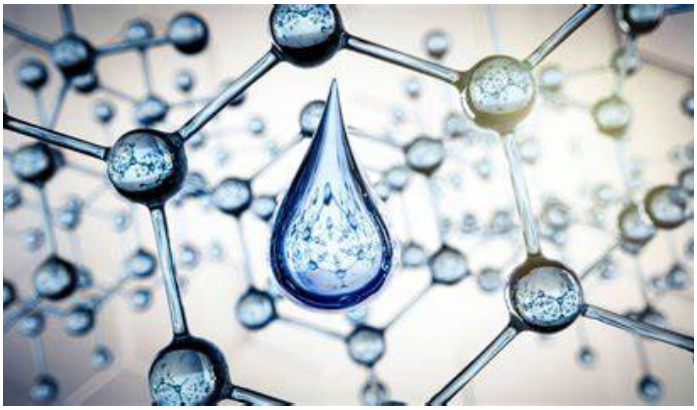
Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry



UCT Part Numbers

ECUNI525 1500 mg 525 C18, 83 mL cartridge	Or	**EC525006-P** 1500 mg 525 C18, 6 mL cartridge, PE Frit

Summary:

The analyte list for this method is comprised of over 120 compounds representative of several classes of pesticides, polynuclear aromatic hydrocarbons, PCBs, phthalates and adipates and other drinking water pollutants. Recovery ranges from 70-130%. Refer to the published method for compound specific MDL's.

The validation data presented herein were determined on independent lots of UCT ENVIRO-CLEAN® Universal Cartridges. MDLs were not determined on all analytes as part of this validation. In addition to the listed method analytes, recovery data for an extended list of analytes is also included.

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Table of Compounds
Tested using the UCT ENVIRO-CLEAN® Universal Cartridge 525

Analyte	Average 3 Replicates (% Recovery)	Std Dev
Acenaphthene	100	0.0
2,4-dinitrotoluene	83	NA
2,6-dinitrotoluene	78	NA
4,4″-DDE	91	4.2
4,4″-DDT	94	3.5
4,4′-DDD	94	4.5
Acenaphthylene	96.0	0.012
Acenaphthene	99.1	0.013
Acetochlor	115	0.01
Alachlor	99	0.007
Aldrin	77	4.4
Ametryn	95	4.6
Anthracene	80	0.0
Atraton	84	17.3
Atrazine	111	0.011
Benzo(a) anthracene	75.4	0.049
Benzo(a)pyrene	105	9.9
Benzo(b)fluoranthene	184.9	0.022
Benzo(k)fluoranthene	95.7	0.029
Benzo[g,h,i]perylene	83.1	0.05
BHC, alpha	108	6.9
BHC, beta	97	3.1
BHC,delta	109	7.9
BHC,gamma	102	11.9
bis- (2-ethylhexyl) adipate	95.1	0.033
bis 2 ethylhexyl phthalate	104	0.029
Bromacil	126	0.012
Butachlor	113	0.005
Butylate	103	4.6
Butylbenzylphthalate	97.1	0.02
Caffeine	90.0	0
Captan	86.9	0.273
Carboxin	103	12.9
Chlordane, alpha	97	4.6
Chlordane, gamma	94	2.5
Chlordane, trans nonachlor	115	11.0

Analyte	Average 3 Replicates (% Recovery)	Std Dev
Chlorneb	113	11.0
Chlorobenzilate	118	10.0
Chlorpropham	130	0
Chlorpyrifos (Dursban)	107	5.0
Chlorothalonil	117	12.1
Chrysene	100	0.012
Cyanazine (Bladex)	126	0.008
Cycloate	111	15.0
Dacthal (DCPA) methyl ester	118	13.1
Diazinon	135	0.031
Dibenzo[a,h]anthracene	77.4	0.051
Dichlorvos (DDVP)	127	9.5
Dieldrin	96	6.8
Diethylphthalate	99.1	0.071
Dimethoate	106	0.008
Dimethylphthlate	78.6	0.022
Di-n-butylphthalate	113	0.12
Diphenamid	119	0.008
Disulfoton	92.1	0.01
Disulfoton Sulfone	108	12.5
Endosulfan I	116	11.1
Endosulfan sulfate	114	6.8
Endrin	88	0.0
Endrin Aldehyde	97	3.6
Endrin Ketone	90	3.8
EPTC	102	0.005
Ethion	112	0.005
Ethoprophos	109	5.8
Etridiazole (terrazole)	97	1.2
Fenarimol	70	0.0
Fluoranthene	100	0.018
Fluorene	99.7	0.012
Heptachlor	79	8.2
Heptachlor Epoxide Iso A	116	16.3
Hexachlorobenzene	94	17.4
Hexachlorocyclopentadiene	82	8.4
Hexazinone (Velpar)	105	8.1
Indeno[1,2,3-cd]pyrene	77.4	0.16
Isophorone	91	NA
Lindane	127	4.8

