

Analysis of 1,4-Dioxane in Drinking Water with Semi-Automated Solid Phase Extraction (SPE) Using EPA Method 522

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Introduction

EPA 522 defines a laboratory protocol for the extraction for analysis of 1,4-Dioxane in drinking water. The method uses Solid Phase Extraction and low resolution GC/MS. Due to the volatility of 1,4-Dioxane, use of a 2 g 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.

To meet demands for a low cost method that requires less financial investment than automated systems, FMS developed a simple semi - automated system which is fast, inexpensive and yields high quality data.

Instrumentation

- FMS EZSpe® System
- FMS SuperVap®
- Vacuum pump
- Thermo DSQ Single Quad GC/MS

Consumables

- FMS, Inc. 2 g Coconut charcoal cartridge
- FMS sodium sulfate column
- Ultra pure DI water
- Fisher Sodium Bisulfate
- Fisher Pesticide Grade Methanol
- Fisher Pesticide Grade Dichloromethane
- 1,4-Dioxane Standard Solution
- 1,4-Dioxane-d8 Standard Solution
- Tetra Hydrofuran-d8 Standard Solution

Sample/Reagent Prep

Procedure

- 6 samples (100 or 500 mL water each) are prepared and acidified with sodium bisulfate till pH < 4
- Spiked with relevant Standards
- Put sample bottles in place and fill automated rinse bottles with 9 mL dichloromethane
- Cartridges are installed in each of the six positions.

Stage 1:

- Vacuum is turned on
- Cartridges are conditioned with: 3 mL dichloromethane, 3 mL methanol, 3 mL methanol (keep wet) and 6 x 3 mL water (keep wet)
- Samples are loaded across cartridges under vacuum at 10 mL/min
- Cartridges are dried under vacuum for 10 min
- Sample bottles are automatically rinsed from the rinse bottles with 9 mL dichloromethane

Stage 2:

- Small amount of dichloromethane is pulled through cartridges, soak 1 min
- Rest of dichloromethane rinses from sample bottles are loaded across the coconut charcoal cartridges and sodium sulfate cartridges and the eluent is collected for analysis into Direct to GC Vial Collection Vessels

Analysis

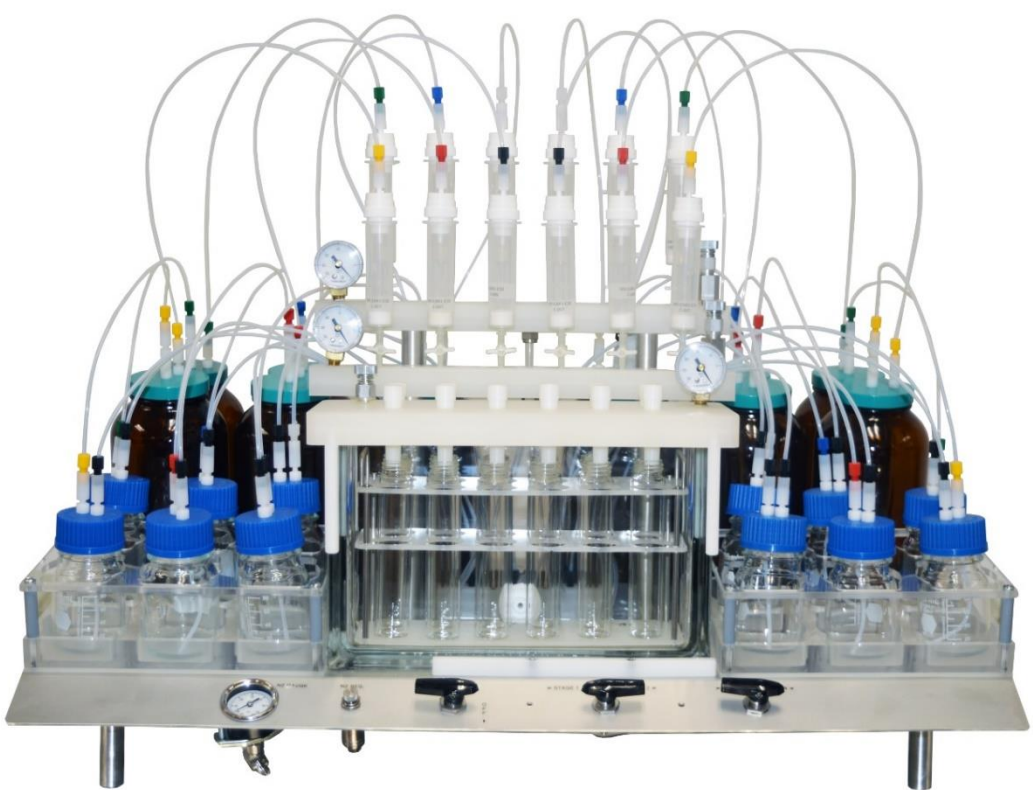
- Analyze aliquot of sample on GC/MS in SIM mode

Results

Table 1 with recoveries spiked at 1 ng/L

1,4-dioxane-d8 recoveries

run # 1	87%
run # 2	84%
run # 3	81%



FMS EZSpe system

Discussion and Conclusion

The results of the samples demonstrate the ability of the FMS EZSpe system to deliver accurate and reliable results. Averages are well within the 80-120% acceptance window. The semi-automated EZSpe is superior to traditional, time-consuming, inconsistent and expensive liquid/liquid extractions.



For additional information please contact:

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Sample
Extraction

Sample
Clean Up

Sample
Concentration