

TN-0147

Robust Analysis of 1,4-Dioxane by Isotopic Dilution using Strata™ Activated Carbon Extraction in Comparison to Competitor Brand

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Introduction

EPA method 8270 is a popular testing method for semi-volatile organic compounds by GC-MS. The analyte list in this method is quite expansive and is often challenging to get good recovery for all classes of compounds in a single extraction. Here, a modified EPA 8270 method that employs activated carbon for the extraction of polar analytes such as 1,4-Dioxane by an isotopic dilution method is used. While dealing with polar small molecules like 1,4-Dioxane, traditional sample preparation using Solid Phase Extraction (SPE) might not be efficient. Since the analyte is polar, a specialized extraction technique is needed. Activated carbon contains porous carbon with defined pore volume and high surface area that are an excellent choice for polar analytes. In this technical note, we have extracted 1,4-Dioxane from water using Strata Activated Carbon cartridges. In addition, multiple batches of Strata Activated Carbon cartridges were evaluated to prove the consistency of the newly developed porous activated carbon material.

GC-MS Conditions

Column: Zebtron™ ZB-624PLUS™

Dimension: 30 meter x 0.25 mm x 1.40 μm

Part No.: [7HG-G040-27](#)

Injection: Splitless, 0.5min @ 175 °C, 1 μL

Recommended Liner: Zebtron PLUS Z-Liner™ (Compatible with Agilent® & Thermo Scientific® GC instrument)

Liner Part No.: [AG2-0A11-05](#)

Carrier Gas: Helium @ 1 ml/min (Constant Flow)

Oven Program	Ramp (°C/min)	Temp (°C)	Time (min)
-	-	35	1.0
12	12	100	0.0
25	25	230	0.0

Detector: MSD

SIM Ions (m/z): 58, 64, 88, 96

Transfer Line Temperature: 300 °C

Sample Preparation

Condition: Strata Activated Carbon, 2 g/6 mL cartridge (Part No. [CS0-9209](#)) or Enviro-Clean® 521 Activated Carbon, 2000 mg/6 mL with 2 washes of 10 mL Methylene Chloride, followed by 2 washes of 10 mL Methanol

Equilibrate: Cartridges with 2 washes of 10 mL Water

Load: 500ml Water sample spiked with internal standard onto cartridges

Dry: Cartridges for 10 min

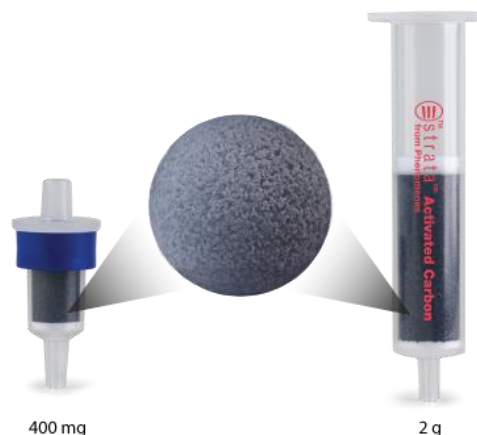
Elute: With 3 mL Methylene Chloride and repeat elution 2 more times (9 mL total volume)

Water Removal: Pass eluent through Sodium Sulfate Giga™ tubes, 5 g/20 mL (Part No. [8B-S124-LEG](#)) and wash with 5mL Methylene Chloride

Evaporate: Solvent to approximately 0.4 mL in a water bath near room temperature (45 °C) under a gentle stream of Nitrogen

Adjust: Final volume to 0.5 mL with Methylene Chloride

Figure 1. Strata Activated Carbon Extraction Cartridges



Ramkumar Dhandapani, PhD

Ramkumar has earned a PhD in Analytical Chemistry and has over 17 years troubleshooting and hands-on experience in chromatography. He loves to write poems, watch, and read Shakespeare's plays.



Results and Discussion

Figure 1 is a representation of the Strata™ Activated Carbon extraction cartridges. The porous carbon is optimized for reproducible extraction of polar analytes. Although the 2 g/6 mL format was employed in this application, there is also a 400 mg format available for smaller sample sizes.

EPA method 522 provides guidelines on determination of 1,4-Dioxane by SPE using activated carbon and recommends Tetrahydrofuran as the internal standard. Since Tetrahydrofuran is quite different in nature from 1,4-Dioxane in terms of structure and properties, the % recovery can vary for the analyte compared to the internal standard. To improve the % recovery from real sample matrices, isotopic dilution was adopted. This can be considered as a modified method for EPA method 8270. As presented in **Figure 2**, the deuterated internal standard provides the least amount of interference with matrix, thereby improving the accuracy of extraction.

