

An aerial photograph of the Colorado River winding through a lush, green landscape. The river is the central focus, reflecting the sky. The surrounding area is filled with dense vegetation, including grasses and shrubs. In the distance, a range of mountains is visible under a clear blue sky. The overall scene is bright and clear, suggesting a sunny day.

***An Experimental Approach for
Students to Learn about Factors
Affecting Retention Time in GLC***

**Scott Donnelly
Arizona Western College
Yuma, AZ**

Colorado River

Presentation Topics

- **Introduction to AWC**
- **Educational Benefits of GC Labs**
- **Prelab Exercise/Questions**
- **Postlab Assessment Questions**
- **Student Comments**



Las Vegas



Grand Canyon

Glen Canyon Dam

San Juan River

Flagstaff



Little Colorado River

Colorado River



Phoenix

ARIZONA

NEW MEXICO



AWC

MEXICO



Tucson

Arizona Western College

- **Public Two-Year College**

- **6,000 FTSE (Full-time Student Equivalents)**

- **Partnerships with NAU & UA**

- **New 40,000ft² Agriculture-Science bldg., Spring 2007**

Driving Forces for GC Labs

- **Earlier work with UV-Visible Spectroscopy**
Chemistry of Color labs
 - Students could mathematically manipulate $A = -\log T$ but had little understanding why red food coloring is red
- **Consistent exposure to instrumentation used in modern scientific inquiry**
- **Why wait until organic to use GC?**

- ***Classes:*** Organic Chemistry, General Chemistry 2, & Environmental Science

- ***GC Prelab:***

- **Conceptual questions**
- **5-10 minutes in length**
- **Four consecutive weeks**

- ***GC Lab:*** runs simultaneously with scheduled lab



- ***GC Postlab Assessment Questions:***

- **Embedded in quizzes and exams**
- **Take home lab exercise(s)**
- **Chromatography/Visible Spectroscopy Exam**

Educational Benefits

- **Demystify instruments- not “magical black boxes”based on known chemical principles**
- **Increase breadth of labs**
- **Expand lecture topic coverage**
- **Illustrate complementary relationship between theory and application**

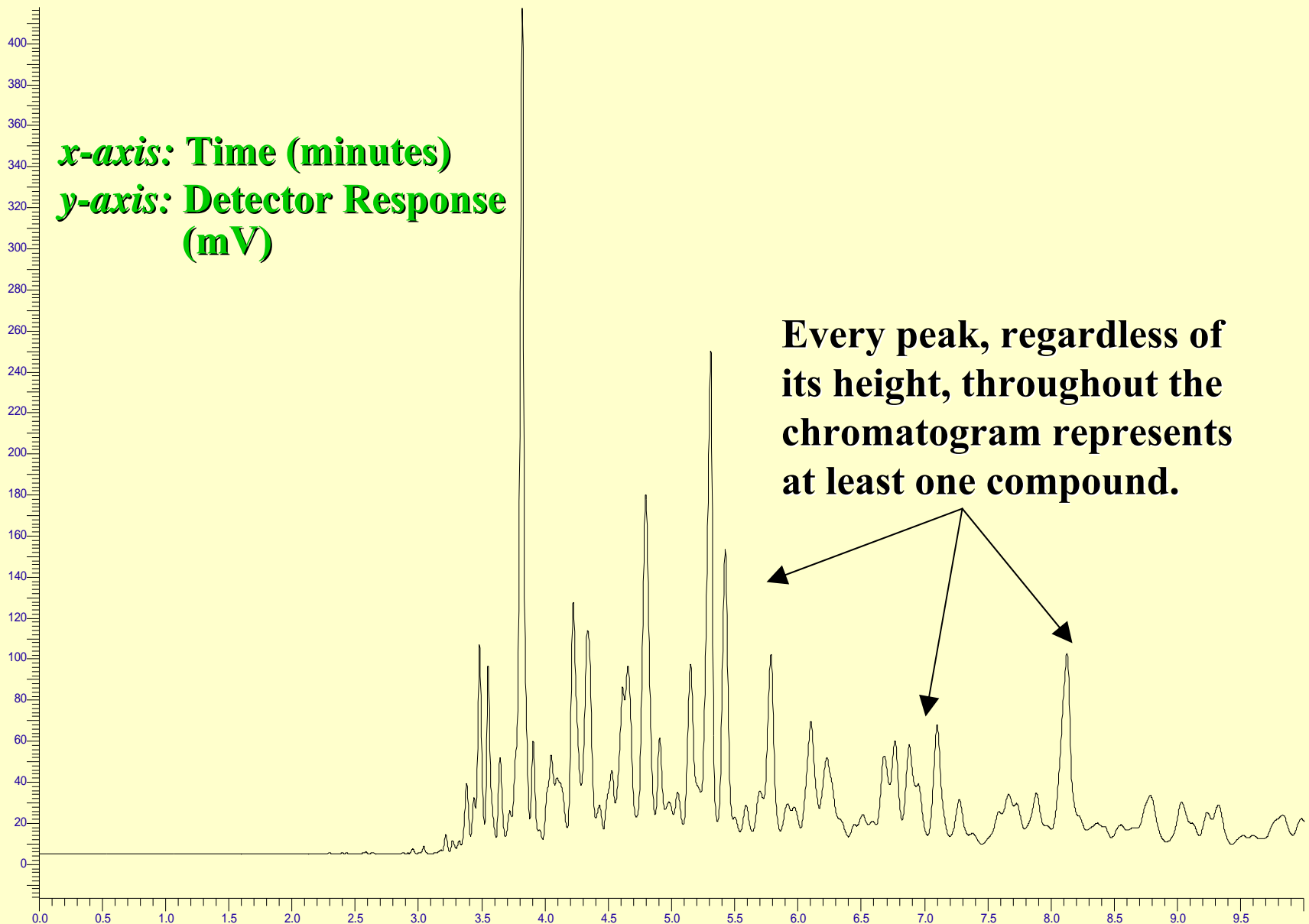
GC Labs: Potential Factors Affecting Retention Time, R_t

