

SIMULTANEOUS EXTRACTION OF PESTICIDES, SEMI-VOLATILES AND PCB CONGENERS USING ONE UNIVERSAL SPE CARTRIDGE

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INTRODUCTION

Pesticides, Semi-volatiles and PCB congeners belong to different groups of contaminants in drinking water. It is difficult to extract them all with one single solid phase extraction as they have a variety of polarities. UCT has successfully developed the universal octadecyl (C₁₁) bonded silica-based sorbent that can simultaneously extract all these compounds in a single extraction, which can make it faster and more cost-effective for environmental laboratories to detect and quantify them.

The EPA is proposing modifications to Method 525.2 including additional analytes of varying polarities, organic acids as preservatives and modified surrogates and internal standards. The UCT universal cartridge (ECUNI525) has demonstrated excellent performance for the proposed method. Extraction of 127 target analytes from reagent water, surface water with high total organic carbon, and groundwater with significant hardness, yielded recoveries between 80-120% and RSDs <10%.

PROCEDURES

1. Weigh 0.1 g L-ascorbic acid, 0.35 g ethyl enediaminetetra acetic acid trisodium salt, and 9.4 g potassium monobasic citrate into a 1 L amber bottle. Fill the bottle with 1 L sample.

3. Wash with 5 mL 1:1 EtOAc:MeCl₂, soak for 1 min, draw the solvent through, keep the vacuum on for 2 mins.

Condition the cartridge with 10 mL MeOH, soak for 1 min, draw most of the MeOH through, leave a thin layer of MeOH on the surface of the cartridge. Condition with 10 mL reagent water, draw most through, leave a thin layer on the surface of the cartridge.

6. Insert a 40 mL vial into the manifold. Elute the dried cartridge with 5 mL EtOAc. Repeat with 5 mL McCl₂. Add 10 g muffled sodium sulfate into the cartridge. Rinse the sample bottle thoroughly with 5 mL EtOAc then pour the rinsed solvent into the cartridge. Repeat with 5 mL McCl₂.

entrate the extract to 1 mL under a gentle N2 stream at 40 °C using ap evaporator. Add internal standard and load to GC/MS for analysis

FLOW CHART:





Universal cartridge with C₁₀ sorbent



MATERIALS

SPE cartridge:
83 m.L UCT Universal cartridge with 1500 mg C18 (ECUNI525)
Vacuum pump: (ECROCKER400)
6-station manifold: (ECUCTVAC6)
Cartridge adaptor: (ECUUCTADP)
Bottle holder: (ECUNISHBID)
20L waste trap: (ECUCTTAP20)
Fritted reservoirs: 50 µm Teflon frit (ERTFT1FUNIP)
Sodium sulfate: anhydrous, ACS, Granular 60 Mesh (ECSS05K)

INSTRUMENTAL

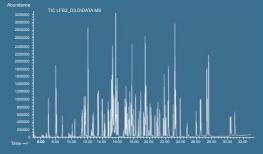
GC/MS: Agilent 6890N GC coupled with 5975C MSD, equipped with 7683 auto sampler. GC capillary column: Restek Rxi-Ssil MS 30m+0.25mm+0.25µm Injector: 1µL splitless injection at 250 °C, with a split delay of 1 min.

Liner: 4 mm splitless gooseneck, 4mmID*6.5mm0D*78.5mm (GCLGN4MM)

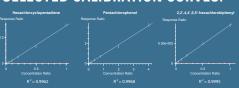
Temperature program: Initial oven temperature of 55 °C, hold for 1 min, ramp at 10 °C/min to 200 °C, ramp at 7 °C/min to a final temperature of 320 °C and hold for 0.36 min. Solvent delay: 5 min.