Figure 3 is a representative chromatogram comparing the extraction of 1,4-Dioxane-d8 on Strata Activated Carbon cartridges and Enviro-Clean® 521 Activated Carbon cartridges. As evident from the chromatogram, the Strata Activated Carbon cartridge extraction provides higher peak response for the analyte of interest. Internal standard reproducibility and variability comparisons are presented in **Figures 4** and **5**, respectively. The Strata Activated Carbon cartridge extraction provides the least variability for seven internal standard extractions.

Water sample recovery and variability in results are presented in **Figures 6** and **7**. **Table 1** shows the comparative data demonstrating consistent recovery of 1,4-Dioxane with percent relative standard deviation (%RSD) as low as 0.27 % when the sample was extracted using the Strata Activated Carbon cartridge. The study was further extended to three different batches of Strata Activated Carbon cartridges to demonstrate the reproducibility of manufacturing as shown in **Figure 8**. The 3 batches demonstrated consistent recovery with %RSD as low as 0.56 % for 6 extractions. Such consistent results are possible because of the narrow range of pore size and surface area that provides least variability between batches.

Figure 2. Blank versus Internal Standard Overlay for 1,4-Dioxane-d8 Extraction from Water using the Strata Activated Carbon Cartridge

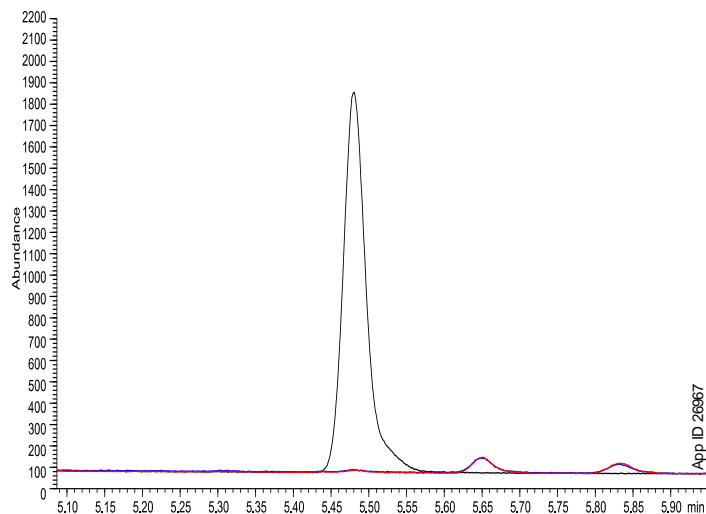


Figure 3. Comparison of 1,4-Dioxane-d8 Extraction from a Water Sample

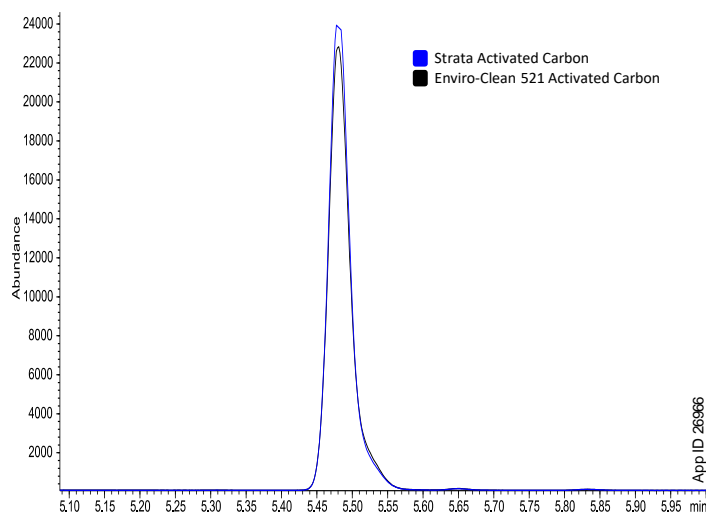


Figure 4. Comparison of Internal Standard 1,4-Dioxane-d8 Reproducibility

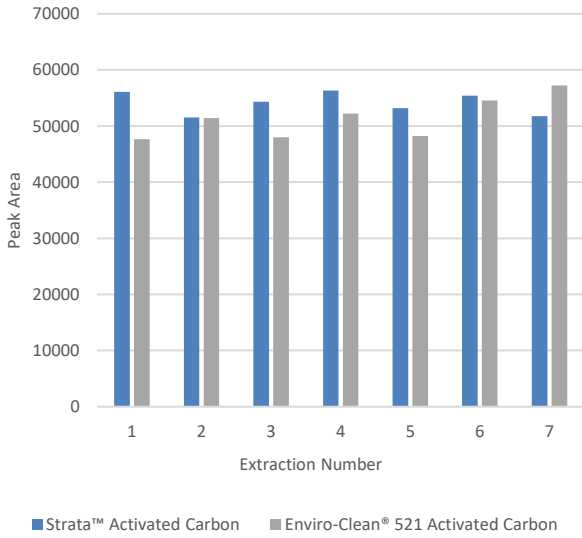


Figure 5. Comparison of Internal Standard 1,4-Dioxane-d8 Variability

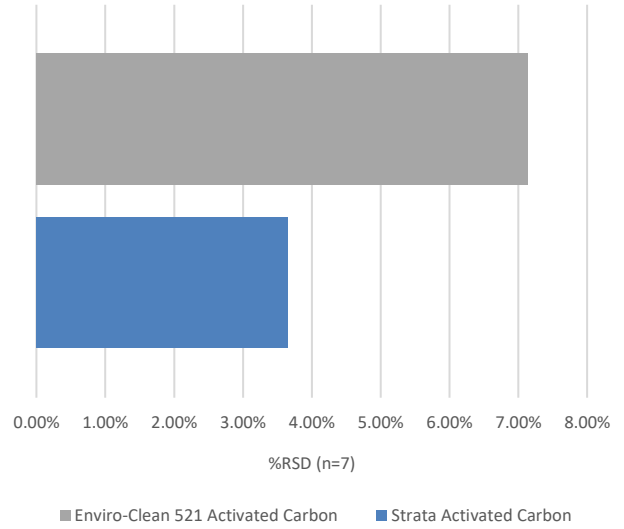


Figure 6. Comparison of 1,4-Dioxane % Recovery from a Water Sample

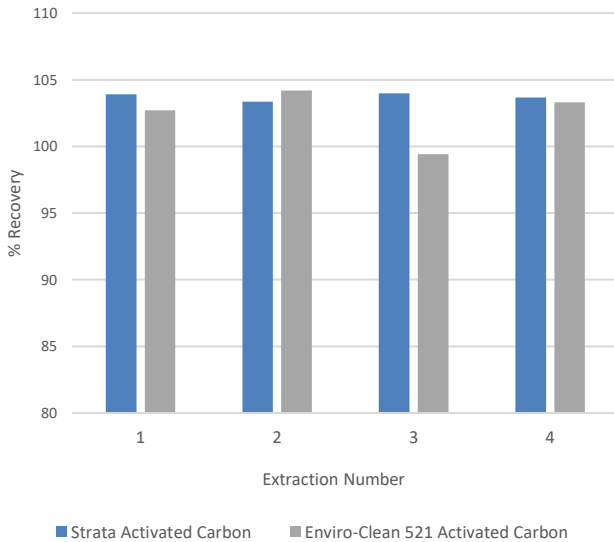


Figure 7. Comparison of 1,4-Dioxane Recovery Variability from a Water Sample

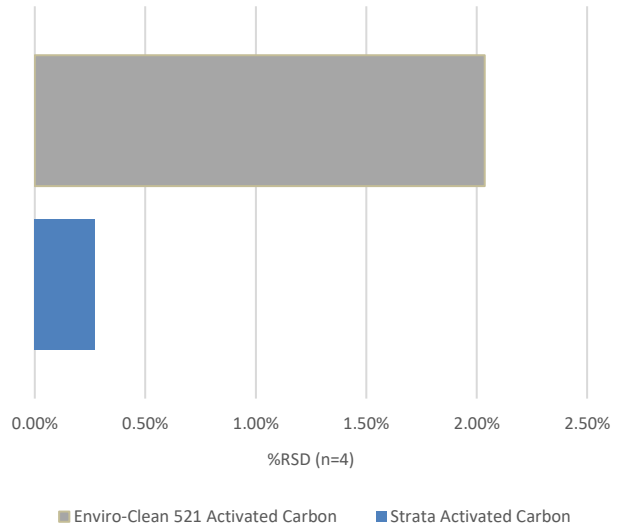
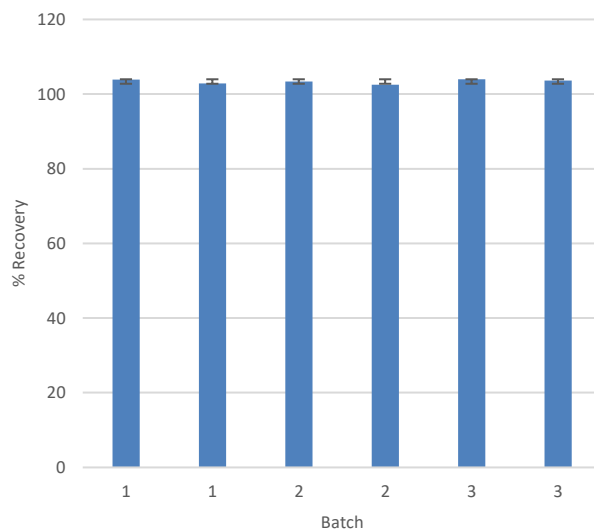