- **Molecular Weight**
- **Boiling Point**
- **Oven Temperature**
- **Injector Temperature**
- **Carrier Gas Flow Rate**
- **Sample Volume Injected**



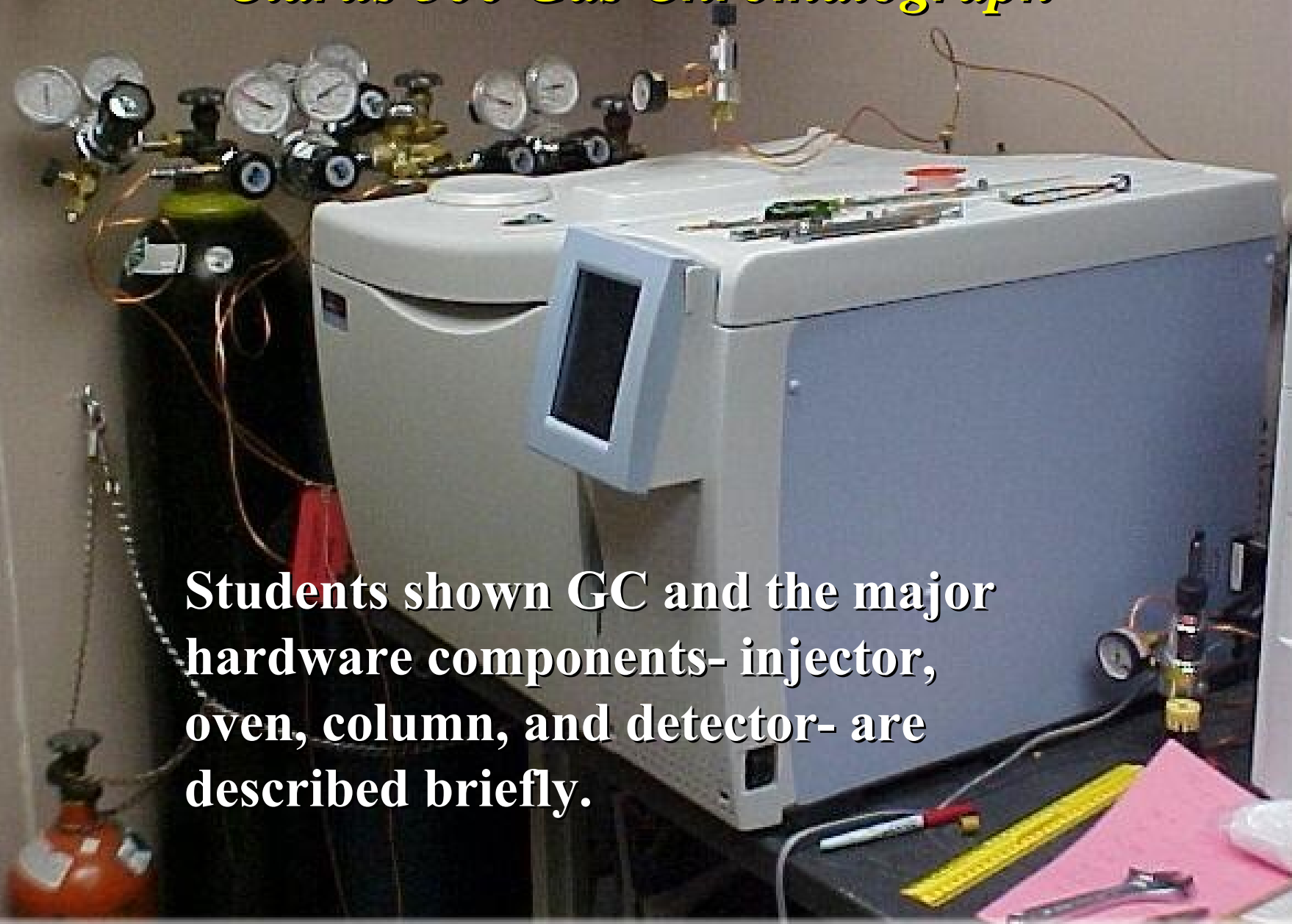
Clarus 500 GC

