Determination of Small Polar Organic Compounds in Drinking Water by Solid Phase Extraction with Activated Carbon Sorbent

UCT Part Numbers:
EU541163 - 600 mg activated carbon in 3 mL cartridges
AD0000AS - Cartridge adaptors
RFV0075P - 75 mL empty reservoirs
VMF024GL – 24-position glass block vacuum manifold
VMF02024 - Stopcocks
ECSS25K - 25 kg sodium sulfate, anhydrous, ACS grade, granular 60 mesh
GCLGN4MM – Agilent style - 4 mm splitless gooseneck GC liner

Summary:

This application describes a solid phase extraction (SPE) method for the determination of EPA method 541 analytes, including 1-butanol, 1,4-dioxane, 2-methoxyethanol and 2-propen-1-ol in finished drinking water. Among these 4 target analytes, 3 are part of the new UCMR4 (Unregulated Contaminant Monitoring Rule 4) compounds. The fourth compound is 1,4-dioxane, a UCMR3 (Unregulated Contaminant Monitoring Rule 3) compound currently being monitored by EPA method 522. The target analytes and surrogates are extracted from 50 mL preserved water samples onto an activated carbon sorbent packed in 3 mL SPE cartridges. The cartridges are dried and eluted with 5% methanol (MeOH, purge and trap grade) in dichloromethane (DCM, pesticide grade). The water residue in the eluate is removed by anhydrous sodium sulfate (Na_2SO_4) and the extract is injected into a GC/MS for analysis without further concentration due to the high volatilities of the analytes. The GC/MS is equipped with a 30 meter WAX column and operated in selected ion monitoring (SIM) mode for analyte separation and detection. Excellent recoveries and relative standard deviations (RSD) were obtained in reagent and tap water samples spiked with known amount of analytes.

Sample Preservation:

2.5 mg of sodium sulfite and 50 mg of sodium bisulfate are added to each empty sample bottle prior to shipment to the field.

SPE Procedure:

1. **SPE Setup**
   a. Connect the 75-mL reservoirs (**RFV0075P**) to the top of the SPE cartridges (**EU541163**) using the cartridge adaptors (**AD0000AS**).
   b. Attach the connected cartridges onto a 24-position glass block vacuum manifold (**VMF024GL**) with control stopcocks (**VMF02024**).

2. **Cartridge Conditioning**
   a. Rinse the SPE cartridges with 5 mL of 5% MeOH in DCM, pass 1/3 through to wet the sorbent. Soak the sorbent for 1 min then draw the remaining solvent to waste. Apply full vacuum and leave on for 1 min.
   b. Repeat Step 2a with 2 mL of MeOH.
   c. Condition the SPE cartridges with 2 mL of MeOH. Allow the MeOH to soak the sorbent for 1 min; then draw to waste. Do not allow the sorbent go completely dry.
   d. Equilibrate the cartridges with 5 mL of DI water. Draw about half of the water through the cartridge.

3. **Sample Extraction**
   a. Add 50 mL water samples into the 75-mL reservoirs, adjust the stopcocks for a slow dropwise flow (about 5 mL/min), and draw the entire sample through.
   b. Rinse the sample bottles with 5 mL of DI water and apply the rinses to the reservoirs. After the rinses pass completely through the cartridges, aspirate for 30 sec, and turn off the stopcocks.
   c. Remove the reservoirs and cartridge adaptors from the SPE cartridges.

4. **Cartridge Drying**
   a. Add 200 µL of MeOH to the SPE cartridges, rinsing the water droplets from the walls of the SPE cartridges, and aspirate at full vacuum for 30 sec.
   b. Transfer the SPE cartridges to a drying manifold. Dry the cartridges with inert nitrogen at 5 L/min for 10 min.

5. **Cartridge Elution**

a. Rinse the stopcocks and tips of the manifold with 2 mL of MeOH to remove any trapped residual water; apply full vacuum for 1 min.

b. Insert a collection rack with small test tubes into the manifold underneath each SPE cartridge.

c. Place the dried SPE cartridges back onto the manifold with stopcocks. Elute the SPE cartridges with 2 x 1.3 mL of 5% MeOH in DCM. Let the elution solvent soak the SPE sorbent for 1 min, then elute by gravity, finally apply a low vacuum to extract all of the elution solvent from the SPE cartridges into the collection tubes. This will yield approximately 2 mL of eluate.

