion of dichloromethane into the evaporation vial.

14. Dry the extract to just below 1 mL under a gentle stream of nitrogen (warm gently). Allow the eluate to come back to room temperature.

Table 2: Overview of the GC and MS parameters and methods.

GC Parameters	
Column:	Restek RXI-5sil-MS (30m x 0.25mm x 0.25µm)
Inlet Temp:	275°C
Transfer Line Temp:	280°C
Injection Mode:	Splitless
Carrier Gas:	He at 40.2 cm s^{-1} (constant flow)
Oven Program:	70°C hold for 1 min, 70 to 200°C at 10°C min^{-1}, 200 to 320°C at 7°C min^{-1}
Mass Spectrometer Parameters	
Solvent Delay:	5 min
Threshold:	0
Mass Range:	5.00-18.90min: 45-380 m/z 18.90-19.90min: 135-415 m/z 19.90-21.65min: 165-365 m/z 21.65-34.00min: 45-450 m/z
Scan Time:	0.5 s

15. Dilute the eluate with ethyl acetate back to the 1 mL mark on the evaporation vial. Transfer this solution to a 1 mL GC-MS vial. Add 4 and 10 µL of PCP and PAH IS respectively to the vial.

16. Repeat this procedure for two other reagent water samples by adding 40 and then 100 µL aliquots of PDS to the sample. Repeat this procedure for one ground water sample adding 40 µL of the PDS to the sample. For all samples, add 10 µL of surrogate. Determine the recovery of each analytical calibration standard using GC-MS.

GC-MS Analysis

The extract analysis was performed on a Shimadzu GC-2010 Gas Chromatograph with a splitless injection port interfaced to a Shimadzu GC-MS QP2010 (Kyoto, Japan) and a 30m x 0.25mm x 0.25µm Restek RXI-5sil-MS GC column. GC-MS parameters are shown below in Table 2:

The GC-MS method described in Table 2 was used on the recommendation of EPA Method 525.3. The mass spectrometry method was modified based on the needs of these experimental conditions. Modifications

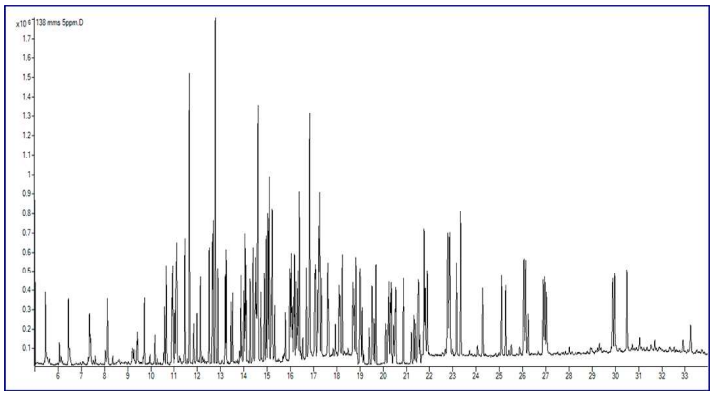

Figure 1. Chromtogram of the separation of 125 analytical calibration standards, 3 surrogate, and 4 IS compounds (132 total).

to mass spectrometry methods are permitted by EPA method 525.3. Ions used for quantification of each standard were selected by those recommended by EPA Method 525.3. A five-point calibration curve was constructed for each analytical calibration standard using concentrations of 0.2, 0.5, 1.0, 2.0, and 5.0 µg L^{-1}. The MS was tuned successfully using DTFPP.

Results and Discussion:

Figure 1 shows the GC chromatogram of the 125 standards, 3 surrogates, and 4 IS compounds under EPA Method 525.3. The compounds are well-separated under the conditions of the GC method described in Table 2.

For the lowest concentration of PDS, 0.2 µg L^{-1}, 118 out of 125 were detected with a recovery ≥ 70%. For these compounds, the average recovery was 89.9% with 7.4% RSD. Of the remaining three water samples (two reagent grade and one tap water), 123 out of 125 compounds were detected with a recovery ≥ 70%. The average recovery of these compounds was between 90.9 and 94.4 with 3.2-5.3% RSD. A summary of this data is located in Table 3. As allowed by EPA Method 525.3, the permissible recovery range is between 70 and 130%, except for hexachloropentadiene (HCCPH) and HCB. For these two compounds, the permissible recovery range is 60-130%.