Chromatogram of Paint Thinner



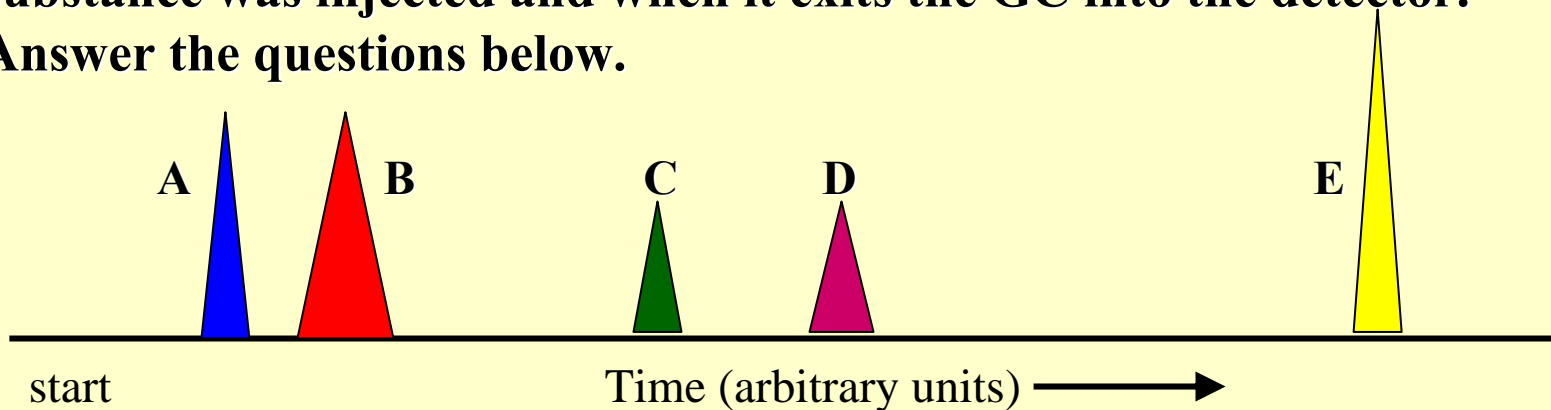
Clarus 500 Gas Chromatograph

Students shown GC and the major hardware components- injector, oven, column, and detector- are described briefly.



