Semivolatile Organic Compounds In Drinking Water By Solid-Phase Extraction and Capillary Column (GC/MS)

UCT Part Numbers

ECUNI525
1500 mg 525 C18,
83 mL cartridge

Or

EC525006-P
1500 mg 525 C18, 6 mL cartridge,
PE Frit

Method Summary:

A 1-liter water sample is fortified with surrogate analytes then extracted using a solid phase extraction (SPE) cartridge (ECUNI525). Analytes are eluted from the solid phase with a small amount of organic solvents. The extract is dried using anhydrous sodium sulfate and concentrated to approximately 0.7 mL using N2. IS are added and the volume adjusted to 1 mL with ethyl acetate. A splitless injection is made into a GC equipped with a capillary column, interfaced to an MS with either scan, SIM or SIS detection used for analysis. The GC/MS may be calibrated using standards prepared in solvent or using matrix-matched standards.

Internal standards are added after the extract concentration step. If the analyte pentachlorophenol is being measured, use IS 13C-pentachlorophenol at 1000 µg/mL.

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Sample Preservation:

Preservation reagents, listed in the table below, are added to each sample bottle as dry solids prior to shipment to the field (or prior to sample collection).

Compound	Amount	Purpose
L-ascorbic acid	0.10 g/L	Dechlorination
Ethylenediaminetetraacetic acid, trisodium salt (EDTA)	0.35 g/L	Inhibit metal-catalyzed hydrolysis of targets
Potassium dihydrogen citrate	9.4 g/L	pH 3.8 buffer, microbial inhibitor

Procedure:

1. Cartridge Cleanup

a) Assemble an extraction system.
b) Rinse bottle holders and cartridges with 5 mL 1:1 EtOAc:DCM (ethylacetate:dichloromethane).
c) Draw half the volume through the cartridge and then soak for 1 min.
d) Draw remaining solvent through the cartridge.
e) Maintain full vacuum for 2 min to dry cartridge.

2. Cartridge Conditioning

a) Add 10 ml of methanol to each cartridge.
b) Soak for 1 minute.
c) Draw through leaving a thin layer of methanol on the cartridge frit.
Note: Do not let the cartridge go dry from this point until elution, otherwise recondition.
d) Add 10 mL of reagent water to each cartridge.
e) Draw through leaving a thin layer of water on the cartridge frit.

3. Sample Extraction

a) All field and QC samples, including LRBs and LFBs, must contain preservatives.
b) Ensure that sample pH is ≤ 4 (use a pH meter for reagent water).
c) Place sample bottle(s) in holder.
d) Adjust vacuum to fast drip flow rate. A flow of 10 mL/min is optimum.
e) After entire sample has extracted, rinse bottle with 10 mL reagent water.
f) Add rinse to cartridge.
g) Rinse cartridge using 10 mL reagent water to remove sample preservatives.
h) Dry cartridge for 10 min under full vacuum or nitrogen positive pressure.

4. Cartridge Elution

a) Insert 40-mL glass vial in manifold.
b) Rinse bottle, holder, and cartridge with 5 mL EtOAc.
c) Pour rinsate into cartridge.
d) Draw 1/2 volume through cartridge, soak 1 min then draw through completely.
e) Repeat using 5 mL DCM rinse.
f) Repeat using 5 mL EtOAc.
g) Repeat using 5 mL DCM.

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5. Extract Drying

a) Pre-rinse a drying tube containing 10-20 g of anhydrous sodium sulfate with DCM.
b) Quantitatively transfer the eluant through the sodium sulfate tube and collect.
c) Rinse the collection tube 2 x 5 mL of DCM.
d) Pass the DCM through the sodium sulfate and collect.

6. Extract Concentration

a) Concentrate extract to about 0.7 mL (not < 0.5 mL) under a gentle stream of N_2 in a water bath at 40 °C.
b) Transfer to a 1-mL volumetric flask, add IS and bring to volume using EtOAc.

