Environmentally Friendly Implementation of EPA 8270E by Solid-Phase Extraction and Hydrogen Carrier Gas GC/MS Analysis

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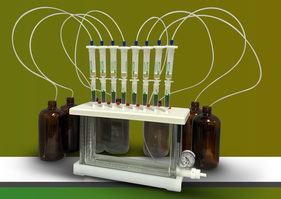

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INTRODUCTION

EPA Method 8270E is a standardized protocol for analyzing semivolatile organic analytes (SVOCs) in various matrices, including water, using gas chromatography/mass spectrometry (GC/MS). Using UCT's proprietary 8270 cartridge blend for solid phase extraction (SPE), this method effectively isolates acidic, basic, and neutral analytes, offering analytical coverage of a wide range of analytes in an aqueous matrix. The method's use of activated carbon cartridges improves the retention of highly polar analytes like 1,4-dioxane. In the wake of the recent restrictions on methylene chloride usage, reducing solvent usage by using SPE is paramount, while adopting hydrogen as a carrier gas provides a sustainable and economical alternative to helium. This alternative procedure achieves recoveries using SPE that are similar to traditional liquid-liquid extraction procedures, with 98% of analytes meeting EPA's recommended 50-150% recovery criteria.

References:

[1] https://www.epa.gov/sites/default/files/2015-12/documents/8000d.pdf
[2] https://www.epa.gov/sites/default/files/2020-10/documents/method_8270e_update_vi_06-2018_0.pdf
[3] https://www.epa.gov/sites/default/files/2015-12/documents/3535a.pdf

SPE PROCEDURE

Pre-Sample Preparation

a) Adjust a 1L sample to pH 2 using 6N H_2SO_4

b) Check for free chlorine and remove with sodium thiosulfate if necessary

c) Fortify with analytes and surrogates

SPE System Setup

a) Connect the carbon cartridge below the 8270 cartridge with an adaptor.

b) Add glass wool to prevent clogging

c) Attach the sample delivery tube to the EC8270 cartridge system.

d) Connect SPE cartridges to the manifold

Cartridge Condition

a) Cartridge rinsing: 15 mL DCM

b) Cartridge conditioning: 15 mL MeOH

c) Cartridge equilibration: 15 mL 0.1N HCl

Sample Processing

a) Load 1L pH 2 sample at 10-15 mL/min

b) Rinse bottle with 10 mL DI water and pass through cartridge system

c) Detach carbon cartridge from 8270 cartridge and dry separately (8270 for 10 min, carbon for 15 min)

Sample Elution

a) 8270 Cartridge: Vial A: 10 mL 9:1 hexane/acetone

b) 8270 Cartridge: Vial B: 10 mL (18:2:80 IPA/NH_4OH/DCM), followed by 15 mL DCM.

c) Carbon Cartridge: Vial C: 15mL DCM

d) Dry eluates by passing through sodium sulfate

Concentration

a) Concentrate to 8-10 mL at 40-45 °C using SPeVAP, or K-D apparatus.

b) Continue to concentrate to 0.6-0.8 mL at 35 °C with nitrogen

c) Rinse tube sides & and adjust to 1 mL with DCM.

d) Transfer to GC vial & add internal standard.

GC/MS PARAMETERS

Instrument	Agilent 6890/5975C
Column	Rxi-5Sil MS, 30m x 250 µm x 0.25 µm
Inlet Temperature	280 °C
Injection Volume	1 µL; Split 10:1
Liner	Restek Topaz Splitless, 4mm
Oven Program	40°C (1 min) to 280 °C at 20 °C/min, 300 °C at 5 °C/min
Carrier Gas	Hydrogen at 1.2 mL/min constant flow rate
MS Parameters	Source at 230 °C; Quad at 150 °C; Transfer line at 280 °C; Electron energy; at 70 eV
Detector	MSD Quadrupole, full scan mode (EI), 50-500 amu
Tune File	DFTPP.U