Carrier gas: Helium at a constant flow of 1.2 mL/min. MSD condition: Aux temperature: 280 °C, MS Source: 230 °C, MS Quad: 150 °C

CHROMATOGRAM: Laboratory reagent blank fortified at 2 µg/L



SELECTED CALIBRATION CURVES:



RESULTS

Table 1. Accuracy and Precision Data obtained for Pesticides: Fortified at Three Concentrations in Reagent Water and One Concentration in Ground Water and Surface Water

	Fortified at 0.25 µg/L*		Fortified at 2.0 µg/L ^b		Fortified at 5.0 µg L*		Fortified at 2.0 µg/L ^b		Fortified at 2.0 µg/L ^b	
Perdicides	Fortined at 0.2 Mean %	SSD SSD		RSD	Hortsmed at 5. Mean %	ISD .	Fortined at 2: Mean %	RED P MB/L-	Fortined at 21 Mean %	RSD RSD
	Decreese	end.	Decreusery	and.	Decouser	104	Darragery M	and.	Recovery ki	and.
Acetochilar	99	2.9	94	2.1	104	2.1	97	2.2	206	4.9
Alachion	100	7.3	90	0.7	93	1.0	94	25	96	2.2
Mária	77	5.0	78	2.9	25	2.4	96	4.0	81	0.6
Ametrya	105	4.8	93	1.3	96	1.1	99	5.7	93	6.2
Atraton	112	2.9	90	41	97	2.2	93	9.2	87	5.3
Atrasine	111	3.5	96	3.2	97	1.5	100	4.6	96	1.5
iromacii	102	9.3	99	0.9	103	2.1	99	2.2	234	4.6
utachlor	107	3.6	86	1.1	99	1.4	96	2.9	96	3.3
utvlate	85	7.1	83	22	84	2.0	*	22	84	1.8
utylated hydroxyanisole	85	2.9	101	10.1	76	9.2	102	1.1	98	2.6
utylated hydroxytoluece	137	4.4	90	2.5	97	1.3	96	1.2	114	1.9
agtan	119	1.7	112	0.5	113	2.9	97	6.6	900	6.6
hiordane, cis	98	53	102	2.5	101	1.3	96	62	98	9.2
hiordane, trans	103	1.9	103	2.3	97	0.7	95	5.9	98	7.3
tilerfenvinghes	113	1.8	110	2.5	111	2.9	93	2.9	111	2.4
Scrobenziste	82	93	100	51	94	1.3	900	5.6	97	43
Soroneb	99	2.2	92	2.9	100	1.4	104	2.0	928	4.0
forethalanii	116	2.8	106	2.8	105	1.5	108	1.5	110	2.2
iorpropham	109	2.5	93	2.5	99	1.1	96	5.6	98	2.4
iorpyrifos	102	5.1	93	2.3	97	2.5	98	5.3	933	4.1
snazine	99	2.9	22	4.9	106	2.4	97	5.2	91	11.4
cloate	102	2.9	97	1.0	29	1.2	92	5.6	95	1.6
cthal	105	26	102	25	101	1.7	92	7.3	937	4.3
00,4,45	107	26	86	0.8	105	1.4	93	2.7	91	2.0
N, 4,4-	99	2.9	82	1.3	101	1.0	90	26	86	2.7
DT, 4,4'-	116	2.8	23	2.8	112	0.8	91	2.7	90	25
thlorvos	104	3.1	92	1.7	20	2.7	90	7.3	89	2.3
Hidrin	103	1.9	87	as	98	0.4	- %	5.4	96	2.6
isopropyl methylphosphorate	112	5.8	90	1.9	94	2.0	90	9.2	900	9.9
phenamid	106	2.2	95	0.9	98	0.7	99	5.6	991	2.3
sulfaton	29	2.5	92	2.9	85	1.5	80	9.2	223	63
doculfan i	95	53	23	1.3	101	1.0	97	7.0	93	45
losulfan II	103	1.9	90	2.5	103	1.0	99	5.7	94	4.1
losulfan sulfate	112	7.1	97	2.4	106	0.7	102	2.8	900	2.1
tis	29	\$.7	83	2.4	91	4.0	98	2.6	88	3.4
95	106	2.2	100	2.7	108	2.1	- %	6.9	99	43
oprophes	110	2.1	91	1.6	- %	1.4	96	5.7	223	2.9
yl Parathion	117	43	98	2.3	105	2.5	98	5.9	933	2.0
diasole	118	2.4	91	2.2	101	1.5	104	2.4	934	3.1
ramiphos	20	5.7	92	1.8	105	5.1	91	5.4	111	5.0
narimol	110	47	27	2.0	92	2.4	94	4.4	97	11
ridone	92	5.0	103	4.9	99	2.9	111	2.9	97	6.0
ptachlor	96	3.4	83	2.0	86	1.2	91	2.0	86	1.5
stachlor eposide		3.1	97	2.0	96	1.6	98	6.2	92	1.6
sachlorocyclohexane, alpha	101	2.0	93	1.3	95	0.5	94	5.7	92	2.4
sachlanocyclohexane, beta sachlanocyclohexane, delta	101 97	3.8 6.2	94	1.3	102 101	1.7 0.5	98	4.2 6.4	98	18
cucromycocexare, cera	97	44	96	2.1	98	1.8	102	6.6	98	2.1
nonurs, colesiare, garana	107	1.9	95 85	1.7	98	2.6	92	2.1	90	2.1 8.1
about the same to		1.9	90	1.7	109	0.7	94	2.1 5.6	97	2.6
the formation	122	1.9		2.1		2.5		2.7		2.5
entration	129	1.8	103	1.1	98	0.4	100 98	2.7 5.6	110 98	2.1
A COLOR	116	2.8	97	0.3	106	2.5	102	2.6	120	0.8
- feebru	115	2.1	96	2.1	97	1.5	90	53	96	4.5
overnos Ex 264tal	94	2.5	26	1.9	**	4.1	93	23	86	41
The second	94 94	2.5	83	0.3	92	0.8	96	41	93	2.9
alinate	88	2.7	29	1.2	89	2.1	95	5.0	89	2.3
N Giethyl-meta columnide	103	1.9	98	32	104	2.0	100	18	229	1.1
acrocamide	105	26	90	2.4	99	1.5	96	46	234	2.1
trofen	129	3.0	106	2.7	113	2.9	111	62	96	5.3
reachlor, trans	119	2.2	103	2.3	- %	1.0	105	4.0	98	53
rflurazon	106	2.2	92	1.6	102	1.6	98	2.8	931	6.4
orfuorfen	129	1.6	94	2.9	111	2.5	92	7.7	96	5.0
bulate	85	8.0	85	1.7	85	2.4		4.4	84	1.0
methón cis	110	26	107	13	107	2.2	93	60	928	4.3
methin, trans	116	2.0	99	2.3	101	1.9	91	7.1	200	2.3
orate	81	4.7	100	2.5	110	1.3	96	2.0	99	4.8
osphamidon	109	25	107	2.5	108	2.1	100	2.3	114	2.8
afenatas	105	65	102	2.6	109	2.8	98	4.4	928	1.7
ometos	112	1.8	92	4.1	95	2.0	97	2.1	88	5.7
ametryo	105	1.9	95	3.2	98	0.9	99	4.9	95	4.9
osamide	109	1.8	94	0.9	100	0.3	95	2.2	97	3.4
rosachlor	107	1.9	93	2.2	106	2.3	106	1.4	110	3.4
nozacine	91	7.5	95	1.6	94	1.8	100	2.8	96	2.5
-Ethyl dipropykhiocarbamate	29	2.2	23	0.8	*	0.6	89	7.2	86	2.0
mazine	113	1.8	99	2.6	101	1.6	100	2.7	96	2.0