7. Analyze by GC/MS

Internal Standards	CASRN	Solvent	PDS conc.
acenaphthene-d10 (IS 1)	15067-26-2	acetone	500 µg/mL
phenanthrene-d10 (IS 2)	1517-22-2	acetone	500 µg/mL
chrysene-d12 (IS 3)	1719-03-5	acetone	500 µg/mL
^{13}C-pentachlorophenol (IS 4)	85380-74-1	methanol	1000 µg/mL

Surrogates	CASRN	Solvent	PDS conc.
1,3-dimethyl-2-nitrobenzene (SUR 1)	81-20-9	acetone	500 µg/mL
triphenyl phosphate (SUR 2)	115-86-6	acetone	500 µg/mL
benzo[a]pyrene-d12 (SUR 3)	63466-71-7	acetone	500 µg/mL

Instrument Conditions for GC Analysis	
Agilent 5975C MSD with 6890N GC	Restek RXI-5sil-MS 30m x 0.25 mm x 0.25 µm column
4-mm i.d. splitless gooseneck injection port liner	UCT#GCLGN4MM
Injection Port	250 °C
Injection Vol	1 µL with 1 min split delay
GC Oven Temp	Initial: 55 °C, hold 1 min Ramp 10 °C/min to 200 °C Ramp 7 °C/min to final T 320 °C Hold 0.36 min

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Precision and Accuracy Data Obtained for Method 525.3 Analytes Fortified in Reagent Water at Three Concentrations and Extracted Using UCT 525 Universal Cartridges; N=4; Full Scan GC/MS Analyses[a]

	Fortified Conc. 0.25 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
Analytes	**Mean Recovery %**	**RSD**	**Mean Recovery %**	**RSD**	**Mean Recovery %**	**RSD**
acenaphthylene	**101**	2.0	**93.6**	0.51	**99.8**	0.88
acetochlor	**99.0**	3.9	**93.6**	2.1	**104**	2.1
alachlor	**100**	7.3	**89.8**	0.72	**92.8**	1.0
aldrin	**77.0**	5.0	**78.4**	2.9	**85.0**	3.4
ametryn	**105**	4.8	**93.1**	1.3	**95.8**	1.1
anthracene	**106**	3.8	**92.3**	1.0	**104**	0.71
atraton	**112**	2.9	**90.3**	4.1	**96.8**	2.2
atrazine	**111**	3.5	**96.1**	3.2	**97.3**	1.5
benzo[a]anthracene	**112**	5.1	**99.1**	3.8	**112**	3.6
benzo[a]pyrene	**109**	5.5	**103**	1.7	**111**	1.2
benzo[b]fluoranthene	**119**	5.0	**102**	1.2	**114**	2.4
benzo[g,h,i]perylene	**112**	2.9	**102**	4.3	**113**	2.8
benzo[k]fluoranthene	**105**	1.9	**103**	2.4	**113**	3.1
BHT	**ND**[e]		**ND**		**ND**	
bromacil	**102**	9.3	**98.9**	0.86	**103**	2.1
butachlor	**107**	3.6	**86.3**	1.1	**99.4**	1.4
butylate	**85.0**	7.1	**83.0**	2.2	**84.0**	3.0
butylbenzylphthalate	**122**	1.9	**95.9**	3.7	**114**	3.6
chlordane, cis	**98.0**	5.3	**102**	2.5	**101**	1.3
chlordane, trans	**103**	1.9	**103**	2.3	**96.6**	0.71
chlorfenvinphos	**113**	1.8	**110**	2.5	**111**	3.9
chlorobenzilate	**82.0**	9.3	**99.8**	5.1	**94.1**	1.3
chloroneb	**93.0**	2.2	**92.0**	2.9	**100**	1.4
chlorothalonil	**116**	2.8	**106**	3.8	**105**	1.5
chlorpropham	**109**	3.5	**93.1**	2.5	**98.6**	1.1
chlorpyrifos	**102**	5.1	**93.4**	3.3	**97.2**	2.5
chrysene	**117**	1.7	**97.1**	1.7	**114**	2.1
cyanazine	**99.0**	3.9	**88.1**	4.9	**106**	2.4
cycloate	**102**	3.9	**87.4**	1.0	**88.8**	1.2
dacthal (DCPA)	**105**	3.6	**102**	3.5	**101**	1.7

