



Empore™ Extraction Disks

Method Summary

EPA Method 508.1

Determination of Chlorinated Pesticides, Herbicides, and Organohalides in Water Using Liquid-Solid Extraction and Electron Capture Gas Chromatography

Method promulgation was announced in the Federal Register Vol. 59, No. 232 on 12/5/94. This method is not currently published in an NTIS manual, and is available directly from EPA: Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268. Telephone: (513) 569-7586.

Summary

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

Method Performance^a

<u>Analyte</u>	<u>Mean %R^b (n=8)</u>	<u>Analyte</u>	<u>Mean %R^b (n=8)</u>
Alachlor	91	Endosulfan I	102
Aldrin	70	Endosulfan II	106
Atrazine	75	Endosulfan sulfate	117
alpha-BHC	109	Endrin	111
beta-BHC	91	Endrin Aldehyde	99
delta-BHC	120	Etridiazole	107
gamma-BHC	111	Heptachlor	83
alpha-Chlordane	98	Heptachlor epoxide	100
gamma-Chlordane	93	Hexachlorobenzene	91
Chlorobenzilate	107	Hexachlorocyclopentadiene	53
Chloroneb	155	Methoxychlor	109
Chlorothalonil	123	Metolachlor	126
Cyanazine	95	Metribuzin	68
DCPA	89	cis-Permethrin	102
4,4'-DDD	108	trans-Permethrin	117
4,4'-DDE	95	Propachlor	102
4,4'-DDT	116	Simazine	82
Dieldrin	101	Trifluralin	90

a = Data reported from the published EPA Method 508.1.

b = Spike levels 0.048 µg/L

Though not included as analytes for this method, the following mixtures were also validated by 3M using the disk procedure and the data shared with the EPA: (Ave % Recovery, spike level 0.5 µg/L, n=10)

Aroclor 1016	78%	Aroclor 1254	84%
Aroclor 1242	70	Aroclor 1260	87
Chlordane (technical)	82	Toxaphene	88

Method

1. Assemble an all glass filtration assembly using a 47mm C18 Empore™ 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) and 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 3-5 mm of methanol on the surface of the disk.
4. Add 5 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 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 the solvent to the disk with a disposable pipette, rinsing down the sides of the filtration reservoir in the process.
8. Pull half of the solvent through disk then release the vacuum. Allow the remaining EtAc 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 MeCl₂.
10. Rinse the reservoir and disk with two 3 mL aliquots of 1:1 EtAc/MeCl₂.
11. Dry the combined eluates with 5-7 grams anhydrous sodium sulfate. Rinse the collection tube and sodium sulfate with two 3 mL portions of EtAc/MeCl₂ and place the combined solvent into a concentrator tube.
12. Concentrate the extract to 0.8 mL under a gentle stream of nitrogen (may be warmed gently – approximately 30 degrees C). Add internal standard fortifying solution and adjust the volume to 1.0 mL with EtAc.
13. Analyze by GC/ECD.

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