Detecting Haloacetic Acids using Chromatography

Overview

Haloacetic Acids, or HAA, is a term describing a collection of halogenated compounds that are commonly found in drinking water, albeit in small amounts. Thus the problem is to detect compounds at low concentrations (ng/L) and achieve separation of multiple components that are structurally similar. Our goal was to adapt methods using gas chromatography (GC) and liquid chromatography (HPLC) to separate the mixture, and mass spectroscopy using EI-ionization at the GC-MS and ESI-ionization at the HPLC with a goal to detect concentrations as high as a few micrograms per liter.

Methods

GC-MS
- 6890 GC with HP—5MS column, 30m x 0.25 mm dia.
- 5973 series MSD
- Relies on volatile solvents, such as DCM, that can easily be transferred in the gas phase through a column
- EI-Ionization: energized electrons collide with compounds to cause fragmentation and ionization

LC-MS
- C-18 LC Column, 100 mm x 2.1 mm
- Relies on liquid, protic solvent to be fed through a column at high pressure to get a signal
- ES-Ionization: a high voltage is applied to a liquid creating an aerosol that is injected. Little fragmentation, forms a molecular ion which is detected

Results

We determined an elution series based on a full scan analysis of stock HAA sample diluted 1:40 with DCM. Using this data, we developed a SIM, or single ion monitoring, method and ions to use for each compound. The ions we monitored for TCAA during El-ionization were m/z 82 and 84. During ESI-ionization it was m/z 141.

Using our LC-MS data, we created a calibration curve according to the mass value 141.

Difficulties

- Possible reaction occurring with TCAA in acetonitrile
- Difficult to pipet with DCM and diethyl ether
- Inconsistent reporting of retention series
- Failed/ difficult derivatization[1]
- Poor details in databases and journals
- Incomplete databases

Acknowledgements

David Cook, Alabama Space Grant Consortium, UAH Office of the Provost, UAH Office of the Vice President for Research and Economic Development, Dean of College of Science and Dr. Tingting Wu for supplying the HAA mixture.

References