	Fortified at 0.25 µg/L*		Fortified at 2.0 µg/L ^b		Fortified at SiD µg/L*		Fortified at 2.0 µg/L ^b		Fortified a	
	Mean %	RSD	Mean %	RSD	Mean %	RSD	Mean %	RSD	Mean %	
	Recovery	504	Recovery	504	Recovery	504	Recovery ht	5114	Recovery M	
Aceraphthylene	101	2.0	94	0.5	200	0.9	99	5.7	96	
Anthracene	106	2.8	92	1.0	234	0.7	992	4.7	304	
Benzja[anthracene	112	5.1	99	3.8	112	3.6	934	1.5	502	
Benzojajpyrene	109	5.5	102	1.7	111	1.2	936	3.2	100	
Benzojbjfluoranthene	119	5.0	102	12	114	2.4	934	3.3	100	
Benzolgh/(perylene	112	2.9	502	43	113	2.8	931	3.9	100	
kenzojk)fluoranthene	105	1.9	503	2.4	113	2.1	923	3.0	100	
Rutyl berzyl phthalate	122	1.9	16	2.7	114	26	937	41	107	
Chrysene	117	1.7	97	1.7	114	2.1	236	2.2	100	
Di(2-ethylhexyl) adigate	112	4.1	97	3.1	111	1.8	992	5.8	106	
D(2-ethylheoji) phthalate	137	2.7	98	1.3	110	2.4	937	2.7	104	
Dibero(a,h)anthracene	110	2.6	95	2.5	939	15	936	62	102	
Dibutyl phthalate	115	2.3	101	1.5	114	2.6	110	2.7	937	
Diethyl Phthalate	111	1.8	111	2.3	114	1.4	110	2.5	107	
Dimethyl phthalate	110	2.6	113	0.3	113	0.8	111	2.4	111	
Dinisratoluene, 2,4-	126	1.8	105	2.6	113	2.5	96	7.0	105	
Dinitrotoluene, 2,6-	121	1.7	106	0.7	111	0.7	96	62	100	
Rustene	106	2.8	98	2.1	931	1.1	931	5.1	104	
Kexachloroberoene	94	5.5	78	2.8	93	1.9	90	40	29	
Kexachlorocyclopentadiene	92	25	66	1.7	68	5.8	65	67	69	
indeno(1,2,3-cd)pyrene	113	4.5	95	2.1	112	2.4	207	6.6	103	
isophorone	108	2.0	108	2.2	932	11	934	3.9	100	
Pentachiorophenol	104	4.1	100	1.3	96	26	98	2.6	97	
Phenanthrene	107	2.6	96	0.7	104	0.5	206	2.7	104	
Pyrece	109	2.5	95	2.4	110	2.9	226	1.2	107	