	Fortified Conc. 0.25 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
Analytes	**Mean Recovery %**	**RSD**	**Mean Recovery %**	**RSD**	**Mean Recovery %**	**RSD**
DDD, 4,4'-	**107**	3.6	**85.8**	0.75	**105**	1.4
DDE, 4,4'-	**99.0**	3.9	**82.3**	1.3	**101**	1.0
DDT, 4,4'-	**116**	2.8	**87.6**	2.8	**112**	0.83
DEET	**103**	1.9	**98.3**	3.2	**104**	2.0
di(2-ethylhexyl)adipate	**112**	4.1	**96.6**	3.1	**111**	1.8
di(2-ethylhexyl)phthalate	**137**	3.7	**97.6**	1.3	**110**	2.4
dibenzo[*a,h*]anthracene	**110**	3.6	**95.4**	2.5	**109**	1.5
dibutyl phthalate	**115**	3.3	**101**	1.5	**114**	2.6
dichlorvos	**104**	3.1	**91.6**	1.7	**88.8**	2.7
dieldrin	**103**	1.9	**87.4**	0.55	**98.1**	0.39
diethylphthalate	**111**	1.8	**111**	2.3	**114**	1.4
dimethipin	**24.0**	14	**29.5**	6.5	**24.9**	2.5
dimethylphthalate	**110**	3.6	**113**	0.25	**113**	0.78
DIMP	**112**	5.8	**90.0**	1.9	**93.7**	3.0
dinitrotoluene, 2,4-	**126**	1.8	**105**	2.6	**113**	2.5
dinitrotoluene, 2,6-	**121**	1.7	**106**	0.71	**111**	0.67
diphenamid	**106**	2.2	**95.1**	0.90	**97.8**	0.69
disulfoton	**79.0**	2.5	**91.5**	8.9	**85.3**	1.5
endosulfan I	**95.0**	5.3	**88.4**	1.3	**101**	1.0
endosulfan II	**103**	1.9	**89.6**	3.5	**103**	1.0
endosulfan sulfate	**112**	7.1	**96.5**	2.4	**106**	0.75
endrin	**89.0**	5.7	**82.9**	3.4	**91.3**	4.0
EPTC	**89.0**	2.2	**88.0**	0.80	**85.8**	0.60
ethion	**106**	2.2	**100**	2.7	**108**	3.1
ethoprop	**110**	2.1	**91.3**	1.6	**96.2**	1.4
ethyl parathion	**117**	4.3	**97.6**	2.3	**105**	3.5
etridiazole	**118**	3.4	**90.6**	2.2	**101**	1.5
fenarimol	**110**	4.7	**87.1**	2.0	**91.7**	3.4
fluorene	**106**	3.8	**97.5**	2.1	**101**	1.1
fluridone	**92.0**	5.0	**103**	4.9	**98.6**	2.9
HCCPD	**92.0**	3.5	**65.6**	1.7	**68.0**	5.8
HCH, α	**101**	3.8	**92.5**	1.3	**95.2**	0.47
HCH, β	**101**	3.8	**94.0**	4.0	**102**	1.8
HCH, δ	**97.0**	6.2	**96.4**	1.3	**101**	0.49

