

Method Summary

EPA Method 525.2



Determination

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

Method promulgation was announced in the Federal Register Volume 59, No. 232 on December 5, 1994. Method authors: J.W. Eichelberger, T.D. Behymer, W.L. Budde; Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

This Drinking Water Methods document is not currently published in an NTIS document and is available directly from US EPA: Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268. Telephone: (513) 569-7586.

Summary of Method

A one liter water sample is passed through a 47mm C18 NuPhase™ disk and eluted with ethyl acetate and methylene chloride, the extract dried and reduced in volume to 1.0 mL and analyzed by GC/MS.

Analytes. The analyte list for this method is comprised of 110 compounds representative of several classes of pesticides, polynuclear aromatic hydrocarbons, PCBs, phthalates and adipates. Method detection limits (MDLs) as published in the method range from 0.03-2.4 µg/L and recoveries from 0-180%. Refer to the published method for compound specific MDLs and recoveries. By compound class, average recoveries are:

Pesticides 108%
PCBs 108%
Phthalates & Adipates 116%
PAHs 112%

The validation data presented herein were determined on three independent lots of C18 disks. MDLs were not determined as part of this validation. In addition to the listed method analytes, recovery data for an extended list of analytes is also included.

525.2 METHOD PERFORMANCE

Analyte	Ave %R(RSD) (n=9)	Analyte	Ave %R(RSD) (n=9)
Acenaphthylene	109(3.2)	Endosulfan I	102(5.2)
Alachlor	113(2.3)	Endosulfan II	118(3.4)
Aldrin	113(3.7)	Endosulfan sulfate	117(3.3)
Ametryn	123(6.5)	Endrin	126(3.0)
Anthracene	120(3.4)	Endrin aldehyde	119(9.2)
Atraton I	44(22)	EPTC	112(8.4)
Atrazine	123(4.6)	Ethoprop	110(4.2)
Benz[a]anthracene	105(3.4)	Etridiazole	116(3.7)
Benzo[b]fluoranthene	112(4.8)	Fenamiphos	99(2.2)
Benzo[k]fluoranthene	109(10)	Fenarimol2	150(5.5)
Benzo[g,h,i]perylene	120(3.9)	Fluorene	108(3.0)
Benzo[a]pyrene	106(2.8)	Fluridone2	114(4.5)
BHC, alpha	122(4.2)	Heptachlor	112(4.1)
BHC, beta	112(4.3)	Heptachlor epoxide	109(2.9)
BHC, delta	119(3.4)	Hexachlorobenzene	104(4.1)
BHC, gamma (Lindane)	118(2.7)	Hexachlorocyclopentadiene	103(7.5)
Bromacil	115(7.1)	Hexazinone	125(4.6)
Butachlor	105(2.7)	Indeno [1,2,3, c, d] pyrene	121 (3.7)
Butylate	115(6.0)	Isophorone	87(11)
Butylbenzylphthalate	121 (3.4)	Methoxychlor	107(2 1)
Carboxin	100(4.9)	Methyl paraoxon	106(4 2)
Chlordane, alpha	97(3.1)	Metolachlor	109(2 2)
Chlordane, gamma	119(3.5)	MetribuzinI	81 (6.0)
Chlordane, bans nonachlor	98(4.2)	Mevinphos	108(7.9)
Chlorneb	123(4.7)	MGK 264	116(4.0)
Chlorobenzilate	107(2 8)	Molinate	121(5.6)
Chlorpropham	100(3 2)	Napropamide	97(3.2)
Chlorpyrifos	120(5.9)	Norflurazon2	141(4.2)
Chlorthalonil	110(3.2)	Pebulate	119(5.4)
Chrysene	104(3.1)	Pentachlorophenol	81 (4.9)
Cyanazine	77(3.2)	Permethnn, cis	110(4.4)
Cycloate	124(3.1)	Permethrin, trans	114(3.8)
DCPA	122(4 1)	Phenanthrene	113(1.9)
4,4'-DDD	112(3.8)	PrometonI	49(37)
4,4'-DDE	99(7 0)	Prometryn	110(5.2)
4,4'-DDT	110(7.4)	Pronamide	120(4.7)
Diazinon	98(13)	Propachlor	120(5.6)
Dibenz[a,h]anthracene	124(3.7)	Propazine	118(4.4)
di-n-butylphthalate	125(4.4)	Pyrene	109(2.4)
Dichlorvos	109(10)	Simazine	92(7.3)
Dieldrin	98(2.9)	Simetryn	103(15)
di(2-ethylhexyl)adipate	111 (4.0)	Stirofos	106(4.3)
di(2-ethylhexyl)phthalate	122(3.7)	Tebuthiuron2	100(11)
Diethylphthalate	106(3.5)	Terbacil	108(6 8)
Dimethylphthalate	113(4.8)	TerbufosI	124(3.0)
2,4-dinitrotoluene	79(11)	Terbutryn	113(5.5)
2,6-dinitrotoluene	84(9.8)	Triademefon	117(13)
Diphenamid	1 08(2.8)	Tricyclazole2	137(12)
Disulfoton	115(7.6)	Trifluralin	113(5.6)
Disulfoton sulfone2	164(2.8)	Vernolate	102(4.8)
Disulfoton sulfoxide2	136(8.9)		
PCB Congeners			
2,3-Dichlorobiphenyl	116(3.6)	1 n=3	
2-Chlorobiphenyl	99(2.3)	2 Analyte recovery reported is from EPA published method.	
2,2',3,3',4,4',6-heptachlorobiphenyl	110(5.6)	It was not included in the independent validation.	
2,2',4,4',5,6'-hexachlorobiphenyl	106(3.3)		
2,2',3,3',4,5',6,6'-octachlorobiphenyl	106(6.2)	Spike levels = 2.0 µg/L	
2,2',3',4,6-pentachlorobiphenyl	105(3.2)		
2,2',4,4'-tetrachlorobiphenyl	114(2.9)		
2,4,5-trichlorobiphenyl	114(3.1)		