Table 1. Comparison of % Recovery of 1,4-Dioxane from a Water Sample

Extraction	% Recovery Strata™ Activated Carbon	% Recovery Enviro-Clean® 521 Activated Carbon
1	103.90	102.70
2	103.36	104.19
3	103.99	99.41
4	103.66	103.30
Average	103.73	102.40
Standard Deviation	0.28	2.09
%RSD	0.27%	2.04%

Figure 8. Batch to Batch Reproducibility on Strata Activated Carbon



Conclusions

Strata Activated Carbon provided reproducible extraction of 1,4-Dioxane from water samples. Results demonstrated low variability from across batches.



Strata™ Tubes and Cartridges Ordering Information

Strata Activated Carbon				
Part	Sorbent Mass	Volume	Format	Units/pk
CS0-9209	2 g	6 mL	Tube	30
CS0-9210	400 mg	Pass through	Cartridge	50

Zebtron™ ZB-624PLUS™ GC Columns Ordering Information

3 µm Analytical Columns (mm)			
ID(mm)	df(µm)	Temp. Limits °	Part No.
0.18	1	-20 to 300/320	7FD-G040-22
0.25	1.4	-20 to 300/320	7FG-G040-27
30-Meter			
0.25	1.4	-20 to 300/320	7HG-G040-27
0.32	1.8	-20 to 300/320	7HM-G040-31
0.53	3	-20 to 300/320	7HK-G040-36
60-Meter			
0.25	1.4	-20 to 300/320	7KG-G040-27
0.32	1.8	-20 to 300/320	7KM-G040-31
0.53	3	-20 to 300/320	7KK-G040-36
75-Meter			
0.53	3	-20 to 300/320	7LK-G040-36

Liners Compatible with Thermo Scientific® GC Systems Ordering Information

Zebtron PLUS Liners for For 5890, 6890 and 7890 Models						
Description	Application	Inlet Style	Dimensions	Deactivation	Part No.	Unit
Direct Connect	Trace analysis, Splitless injections	S/SL	4 x 78.5	PLUS Inert	AG2-0A50-01 AG2-0A50-05 AG2-0A50-25	ea 5/pk 25/pk
Single Taper	Pesticides	S/SL	4 x 78.5	PLUS Inert	AG2-0A10-01 AG2-0A10-05 AG2-0A10-25	ea 5/pk 25/pk
Single Taper Z-Liner™	Semi-volatiles, Dirty samples	S/SL	4 x 78.5	PLUS Inert	AG2-0A13-01 AG2-0A13-05 AG2-0A13-25	ea 5/pk 25/pk
Single Taper with Wool	Semi-volatiles	S/SL	4 x 78.5	PLUS Inert	AG2-0A11-01 AG2-0A11-05 AG2-0A11-25	ea 5/pk 25/pk
Straight	Volatiles	S/SL	4 x 78.5	PLUS Inert	AG2-0A00-01 AG2-0A00-05 AG2-0A00-25	ea 5/pk 25/pk
Straight Z-Liner	Dirty samples, Volatiles, High initial oven temperatures	S/SL	4 x 78.5	PLUS Inert	AG2-0A03-01 AG2-0A03-05 AG2-0A03-25	ea 5/pk 25/pk
Straight Single Baffle	Semi-volatiles, Pesticides	PTV	1.8 x 71	PLUS Inert	AG2-1F06-01 AG2-1F06-05 AG2-1F06-25	ea 5/pk 25/pk

Sodium Sulfate Tubes Ordering Information

Strata			
Format	Sorbent Mass	Part Number	Unit
Tube	1 g	8B-S124-JCH	6 mL (30/box)
Giga™ Tube	5 g	8B-S124-LEG	20 mL (20/box)



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