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	Fortified Conc. 0.25 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
Analytes	**Mean Recovery %**	**RSD**	**Mean Recovery %**	**RSD**	**Mean Recovery %**	**RSD**
HCH, γ (lindane)	**90.0**	4.4	**95.6**	2.1	**97.9**	1.8
heptachlor	**96.0**	3.4	**83.1**	2.0	**86.2**	1.2
heptachlor epoxide	**104**	3.1	**86.9**	2.0	**95.9**	1.6
hexachlorobenzene	**94.0**	5.5	**78.4**	3.8	**93.0**	1.9
hexazinone	**107**	1.9	**84.6**	1.7	**94.6**	2.6
indeno[1,2,3-*c,d*]pyrene	**113**	4.5	**95.0**	2.1	**112**	2.4
isophorone	**108**	3.0	**108**	3.2	**102**	1.1
methoxychlor	**122**	1.9	**89.9**	1.5	**109**	0.72
methyl parathion	**129**	3.0	**103**	2.1	**112**	2.5
metolachlor	**109**	1.8	**93.1**	1.1	**97.8**	0.42
metribuzin	**116**	2.8	**97.3**	0.30	**106**	2.5
mevinphos	**115**	3.3	**96.1**	3.1	**97.0**	1.5
MGK 264(a)	**94.0**	2.5	**75.5**	1.9	**88.3**	4.1
MGK 264(b)	**94.0**	2.5	**82.8**	0.35	**92.3**	0.80
molinate	**88.0**	3.7	**89.4**	1.2	**88.9**	2.1
napropamide	**105**	3.6	**89.9**	2.4	**99.2**	1.5
nitrofen	**129**	3.0	**106**	2.7	**113**	3.9
nonachlor, trans	**119**	3.2	**103**	2.3	**96.2**	1.0
norflurazon	**106**	2.2	**91.9**	1.6	**102**	1.6
oxyfluorfen	**129**	1.6	**93.9**	2.9	**111**	3.5
pebulate	**85.0**	8.0	**84.5**	1.7	**84.7**	2.4
pentachlorophenol	**104**	4.1	**100**	1.3	**96.0**	3.6
permethrin, cis	**110**	3.6	**107**	1.3	**107**	2.2
permethrin, trans	**115**	3.3	**96.1**	3.1	**97.0**	1.5
phenanthrene	**94.0**	2.5	**75.5**	1.9	**88.3**	4.1
phorate	**94.0**	2.5	**82.8**	0.35	**92.3**	0.80
phosphamidon	**88.0**	3.7	**89.4**	1.2	**88.9**	2.1
profenofos	**105**	3.6	**89.9**	2.4	**99.2**	1.5
prometon	**129**	3.0	**106**	2.7	**113**	3.9
prometryn	**119**	3.2	**103**	2.3	**96.2**	1.0
pronamide	**106**	2.2	**91.9**	1.6	**102**	1.6
propachlor	**129**	1.6	**93.9**	2.9	**111**	3.5
propazine	**85.0**	8.0	**84.5**	1.7	**84.7**	2.4
pyrene	**104**	4.1	**100**	1.3	**96.0**	3.6

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	Fortified Conc. 0.25 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
Analytes	**Mean Recovery %**	**RSD**	**Mean Recovery %**	**RSD**	**Mean Recovery %**	**RSD**
simazine	**110**	3.6	**107**	1.3	**107**	2.2
simetryn	**115**	3.3	**96.1**	3.1	**97.0**	1.5
tebuconazole	**94.0**	2.5	**75.5**	1.9	**88.3**	4.1
tebuthiuron	**94.0**	2.5	**82.8**	0.35	**92.3**	0.80
terbacil	**88.0**	3.7	**89.4**	1.2	**88.9**	2.1
terbutryn	**105**	3.6	**89.9**	2.4	**99.2**	1.5
tetrachlorvinphos	**129**	3.0	**106**	2.7	**113**	3.9
triadimefon	**119**	3.2	**103**	2.3	**96.2**	1.0
tribufos+merphos	**106**	2.2	**91.9**	1.6	**102**	1.6
trifluralin	**129**	1.6	**93.9**	2.9	**111**	3.5
vernolate	**85.0**	8.0	**84.5**	1.7	**84.7**	2.4
vinclozolin	**104**	4.1	**100**	1.3	**96.0**	3.6

