

Using Gas Chromatography: Identifying an Unknown Compound

In this experiment, students explore the process of gas chromatography to identify one or more unknown species. First, they practice using a gas chromatograph by testing several known substances. Then, they test an unknown mixture that you provide. They will use the information they learned from the known substances to ascertain what compounds are present in the unknown mixture.

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. 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.
3. 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.
4. Vials must be kept tightly sealed when not in use, as the compounds used in this experiment are highly volatile and will evaporate quickly.
5. 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.
6. Four unknown mixtures are recommended in Table 1. The chart lists the compounds and the volumes.

Table 1				
Substance	Unknown mixture 1	Unknown mixture 2	Unknown mixture 3	Unknown mixture 4
pentane	1.0 mL	1.0 mL	1.0 mL	X
n-hexane	1.0 mL	1.0 mL	1.0 mL	X
ethyl acetate	0.5 mL	X	X	0.5 mL
cyclohexane	X	1.5 mL	1.5 mL	1.5 mL
toluene	X	1.0 mL	X	1.0 mL
butyl acetate	0.3 mL	X	0.5 mL	0.5 mL

- As an alternative to using the Peak Integral feature of the software, students can manually determine the retention time for a compound by using the Examine feature (simply click or tap the graph to view the x- and y-values for each data point). 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.
- The temperature-pressure profile is set so that each data collection run will last 14 minutes. You may remind your students that some of the substances will have passed through the column and detector well before 14 minutes and they can stop the data collection early to save time.
- 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.
- It is important for your students to inject a consistent volume for each test. **Note:** The injected volume of the unknown mixture is two times greater than the injected volume of a single, known substance.

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, $\text{CH}_3\text{COOC}_2\text{H}_5$: **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.

n-Pentane, $\text{CH}_3(\text{CH}_2)_3\text{CH}_3$: **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 or in contact with skin. Avoid breathing mist, vapors or spray. May cause drowsiness or dizziness.

Toluene, $\text{C}_6\text{H}_5\text{CH}_3$, **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.

PRE-LAB ACTIVITY

Compound	Boiling temperature (°C)	Molar mass (g/mol)	Bonding functional group
pentane	36.1	72.15	alkane
n-hexane	68	86.18	alkane
ethyl acetate	77.1	88.11	ester
butyl acetate	80.1	78.11	aromatic hydrocarbon
cyclohexane	81	84.16	aromatic hydrocarbon
toluene	110.6	92.14	aromatic hydrocarbon

SAMPLE DATA

Compound	Retention time (min)
pentane	0.69
n-hexane	1.22
ethyl acetate	1.85
butyl acetate	6.31
cyclohexane	2.27
toluene	5.05