Prelab 1: Retention Time, R_t

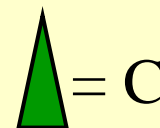
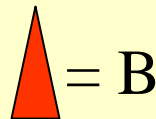
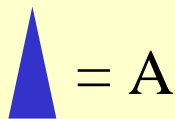
Gas-liquid chromatography, or GC for short, physically separates liquids in a mixture according to a number of factors. Suppose you carry out a GC analysis of an unlabeled bottle. Using a small volume syringe you withdraw some sample from the bottle and inject it into the GC. The chromatogram produced is shown below. *Retention time* (R_t) is the difference in time between when a substance was injected and when it exits the GC into the detector. Answer the questions below.



- A) Which peak represents the compound that travels through the GC instrument in the least amount of time?**
- B) Which peak has the shortest R_t ? the longest?**

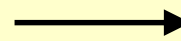
Prelab 1: Molecular Weight

Gas-liquid chromatography, or GC for short, physically separates liquids in a mixture according to a number of factors, one of which is molecular weight (MW). Suppose a mixture of three liquid hydrocarbons- **A**, **B**, and **C**- was injected into a GC with constant oven temperature, T_{oven} . The relative MWs of the three hydrocarbons are $\mathbf{B} > \mathbf{A} > \mathbf{C}$. Based on this information arrange the three compounds in the order that they elute or exit from the column. Then explain your reasoning.



start

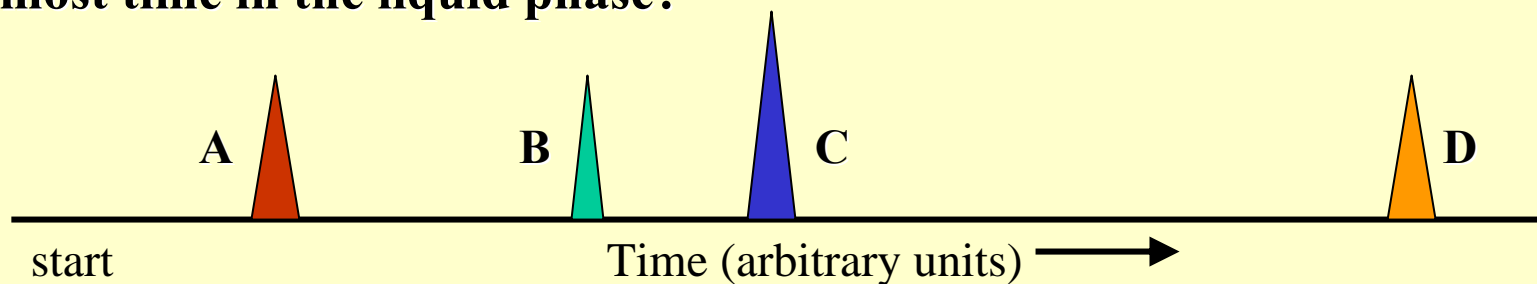
Time (arbitrary units)



Postlab 1 Assessment Questions: R_t and Molecular Weight

1) Suppose there are four colorless alcohols labeled A, B, C & D, all of which are liquids at room temperature. With respect to their molecular weights $B > D > C > A$. GC analysis was done at $T_{\text{oven}} = 115^{\circ}\text{C}$. Arrange these four alcohols with respect to their retention times.

2) A mixture of four hydrocarbons was injected into a GC at $T_{\text{oven}} = 85^{\circ}\text{C}$, giving the chromatogram below. Which compound spends the most time in the liquid phase?