6. **Eluate Drying**

a. Add 10 µL of the 50 ppm internal standard solution to each extract and 1-2 grams of anhydrous sodium sulfate (**ECSS25K**), vortex for 1 min using a multi-tube vortexer.

b. Let the salt and extract remain in contact for 15 min before transferring the dried extract to a 2 mL auto-sampler vial. Adjust the final volume to 2 mL using 5% MeOH in DCM.

c. The samples are ready for GC/MS analysis.

GC/MS Conditions

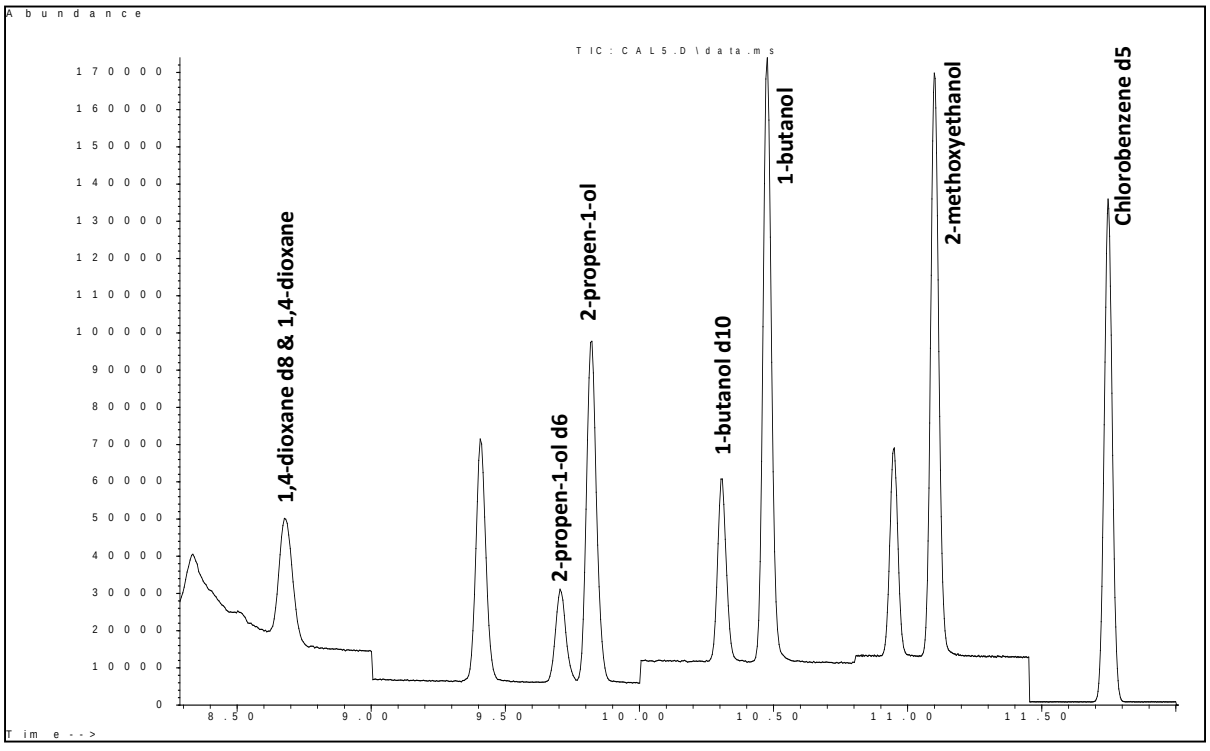
Parameter	Conditions
Column	Phenomenex ZB-WAX *plus*: 30m x 0.25mm x 0.5µm
Inlet liner (**GCLGN4MM**)	4mm, single gooseneck, with deactivated glass wool
Injection	1 µL at 200 °C, splitless injection, purge flow of 50 mL/min at 0.5 min
Carrier gas and flow rate	Ultra high purity Helium at 0.9 mL/min (constant flow)
GC temperature program	35 °C for 5 min, ramp 10 °C/min to 105 °C, ramp 30 °C/min to 240 °C, hold 3.5 min
Solvent delay	8.2 min (MS filament on from 8.2 to 12 min)

