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[2] 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 EI-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

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References