Applications of Gas Chromatography with Headspace Autosampler

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Introduction

Gas chromatography (GC) is a separation technique for volatile compounds that involves the vaporization of a sample through an injector. Then, the sample is carried by a gas moving through a stationary phase (column) packed with a gel or a solid until reaching a detector. The CME Department recently acquired a GC system with exchangeable detectors: Thermal conductivity detector (TCD) and flame ionization detector (FID). *To enhance interdisciplinary collaboration across Units and Departments within the University of Dayton, this work seeks to develop GC and Headspace-GC (HS-GC) analytical methods for use both in the classroom and in research.*

Objectives

- To understand the variables manipulated in GC and HS-GC and apply this knowledge to developing characterization methods
- To generate a characterization method to determine the concentration of *ethanol in aqueous mixtures obtained from a distillation process*

Methods

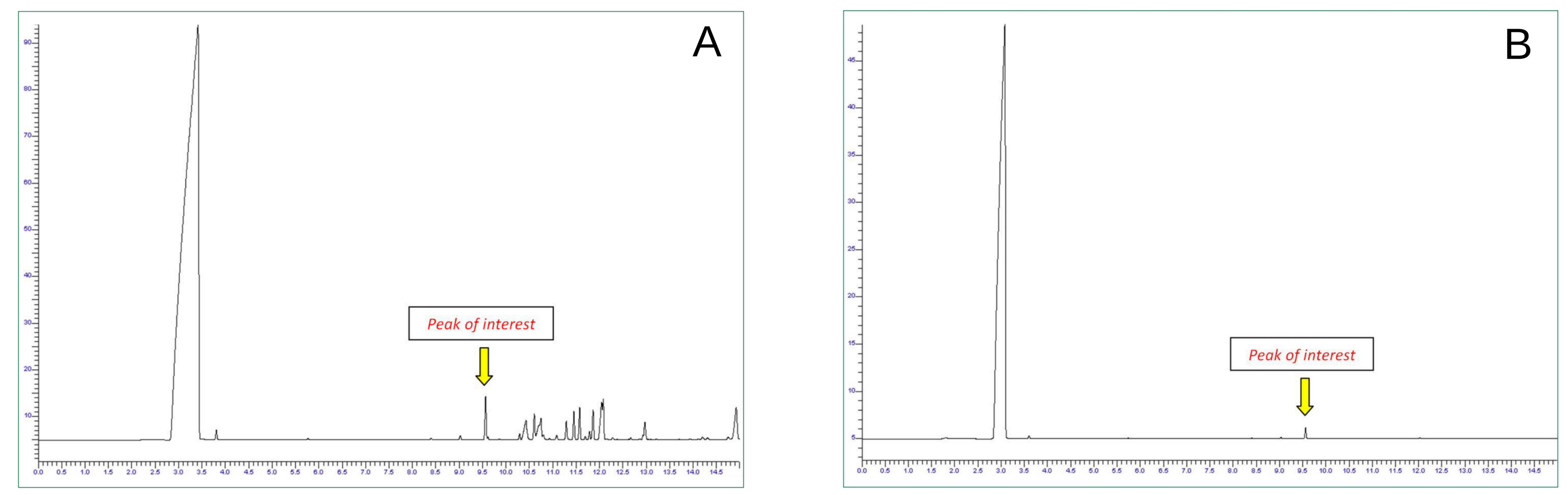
An extensive literature review has been conducted through Journal articles, books, and webinars before starting with the Summer Undergraduate Research Experience(SURE) program with the SoE this summer.

Equipment

- Thermo Scientific GC w/TCD and FID detector
- Autosampler (Triplus RSH)
- Capillary Column [TG-BOND Q+]
- HS and GC vials