SAMPLE CHROMATOGRAMS

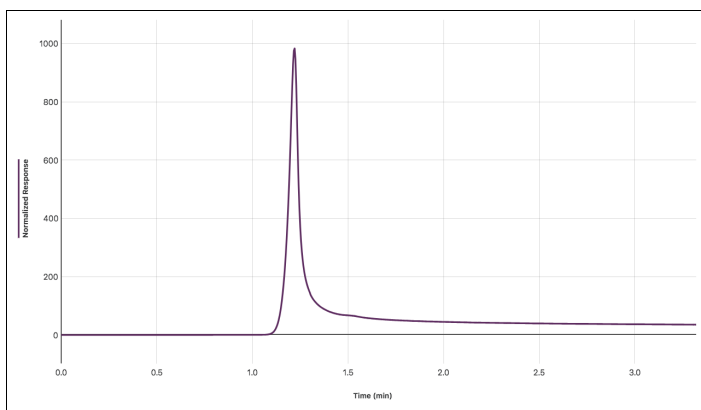


Figure 1 Sample chromatogram of n-hexane

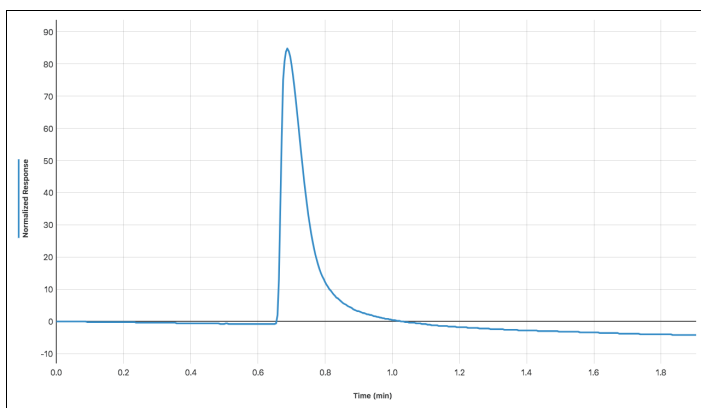


Figure 2 Sample chromatogram of pentane

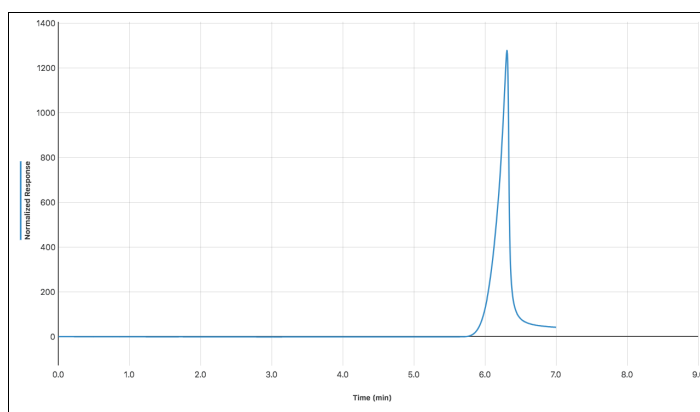


Figure 3 Sample chromatogram of butyl acetate

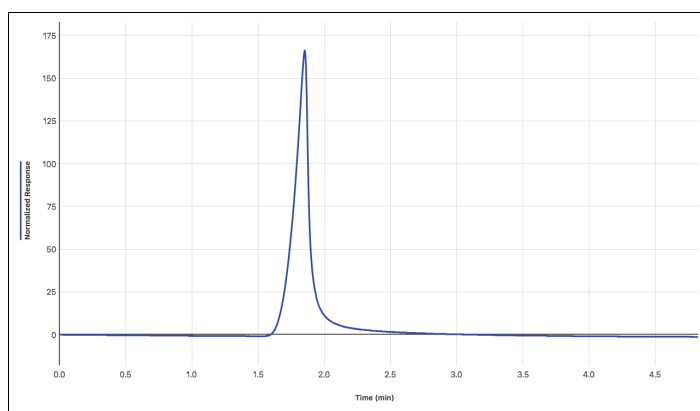


Figure 4 Sample chromatogram of ethyl acetate

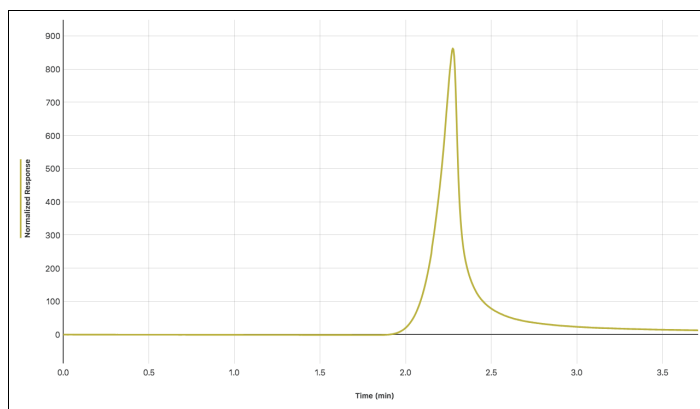


Figure 5 Sample chromatogram of cyclohexane

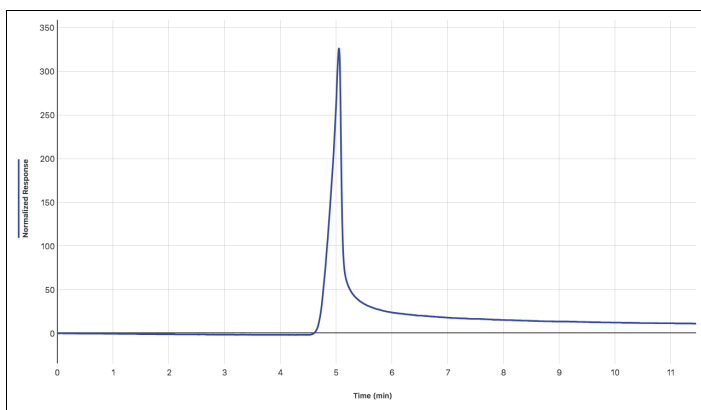


Figure 6 Sample chromatogram of toluene

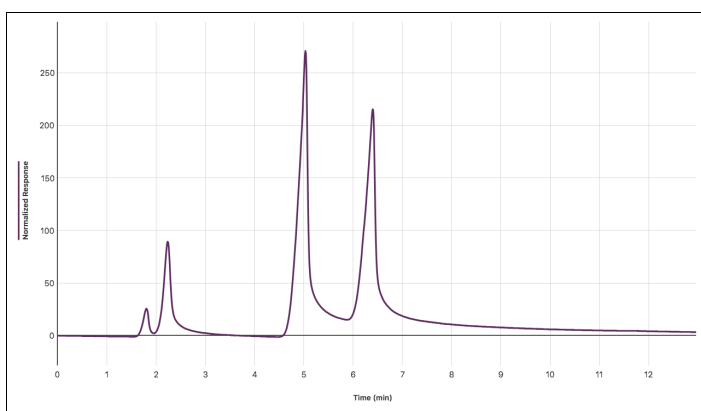


Figure 7 Sample chromatogram of unknown mixture #4

ANSWERS TO ANALYSIS QUESTIONS

1. In this set of substances, molecular mass alone does not suggest a predictable pattern with regard to retention time. The bonding functional group is the most important factor. Within a family of organic compound, possessing a specific bonding functional group, the substance with the greater molecular mass had a longer retention time. This pattern is evident with all three families of substances: the alkanes (pentane and n-hexane), the aromatic hydrocarbons (cyclohexane and toluene), and the esters (ethyl acetate and butyl acetate).
2. Typical answers to Question 2 will be similar to Question 1. In comparing two compounds within a given organic family, the compound with the higher boiling temperature will have a longer retention time.
3. The answer will, of course, vary according to the unknown mixtures that are offered to the student. Typical identifications will be supported by retention time correlation of the unknown compounds to the known compounds. Some students may note that the retention time of a substance alone can be slightly different when the substance is part of a mixture.