Method

1. Assemble an all glass filtration assembly using a 47 mm C18 NuPhase™ disk. Use of a manifold for multiple extractions is acceptable.
2. Wash the extraction apparatus and disk by adding 5 ml of a 1:1 mixture of ethyl acetate (EtAc): methylene chloride (MeCl₂) to the reservoir. Pull a small amount through the disk with a vacuum; turn off the vacuum and allow the disk to soak for about one minute. Pull the remaining solvent through the disk and allow the disk to dry.
3. Condition the disk by adding approximately 5 ml of methanol to the reservoir, pulling a small amount through the disk then letting it soak for about one minute. Pull most of the remaining methanol through the disk, leaving 3 to 5 mm of methanol on the surface of the disk.
4. Add 5 ml of reagent water to the disk and using the vacuum pull most through, again leaving 3 to 5 mm of water on the surface of the disk.
5. Add 5 ml of methanol to the water sample and mix well. Add the water sample to the reservoir and, under vacuum, filter as quickly as the vacuum will allow. Drain as much water from sample bottle as possible.
6. Remove filter assembly and insert suitable sample tube for eluate collection.
7. Add 5 ml of EtAc to the sample bottle. Rinse bottle thoroughly and transfer solvent to the disk with dispo-pipet, rinsing sides of filtration reservoir in the process.
8. Pull half of solvent through disk then release the vacuum. Allow the remaining solvent to soak the disk for about one minute, then draw remainder through under vacuum.
9. Repeat the solvent rinse of the sample bottle and apparatus using 5 mL of MeCl₂.
10. Using a disposable pipette, rinse down the sides of the filtration glassware with two 3 mL aliquots of 1:1 EtAc/MeCl₂.
11. Dry the combined eluant with 5-7 grams granular anhydrous sodium sulfate. Rinse the collection tube and sodium sulfate with two 3 mL portions of 1:1 EtAc/MeCl₂ and place combined solvent into a concentrator tube.
12. Concentrate extract to 1 ml under gentle stream of nitrogen (may be warmed gently). Do not concentrate to <0.5 ml or loss of analytes could occur.
13. Analyze by GC/MS.

Additional compounds validated by this method include:

Analyte	Ave %R (RSD)¹	Analyte	Ave %R (RSD)¹
Aspon	121(3.8)	Famphur	107(5.3)
Azinphos-methyl	93(3.2)	Fenthion	124(4.1)
Benfluralin	115(4.5)	Fluazifop-butyl	123(3.1)
Bolstar	117(4.1)	Fluchloralin	123(3.5)
Chloropropylate	103(3.0)	Fluometuron	99(3.9)
Clomazone	104(4.1)	Malathion	113(3.9)
Coumaphos	100(2.3)	Merphos	100(17)
Demeton	61(4.5)	1-methyl Naphthalene	93(4.5)
Desethylatrazine	26(3.0)	MGK-326	120(5.1)
Desisopropylatrazine ²	102(NA)	Oxadiazon	115(2-9)
Dichlobenil	99(7.2)	Pendimethalin	123(7.1)
Dichlofenthion	123(3.2)	Phorate	97(4.1)
Dichloran	114(12)	Profluralin	127(11)
Dyfonate	104(3.5)	Propanil	100(4.2)
Ethelfluralin	97(2.3)	Tribufos (DEF)	107(3.8)
Ethion	115(3.6)		

1 Spike levels 2.0 µg/L, n=9

2 n=2

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