High Recoveries of Phenols from Water with the Polymeric SPE Sorbent – strata™ X

Sky Countryman, David Shock and James Teuscher
Phenomenex Inc., Torrance, CA, USA

Phenol and substituted phenols are small polar organic molecules with low log k_w values. These chemicals have a wide range of industrial applications ranging from the production of adhesives to serving as intermediates in numerous synthetic processes. Due to their wide usage, water and soil samples are routinely monitored to detect for their presence as contaminants. The chemical nature of phenols makes their extraction from water a significant challenge in environmental analysis.

This application demonstrates a Solid Phase Extraction (SPE) method that uses strata-X to extract and concentrate phenols from water. strata-X is an innovative, patent-pending polymeric SPE sorbent developed by Phenomenex. With a surface area of >800m²/g, strata-X has a higher capacity for polar solutes than silica-based C18 or C8 SPE sorbents. The functionalized styrene-divinylbenzene surface of strata-X has numerous retention mechanisms, including hydrophobic, π–π and hydrophilic interactions that yield high recoveries for a wide range of compounds, including phenols from water. These unique surface properties give strata-X a significant advantage when compared with unmodified styrene-divinylbenzene (SDB), which is typically used in extracting organic molecules from water.[1]

Instrumentation and Equipment

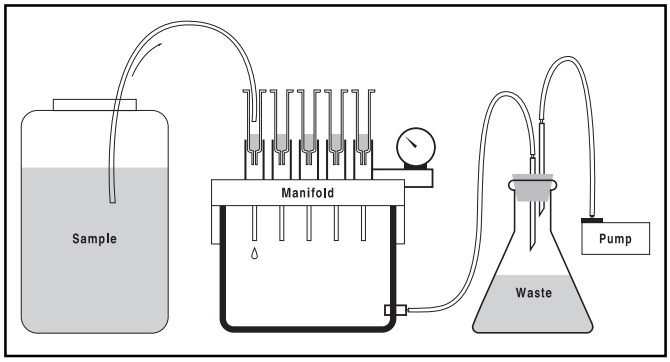
Solid Phase Extraction

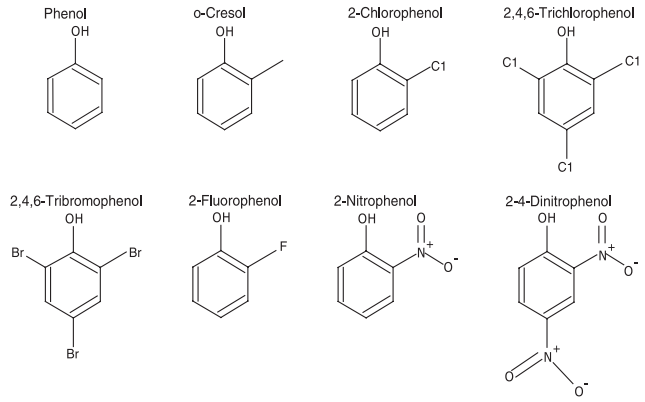
strata-X 500mg/6mL and Strata SDB-L 500mg/6mL syringe-barrel tubes were used. Multiple SPE tubes were processed simultaneously with a 12-position SPE vacuum manifold supplied by Phenomenex. The sample was continuously drawn through the tube using the apparatus shown in ***Figure 1.***

Gas Chromatography

The samples were analyzed using an HP 6890N GC system (Agilent Technologies, Palo Alto, CA) equipped with the HP 5973 Mass Selective Detector (MSD). The GC column was a Phenomenex Zebron ZB-5 (30.0m x 0.25mm x 0.25µm). The data was analyzed with HP ChemStation software.

Figure 1. Schematic of Apparatus used in the Extraction of Phenols from Water.





Experimental Conditions

Specimen Preparation

A 500mL water sample spiked with the target compounds was acidified with glacial acetic acid (pH = 3.5). The concentration range of 30-75ppm, consistant with practical quantitation limits for waste water in EPA method 8270.

SPE Extraction Method

Condition: The sorbent was conditioned with 5mL methanol, followed by 5mL deionized water.

Load: The 500mL sample was continually loaded onto the column using the vacuum manifold apparatus as shown in ***Figure 1.***

Elution: The compounds were eluted with 5mL acetone, followed by 5mL methylene chloride. All elution solvents were collected in a single test tube.

High Recoveries of Phenols from Water with the Polymeric SPE Sorbent – strata™ X

Analysis

Extracts were dried using anhydrous sodium sulfate and concentrated to 0.5mL.

GC Experimental Conditions: 1µL of sample was injected at 250°C in 11:1 split ratio. The initial oven temperature was set at 40°C and held for 3 min. The temperature was ramped to 325°C at 9°C/min (held for 2 minutes at final temperature). The flow rate of helium was 1.2mL/min.

Conclusion

As shown in ***Table 1***, strata-X successfully extracted ppm concentration levels of phenols from water with recoveries of >90%. To illustrate the high retention character of strata-X, percent recoveries using strata-X are compared with those using SDB. In all cases, the recoveries of these polar organic compounds are higher when strata-X was used in the extraction. In particular, the retention of phenol and 2-fluorophenol is significantly improved on the functionalized polymer. While the concentration levels fall in the range for the practical quantitation limits for waste water, similar recoveries for lower concentration levels are expected. The versatility and selectivity of strata-X make it a powerful tool in the extraction and concentration of polar and nonpolar compounds from water.

Reference

1. Solid Phase Extraction Principles and Practice
by Thurman and Mills, John Wiley & Sons, 1998, pages 189-190

Ordering Information:

Order No.	Description
8B-S100-TAK	strata-X Tubes (30mg/1mL)
8B-S100-UBJ	strata-X Tubes (60mg/3mL)
8B-S100-ECH	strata-X Tubes (100mg/6mL)
8B-S100-FCH	strata-X Tubes (200mg/6mL)
8B-S100-HCH	strata-X Tubes (500mg/6mL)
8B-S100-HDG	strata-X Giga Tubes (500mg/12mL)
8B-S100-JEG	strata-X Giga Tubes (1g/20mL)
8E-S100-AGB	strata-X 96-Well Plate (10mg/well)
8E-S100-TGB	strata-X 96-Well Plate (30mg/well)
8B-S014-EAK	Strata SDB-L Tubes (100mg/1mL)
8B-S014-FBJ	Strata SDB-L Tubes (200mg/3mL)
8B-S014-HBJ	Strata SDB-L Tubes (500mg/3mL)
8B-S014-HCH	Strata SDB-L Tubes (500mg/6mL)
8E-S014-DGB	Strata SDB-L 96-Well Plate (50mg/well)
AH0-6023	12-position SPE manifold
AH0-6024	24-position SPE manifold
7HG-G002-11	Zebron ZB-5 GC Column

Table 1. Average % recoveries of phenols from water

Compound	% Recovery using strata-X	% Recovery using SDB
Phenol	95	16
2,4,6-tribromophenol	101	79
2-fluorophenol	95	27
2-chlorophenol	97	83
2,4,6-trichlorophenol	102	93
2-nitrophenol	103	92
2,4-dinitrophenol	97	81
o-cresol	101	78

2136_1