	Fortified Conc. 0.25 µg/L[b]		Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
PCB Congeners by UPAC#	**Mean Recovery %**	**RSD**	**Mean Recovery %**	**RSD**	**Mean Recovery %**	**RSD**
2-chlorobiphenyl (1)	**75.0**	2.7	**81.0**	2.6	**85.0**	1.1
4-chlorobiphenyl (3)	**85.0**	2.4	**84.4**	2.8	**88.9**	1.5
2,4′-dichlorobiphenyl (8)	**85.0**	2.4	**82.5**	2.2	**87.1**	0.51
2,2′,5-trichlorobiphenyl(18)	**104**	3.1	**89.3**	2.9	**95.3**	4.0
2,4,4′-trichlorobiphenyl (28)	**81.0**	2.5	**88.6**	3.3	**92.4**	0.48
2,2′,3,5′-tetrachlorobiphenyl(44)	**85.0**	9.7	**91.1**	3.1	**93.4**	1.6
2,2′,5,5′-tetrachlorobiphenyl(52)	**84.0**	3.9	**90.3**	4.4	**96.4**	1.1
2,3′,4′,5-tetrachloroobiphenyl (70)	**84.0**	3.9	**92.4**	3.4	**97.0**	0.94
2,3,3′,4′,6-pentachlorobiphenyl (110)	**81.0**	2.5	**94.3**	3.2	**97.1**	0.90
2,3′,4,4′,5-pentachlorobiphenyl (118)	**90.0**	2.6	**94.5**	3.7	**98.8**	1.1
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	**86.0**	4.7	**99.0**	3.2	**105**	1.3
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	**87.0**	5.8	**96.5**	4.3	**101**	1.2
2,2′,4,4′,5,5′-hexachlorobiphenyl (153)	**79.0**	2.5	**96.9**	2.8	**101**	1.1
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	**101**	2.0	**93.1**	2.9	**91.6**	2.2
Surrogate Analytes						
1,3-dimethyl-2-nitrobenzene	**92.9**	4.0	**98.9**	3.1	**88.8**	4.0
benzo[*a*]pyrene-*d*12	**112**	2.1	**101**	2.6	**101**	4.9
triphenyl phosphate	**107**	2.5	**97.9**	4.1	**104**	3.2

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a. Data obtained on the instrumentation described in Sect. 13.1.1.4

b. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 1.0 µg/L, c-permethrin is 0.13 µg/L, t-permethrin is 0.38 µg/L, MGK 264 (a) is 0.085 µg/L and MGK 264 (b) is 0.17 µg/L.

c. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 8.0 µg/L, c-permethrin is 1.0 µg/L, t-permethrin is 3.0 µg/L, MGK 264 (a) is 0.67 µg/L and MGK 264 (b) is 1.3 µg/L.

d. Exceptions to the stated concentration are as follows: Surrogate concentrations are 5.0 µg/L, pentachlorophenol is 20.0 µg/L, c-permethrin is 2.5 µg/L, and t-permethrin is 7.5 µg/L, MGK 264 (a) is 1.7 µg/L and MGK 264 (b) is 3.3 µg/L.

e. ND = Not determined.

Precision and Accuracy Data Obtained for Method Analytes Fortified into Finished Drinking Waters from Ground and Surface Water Sources, and Extracted Using UCT