The recovery of all 125 compounds is shown in Table 4 for three different concentrations of PDS in reagent grade water and one concentration of PDS in ground water. The R^2 of the calibration curve for each analytical calibration standard can be found in the final column of Table 4. The average R^2 was 0.999 with 0.3% RSD.

Compounds yielding notably low recoveries include dimethipin and disulfoton. Dimethipin, for example, is also noted to have remarkably low recoveries in

EPA Method 525.3 as well. The average recovery of dimethipin and disulfoton in this experimental work is 31.5 and 39.1% respectively. Other compounds, such as HCCPD, phorate, benzy butyl phthalate, bis(2-ethylhexyl)adipate, and di(2-ethylhexyl)phthalate yielded recoveries outside the acceptable recovery range for the reagent grade water sample containing the lowest concentration of PDS. For higher concentrations of PDS, the recovery of these compounds were within the acceptable recovery limits.

The number of compounds having recoveries exceeding 100% was minimized in this experimental work. At 0.2 µg L^{-1} PDS, only 11 of 125 compoundsproduced recoveries greater than 100% with a maximum recovery of 105.8%. For concentrations of 2.0 and 5.0 µg L^{-1}, this number is reduced to 1 compound with recovery greater than 100%. No compounds had a recovery exceeding 130%.

Compounds such as phthlatates are cited by EPA Method 525.3 as potential candidates for contamination from plastics. A high background concentration of phthalates would likely result in low recoveries of phthalate standards. However, phthalate recoveries were not low in this experimentall work, suggesting that their background concentrations were low and the system was clean. This result indicates that the Empore™ EZ-Trace is excellent for performing clean extraction procedures in addition to significantly reducing the time needed for performing replicate experimental measurements.

Conclusions:

The performance of the Empore™ EZ-Trace was demonstrated as part of this experimental work, performing 4 extractions of 125 standard semi-volatile organic compounds simultaneously on the Empore™ SDB-XC SPE disk. As few as 118 of 125 compounds were detected with ≥ 70% at 0.2 µg L^{-1} of PDS in reagent grade water. For concentrations of PDS greater than 0.2 µg L^{-1}, 123 of 125 compounds were detected with ≥ 70% recovery. For five of the compounds whose recoveries were outside the acceptable limits at 0.2 µg L^{-1}, their recoveries fall within the acceptable limits for the higher concentrations of PDS. Additionally, of the 11 compounds whose recoveries were greater than 100% at 0.2 µg L^{-1}, the maximum observed recovery was 105.8%. Common contaminants, such as phthalates from plastics did not produce low recoveries, indicating their background concentrations were low and the extractions were clean.

Table 3: Summary of results for three different concentrations of PDS in reagent grade water and one sample of tap water.

Water	Reagent Grade			Ground
µg L^{-1}	0.2	2.0	5.0	2.0
≥ 70%	118	123	123	123
avg Rec. (%RSD)	89.9 (7.4)	90.9 (5.8)	94.4 (3.4)	93.4 (4.5)

In summary, the CDS Empore™ EZ-Trace, combined with the Empore™ SDB-XC SPE disk, provide a clean and efficient screening method while still produced high analyte recoveries for the compounds listed by EPA Method 525.3.

References:

1. Environmental Protection Agency. Method 525.3. Determination of Semivolatile Organic Chemicals in Drinking Water by Solid Phase Extraction and Capillary Column Gas Chromatography / Mass Spectrometry (GC/MS), 2012.
2. Grimmett, P.E.; Munch, J.W.; Development of EPA Method 525.3 for the analysis of semivolatiles in drinking water, *R. Soc. Chem.*, 2013.

Table 4: Average recovery of 125 analytical calibration standards along with 3 surrogate compounds for 3 samples of reagent water containing different concentrations of standards and one sample of ground water. Shown in parentheses are the standard deviations (s). For each water sample, there are four replicate measurements. R^2 values were determined determined from a 5-point calibration curve of each standard.