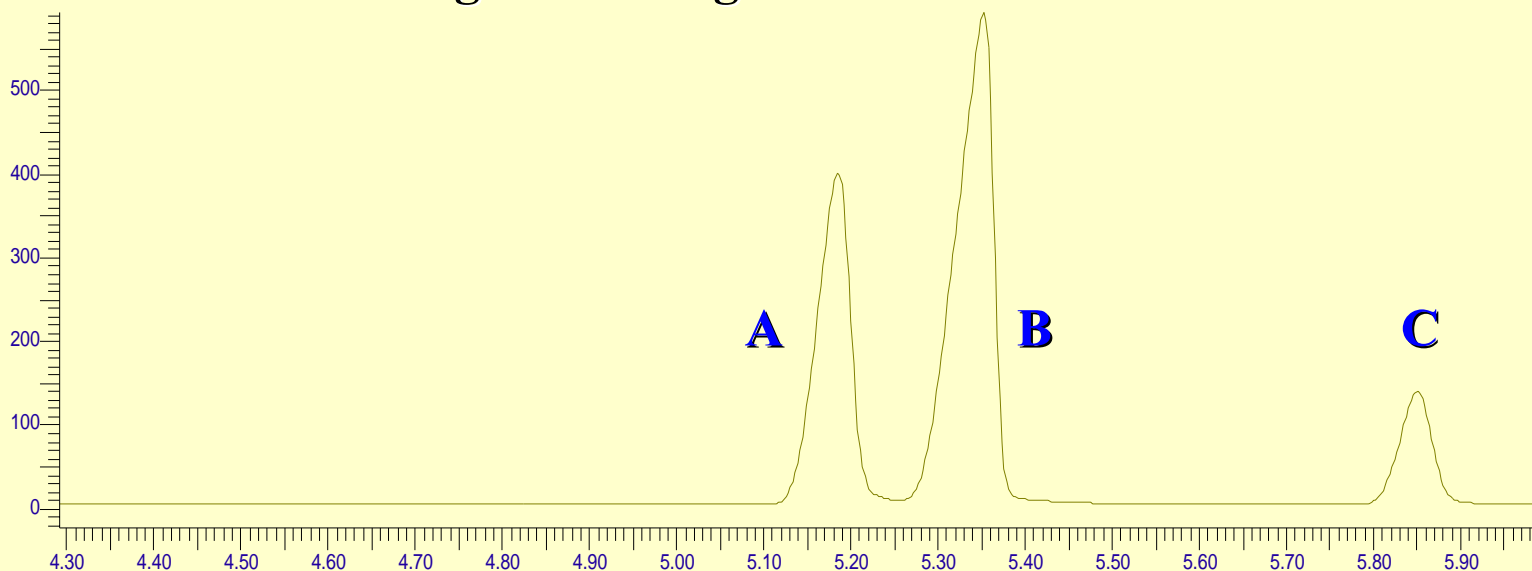


3) Of the choices below, which is predicted to have the longest R_t ?

- a. CH_3CH_3 b. $\text{CH}_3\text{CH}_2\text{Cl}$ c. $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$
d. $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_3$ e. CH_3CHO

Postlab Assessment Question: Molecular Weight

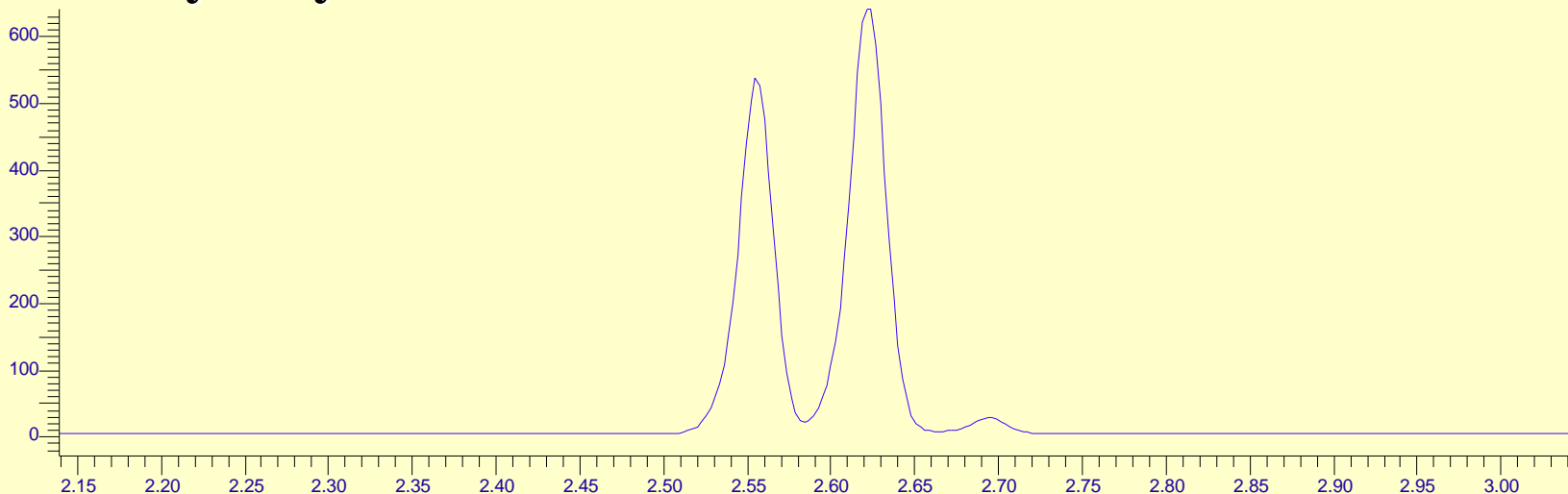
A mixture of three liquid saturated hydrocarbons- **A**, **B**, and **C**- was injected into a GC under isothermal heating conditions at 85°C. The GC was outfitted with a 30m, 0.250mm i.d. capillary column. The resulting chromatogram is shown below.



