Gas Chromatography Basics: Column Temperature and Loading

This experiment is designed to help students navigate what can often be the daunting world of modifying instrumental parameters. Students will observe how changes in instrumental settings alter compound separation by the gas chromatograph. Then they will use temperature ramping to improve a chromatogram's appearance. In addition, they will learn how to adjust experimental parameters to improve a chromatogram's appearance rather than injecting more analyte.

ESTIMATED TIME

We estimate that this experiment can be completed in one 3-hour class period.

TIPS

1. In the Electronic Resources you will find PDF and word-processing files of the student experiment. You can print the PDF, distribute it to students electronically, or post the file to a password-protected class web page or learning management system. Edit the word-processing file if you would like to tailor the experiment to suit your equipment and students. Sign in to your account at **www.vernier.com/account** to access the Electronic Resources.

2. To make the sample mixture 1, combine 1.0 mL cyclohexane, 1.0 mL toluene, 1.0 mL ethyl acetate, and 0.5 mL butyl acetate.

3. An alternate mixture 1 could be cyclohexane, toluene, pentane, and n-hexane. You can prepare these by adding 1.0 mL of each compound to create the mixture.

4. Mixture 2 should be prepared with 1.0 mL of cyclohexane and 1.0 mL of 1-propanol.

5. We recommend keeping back-up vials of each compound in addition to the vials that are made available to the students; if a student contaminates a vial by mistake or spills a compound, the back-up vials can be used as replacements.

6. We strongly recommend using reagent grade compounds for the best, most reliable results. The substances used in this experiment may be purchased from Flinn Scientific.

7. Vials must be kept tightly sealed when not in use, as the compounds used in this experiment are highly volatile and will evaporate quickly.

8. All of these compounds should be used in a well-ventilated area. Be familiar with the SDS information for each compound and follow safe handling practices..

9. As an alternative to using the Peak Analysis feature of the software, your students can manually determine the retention time for a compound by using the Examine feature. Because of the manner in which the Mini GC operates, the retention time is the x-value, in minutes, at

the maximum y-value of the peak. If you want students to use the Examine feature, consider modifying the student version of the experiment.

10. The temperature-pressure profile is set so that each data collection run will last 10 to 30 minutes. You may remind your students that some of the substances will have passed through the column and detector well before the allotted time and they can stop the data collection early to save time.

11. Note that this experiment requires the Go Direct Mini GC (order code: GDX-GC). The compounds used in this experiment are not detectable on the Mini GC (order code: GC-MINI) or Mini GC Plus (order code: GC2-MINI).

12. To optimize the reproducibility of your retention times, it is best practice to let the Go Direct Mini GC return to 45°C between each trial.

HAZARD ALERTS

The chemical safety signal words used in this experiment (**DANGER** and **WARNING**) are part of the Globally Harmonized System of Classification and Labeling of Chemicals (GHS). Refer to the Safety Data Sheet (SDS) that came with the chemical. These can also be found online from the manufacturer. See the Preface for additional chemical safety information.

Butyl acetate, $C_6H_{12}O_2$: **WARNING**: Flammable liquids. Flammable liquid and vapor. Keep away from heat, sparks, open flames, and hot surfaces. No smoking. May cause drowsiness or dizziness. Avoid breathing mist, vapors or spray.

Cyclohexane, C_6H_{12}: **DANGER**: Highly flammable liquid and vapor. Keep away from heat, sparks, open flames, and hot surfaces. May be fatal if swallowed and enters airways. Avoid breathing dust or fumes. May cause drowsiness or dizziness.

Ethyl acetate, $CH_3COOC_2H_5$: **DANGER**: Keep away from heat, sparks, open flames, and hot surfaces—flammable liquid and vapor. Causes serious eye irritation. Avoid breathing mist, vapors, or spray. May cause drowsiness or dizziness.

n-Hexane, C_6H_{14}: **DANGER**: Keep away from heat, sparks, open flames, and hot surfaces—highly flammable liquid and vapor. Do not eat or drink when using this product. Avoid breathing mist, vapors, or spray. May be fatal if swallowed and enters airways. May cause damage to organs. Causes skin and eye irritation. May cause drowsiness or dizziness. Suspected of damaging fertility or the unborn child. Do not handle until all safety precautions have been understood. Use personal protective equipment as required.

1-Propanol, C_3H_8O: **DANGER**: Keep away from heat, sparks, open flames, and hot surfaces—highly flammable liquid and vapor. Do not eat or drink when using this product—harmful if swallowed. Causes mild skin irritation and serious eye damage. May be harmful if inhaled. May cause drowsiness or dizziness.

Toluene, $C_6H_5CH_3$, **DANGER**: Flammable liquids. Highly flammable liquid and vapor. Keep away from heat, sparks, open flames, and hot surfaces. Acute toxicity, oral. Harmful if swallowed. Aspiration hazard. May be fatal if swallowed and enters airways. Causes skin and eye irritation.

May cause drowsiness or dizziness. Avoid breathing mist, vapors or spray. Use personal protective equipment as required. Specific target organ toxicity, repeated exposure.

