



Empore™ Extraction Disks

Method Summary

EPA Method 553

Determination of Benzidines and Nitrogen-Containing Pesticides in Water by Liquid-Liquid Extraction or Liquid-Solid Extraction and Reverse Phase High Performance Liquid Chromatography/Particle Beam/Mass Spectrometry

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The complete method is available as a part of Supplement II from National Technical Information Service (NTIS), Springfield, VA 22161; publication PB 92 207703, (800) 553-6847.

Summary

This method determines certain benzidines and nitrogen-containing pesticides in water. The analytes are extracted from the water with a C18 or poly(styrene-divinylbenzene) disk and eluted with methanol. The final analysis uses particle beam HPLC/MS.

Analytes

Benzidine	3,3'-dimethylbenzidine
Benzoylprop ethyl	Diuron
Caffeine*	Ethylene thiourea*
Carbaryl	Linuron
o-chlorophenyl thiourea*	Monuron
3,3'-dichlorobenzidine	Rotenone
3,3'-dimethoxybenzidine	Siduron

Compounds which may also be amenable to this method include Aldicarb sulfone, Carbofuran, Methiocarb, Methomyl, Mexacarbate, and N-(1-Naphthyl) thiourea.

*These compounds have been successfully analyzed by HPLC/PB/MS, but not successfully extracted from water.

Method Detection Limits

Method detection limits (MDLs) in reagent water range from 1.6 µg/L to 29.3 µg/L with an average of 10.7 µg/L.

Method

1. Assemble Empore™ 47 mm C18 or SDB extraction disks in an all-glass filtration apparatus (manifolds are appropriate for multiple samples). Although the method offers a choice of sorbents, the SDB provides higher recoveries for benzidine.
2. Wash and condition the disk by adding 10 ml of methanol to the reservoir, pulling about half of the methanol through the disk and allowing the disk to soak for approximately one minute. Using the vacuum, pull most of the remaining methanol through the disk, leaving 3 to 5 mm of methanol on the surface of the disk.

3. Add 10 ml reagent water to the disk and, using the vacuum, pull most of the water through the disk to eliminate the methanol, leaving 3 to 5 mm of water on the surface of the disk.
4. Add the water sample to the reservoir and start the vacuum. Pull the sample through the disk as fast as the vacuum will allow. Drain as much of the water from the sample bottle as possible.
5. Remove the filtration assembly from the filter flask or manifold, discard the water sample, and place an appropriately sized sample collection tube into the filter flask or manifold.
6. Add 5 ml methanol to the sample bottle and rinse it thoroughly. Allow the methanol to collect on the bottom of the sample bottle; transfer it to the disk reservoir using a dispo-pipet, rinsing the sides of the reservoir in the process.
7. Pull half of the methanol through the disk and release the vacuum. Allow the remaining methanol to sit on the disk for about one minute; then pull the remaining methanol through the disk, collecting the methanol in the test tube.
8. Repeat the solvent rinse of the sample bottle and filter apparatus twice using 5 ml aliquots of methylene chloride.
9. Concentrate the combined extracts to the desired volume under a gentle stream of nitrogen (no volume suggested in the method). Acetonitrile is suggested as a better extraction solvent for rotenone when the water sample contains high levels of particulate matter.
11. Analyze by HPLC/PB/MS.

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