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Analyte	Average 3 Replicates (% Recovery)	Std Dev
methoxychlor	123	7.6
Methyl Paraxon (Parathion)	115	5.0
Metolachlor	111	0.004
Metribuzin	109	0.005
Mevinphos (phosdrin)	117	12.1
MGK 264	121	5.8
Molinate	114	0.013
Naphthalene	90.3	0.013
Napropamide (Devrinol)	115	2.3
Nonachlor, trans	116	11.1
Norflurazon	133	6.1
PCNB (carbaryl)	91.4	0.021
Pebulate	101	1.7
Pentachlorophenol	80	0.017
Permethrin, cis	124	2.1
Permethrin, trans	123	3.1
Perylene-d12	119	0.0
Phenanthrene	96.9	0.014
Phenanthrene-d10	99	6.6
Prometon	78.6	0.008
Prometryn	110	0.012
Pronamide (propyzamide)	101	1.2
Propachlor	113	15.0
Propazine	105	4.6
Pyrene	94.6	0.022
Simazine	91.4	0.005
Simetryn	93	4.6
Stirofos (tetrachlorvinphos)	126	6.9
Thiobencarb	112	0.008
Tebuthiuron	85	33.5
Terbacil	120	3.5
Terbutryn	103	2.3
Triademefon	98	6.9
Tricyclazole	107	5.0
Trifluralin	82	9.7
Trifluran	83	9.2
Trithion (carbofenothion)	101	0.004
Terbufos	95	7.0
Vernolate	107	1.2

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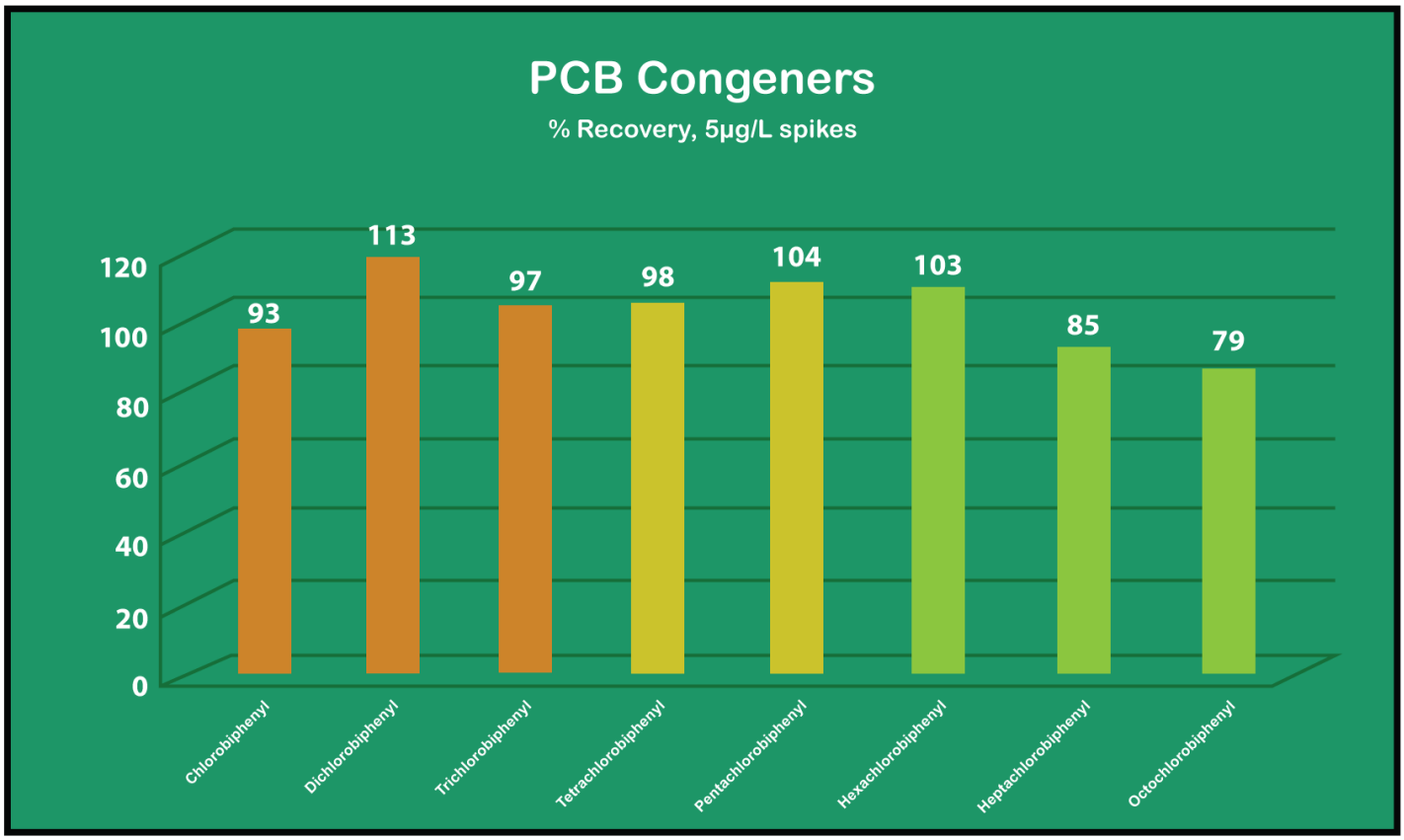
PCB Congeners	Average	Std Dev
2-chlorobiphenyl	93	2.3
2.3-Dichlorobiphenyl	113	15.0
2,4,5-trichlorobiphenyl	97	3.1
2,2,4,4-tetrachlorobiphenyl	98	5.3
2,2,3,4,6-pentachlorobiphenyl	104	2.0
2,2,4,4,5,6-hexachlorobiphenyl	103	3.1
2,2,3,3,4,4,6-heptachlorobiphenyl	85	1.2
Octachlorobiphenyl (BZ#200)	79	1.2