	Reagent Grade Water			Ground Water	
Concentration (μg L^{-1})	**0.2**	**2.0**	**5.0**	**2.0**	
	%Rec. (s)	**%Rec. (s)**	**%Rec. (s)**	**%Rec. (s)**	**R^2**
2,2',3,4,4',5,5'-Heptachlorobiphenyl (PCB 180)	87.1 (4.2)	91.8 (3.2)	95.2 (2.3)	98.4 (2)	0.9976
2,2',3,4,4',5'-Hexachlorobiphenyl (PCB 138)	84.6 (3.4)	91.8 (4.3)	95.7 (1.6)	98.0 (2.3)	0.9973
2,2',3,4',5',6-Hexachlorobiphenyl (PCB 149)	86.9 (4.4)	90.2 (3.3)	95.5 (2.3)	95.5 (1.4)	0.9978
2,2',3,5'-Tetrachlorobiphenyl (PCB 44)	80.2 (6.3)	91.4 (2.5)	94.7 (1.7)	93.5 (1.8)	0.9987
2,2',4,4',5,5'-Hexachlorobiphenyl (PCB 153)	84.1 (7.6)	92.5 (4.6)	96.1 (2)	98.7 (1.6)	0.9975
2,2',5,5'-Tetrachlorobiphenyl (PCB 52)	83.1 (8.2)	91.8 (2.6)	95.3 (1.3)	92.3 (1.4)	0.9979
2,2',5-Trichlorobiphenyl (PCB 18)	82.6 (3.6)	88.9 (2.8)	94.8 (1.2)	91.4 (1.5)	0.9986
2,3,3',4',6-Pentachlorobiphenyl (PCB 110)	86.3 (5.7)	90.8 (2.8)	96.2 (2.6)	94.6 (1.1)	0.9972
2,3',4,4',5-Pentachlorobiphenyl (PCB 118)	83.7 (5.8)	92.5 (3.8)	95.3 (2.2)	97.9 (2.3)	0.9983
2,3',4',5-Tetrachlorobiphenyl (PCB 70)	85.4 (5)	91.9 (2.5)	95.2 (1.6)	94.3 (1.4)	0.9991
2,4,4'-Trichlorobiphenyl (PCB 28)	87.5 (6)	89.5 (3.6)	93.9 (1.3)	92.6 (2.2)	0.9990
2,4'-Dichlorobiphenyl (PCB 8)	79.3 (0.8)	90.4 (3)	94.6 (1.1)	91.1 (1.3)	0.9990
2,4-Dinitrotoluene	88.9 (5.3)	88.9 (2.2)	94.7 (2.3)	91.3 (1.9)	0.9999
2,6-Dinitrotoluene	90.5 (4)	86.7 (2)	94.3 (2)	91.1 (1.9)	0.9996
2-Chlorobiphenyl (PCB 1)	73.3 (4.9)	83.5 (2.5)	91.6 (2)	90.6 (1.4)	0.9988
4,4'-DDD	85.7 (3.8)	94.3 (3.9)	95.5 (2.1)	97.1 (2.1)	0.9985
4,4'-DDE	84.4 (4.5)	92.1 (2.5)	96.6 (1.7)	94.5 (1.5)	0.9981
4,4'-DDT	87.2 (4.6)	89.6 (3.3)	96.0 (2.1)	98.7 (1.8)	0.9990
4-Chlorobiphenyl (PCB 3)	79.2 (3)	83.9 (2.2)	90.9 (2)	92.8 (0.8)	0.9996
Acenaphthylene	83.7 (0.7)	84.3 (2.3)	91.4 (2.2)	91.3 (1.1)	0.9995
Acetochlor	94.1 (4)	93.5 (2.7)	94.2 (1.7)	95.1 (1.2)	0.9991
Alachlor	92.3 (3.1)	94.2 (2.7)	95.1 (1.3)	94.6 (2.5)	0.9990
Aldrin	78.0 (11.3)	81.2 (3)	90.6 (1.5)	90.0 (1.1)	0.9995
Ametryn	84.8 (3.9)	92.8 (2.5)	97.9 (3.1)	90.6 (2.2)	0.9973
Anthracene	89.1 (3.6)	95.4 (2.5)	96.8 (0.8)	92.4 (1.1)	0.9990
Atraton	89.1 (4.7)	90.2 (2.6)	98.1 (1.4)	94.7 (2.8)	0.9980
Atrazine	93.7 (2.5)	96.1 (2.7)	95.2 (1.5)	98.7 (1.6)	0.9990

