Experiment 2

Gas Chromatography Basics: Column Temperature and Loading

Gas chromatography is an essential technique for a chemist. While the basic operation of a gas chromatograph is fairly easy to master, with practice, you will be able to analyze a chromatogram and adjust operational settings in order to tease out the identity of similar compounds.

Column temperature is one setting that can be adjusted to optimize your chromatogram. In this experiment, you will explore how changes in the instrument's temperature setting alter compound separation by the gas chromatograph. You will also investigate how temperature ramping and pressure can be used to improve peak shapes and separations in a chromatogram.

Another important parameter to understand is *column loading*, or how much analyte is injected into the column. Optimum peak shape is obtained when the sample aliquot does not saturate the column coating. Using too little analyte introduces error because the noise becomes a significant contribution to the desired measurement. Using too much analyte can overload the absorption capabilities of the polymer lining the separatory column and/or overwhelm the detection range of the detector. In the second half of the experiment, you will explore how increasing the volume of analyte affects the shape of the peaks on the chromatogram.

OBJECTIVES

- Observe how changes in instrumental settings alter compound separation by the gas chromatograph.
- Use temperature ramping to improve a chromatogram's appearance.
- Learn how to adjust experimental parameters, including pressure and volume of analyte, to improve the appearance of a chromatogram.

MATERIALS

Chromebook, computer, or mobile device
Instrumental Analysis app
Go Direct Mini GC
1 µL Hamilton syringe
paper towel or lint-free wipe
goggles and gloves
hexane
mixture 1, containing cyclohexane, toluene, ethyl acetate, and butyl acetate (Part I and Part II)
mixture 2, containing cyclohexane and ethyl acetate (Part III)

PROCEDURE

Part I Effect of column temperature

1. Put on safety goggles and gloves.
2. Set up the Go Direct Mini GC and Instrumental Analysis. **Important**: The Mini GC should be off for now; you will turn on the Mini GC during part c of this step.
   a. Launch Instrumental Analysis.
   b. Click or tap Connect an Instrument.
   c. Turn on the Mini GC. Then, connect the Mini GC to your Chromebook, computer, or mobile device via USB or Bluetooth wireless technology.
   d. Click or tap Sensor Channels. Enable the Temperature channel (leave the Normalized Response channel selected). Then, click or tap Done.
   e. Click or tap Gas Chromatography.
   f. Set the temperature and pressure profile to the parameters listed for Data Set 1 in Table 1. Then, click or tap Apply to initiate the profile.

Table 1

	Data Set 1	Data Set 2	Data Set 3
Start temperature (°C)	140	45	45
Hold time (min)	5	5	2
Ramp rate (°C/min)	0	0	2
Final temperature (°C)	140	45	75
Final hold time (min)	0	5	2
Total time (min)	5.0	10.0	19.0
Pressure (kPa)	21	21	21
Injection volume (µL)	0.4	0.4	0.4

3. While the Mini GC is warming up, follow the procedure in this step to clean and flush the syringe with n-hexane. **Important**: The glass syringe is fragile. Be careful not to bend the needle or bend the plunger. Never pull the plunger back more than 50% of its total volume.
   a. Depress the plunger fully.
   b. Submerge the tip of the syringe needle into the vial of n-hexane. **DANGER**: *n-Hexane,* C_6H_{14}: *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, drowsiness, or dizziness. Suspected of damaging fertility or the unborn child. Do not handle until safety precautions have been understood.*
   c. Pull back the plunger to fill the barrel with 0.4 µL of n-hexane.
   d. Expel the liquid onto a lint-free wipe or a paper towel.
   e. Repeat Steps a–d at least two times, or until you are comfortable pulling up a liquid into the syringe and measuring the volume in the syringe barrel.

4. Follow the process in Step 3 to clean and flush the syringe with mixture 1, the first sample to be injected into the Mini GC. **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.*

5. Once the Mini GC has reached the correct start temperature and pressure, the LED will turn green. Collect 0.40 µL of mixture 1 for injection. Insert the needle into the injection port of the Mini GC. Depress the syringe plunger; immediately following, press the button on the Mini GC to initiate data collection. Be careful not to bend the plunger as you press it down. Pull the needle out of the injection port immediately. Data collection will automatically stop after the total time, or you can choose to end it early if you are satisfied that your species has eluted completely.