CHROMATOGRAPHY

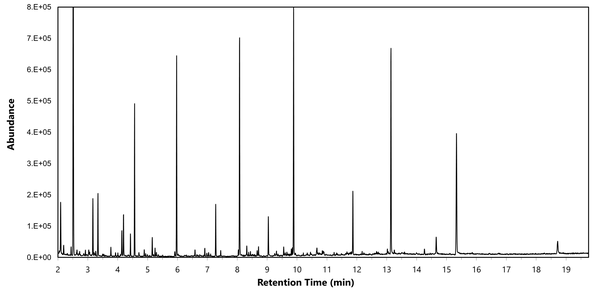


Figure 1. Total Ion Chromatogram (TIC) of a method blank with surrogates at 10 and 20 µg/L and internal standard at 40 µg/L.

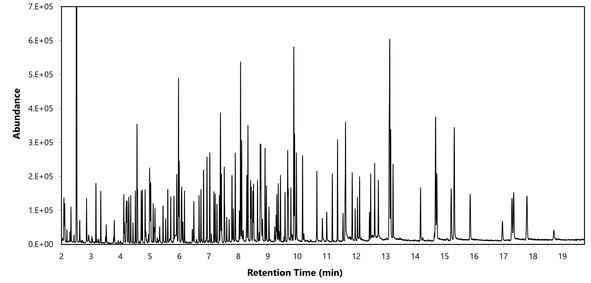


Figure 2. Total Ion Chromatogram (TIC) of the 1L fortified sample at 20 µg/L (with internal standards at 40 µg/L). (133 Compounds)

DISCUSSION & CONCLUSION

Initial demonstration of proficiency (IDP) was performed by fortifying 4 replicates of reagent water samples (133 analytes) at different concentration levels (1 µg/L, 5 µg/L, and 20 µg/L). The IDP data shown represents the 20 µg/L study (**Figure 3**). At this concentration, recovery values for 131 analytes ranged from 56% to 121%, with RSD values ≤20%. The analytes 1,4-phenylenediamine (34%) and hexachloropropene (41%) did not meet acceptable recovery criteria. These analytes are either known to pose challenges in extraction methods or lack historical data for aqueous extraction techniques.

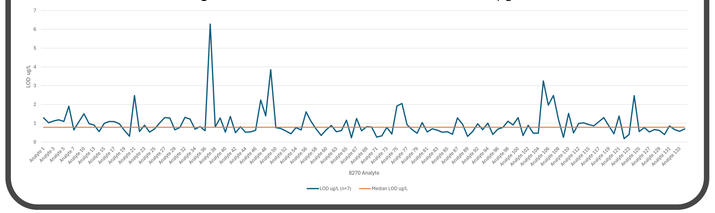
Additional low-level concentration extractions were performed over three days to generate seven replicate data points per analyte. Using statistical analysis, these data were used to calculate the limit of detection (LOD) for each analyte. The LOD data shown represents the 5 µg/L study (**Figure 4**). At this concentration, LODs ranged from 0.25 µg/L to 6.28 µg/L, with a median LOD of 0.78 µg/L.

According to EPA Method 8270E, recovery values between 50–150% with ≤20% RSD are considered acceptable to validate the extraction method; laboratories are encouraged to establish in-house recovery limits based on sufficient ongoing data, as outlined in Method 8000D Section 9.6. This study demonstrates that the SPE method using EC8270 and EU521 cartridges, in conjunction with analysis by GC/MS with hydrogen carrier gas, provides accurate and precise recovery of a wide range of acidic, basic, and neutral semi volatile analytes. The results met these criteria across multiple concentration levels for 131 out of 133 compounds.

This approach offers a reliable alternative to traditional aqueous extraction methods, with added benefits of increased sample throughput, reduced solvent usage, and lower analyst exposure to hazardous chemicals, enhancing both laboratory efficiency and environmental safety. Furthermore, using hydrogen as the carrier gas in GC/MS analysis reduces operational costs and offers a more environmentally sustainable alternative to traditional helium-based methods.

RESULTS

Figure 4. Calculated Limit Of Detection (LOD) in µg/L



RESULTS

Figure 3. Accuracy & Precision Study (IDP) at 20 µg/L