	Reagent Grade Water			Ground Water	
Concentration (μg L^{-1})	**0.2**	**2.0**	**5.0**	**2.0**	
	%Rec. (s)	**%Rec. (s)**	**%Rec. (s)**	**%Rec. (s)**	**R^2**
Benzo[a]pyrene	93.6 (4.1)	95.9 (2.3)	96.4 (0.9)	95.7 (1.1)	0.9992
Benzo[b]fluoranthene	94.1 (3.5)	91.3 (2.6)	93.0 (1.1)	91.2 (0.8)	0.9988
Benzo[g,h,i]perylene	92.1 (3.3)	91.6 (2.2)	94.3 (0.6)	93.4 (0.8)	0.9998
Benzo[k]fluoranthene	95.9 (5.4)	92.0 (2.7)	93.3 (1.8)	92.5 (2.1)	0.9994
Benzyl butyl phthalate	94.3 (2.8)	91.8 (3)	91.9 (2.1)	93.5 (0.8)	0.9929
Bis(2-ethylhexyl)adipate	35.6 (2.1)	95.8 (3.9)	99.5 (2)	96.1 (3.1)	0.9975
Bromacil	67.4 (4.6)	85.3 (3.3)	93.3 (2.4)	98.8 (10.5)	0.9959
Butachlor	103.0 (4.6)	79.6 (2.7)	94.6 (1)	77.6 (2)	0.9989
Butylate	90.9 (3.3)	93.4 (3)	95.9 (1.8)	93.7 (1.6)	0.9997
Butylated hydroxytoluene (BHT)	90.2 (7.2)	85.2 (2)	92.8 (1.7)	90.1 (1.4)	0.9998
Chlofenvinphos	79.8 (3.7)	83.5 (5.2)	90.1 (1.8)	89.0 (2.1)	0.9990
Chlorobenzilate	91.2 (6)	92.9 (2.8)	94.7 (1.8)	94.0 (1.5)	0.9982
Chloroneb	87.9 (2.4)	93.2 (4.4)	94.8 (2.2)	96.8 (2.6)	0.9995
Chlorothalonil	96.7 (9.7)	88.5 (1.6)	94.6 (1.9)	96.5 (1.1)	0.9997
Chlorpropham	91.3 (4.6)	96.8 (2.8)	94.7 (1.5)	91.9 (1.7)	0.9994
Chlorpyrifos	97.0 (6.9)	94.3 (3.3)	96.6 (3.6)	99.1 (2.8)	0.9992
Chrysene	96.1 (9.8)	90.0 (2.9)	92.9 (1.8)	94.4 (1.7)	0.9997
cis-Chlordane	90.9 (3.7)	96.0 (2.4)	96.4 (0.9)	95.6 (1.1)	0.9989
cis-Permethrin	87.0 (8.1)	91.7 (2.6)	90.4 (3.2)	96.7 (0.8)	0.9987
Cyanazine	87.2 (2.9)	88.2 (2.7)	92.7 (2)	94.2 (2)	0.9989
Cycloate	100.1 (10)	82.1 (2.7)	77.3 (1.7)	86.3 (1.2)	0.9995
DCPA	88.8 (3.1)	95.9 (1.6)	97.8 (1.5)	94.2 (1.9)	0.9978
Di(2-ethylhexyl)phthalate	91.7 (5.8)	94.7 (2.9)	95.4 (2.6)	95.5 (1.7)	0.9651
Dibenzo[a,h]anthracene	49.4 (6.2)	90.2 (4)	102.4 (2.5)	90.3 (5.2)	0.9999
Dibutyl phthalate	101.5 (4.5)	93.7 (3)	94.9 (1.4)	91.9 (2.5)	0.9954
Dichlorvos	78.0 (0.9)	82.2 (2.8)	90.9 (1.8)	83.2 (3.8)	0.9999
Dieldrin	95.7 (3.1)	88.0 (2.1)	93.9 (2.1)	91.9 (1.6)	0.9983
Diethyl Phthalate	89.8 (8.6)	95.4 (2.4)	96.3 (2.2)	96.1 (1.2)	0.9995
Dimethipin	75.7 (6.4)	86.8 (2)	92.8 (2.5)	91.4 (1.7)	0.9986
Dimethyl phthalate	36.9 (10.7)	32.5 (4.1)	30.3 (3.2)	26.4 (15.3)	0.9995
Diphenamid	90.8 (4.1)	91.7 (1.7)	95.5 (1.9)	94.6 (1.7)	0.9993
Disopropyl methylphosphonate (DIMP)	92.9 (3.3)	96.9 (2.9)	96.5 (1.5)	93.9 (2.1)	0.9998
Disulfoton	92.7 (3.6)	72.7 (2.4)	89.7 (1.9)	90.6 (1.3)	0.9969
Endosulfan	28.2 (16.2)	23.1 (33.4)	54.8 (5.4)	50.4 (25)	0.9992
Endosulfan II	90.2 (10.9)	96.2 (3.6)	96.8 (3)	97.8 (2)	0.9987
Endosulfan sulfate	89.4 (8.5)	98.0 (3.8)	96.1 (2)	98.9 (3.1)	0.9991
Endrin	105.5 (5.5)	96.9 (4.9)	96.7 (2.2)	99.5 (2.6)	0.9980

