



Empore™ Extraction Disks

Method Summary

EPA Method 550.1

Determination of Polycyclic Aromatic Hydrocarbons in Drinking Water by Liquid-Solid Extraction and HPLC with Coupled Ultraviolet and Fluorescence Detection

Method acceptance 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." Author: 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 Services (NTIS), Springfield, VA 22161; publication PB 91-108266, (800) 553-6847.

Summary

A one liter water sample is passed through a 47 mm C18 Empore™ disk to remove the analytes. The disks are eluted with acetonitrile and methylene chloride, the extract dried and reduced to 0.5 ml and analyzed by HPLC using ultraviolet and fluorescence detectors.

Analytes

Acenaphthene	Acenaphthylene
Anthracene	Benzo(a)anthracene
Benzo(a)pyrene	Benzo(b)fluoranthene
Benzo(g,h,i)perylene	Benzo(k)fluoranthene
Chrysene	Dibenzo(a,h)anthracene
Fluoranthene	Fluorene
Indeno(1,2,3cd)pyrene	Napthalene
Phenanthrene	Pyrene

Method Detection Limits

Method detection limits (MDLs) in water samples range from 0.126 to 2.20 µg/L for the compounds determined using a UV detector; and for compounds measured by fluorescence detection, from 0.003 to 0.160 µg/L. MDLs for this method were not differentiated for LLE versus SPE disks. In general, disks successfully produce lower detection limits than liquid-liquid extractions.

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 water on the surface of the disk.
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 into the sample collection tube.
9. Repeat the solvent rinse of the sample bottle and filter apparatus twice using 5 ml aliquots of methylene chloride instead of acetonitrile.
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 HPLC with coupled UV and fluorescence detection.

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**New Products Department
3M Industrial and Consumer Sector**

3M Center, Building 220-9E-10
St. Paul, MN 55144-1000

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