Empore™
Extraction Disks

Method Summary
SPE Disk Modification to EPA Method 507

This method is an alternative to EPA Method 507 – Determination of Nitrogen – and Phosphorus-Containing Pesticides in Water by Gas Chromatography with a Nitrogen-Phosphorus Detector which incorporates the use of C18 Disks for the extraction of the compounds of interest. This modification has not been written or approved by the EPA; but some of the analytes may be analyzed by Method 508.1 which also incorporates Empore™ disks.

Although this modification is not included in the currently available publications, the reference methods are included in the following documents:

Methods for the Determination of Organic Compounds in Drinking Water – publication PB 91-231480. Supplement I – publication PB91-146027; Supplement II – publication PB92-207703

The Drinking Water Methods documents are available from National Technical Information Service (NTIS), Springfield, VA 22161; (800) 553-6847

Summary

A one liter water sample is passed through a 47 mm Cl8 Empore disk and eluted with ethyl acetate and methylene chloride. The extract is dried, reduced in volume[a] to about 2.0 ml, solvent exchanged to methyl tert-butyl ether (MTBE), reconcentrated to 5.0 ml, and analyzed by GC/NPD.

Analyte	Calc. MDL (µg/L)	Mean %R[c] n=10	Analyte	Calc. MDL (µg/L)	Mean %R[c] n=10
Alachlor[b]	0.27	73.6	Methyl paraoxon	0.53	90.4
Ametryn	0.23	109.0	Metolachlor	1.00	79.0
Atraton	0.80	128.0	Metribuzin	0.24	68.8
Atrazine[b]	0.19	73.6	Mevinphos	1.00	97.7
Bromacil	1.98	75.2	MGK-264	1.68	76.0
Butachlor	0.74	75.0	Molinate	0.23	32.0
Butylate	0.58	61.9	Napropamide	0.93	77.9
Carboxin	2.50	87.7	Norflurazon	0.77	114.0
Chlorpropham	1.00	93.5	Pebulate	0.41	64.3
Cycloate	0.54	73.4	Prometon	0.56	97.3
Diazinon	0.18	73.0	Prometryn	0.33	112.0
Dichlorvos	0.91	118.0	Pronamide	0.93	52.9
Diphenamid	0.60	78.3	Propazine	0.21	62.2
Disulfoton	0.53	63.0	Simazine[b]	0.09	72.6
Disulfoton sulfoxide	2.88	73.2	Simetryn	0.23	107.0
Disulfoton sulfone	0.01	112.0	Stirofos	0.34	63.9
EPTC	0.75	74.0	Terbacil	1.39	75.7
Ethoprop	0.17	75.8	Terbufos	0.10	65.1
Fenarimol	0.70	84.1	Terbutryn	0.31	92.5
Fenamiphos	0.23	78.7	Triademefon	0.74	132.0
Fluridone	5.90	73.6	Tricyclazole	2.29	94.0
Hexazinone	0.29	78.7	Vernolate	0.25	70.9
Merphos	0.07	75.7			

[a] = These data were generated using the attached method which included a nitrogen blowdown with gentle heating. Recovery of some of the more volatile compounds may be increased by using the micro KD concentration as outlined in EPA Method 507.

[b] = Regulated Compound

[c] = Spiking levels range 0.3-5.1 µg/L

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Method

1. Assemble an all glass filtration apparatus 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 a 1:1 mixture of ethyl acetate/methylene chloride to the reservoir. Pull a small amount through the disk with a vacuum; turn off the vacuum and allow disk to soak for about one minute. Pull the remaining solvent through 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 and 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 ethyl acetate to sample bottle. Rinse bottle thoroughly and transfer solvent to the disk with dispo-pipette, rinsing sides of filtration reservoir in the process.
8. Pull half of solvent through disk then release the vacuum. Allow the remaining ethyl acetate 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 a 1:1 mixture of ethyl acetate and methylene chloride and repeat a third time with 5 ml methylene chloride.
11. Dry the combined eluates with anhydrous sodium sulfate. Rinse the collection tube and sodium sulfate with two 5 ml portions of methylene chloride and place combined solvent into a concentrator tube.
12. Concentrate extract to approximately 2 ml under gentle stream of nitrogen (may be warmed gently at approximately 30 degrees C). Add about 8 ml MTBE to bring volume to 10 ml. Concentrate extract to approximately 4 ml and adjust to 5 ml final volume with MTBE.
13. Analyze by GC/NPD.

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