EPA 522; Analysis of 1,4-Dioxane In Drinking Water by Solid Phase Extraction (SPE)

Introduction
EPA 522 defines a laboratory protocol for the extraction for analysis of 1,4-Dioxane (CASRN 123-91-1) in drinking water. The method uses solid phase extraction paired with GC/MS analysis. The usage of selective ion monitoring (SIM) is often utilized to achieve minimum detection levels.

Due to the relatively high volatility of 1,4-Dioxane, the use of a 2 gram coconut charcoal cartridge is required for the retention of 1,4-dioxane from aqueous samples. Cartridges are then eluted with a small portion of methylene chloride, and evaporated to 1 mL. The procedure requires a slow sample loading process where low rates and times must be precise and consistent. By implementing an automated SPE system like the TurboTrace™ System, samples can be extracted in a fully automated, precise, process that frees up extraction chemists to perform other tasks.

Instrumentation and Consumables

Instrumentation
• FMS, Inc. TurboTrace (Solid Phase Extraction) System
• FMS, Inc. SuperVap™ 12-Position Concentrator
• FMS, Inc. direct-to-vial concentrator tubes
• Thermo Trace Ultra GC with SSL Injection Port
• Thermo DSQ Mass Spectrometer

Consumables
• Restek 2g Coconut Charcoal column (Cat# 26032)
• Restek 1,4-dioxane-d8 Standard (Cat# 30614)
• Restek 1,4-dioxane Standard (Cat# 30287)
• Restek Tetrahydrofuran-D8 Standard (Cat# 30112)
• Fisher Optima Grade Methanol
• Fisher Optima Grade Methylene Chloride
• Fisher HPLC Grade Water
• Fisher Anhydrous Sodium Sulfate

Procedure

TurboTrace SPE system
1. Cartridges conditioned with 10 mL MeOH
2. Cartridges conditioned with 10 mL H2O
3. Samples loaded across cartridges via vacuum (5-10 mL/min)
4. Cartridges dried under nitrogen for 5 minutes.
5. Sample bottle sprayed with 20 mL methylene chloride.
6. 20 mL methylene chloride spray loaded across cartridge and collected.
7. Cartridges eluted with 5 mL methylene chloride
8. Eluted solvent nitrogen purged into collection vials.

**Removal of residual water from extract by sulfate filtration.

SuperVap Concentrator
1. Preheat temp: 20 minutes at 35 °C
2. Evap mode w/Sensor temp: 35 °C
3. Nitrogen Pressure: 5 PSI

Figure 1: Chemical structure of 1,4-dioxane
Results

Table 1: Results for .5 liter replicates spiked at 1 µg/L

<table>
<thead>
<tr>
<th>Replicate</th>
<th>Recovery</th>
<th>1,4-dioxane recovered %</th>
<th>1,4-dioxane conc. (µg/L)</th>
<th>STD DEV</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>89.59%</td>
<td>1.056</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>84.59%</td>
<td>0.962</td>
<td></td>
<td>0.05</td>
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<tr>
<td>3</td>
<td>82.15%</td>
<td>1.03</td>
<td></td>
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</tr>
</tbody>
</table>

Conclusions

Extraction method performance of the FMS TurboTrace system and SuperVap Concentrator resulted in all replicates yielding 1,4-dioxane-d8 recoveries within the 70-130 percent recovery window. Native 1,4-dioxane calculated concentration showed excellent precision and accuracy. The usage of independent sample loading sensors to stop sample loading of individual runs was a critical component to ensuring that no analyte was lost from over drying the cartridges.

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