525 Universal Cartridges; N=4; Full Scan GC/MS Analyses[a]

		Ground Water[b]		Surface Water[c]	
Analytes	**Fortified Conc. (µg/L)**	**Mean Recovery[d] %**	**RSD**	**Mean Recovery[d] %**	**RSD**
acenaphthylene	2.0	**99.3**	5.7	**95.1**	1.8
acetochlor	2.0	**97.4**	5.5	**106**	4.9
alachlor	2.0	**93.5**	3.5	**95.0**	3.3
aldrin	2.0	**94.9**	4.0	**81.1**	0.59
ametryn	2.0	**98.9**	5.7	**93.4**	6.2
anthracene	2.0	**102**	4.7	**101**	1.6
atraton	2.0	**92.6**	5.9	**87.3**	5.3
atrazine	2.0	**100**	4.6	**95.6**	1.5
benzo[a]anthracene	2.0	**104**	1.5	**102**	2.4
benzo[a]pyrene	2.0	**106**	3.2	**100**	2.7
benzo[b]fluoranthene	2.0	**104**	3.2	**100**	3.9
benzo[g,h,i]perylene	2.0	**101**	3.9	**101**	5.3
benzo[k]fluoranthene	2.0	**103**	3.0	**101**	2.6
BHT	2.0	**95.5**	1.2	**114**	1.9
bromacil	2.0	**98.9**	5.5	**104**	4.6
butachlor	2.0	**96.4**	2.9	**95.6**	3.3
butylate	2.0	**87.6**	3.2	**83.6**	1.8
butylbenzylphthalate	2.0	**107**	4.1	**107**	2.9
chlordane, cis-	2.0	**96.1**	6.2	**98.0**	5.9
chlordane, trans	2.0	**94.5**	5.9	**98.3**	7.3
chlorfenvinphos	2.0	**93.1**	3.9	**111**	3.4
chlorobenzilate	2.0	**101**	5.6	**97.1**	4.3
chloroneb	2.0	**104**	2.0	**108**	4.0

		Ground Water[b]		Surface Water[c]	
Analytes	**Fortified Conc. (µg/L)**	**Mean Recovery[d] %**	**RSD**	**Mean Recovery[d] %**	**RSD**
chlorothalonil	2.0	**108**	1.5	**110**	2.2
chlorpropham	2.0	**95.9**	5.6	**98.1**	2.4
chlorpyrifos	2.0	**97.8**	5.3	**103**	4.1
chrysene	2.0	**106**	2.2	**100**	3.2
cyanazine	2.0	**97.4**	5.2	**91.0**	11
cycloate	2.0	**92.3**	5.6	**95.1**	1.6
dacthal (DCPA)	2.0	**92.1**	7.3	**107**	4.3
DDD, 4,4'-	2.0	**93.1**	3.7	**91.0**	2.0
DDE, 4,4'-	2.0	**90.0**	3.6	**85.6**	2.7
DDT, 4,4'-	2.0	**91.4**	3.7	**90.4**	3.5
DEET	2.0	**101**	1.8	**109**	1.1
di(2-ethylhexyl)adipate	2.0	**102**	5.8	**106**	3.4
di(2-ethylhexyl)phthalate	2.0	**107**	2.7	**104**	2.2
dibenzo[a,h]anthracene	2.0	**106**	6.2	**102**	2.2
dibutyl phthalate	2.0	**110**	3.7	**107**	1.5
dichlorvos	2.0	**90.4**	7.3	**88.8**	2.3
dieldrin	2.0	**96.0**	5.4	**96.3**	2.6
diethylphthalate	2.0	**110**	2.5	**107**	1.1
dimethipin	2.0	**29.4**	8.7	**38.1**	4.2
dimethylphthalate	2.0	**111**	2.4	**111**	1.0
DIMP	2.0	**89.6**	5.9	**99.6**	5.9
dinitrotoluene, 2,4-	2.0	**95.6**	6.2	**101**	5.0
dinitrotoluene, 2,6-	2.0	**104**	2.0	**108**	4.0
diphenamid	2.0	**98.5**	5.6	**101**	2.3
disulfoton	2.0	**79.6**	5.9	**103**	6.3
endosulfan I	2.0	**96.9**	7.0	**93.0**	4.5
endosulfan II	2.0	**98.5**	5.7	**94.3**	4.1
endosulfan sulfate	2.0	**102**	2.8	**100**	2.1
endrin	2.0	**97.9**	2.6	**88.1**	3.4
EPTC	2.0	**89.0**	7.2	**85.8**	3.0
ethion	2.0	**95.8**	6.9	**98.9**	4.3
ethoprop	2.0	**94.9**	5.7	**103**	3.9
ethyl parathion	2.0	**97.6**	5.9	**103**	3.9
etridiazole	2.0	**104**	2.4	**104**	3.1
fenarimol	2.0	**93.5**	4.4	**86.6**	3.3
fluorene	2.0	**101**	5.1	**104**	1.8
fluridone	2.0	**111**	3.9	**97.4**	6.0