MS source temperature	250 °C
MS quadrupole temperature	150 °C
GC/MS interface	240 °C

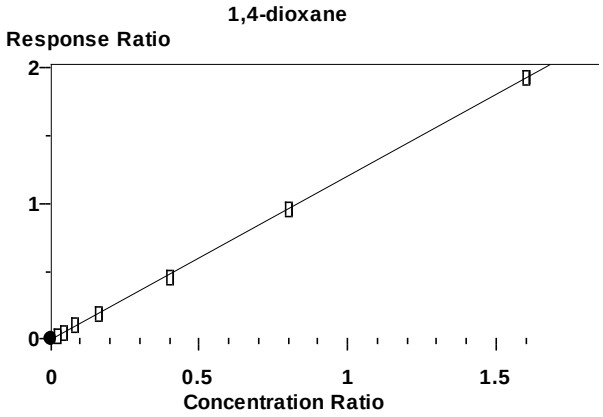
Accuracy and Precision Data

Compound	Fortified reagent water		Fortified tap water	
	Recovery%	RSD% (n=5)	Recovery%	RSD% (n=5)
1,4-dioxane	**89.5**	5.2	**87.7**	1.9
2-propen-1-ol d6	**92.9**	4.6	**82.5**	4.8
2-propen-1-ol	**85.5**	3.4	**84.1**	2.8
1-butanol d10	**101.0**	3.6	**88.0**	5.3
1-butanol	**96.4**	3.3	**88.4**	2.1
2-methoxyethanol	**97.6**	4.0	**92.9**	2.6

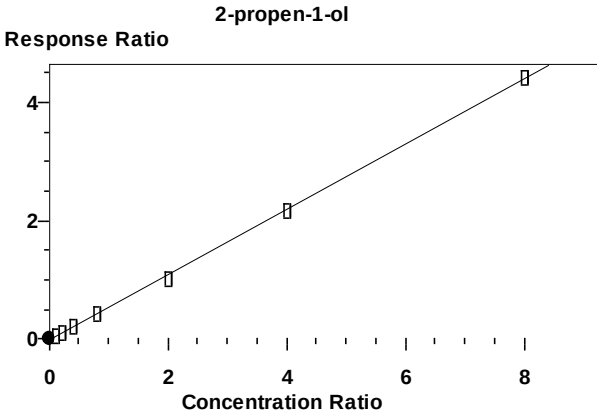
Chromatogram



Calibration Curves



$R^2 = 0.9998$



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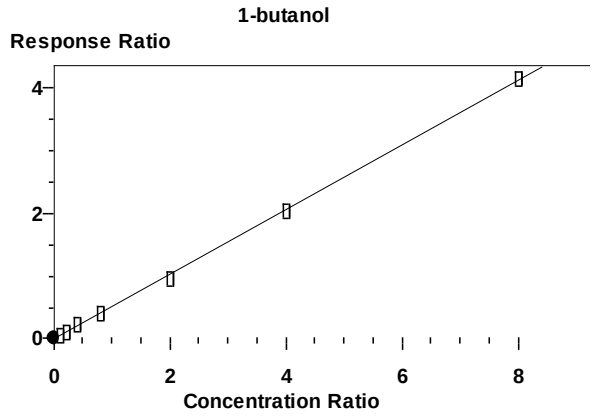

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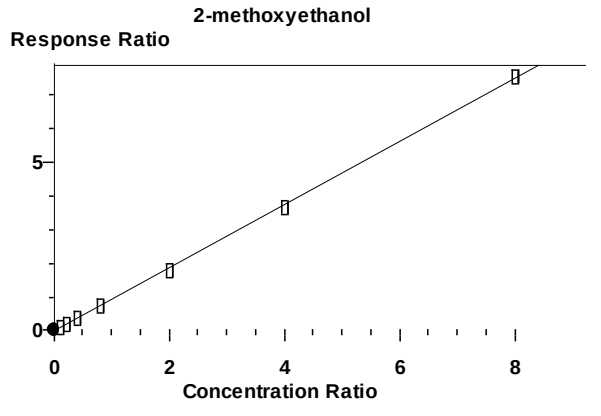

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