Automated Solid-Phase Extraction of Triclosan in Tap Water Using Hydrophilic Reversed-Phase Cartridges and HPLC with UV Detection

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Key Words
Liquid hand soap, SolEx SPE HRPHS cartridges, Dionex AutoTrace 280, Recovery, Antifungal and antimicrobial agent

Goal
Analysis of triclosan in Sunnyvale tap water by automated SPE using the Dionex AutoTrace 280 instrument and a new polymeric sorbent Thermo Scientific™ Dionex™ SolEx™ SPE HRPHS cartridge

Introduction
Triclosan is a powerful antibacterial and antifungal agent that is an ingredient added to many consumer products to reduce or prevent bacterial contamination. This ingredient may be found in certain antibacterial soaps, body washes, toothpastes, and cosmetic products. Triclosan safety is currently under review by the U.S. FDA based on recent studies that indicate it may cause endocrine disruption in the body and possibly lead to the emergence of drug-resistant "super" bacteria. The Thermo Scientific™ Dionex™ AutoTrace™ 280 Solid-Phase Extraction (SPE) instrument performs automated SPE of large-volume liquid samples for organic analysis. In this application note, triclosan-contaminated samples of tap water were simulated by adding known amounts of triclosan-containing liquid hand soap to Sunnyvale tap water. The Dionex AutoTrace 280 instrument along with the high-surface-area, hydrophilic, reversed-phase cartridges were used to concentrate triclosan. The HRPHS stationary phase has a neutral resin comprised of a 22 µm, high-surface-area, divinylbenzene-based particle grafted with polyvinyl-pyrrolidone polymer. This material has properties of a hydrophilic, reversed-phase resin and also has high capacity for polyphenolics, including humic acids and azo-containing substance such as azo dyes. The hydrophilic, reversed-phase properties allow high recovery of a hydrophobic target such as triclosan.

Experimental
- Dionex AutoTrace 280 system for 6 mL Cartridges
- Thermo Scientific™ Dionex™ UltiMate™ 3000 HPLC system including:
  - DGP 3600M Dual-Gradient Micro Pump
  - SRD 3600 Integrated Solvent and Degasser Rack, 6 Channels
  - TCC-3000 Thermostatted Column Compartment
  - WPS-3000 Wellplate Sampler equipped with 5 µL loop
  - DAD-3000 Diode Array Detector
  - Semi-Micro Flow Cell for DAD-3000 and MWD-3000 Series, SST, 2.5 µL Volume, 7 mm Path Length
- Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System software, version 6.80 SP4 and higher

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SPE Conditions	
Cartridge:	Dionex SolEx HRPHS phase, Polymer-Based Solid-Phase Extraction Cartridge, 6 mL, 200 mg, Package of 48
Flow Rate:	5 mL/min
Elution Solvent:	Acetonitrile or methanol

Table 1. Dionex AutoTrace 280 instrument method.

No.	Method	SPE Steps
1	Condition cartridge with 5.0 mL of methanol into solvent waste	Condition
2	Condition cartridge with 10.0 mL of water into aqueous waste	Condition
3	Dry cartridge with gas for 5.0 minutes	Dry
4	Load 100.0 mL of sample onto cartridge at 10 mL/min	Load
5	Rinse cartridge with 5.0 mL of water into aqueous waste	Wash
6	Collect 5.0 mL fraction into sample tube using acetonitrile or methanol	Elute

Analytical Conditions	
Column:	Thermo Scientific™ Acclaim™ 120, C18, 5 µm, 2.1 × 50 mm
Eluent:	65/35 v/v acetonitrile/water
Flow Rate:	0.6 mL/min
Injection Volume:	5 µL
Temperature:	30 °C
Detection:	UV at 254 nm
Peaks:	Triclosan

Results and Discussion

Figure 1 is a chromatogram that shows the separation of triclosan in the tap water analyzed. The sample liquid antibacterial hand soap contains 0.15% triclosan. This antibacterial hand soap was diluted by 1:3000 in Sunnyvale tap water and 100 mL of this diluted hand soap was concentrated by an HRPHS SPE phase using the Dionex AutoTrace 280 instrument. The HRPHS SPE phase was packed into a 6 mL barrel with a bed weight of 500 mg. In the Dionex AutoTrace 280 SPE method, HRPHS sorbent is preconditioned by methanol followed by water. After drying under nitrogen for 5 min, 100 mL 1/3000 diluted liquid hand soap in Sunnyvale tap water was loaded at a flow rate of 10 mL/min. The hydrophilic matrix was removed by water rinse of the cartridge. Triclosan was eluted in the final step by an organic solvent such as methanol or acetonitrile (good recoveries of triclosan were achieved using both). The recoveries for triclosan using methanol was 96% and using acetonitrile was 103%.

Sample Prep
Instrument: Dionex AutoTrace 280 for 6 mL Cartridges HRPHS Resin
Sample: 100 mL 0.033 liquid antibacterial hand soap in Sunnyvale tap water
Flow Rate: 10 mL/min
Elution: Acetonitrile or methanol

Analytical
Column: Acclaim 120, 2 × 50 mm
Eluent: 65/35 Acetonitrile/water
Flow Rate: 0.21 mL/min
Detection: UV 254 nm

Peak: 1. Triclosan 10 mg/L*
* Final concentrated concentration

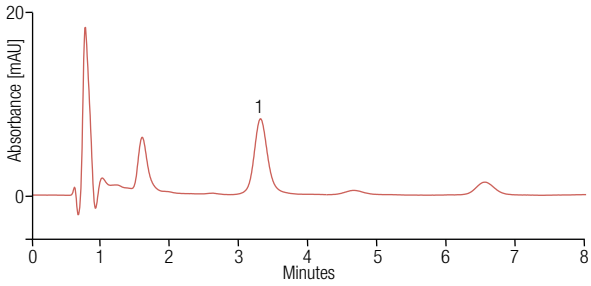

Figure 1. Chromatogram showing the separation of triclosan using the Dionex Acclaim 120 column after first using the Dionex SolEx HRPHS for sample preparation with the Dionex AutoTrace 280 instrument for automated SPE.

Conclusion

Shown here is the successful recovery of triclosan in tap water by automated SPE using the Dionex AutoTrace 280 instrument and Dionex SolEx HRPHS cartridge in two different elution solvents without laborious sample preparation followed by HPLC analysis.

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