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		Ground Water[b]		Surface Water[c]	
Analytes	**Fortified Conc. (µg/L)**	**Mean Recovery[d] %**	**RSD**	**Mean Recovery[d] %**	**RSD**
HCCPD	2.0	**64.6**	6.7	**69.1**	2.8
HCH, α	2.0	**93.5**	5.7	**91.8**	2.4
HCH, β	2.0	**97.9**	4.2	**97.8**	1.8
HCH, δ	2.0	**102**	6.4	**94.3**	3.7
HCH, γ (lindane)	2.0	**100**	6.8	**90.0**	2.1
heptachlor	2.0	**91.0**	3.0	**86.1**	1.5
heptachlor epoxide	2.0	**98.3**	6.2	**91.6**	1.6
hexachlorobenzene	2.0	**90.4**	4.0	**89.0**	2.7
hexazinone	2.0	**93.4**	3.1	**97.0**	8.1
indeno[1,2,3-c,d]pyrene	2.0	**107**	6.6	**103**	4.6
isophorone	2.0	**104**	3.9	**101**	2.6
methoxychlor	2.0	**94.0**	5.6	**94.0**	2.6
methyl parathion	2.0	**100**	3.7	**110**	2.5
metolachlor	2.0	**97.5**	5.6	**97.8**	2.1
metribuzin	2.0	**102**	2.6	**120**	0.77
mevinphos	2.0	**89.9**	5.3	**96.3**	4.5
MGK 264(a)	1.3	**92.9**	2.3	**86.4**	4.1
MGK 264(b)	0.67	**95.9**	4.1	**92.6**	3.9
molinate	2.0	**94.9**	5.0	**89.1**	2.3
napropamide	2.0	**95.9**	4.6	**104**	3.1
nitrofen	2.0	**111**	6.2	**96.4**	5.3
nonachlor, trans	2.0	**105**	4.0	**98.4**	5.3
norflurazon	2.0	**98.1**	3.8	**101**	6.4
oxyfluorfen	2.0	**91.8**	7.7	**94.9**	5.0
pebulate	2.0	**89.3**	4.4	**84.3**	1.0
pentachlorophenol	8.0	**98.3**	2.6	**97.3**	2.9
permethrin, cis	1.0	**92.6**	6.0	**108**	4.3
permethrin, trans	3.0	**91.4**	7.1	**100**	2.3
phenanthrene	2.0	**106**	3.7	**104**	2.5
phorate	2.0	**95.5**	2.0	**98.9**	4.8
phosphamidon	2.0	**100**	3.3	**114**	2.8
profenofos	2.0	**98.4**	4.4	**108**	1.7
prometon	2.0	**96.9**	2.1	**88.4**	5.7
prometryn	2.0	**98.8**	4.9	**94.6**	4.9
pronamide	2.0	**95.1**	5.5	**96.6**	3.4
propachlor	2.0	**106**	1.4	**110**	3.4
propazine	2.0	**101**	2.8	**96.4**	2.5

		Ground Water[b]		Surface Water[c]	
Analytes	**Fortified Conc. (µg/L)**	**Mean Recovery[d] %**	**RSD**	**Mean Recovery[d] %**	**RSD**
pyrene	2.0	**106**	1.2	**107**	2.2
simazine	2.0	**101**	3.7	**96.1**	3.0
simetryn	2.0	**98.6**	4.2	**87.9**	3.8
tebuconazole	2.0	**94.4**	5.4	**96.0**	5.2
tebuthiuron	2.0	**88.5**	7.1	**101**	1.8
terbacil	2.0	**101**	5.6	**95.8**	10
terbutryn	2.0	**97.3**	5.1	**87.4**	5.1
tetrachlorvinphos	2.0	**97.5**	6.6	**104**	5.4
triadimefon	2.0	**99.3**	2.1	**101**	3.0
tribufos+merphos	4.0	**96.6**	7.3	**107**	2.0
trifluralin	2.0	**90.8**	2.2	**90.0**	4.5
vernolate	2.0	**88.0**	6.0	**88.3**	1.8
vinclozolin	2.0	**98.6**	7.0	**111**	1.7

