Empore™
Extraction Disks

Method Summary
EPA Method 506

Determination of Phthalate and Adipate Esters in Drinking Water by Liquid-Liquid Extraction or Liquid-Solid Extraction and Gas Chromatography with Photoionization Detection

Method approval announced in Federal Register Volume 57, No. 138, Page 13779 (July 17, 1992). Issued July 1990 as a part of the "Methods for the Determination of Organic Compounds in Drinking Water, Supplement I" Authors: F.K. Kawahara and J.W. Hodgeson, Environmental Monitoring Systems Laboratory, US Environmental Protection Agency, Cincinnati, Ohio 45268.

This method is part of Supplement I and is available from National Technical Information Service (NTIS) Springfield, VA 22161; publication PB91-108266, (800) 533-6847.

Summary

This method is used for the determination of certain phthalate and adipate esters in drinking water. This summary describes the solid phase extraction technique utilizing Empore™ extraction disks. A liter of water is extracted with a 47 mm C18 extraction disk, eluted with methylene chloride, concentrated under a nitrogen stream, and analyzed by gas chromatography.

Analytes

Bis (2-ethylhexyl) phthalate
Butylbenzyl phthalate
Di-n-butyl phthalate
Diethyl phthalate
Dimethyl phthalate
Bis (2-ethlhexyl) adipate
Di-n-octyl phthalate

Method Detection Limits

The published method detection limits (MDLs) for this method range from 0.84 to 11.82 µg/L, and were determined using liquid-liquid extractions (LLE). In general, disks produce lower detection limits than LLE.

Method

1. Assemble an all glass filtration assembly using a 47 mm C18 extraction disk. Use of a manifold for multiple extractions is acceptable.
2. Wash the extraction disk by adding 5 ml of methylene chloride to the reservoir, pulling about half through the disk and allowing the disk to soak for approximately one minute. Using the vacuum, pull the remaining methylene chloride through the disk and allow the disk to dry.
3. Condition the disk by adding approximately 5 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.
4. Add 5 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.

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5. 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.
6. 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.
7. Add 5 ml acetonitrile to the sample bottle and rinse it thoroughly. Allow the acetonitrile 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.
8. Pull half of the acetonitrile through the disk and release the vacuum. Allow the remaining acetonitrile to sit on the disk for about one minute; then pull the remaining acetonitrile through the disk, collecting the acetonitrile in the test tube.
9. Repeat the solvent rinse of the sample bottle and filter apparatus twice using 5 ml aliquots of methylene chloride.
10. Dry the combined extracts with sodium sulfate. Rinse the test tube and sodium sulfate with two 5 ml portions of methylene chloride and place combined extract into a concentrator tube. Evaporate the eluate to 0.5 ml under nitrogen at 28°C.
11. Analyze by gas chromatography.

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