6. Name your sample appropriately. To do this, click or tap the y-axis label. Click or tap ⋯ next to the data set you wish to rename, and select Rename Data Set. Enter a name for the data set, and click or tap Rename.

7. Collect data for Data Set 2.
   a. Click or tap Initiate Data Collection to open the temperature and pressure profile.
   b. Change the parameters to those listed in Table 1 for Data Set 2. Click or tap Apply to initiate the new profile.
   c. Follow the process in Step 3 to clean and flush the syringe with mixture 1.
   d. Repeat Steps 5 and 6.

8. Collect data for Data Set 3.
   a. Click or tap Initiate Data Collection to open the temperature and pressure profile.
   b. Change the parameters to those listed in Table 1 for Data Set 3. Click or tap Apply to initiate the new profile.
   c. Follow the process in Step 3 to clean and flush the syringe with mixture 1.
   d. Repeat Steps 5 and 6.

Part II Effect of pressure

9. Click or tap Initiate Data Collection to open the temperature and pressure profile. Change the parameters to those listed in Table 2 for Data Set 4. Click or tap Apply.

Table 2

	Data Set 4	Data Set 5
Start temperature (°C)	45	45
Hold time (min)	2	2
Ramp rate (°C/min)	2	2
Final temperature (°C)	75	75
Final hold time (min)	2	2
Total time (min)	24	24
Pressure (kPa)	5	11
Injection volume (µL)	0.4	0.4

10. Follow the process in Step 3 to clean and flush the syringe with mixture 1, the next sample to be injected into the Mini GC. **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.*

11. Once the Mini GC has reached the correct start temperature and pressure, the LED will turn green. Collect the designated volume of mixture 1 for injection. Insert the needle into the injection port of the Mini GC. Depress the syringe plunger; immediately following, press the button on the Mini GC to initiate data collection. Be careful not to bend the plunger as you press it down. Pull the needle out of the injection port immediately. Data collection will automatically stop after the total time, or you can choose to end it early if you are satisfied that your species has eluted completely.

12. Name your sample appropriately.

13. Repeat Steps 9–12 for Data Set 5.

Part III Effect of column loading

14. Click or tap Initiate Data Collection to open the temperature and pressure profile. Change the parameters to those listed in Table 3 for Data Set 6. Click or tap Apply.

Table 3

	Data Set 6	Data Set 7	Data Set 8
Start temperature (°C)	35	35	35
Hold time (min)	10	10	10
Ramp rate (°C/min)	0	0	0
Final temperature (°C)	35	35	35
Final hold time (min)	0	0	0
Total time (min)	10	10	10
Pressure (kPa)	11	11	11
Injection volume (µL)	0.6	0.4	0.2

15. Follow the process in Step 3 to clean and flush the syringe with mixture 2, the next sample to be injected into the Mini GC. **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.*

16. Once the Mini GC has reached the correct start temperature and pressure, the LED will turn green. Collect the designated volume of mixture 2 for injection (see Table 3). Insert the needle into the injection port of the Mini GC. Depress the syringe plunger; immediately following, press the button on the Mini GC to initiate data collection. Be careful not to bend the plunger as you press it down. Pull the needle out of the injection port immediately. Data collection will automatically stop after the total time, or you can choose to end it early if you are satisfied that your species has eluted completely.

17. Name your sample appropriately.

18. Repeat Steps 14–17 for Data Sets 7 and 8.

19. Save the file as directed by your instructor. Keep your test results open in Instrumental Analysis; you will need to refer to the various chromatograms to answer the Data Analysis questions.

20. When you are done with data collection, make sure to turn the Mini GC power switch to the off position and disconnect the Mini GC from AC power.

DATA ANALYSIS

1. Which temperature/pressure profile resulted in the best peak definition?

2. Comment on how higher temperature and ramping affected the separation of the peaks.
   Note: You may find it helpful to set up a second graph showing temperature on the y-axis and time on the x-axis.

3. Comment on how pressure affected the separation of the peaks.

4. Comment on how injection volume affected the appearance and separation of the peaks.

5. Summarize how experiment parameters can be adjusted to improve your ability to use a gas chromatograph to identify compounds in a solution.