

Solid Phase Extraction of Nitroanilines from Water

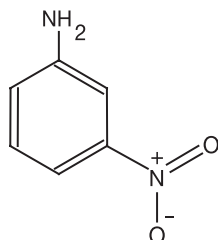
A comparison of strata™ X vs. styrene-divinylbenzene polymeric sorbents

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Nitroanilines are small polar organic molecules with a wide range of industrial applications. They serve as intermediates in numerous synthetic processes, including the production of dyes and plastics. Due to their toxicity, water and soil samples are routinely monitored to detect their presence as pollutants. The high solubility of the nitroanilines (small $\log k_w$ values)¹ makes the extraction from water a difficult challenge in environmental analyses.²

This application compares the solid phase extraction (SPE) of nitroanilines from water for two polymeric sorbents - strata-X and styrene-divinylbenzene (SDB). **Table 1** lists the physical and chemical properties of the polymeric sorbents. strata-X is a revolutionary, patent-pending polymeric SPE sorbent developed by Phenomenex. The functionalized styrene-divinylbenzene surface of strata-X has numerous retention mechanisms, including π - π and hydrophilic interactions that yield high recoveries for a wide range of small polar organic compounds from water such as nitroanilines. Thus, the aim of the study was to determine whether strata-X demonstrates higher recoveries when compared with unmodified SDB, which is typically used in extracting organic molecules from water.³

2-nitroaniline ($\log P = 1.85$)



4-nitroaniline ($\log P = 1.36$)

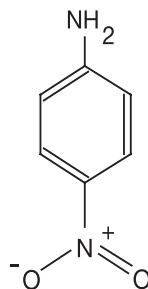


Table 1.
Physical and chemical properties of strata-X and SDB

	strata-X	SDB
Particle size (μm)	33	100
Pore size (\AA)	85	260
Surface area (m^2/g)	800	500

Instrumentation and Equipment

Solid phase extraction:

Strata SDB-L (500mg/6mL) and strata-X (500mg/6mL) 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 cartridge 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 0.25 μm). The data was analyzed with HP ChemStation software.

Experimental Conditions

Specimen preparation:

A 500mL water sample spiked with the target compounds (60ppm) was acidified with glacial acetic acid ($\text{pH} = 3.5$). This concentration level is consistent with practical quantification limits for wastewater as specified in EPA method 625.

SPE extraction method:

Same method used for both sorbents

Condition: The sorbent was conditioned with 5mL methanol, followed by 5mL of DI 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 of dichloromethane. All elution solvents were collected in a single test tube.

Analysis:

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

GC experimental conditions:

1 μL 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.



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Conclusion

Table 2 compares the recoveries of nitroanilines using strata-X vs. SDB. Styrene-divinylbenzene polymer was chosen as the benchmark as it was previously shown to have a high capacity for polar analytes.³ The percent recovery of 2-nitroaniline is >90% for SDB and strata-X. In the case of 4-nitroaniline (which is slightly more polar than 2-nitroaniline), the percent recovery is significantly improved when the functionalized polymer is used. This enhanced recovery is due to the larger surface area plus the unique hydrophilic-hydrophobic retention mechanism of strata-X. For both compounds, the recoveries were more consistent when strata-X was used. The recoveries ranged from 83-102% and 36-55% for 2-nitroaniline and 4-nitroaniline, respectively. Interestingly, the high recoveries using strata-X were obtained without adding salt to the extraction protocol, which is a common practice.²

While the concentration levels fall in the range for practical quantitation limits for wastewater, similar recoveries for lower concentration levels are expected. The versatility and selectivity of strata-X makes it a powerful tool in the simultaneous extraction and concentration of polar compounds from water that are problematic for conventional styrene-divinylbenzene polymer.

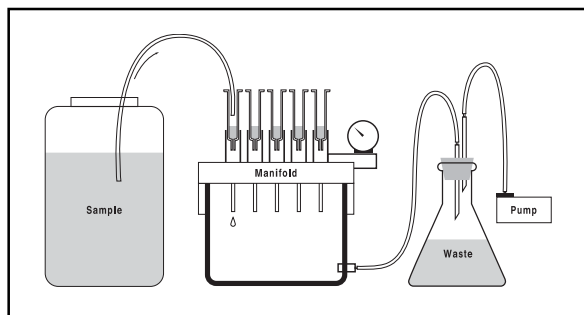
Reference

1. *Analytical Solid-Phase Extraction* by James S. Fritz, John Wiley & Sons, 1999, page 74.
2. *Solid-Phase Extraction Principles, Techniques and Applications* edited by Nigel Simpson, Marcel Dekker, Inc. 2000, pages 97-124.
3. *Solid Phase Extraction Principles and Practice* by Thurman and Mills, John Wiley & Sons, 1998, page 313.

Table 2. Average % recoveries of nitroanilines from water

compound	% recovery using strata-X	RSD	% recovery using SDB	RSD
2-nitroaniline	93	0.7	94	11.5
4-nitroaniline	94	1.4	42	10.4

Figure 1. Schematic of apparatus used in the extraction of nitroanilines from a large water sample.



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