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Procedure

1. Wash the extraction apparatus and cartridge

a) Add 10 mL of a 1: 1 mixture of ethyl acetate: methylene chloride (EtOAc: $MeCl_2$) to the reservoir.
b) Draw a small amount through the cartridge with vacuum
c) Turn off the vacuum and allow the cartridge to soak for about one minute
d) Draw the remaining solvent through the cartridge to waste
e) Allow the cartridge to dry for 2 minutes under full vacuum

2. Condition Cartridge

a) Add 10 mL of methanol
b) Draw a small amount through the cartridge
c) Let soak for about one minute
d) Draw most of the remaining methanol through the cartridge, leaving 3 to 5 mm of methanol on the surface of the cartridge frit
e) Immediately add 20 mL of reagent water to the cartridge and draw most of the water through leaving 3 to 5 mm on the top of the cartridge frit

Note: Do not let the cartridge dry out after the addition of water

f) Add 5 ml of methanol to the water sample (dechlorinated and pH adjusted to ≤2) and mix well
g) Add the water sample to the cartridge and under vacuum, filter at a rate of approximately 50 mL per minute
h) Drain as much water from sample bottle as possible
i) Dry the cartridge under vacuum for 10 minutes

Note: Exceeding a 10-minute dry time could result in low recoveries. For faster drying, remove the cartridge and tapping the excess moisture from the bottom of the cartridge before continuing vacuum drying

3. Elution

a) Insert a suitable sample tube for eluate collection
b) Add 10 mL of EtOAc to the sample bottle
c) Rinse the sample bottle thoroughly
d) Transfer the solvent to the cartridge with a disposable pipette, rinsing sides of filtration reservoir
e) Draw half of solvent through cartridge then release the vacuum. Allow the remaining solvent to soak the cartridge for about one minute
f) Draw remainder through under vacuum
g) Repeat the solvent rinse of the sample bottle and apparatus using 10 mL of 1:1 EtOAc:$MeCl_2$
h) Using a disposable pipette, rinse down the sides of the cartridge and bottle holder with another 10 mL aliquot of 1: 1 EtOAc:$MeCl_2$
i) Add the rinse to the cartridge, then draw through

4. Dry the combined eluant

a) Use granular anhydrous sodium sulfate
b) Rinse the collection tube and sodium sulfate with 2 x 5 mL portions of $MeCl_2$ and place combined solvent in a concentrator tube
c) Draw through using vacuum
d) Concentrate the extract to 1 mL under gentle stream of nitrogen (may be warmed gently) being careful not to spatter the contents

Note: Do not concentrate to <0.5 mL or loss of analytes could occur. Rapid extract concentration could result in loss of low molecular weight analytes

5. Analyze by GC/MS

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Revision 2.0, 1995. Method authors: Eichelberger, J. W., Behymer,T .D. Budde, W L., Munch, J., National Exposure Research Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268

This summary highlights major steps in the 525.2 method. Complete details about the preparation and composition of reagent solutions can be found in method and should be referenced by anyone needing complete details. It is available as a part of Supplement 11 from National Technical Information Service (NTIS), Springfield, VA 22161; publication PB 92 207703. (800) 553-6847 or at www.epa.gov/safewater/methods/methods.html

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