Automated Solid-Phase Extraction (SPE) of Dioxins and Furans in Surface Water

Introduction

U.S. EPA Methods 8260 and 8270 are the basis of the sample preparation of polychlorinated dibenzo-p-dioxins, polychlorinated dibenzofurans and polychlorinated biphenyls (PCBs) in surface and ground water. The clean up described in this Application Brief also recovers 1,4-Dioxan. The addition of a 1% v/v isopropanol solution to the sample bottle reduces losses due to adhesion to glass and tubing. The use of HCL 0.1 M to acidify the sample to pH 2 prevents the humic acids often present in the water from allowing dioxins to pass through the Thermo Scienficic™ Dionex™ SolEx™ SPE C18 Cartridge.

Instrumentation Used for Sample Preparation

Thermo Scientific™ Dionex™ AutoTrace™ 280 Solid-Phase Extraction instrument

Sample Preparation

- A 500 mL sample of river water is taken for the analysis and mixed with 5 mL of isopropanol to prevent the components from sticking to the glass bottle.
- The use of HCL 0.1 M is important to adjust the water to pH 2 to enhance recoveries
- A standard Dionex SolEx SPE C18 cartridge end capped SPE silica column, 6 mL, 1 g, can be used for the clean up. The elution solvent is ethanol/toluene (70:30) mL and six sample collection positions for the eluent.

Method and Conclusion

All solvent lines are purged and primed with solvent first. A maximum of five solvents can be used to run a wide range of methods. Independent lines separate the aqueous and organic waste solvents. The instrument sample rack has six positions that can take volumes from 10 mL to 2000 mL and six sample collection positions for the eluent. This method offers an automated rugged and reproducible solution for cleaning up the samples to concentrate and remove interferences.

No.	Method: Estimated Time 1 h 30 min
1	Process six samples using the following method steps:
2	Condition column with 5.0 mL of CH ₃ OH into solvent waste.
3	Condition column with 5.0 mL of water into aqueous waste.
4	Load 500.0 mL of sample into column.
5	Rinse column with 3.0 mL of CH ₃ OH/water 40:60 into solvent waste.
6	Wash syringe with 5.0 mL of 70%EtOH/toluene.
7	Dry column with gas for 10 min.
8	Collect 5.0 mL fraction into sample tube using 70%EtOH/toluene.
9	Wash syringe with 5.0 mL of CH ₃ 0H.
10	End.



Parameters

Flow Rates		
Cond Flow:	15.0 mL/min	
Load Flow:	10.0 mL/min	
Rinse Flow:	20.0 mL/min	
Elute Flow:	5.0 mL/min	
Cond Air Push:	15.0 mL/min	
Rinse Air Push:	20.0 mL/min	
Elute Air Push:	5.0 mL/min	
SPE Parameters		
Push Delay:	5 sec	
Air Factor:	1.0	
Autowash Vol.:	1.00 mL	
Instrument Parameters		
Max. Elution Vol.:	12.0 mL	
Exhaust Fan On:	Yes	
Beeper On:	Yes	

www.thermoscientific.com/autotrace

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