	Reagent Grade Water			Ground Water	
Concentration (μg L^{-1})	**0.2**	**2.0**	**5.0**	**2.0**	
	%Rec. (s)	**%Rec. (s)**	**%Rec. (s)**	**%Rec. (s)**	**R^2**
Eptam	88.1 (4.1)	87.5 (2.1)	94.8 (2.3)	90.5 (0.9)	0.9994
Ethion	92.2 (2.5)	91.3 (3.8)	94.2 (2.2)	95.6 (2.2)	0.9988
Ethoprophos	95.0 (4.7)	85.4 (1.7)	92.0 (2)	97.5 (1.6)	0.9999
Etridiazole	89.3 (5.2)	83.9 (2.4)	92.5 (2.1)	92.4 (2)	1.0000
Fenarimol	86.6 (5)	89.6 (3.1)	92.2 (1.5)	94.5 (2.1)	0.9986
Fluorene	85.1 (1.1)	98.9 (2)	97.8 (1.4)	103.2 (1.2)	0.9995
Fluridone	102.3 (3.5)	84.6 (2.5)	89.4 (1.6)	92.8 (1.9)	0.9995
Heptachlor	79.4 (4.6)	81.5 (2.9)	90.9 (1.6)	91.5 (0.9)	0.9996
Heptachlor epoxide	95.1 (4.6)	93.4 (3.4)	95.1 (2.9)	94.8 (2.2)	0.9978
Hexachlorobenzene (HCB)	79.2 (3.3)	83.7 (3.2)	90.3 (3.4)	91.5 (1.6)	0.9993
Hexachlorocyclopentadiene (HCCPD)	21.2 (27.4)	72.4 (9.1)	77.1 (1.6)	70.7 (4.5)	0.9961
Hexazinone	89.2 (1.7)	92.5 (3.5)	94.7 (1.5)	94.2 (2.1)	0.9986
Indeno[1,2,3-cd]pyrene	100.0 (4.2)	93.1 (3)	95.2 (1)	91.7 (1.7)	0.9998
Isophorone	90.4 (1.3)	88.5 (2.3)	96.3 (2.6)	87.0 (1.1)	0.9988
Methoxychlor	89.7 (2.2)	90.0 (2.9)	94.9 (1.9)	96.7 (2)	0.9985
Methyl parathion	70.5 (4.1)	88.4 (2.3)	93.2 (1.3)	88.1 (2)	0.9998
Metolachlor	90.7 (3.2)	97.3 (3)	97.1 (1.7)	99.3 (2.5)	0.9988
Metribuzin	91.4 (4.2)	94.0 (12.5)	91.5 (6.8)	91.4 (8.1)	0.9983
Mevinphos I	84.1 (39.3)	80.9 (38.6)	95.1 (2)	94.0 (2)	0.9999
Mevinphos II	97.0 (3)	93.3 (2.3)	95.1 (2)	94.0 (2)	0.9999
MGK 264 (a)	86.4 (4.8)	97.5 (2.8)	97.3 (1.5)	93.7 (2)	0.9990
MGK 264 (b)	89.1 (6.1)	95.8 (2.4)	97.3 (1.5)	93.7 (2.2)	0.9991
Molinate	92.0 (2.1)	90.4 (2.1)	96.1 (2.1)	92.9 (1.2)	0.9994
N,N-Diethyl-meta-toluamide (DEET)	91.7 (3.7)	95.9 (1.8)	96.9 (1.6)	96.9 (2)	0.9996
Napropamide	90.1 (6.3)	93.5 (3)	96.1 (2.1)	92.2 (3)	0.9967
Nitrofen	85.2 (5.5)	86.6 (2.4)	94.9 (2.5)	89.5 (1.9)	0.9985
Norflurazon	95.4 (2.5)	90.2 (2.7)	91.9 (1.7)	92.8 (1.7)	0.9995
Oxyfluorfen	85.8 (5.9)	80.1 (3)	93.1 (2)	89.4 (2.2)	0.9983
Parathion	96.9 (5.8)	90.7 (2.8)	94.3 (1.5)	92.4 (2.3)	0.9988
Pebulate	87.9 (1.8)	88.5 (2.2)	94.6 (1.9)	91.9 (1.7)	0.9995
Pentachlorophenol	95.4 (3.6)	93.8 (3.3)	95.4 (2.2)	98.8 (2.2)	1.0000
Phenanthrene	84.3 (2.6)	94.5 (2.5)	97.0 (1)	92.1 (1.1)	0.9985
Phorate	39.0 (70.8)	73.9 (5.8)	86.9 (1.6)	78.7 (7.2)	0.9977
Phosphamidon	101.7 (5.3)	93.7 (2.9)	95.0 (1.7)	95.5 (2.2)	0.9999
Profenofos	102.4 (6.7)	92.3 (2.6)	95.1 (2.6)	96.6 (1.5)	0.9998
Prometon	89.3 (4.5)	92.5 (2.6)	96.2 (1.3)	93.6 (2.5)	0.9984
Prometryn	82.7 (2.6)	92.8 (2.6)	96.2 (1.6)	91.3 (2.6)	0.9973