SAMPLE DATA

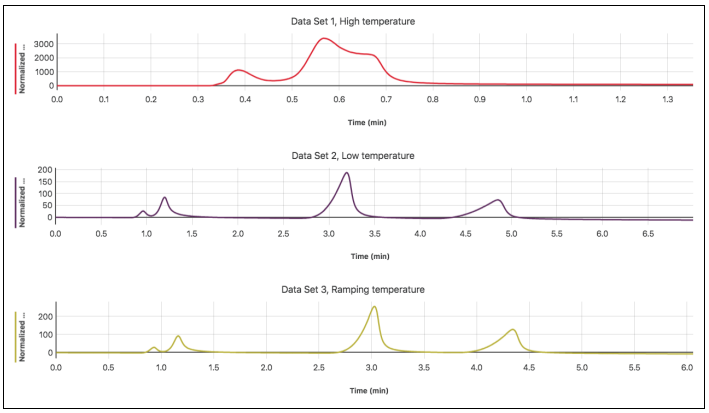

Figure 1 Sample chromatogram resultant from Part I: Effect of Column Temperature

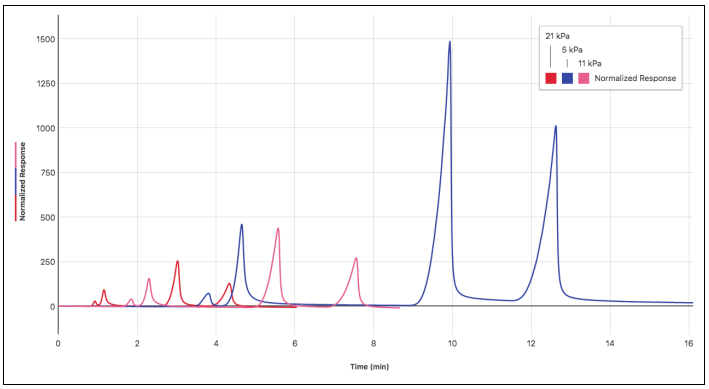

Figure 2 Sample chromatogram resultant from Part II with mixture of cyclohexane, toluene, ethyl acetate, and butyl acetate under varied pressure conditions

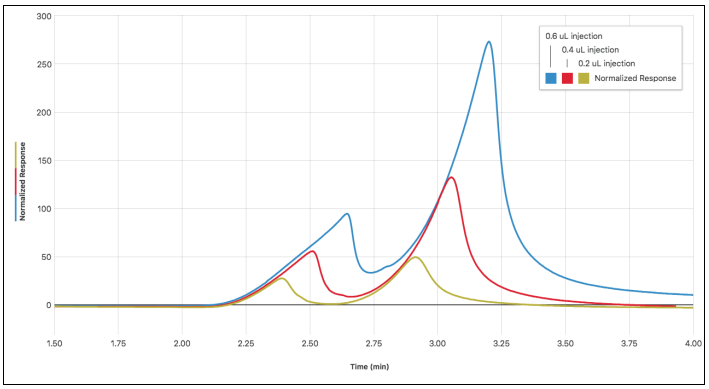

Figure 3 Sample chromatogram of column loading with cyclohexane and ethyl acetate

ANSWERS TO ANALYSIS QUESTIONS

1. Data Set 3 should result in the best peak definition. Some students might find Data Set 4 gives the best definition, but it comes at a trade-off in time and gaussian shape.

2. Note that the peaks become better defined (more nearly Gaussian) with higher temperature; a higher temperature frequently helps to eliminate "shark fin" shape, but peaks may become overlapped. Thus, optimal peak shapes are often best obtained by temperature ramping. Ramping allows the early peaks to elute relatively slowly and increasingly speeds up later peaks to give better peak shape and overall faster analyses than operating the instrument isothermally. Remember that because of gaseous diffusion, the peaks tend to become wider as the analytes elute at later times from the column. Higher temperatures facilitate faster elution; thus, higher temperature allows faster elution of analytes, which works toward delivering narrower peaks. Temperature ramping, in general, allows earlier peaks to separate effectively while speeding up elution times for late-eluting analytes. Higher temperature causes analytes to elute faster, which yields sharper peaks but reduces the separation efficiency such that the peaks in the chromatogram may overlap.

3. Increasing pressure causes the chemicals to elute faster, but can be at the cost of peak resolution. In addition, setting the pressure too low can cause data collection to last a long time. The balance between temperature, ramp, and pressure to get a resolved chromatogram, along with reasonable data collection times, is important.

4. While using the larger injection creates a larger peak, the larger peak can interfere with, and mask, peaks eluting later in the run. Thus, it is often preferred to make adjustments to operating parameters of the Mini GC to increase sensitivity rather than increasing injection volumes to detect small amounts of poorly detected analytes.

5. The experiment parameters can be adjusted to improve your ability to use a gas chromatograph to identify compounds in a solution. Options to achieve better peak detection include use of lower carrier gas pressure and use of higher temperatures.