



Empore™ Extraction Disks

Method Summary

Proposed SPE Disk Method for Aqueous Phase EPA Quick Turnaround Methods (QTM):
Pesticides and Polychlorinated Biphenyls (PCBs)

The pesticide and PCB analyses for the QTM's are performed on separate fractions. The sample extraction is the same; however, the clean-up protocols vary. Because the sample extractions are identical, this method summary applies to both analyte groups. Please refer to the appropriate QTM for details on clean-up protocols.

Summary

For each group, a water sample (100 ml) is passed through a 47 mm C18 Empore™ disk and eluted with methylene chloride. The extract is dried, reduced in volume, solvent exchanged to hexane and analyzed by GC/ECD for Pesticides/PCBs. If interfering compounds are present, clean-up steps are described in the EPA QTM method.

<u>Analyte</u>	PERFORMANCE DATA			
	High Level^a		Low Level^b	
	Ave	%	Ave	%
	% R	RSD	% R	RSD
PESTICIDES:				
alpha-BHC	68.8	3.8	80.7	3.0
beta-BHC	78.2	2.2	84.2	3.4
gamma-BHC	72.9	3.4	83.9	2.7
delta-BHC	77.6	1.4	120	16.5
Heptachlor	68.0	5.5	89.3	3.3
Aldrin	48.3	6.4	80.9	5.4
Heptachlor epoxide	79.2	2.1	88.7	2.3
gamma-Chlordane	72.9	2.4	80.6	3.0
Endosulfan I	81.5	2.9	86.0	3.2
alpha-Chlordane	75.7	2.1	82.1	3.1
4,4'-DDE/Dieldrin	70.6	3.6	75.0	5.4
Endrin	91.6	6.7	110	1.5
Endosulfan II	78.8	3.0	89.6	2.4
4,4'-DDD	78.3	1.0	84.2	1.8
Endrin aldehyde	75.9	3.4	79.1	2.9
Endosulfan sulfate	82.7	1.7	86.1	1.0
4,4'-DDT	71.1	3.5	93.2	^d
Endrin ketone	80.2	1.2	83.4	2.5
Methoxychlor	93.2	5.1	108	6.9
Decachlorobiphenyl ^c	88.0	1.9	80.2	6.0
PCB's: AR-1016	60.6	3.5	62.1	18.2
AR-1260	75.6	5.8	79.4	5.9
Decachlorobiphenyl ^c	85.7	6.3	86.7	5.6

^a Compounds spiked at 0.50 ppb (Pest) and 5.0 ppb (PCB) into groundwaters obtained from two different hazardous waste sites. n=6.

^b Compounds spiked at 0.10 ppb (Pest) and 1.0 ppb (PCB) into groundwater from one site. n=3.

^c Surrogate compound.

^d Due to background interference encountered in one replicate sample RSD was not determined.

Method

1. Assemble an all glass filtration assembly using a 47 mm C18 Empore disk. Use of a manifold for multiple extractions is acceptable.
2. Wash the extraction apparatus and disk by adding 5 ml of methylene chloride 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 3-5 mm of methanol on the surface of the disk.
4. Add 10 ml of reagent water to the disk. Using the vacuum pull most through, again leaving 3-5 mm of water on the surface of the disk.
5. Add 0.5 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 the sample bottle as possible.
6. Remove filter assembly and insert suitable sample tube for eluate collection.
7. Add 10 ml of methylene chloride to sample bottle. Rinse bottle thoroughly and set aside momentarily.
8. Wet the disk with a small amount of acetone – just enough to wet the surface (approximately 0.5 ml or less) and immediately transfer the methylene chloride from the sample bottle to the disk with dispo-pipette, rinsing the sides of the filtration reservoir in the process.
9. Pull half of the solvent through the disk then release the vacuum. Allow the remaining methylene chloride to soak the disk for about one minute then draw remainder through under vacuum.
10. Repeat the solvent rinse of the sample bottle using 5 ml methylene chloride and transfer to the apparatus, rinsing down the sides of the reservoir. Add 5 ml methylene chloride directly on the disk, let soak for about one minute, and draw through under vacuum.
11. Dry the combined eluate with anhydrous sodium sulfate. Rinse the collection tube and sodium sulfate with two 5 ml aliquots of methylene chloride and place combined solvent into a concentrator tube.
12. Concentrate extract to approximately 2 ml with gentle stream of nitrogen. Add hexane to bring volume to 10 ml., concentrate to approximately 4 ml and adjust to 5 ml final volume with hexane.
13. Analyze by GC/ECD.

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