	Reagent Grade Water			Ground Water	
Concentration (μg L^{-1})	**0.2**	**2.0**	**5.0**	**2.0**	
	%Rec. (s)	**%Rec. (s)**	**%Rec. (s)**	**%Rec. (s)**	**R^2**
Pronamide	91.1 (4.3)	97.3 (2.6)	96.4 (1.4)	93.6 (2.4)	0.9984
Propachlor	93.1 (3.2)	96.0 (1.5)	96.9 (1.4)	95.8 (1.9)	0.9997
Propazine	89.8 (5)	98.1 (2.7)	96.6 (1.4)	95.3 (2.4)	0.9984
Pyrene	89.5 (3.3)	94.4 (2.6)	96.6 (1.5)	94.6 (1.3)	0.9993
Simazine	95.1 (3.3)	93.1 (2.4)	94.9 (1)	95.6 (1.4)	0.9993
Simetryn	83.9 (3.7)	93.5 (3.3)	96.8 (1.1)	95.5 (4)	0.9991
Tebuconazole	102.4 (14.9)	88.1 (3.6)	92.2 (1.4)	94.1 (2.8)	0.9984
Tebuthiuron	93.8 (4.2)	89.5 (2.3)	91.8 (0.9)	95.1 (2.5)	0.9997
Terbacil	96.3 (5.3)	99.1 (2.5)	95.1 (1.7)	93.5 (2.4)	0.9995
Terbutryn	88.1 (2.5)	91.5 (2.8)	96.1 (1.3)	92.8 (2.1)	0.9981
Tetrachlorvinphos	105.8 (7.2)	97.7 (2.7)	96.5 (1.7)	98.2 (2.3)	0.9997
trans-Chlordane	85.2 (6.5)	88.6 (3.6)	93.1 (4)	94.9 (1.1)	0.9992
trans-Nonachlor	86.3 (6)	91.6 (4.1)	94.6 (2.5)	96.4 (2.3)	0.9992
trans-Permethrin	89.6 (3.5)	88.6 (2.9)	92.9 (2.2)	94.0 (1.4)	0.9988
Triadimefon	87.3 (6)	94.1 (3.2)	95.2 (1.4)	93.3 (2.1)	0.9990
Tribufos	89.7 (5.1)	93.7 (3)	98.6 (2.3)	93.1 (1.4)	0.9979
Trifluralin	84.5 (5.2)	88.3 (2.5)	93.2 (0.8)	90.6 (2.6)	0.9993
Vernolate	88.4 (2.6)	88.1 (2.4)	94.7 (2.1)	91.4 (1)	0.9995
Vinclozoline	97.4 (4.8)	97.1 (2.8)	96.6 (1)	93.6 (3)	0.9982
α-HCH	101.8 (6.1)	96.4 (2.2)	94.8 (2.5)	89.3 (2)	0.9989
β-HCH	82.4 (3.8)	99.8 (3.7)	89.3 (1.8)	87.3 (2)	0.9982
γ-HCH (Lindane)	86.9 (5.8)	96.0 (2.8)	97.0 (1.3)	93.7 (2.2)	0.9992
δ-HCH	100.1 (5.7)	97.2 (2.7)	96.0 (1.2)	94.5 (2.3)	0.9993