

Analysis of Polynuclear Aromatic Hydrocarbons Using US EPA Quick Turnaround Method (QTM) by CDS Empore Disk

Application Note

Environmental

Abstract

This application note shows Empore™ C18 SPE Disk to effectively extract Polynuclear Aromatic Hydrocarbons (PAHs) from aqueous sample and quantified by GC-FID as the EPA QTM method.

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Introduction

Polycyclic aromatic hydrocarbons (PAHs) are well-known as one of the most hazardous organic pollutants to environment. PAHs are further defined by a group of over 100 different compounds with fused aromatic rings and are normally formed during the incomplete burning of carbon based fuels (wood, coal, diesel, oil and gas, and other organic compounds), as well as directly residing in crude oil, rubbers, plastics, lubricants, paints, leather and other products. Seven PAH compounds: benz[a]anthracene, benzo[a]pyrene, benzo[b]fluoranthene, benzo[k]fluoranthene, chrysene, dibenz[a,h]anthracene, and indeno[1,2,3-cd]pyrene have been classified as being potentially carcinogenic, mutagenic and teratogenic by the United States Environmental Protection Agency (US EPA). PAHs need to be closely monitored in environments, especially in drinking water to ensure the public safety. In this application note, a simple but effective method to extract PAHs from large volume water sample (100 ml) via a 47mm C18 CDS Empore™ disk and eluted with methylene chloride has been developed. The extract is then concentrated and analyzed by GC/FID. If interfering compounds are present, clean-up protocols are described in the EPA QTM method.

Method

- Assemble an all glass filtration assembly using a 47mm C18 Empore disk. Use of a manifold for multiple extractions is acceptable.
- Wash the extraction apparatus and disk by adding 5 ml of methylene chloride 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.
- 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.
- Add 10 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.
- Add 0.5 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 the sample bottle as possible.
- Remove filter assembly and insert suitable sample tube for eluate collection.



- Add 10 ml of methylene chloride to sample bottle. Rinse bottle thoroughly and set aside momentarily.
- Wet the disk with a small amount of acetone – just enough to wet the surface (approximately 0.5 ml or less) and immediately transfer the methylene chloride from the sample bottle to the disk with a dispo-pipette, rinsing the sides of the filtration reservoir in the process.combined solvent into a concentrator tube.
- Pull half of the solvent through the disk then release the vacuum. Allow the remaining methylene chloride to soak the disk for about one minute then draw remainder through under vacuum.
- Repeat the solvent rinse of the sample bottle using 5 ml methylene chloride and transfer to the apparatus, rinsing down the sides of the reservoir. Add 5 ml methylene chloride directly on the disk, let soak for about one minute, and draw through under vacuum.
- Dry the combined eluate with anhydrous sodium sulfate. Rinse the collection tube and sodium sulfate with two 5 ml aliquots of methylene chloride and place combined solvent into a concentrator tube.
- Concentrate extract to 1.0 ml under a gentle stream of nitrogen (may be warmed gently at approximately 30°C).
- Analyze by GC/FID.

The CDS Empore™ C18 47mm disk demonstrates its advantage on large volume (100 ml) aqueous samples analysis than the traditional SPE cartridge with filled media. This method could be used as a screening tool for environmental testing labs to analyze PAHs' concentrations from aqueous samples.

Results

Analyte	High Level ^a		Low Level ^b	
	Ave % R	% RSD	Ave % R	% RSD
Naphthalene	47.0	4.2	48.2	0.4
Acenaphthylene	58.8	3.9	59.2	1.8
Acenaphthene	57.7	3.8	60.0	2.3
Fluorene	62.7	4.1	69.5	6.8
Phenanthrene	67.0	4.6	80.6	7.6
Anthracene	64.9	4.5	81.4	7.9
Fluoranthene	70.9	4.7	86.5	7.1
Pyrene	70.6	5.2	86.1	6.8
Benzo(a)anthracene	67.4	6.6	91.6	5.4
Chrysene	67.5	7.0	90.9	5.0
Benzo(b&k)fluoranthene	69.9	6.4	87.5	6.7
Benzo(a)pyrene	69.5	7.2	91.7	5.8
Indeno(1,2,3,c,d)pyrene	69.0	9.7	94.0	5.9
Dibenzo(a,h)anthracene	72.0	8.6	88.4	7.4
Benzo(g,h,i)perylene	74.4	7.5	91.7	7.3
Bromofluorene ^c	73.1	5.9	58.4	14.4

^a Compounds spiked at 200 ppb into groundwaters from two different hazardous waste sites. n=6.

^b Compounds spiked at 20 ppb into groundwater from a hazardous waste site. n=3.

^c Surrogate compound

Conclusion

A simple and effective method to extract PAHs from large volume water sample (100 ml) by CDS Empore™ C18 47mm disk has been developed. 16 PAHs extracted from groundwaters were then quantified by GC-FID with concentration at 20ppb and 200ppb with an average RSD of 5.9% and 6.2% respectively.