	Fortified Conc. 0.25 µg/L[b]	Fortified Conc. 2.0 µg/L[c]		Fortified Conc. 5.0 µg/L[d]	
PCB Congeners by IUPAC#	**Mean Recovery %**	**Mean Recovery %**	**RSD**	**Mean Recovery %**	**RSD**
2-chlorobiphenyl (1)	2.0	**90.8**	1.7	**96.3**	5.6
4-chlorobiphenyl (3)	2.0	**96.1**	0.65	**99.9**	5.4
2,4′-dichlorobiphenyl (8)	2.0	**97.9**	0.87	**86.6**	6.3
2,2′,5-trichlorobiphenyl(18)	2.0	**101**	3.8	**90.9**	7.5
2,4,4′-trichlorobiphenyl (28)	2.0	**101**	2.2	**86.1**	4.9
2,2′,3,5′-tetrachlorobiphenyl(44)	2.0	**93.0**	3.0	**88.1**	6.9
2,2′,5,5′-tetrachlorobiphenyl(52)	2.0	**97.1**	3.1	**87.8**	6.6
2,3′,4′,5-tetrachloroobiphenyl (70)	2.0	**107**	1.5	**88.1**	5.2
2,3,3′,4′,6-pentachlorobiphenyl (110)	2.0	**107**	1.3	**92.0**	6.3
2,3′,4,4′,5-pentachlorobiphenyl (118)	2.0	**108**	1.2	**91.3**	6.4
2,2′,3,4,4′,5′-hexachlorobiphenyl (138)	2.0	**110**	2.0	**93.8**	6.4
2,2′,3,4′,5′,6-hexachlorobiphenyl (149)	2.0	**106**	1.4	**91.5**	6.0
2,2′,4,4′,5,5′-hexachlorobiphenyl (153)	2.0	**108**	0.89	**91.3**	6.3
2,2′,3,4,4′,5,5′-heptachlorobiphenyl (180)	2.0	**99.6**	1.0	**87.6**	7.2
Surrogate Analytes					
1,3-dimethyl-2-nitrobenzene	5.0	**91.7**	7.5	**89.0**	6.5
benzo[*a*]pyrene-*d*12	5.0	**103**	2.7	**104**	3.0
triphenyl phosphate	5.0	**104**	0.58	**112**	3.5

a. Data obtained on the instrumentation described in Sect. 13.1.1.4.
b. Tap water from a ground water source with high mineral content. Tap water hardness was 300mg/L as calcium carbonate
c. Tap water from a surface water source. TOC of 2.4 mg/L
d. Recoveries have been corrected to reflect the native amount in the unfortified matrix water.

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Toxaphene from Fortified Reagent Water Precision and Accuracy: Extracts Analyzed by SIM

Fortified Concentration 10 µg/L (N=4)	Mean % Recovery	RSD
Toxaphene	111	1.8

References:

[1] Method 525.3, "Determination Of Semivolatile Organic Chemicals In Drinking Water By Solid Phase Extraction And Capillary Column Gas Chromatography/ Mass Spectrometry (GC/MS)," Ver 1.0, February 2012, Jean W. Munch and Paul E. Grimmett (U.S. EPA, Office of Research and Development, National Exposure Research Laboratory), David J. Munch and Steven C. Wendelken (U.S. EPA, Office of Water, Office of Ground Water and Drinking Water, Technical Support Center) Mark M. Domino (Industrial and Environmental Services, LLC) Alan D. Zaffiro and Michael L. Zimmerman (Shaw Environmental and Infrastructure, Inc.), National Exposure Research Laboratory Office Of Research And Development, U. S. Environmental Protection Agency, Cincinnati, Ohio 45268

[2] Complete details at www.epa.gov/safewater/methods/methods.html

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