	Reager Fortified at		Reagent Water Fortified at 2.0 up L ^b		Reagent Water Fortified at 5.0 up 6."		Ground Water * Fortified at 2.0 up (L*		Surface Water * Fortified at 2.0 uo/L*	
	Mean % Recovery	RSD 014	Mean % Recovery	850	Mean % Recovery	150	Mean % Recovery N	ESD COM	Mean % Recovery ^{hd}	RSD 0114
2-chlorobiphenyl (1)	75	2.7	81	2.6	85	1.1	91	1.7	96	5.6
4-chlorobipheryl (3)	85	2.4	84	2.8	29	1.5	96	0.7	200	5.4
					87				87	
2,2',5-trichlorobiphenyl (18)	104	3.1	29	2.9	95	4.0	101	3.8	91	7.5
2,4,4"-trichlorobiphenyl (28)	81	2.5	29	3.3	92	0.5	101	2.2	86	49
2,2',3,5'-tetrachlorobiphenyl (44)	25	9.7	91	3.1	93	1.6	93	2.0	88	6.9
2,2',5,5'-tetrachlorobiphenyl (\$2)	24	19	90	4.4	96	1.1	97	31	88	66
2,17,47,5-tetrachloroobiphenyl (70)	24	2.9	92	3.4	97	0.9	107	1.5	88	5.2
2,2,2°,4°,6-pentachionobiphenyl (190)	81	2.5	94	32	97	0.9	107	1.3	92	63
2,2',4,4',5-pentachlorobighenyl (118)	90	2.6	95	2.7	- 00	1.1	508	1.2	91	6.4
2,2',3,4,4',5'-heuschlorobiphenyl (138)	26	4.7	99	3.2	105	1.3	110	2.0	94	5.3
2,2',3,6',5',6-heuschlorobiphenyl (149)	27	8.2	97	43	101	1.2	106	1.4	92	6.0
2,2',4,4',5,5'- hexachlorobiphenyl (153)	79	2.5	97	2.8	101	1.1	108	0.9	91	63
2.2.3.4.6.5.5'-heotachloroblohenyl (180)	101	20	- 93	20	63	- >>	100	10		7.2

CONCLUSION