Background: GC vs. HS-GC

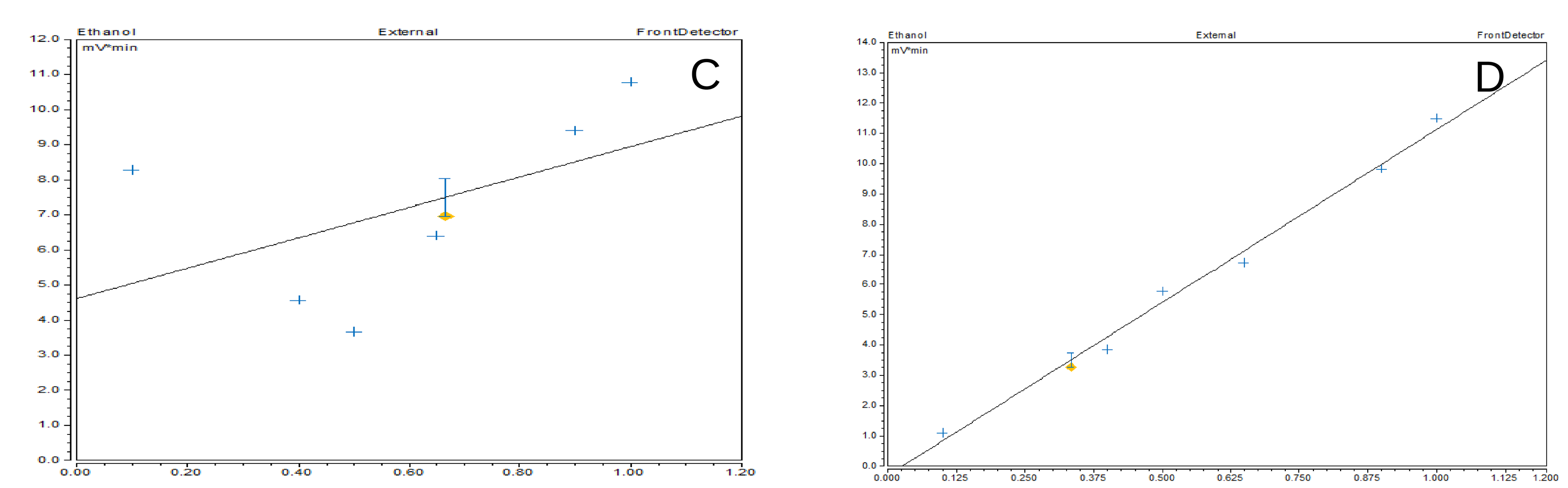
Headspace sampling: The more volatile compounds are extracted from the sample. For example:



Perfume Chromatographs obtained using a direction injection GC (A) method and a Headspace sampling technique (B). (Tipler, 2013)

Results and Discussion

- Two separate calibration curves (C,D) were generated in the laboratory using liquid samples of 10, 40, 50, 65, 90, and 100 % (v/v) ethanol/water using GC.
- Sample preparation was improved between analysis as shown on each calibration



$EtOH/H_2O$ Calibration Curves

Proposed HS-GC-Method

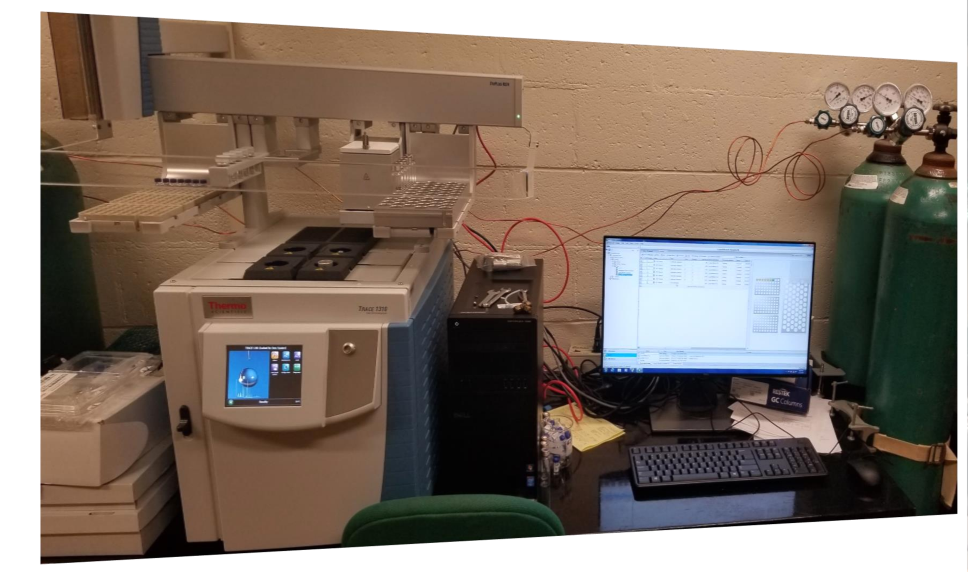
A method proposed by Shan et al., (2011) using a HS-GC with a FID detector will be tested during the summer to evaluate SCFA.

Headspace Explanation

- Partition coefficient: K=Cs/Cg
- Phase Ratio: B=Vg/Vs
- Headspace Concentration: Cg=Co/(K+B)
- As T goes up, K goes down
- Activity coefficient: Pi=Po x Yi x Xs(i)
- Adding salts to $EtOH/H_2O$ samples

Using HS-GC Quantitatively

- Sample volume: Vs=Ve x (Fcol/Fcol+Fs)
- Injection time: Ti=(Vsyr/Fcol+Fs)x(Pi/Pa)
- This combination allows sample concentrations determination



Thermo Scientific GC And Autosampler

Distillation Column

Future Applications

- Analyze ethanol and water samples obtained from the Unit Operations Laboratory.
- Characterize the efficiency of GC analysis of $EtOH/H_2O$/oil samples vs. using HS-GC for ternary mixtures
- Characterize SCFA (short chain fatty acid) content in Listeria metabolites present in mice feces with the SURE program in the SoE during this summer

Acknowledgements

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Selected References

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4. Tiscione, N. B., Alford, I., Yeatman, D. T., & Shan, X. (2011). Ethanol analysis by headspace gas chromatography with simultaneous flame-ionization and mass spectrometry detection. *Journal of analytical toxicology*, *35*(7), 501-511..