

Analysis of Carbonyl Compounds in Drinking Water Using EPA Method 554 with Semi-Automated Solid Phase Extraction (EZSpe®)

Introduction

Low-weight carbonyl compounds are ubiquitous in various fine chemicals, pharmaceuticals and consumer products. Due to their high solubility they frequently leak into water sources, and their buildup is confirmed to pose severe risks to human health, especially formaldehyde.

EPA Method 554 outlines the procedure for the extraction and analysis of various carbonyl compounds in drinking water. The extraction method outlines the use of solid phase extraction for water matrix samples employing both cartridges and disks. Consistent with other EPA 500 series methods, EPA 554 incorporates a rigid set of QC and acceptance criteria requiring precise and reproducible analytical practices. The potential for error and the variability associated with manual extractions makes the benefits of semi-automating these processes apparent.

To meet demands for a low cost method that requires less financial investment than fully 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
- Waters Alliance 2695 HPLC, UV254

Consumables

- FMS, Inc. 500 mg C-18 cartridge
- FMS sodium sulfate cartridge
- Ultra pure DI water
- Fisher 6 N Hydrochloric Acid
- 2,4-dinitrophenyl hydrazine (DNPH) (70% in water)
- Fisher ACS-Grade Absolute Ethanol
- Sodium citrate buffer
- Restek 554 spiking standards

Procedure

- 6 samples (100 mL water each) are prepared and acidified with HCl till pH ~ 3
- Spike with various 554 standards
- Derivatize at 40 °C with DNPH- 1 hour
- Put sample bottles in place and fill rinse bottles with 9 mL ethanol
- Cartridges are installed in each of the six positions.

Stage 1:

- Vacuum is turned on
- Cartridges are conditioned with 10 mL dilute sodium citrate (1M in 250 mL),
- Samples are loaded across cartridges under vacuum, at a rate of 3-5 mL/min
- Cartridges are dried under vacuum for 10 min (no nitrogen)
- Sample bottles are automatically rinsed from the rinse bottles with 9 mL ethanol

Stage 2:

- Ethanol from sample bottles is loaded across the cartridges (9 mL) and the eluent is diluted to 10 mL, across the cartridges and collected for analysis.

FMS SuperVap®

- Pre-heat temp: 45 °C
- Pre-heat time: 15 minutes
- Heat in Sensor mode at 45 °C under nitrogen (7-10 psi)
- Direct to GC Vial Vessel Reduce to 1 mL
- Samples are now ready for analysis

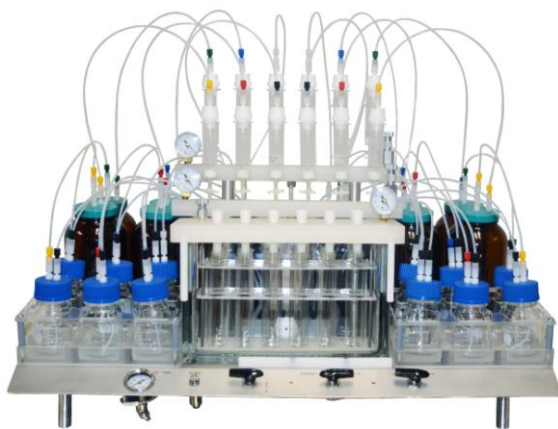


Table 1 with recoveries for the 554 target compounds

Compound	%Recovery	Stdev
Formaldehyde	72.4	1.77
Acetaldehyde	76.8	2.69
Propanal	94.2	1.96
Butanal	88.9	1.24
Pentanal	97.3	0.96
Hexanal	85.5	2.17
Heptanal	100.2	3.75
Octanal	92.2	0.98
Nonanal	96.4	1.44
Decanal	103.9	2.36
Cyclohexanone	90.1	3.81
Crotonaldehyde	100.6	4.22

Conclusions

Reviewing the sample data shows high recoveries for a dozen spiked analytes, demonstrating excellent efficiency for these compounds. Samples can be taken from collection bottle to LC vial in one quick, consistent, reproducible process that will save laboratories both time and money.



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 system