Suppose the three hydrocarbons were *2-methylhexane*, *pentane*, and *2,2,3-trimethylhexane*. Of the three which is represented by **B** in the chromatogram above? *Offer an explanation to support your choice.*

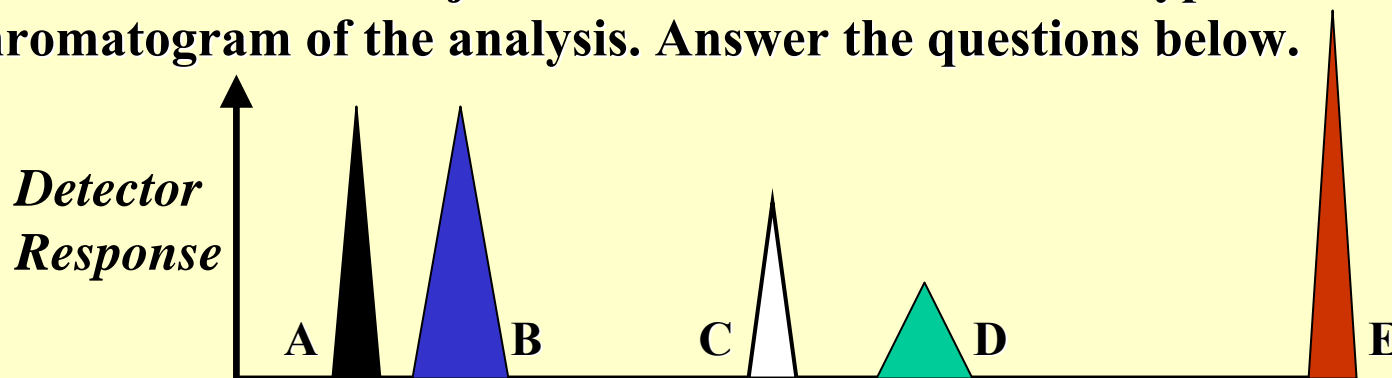
Prelab 2: Boiling Point

The chromatogram below is for a two component liquid mixture, containing the alcohols 1-propanol and 2-propanol. Both alcohols have the molecular formula C_3H_8O and hence the same molecular weight (MW). Yet, as shown the two alcohols have different retention times (R_t) under identical GC conditions. Since their MWs are the same, their differences in R_t cannot be explained on account of a difference in MW. So what then is a plausible explanation for why they have different retention times?



Prelab 2: R_t & Molecular Weight

As you learned last week gas-liquid chromatography physically separates liquids in a mixture according to a number of factors, one of which is molecular weight. Suppose you carry out a GC analysis of a bottle that is not labeled. Using a small volume syringe you withdraw some sample from the bottle and inject it into the GC. Below is a hypothetical chromatogram of the analysis. Answer the questions below.

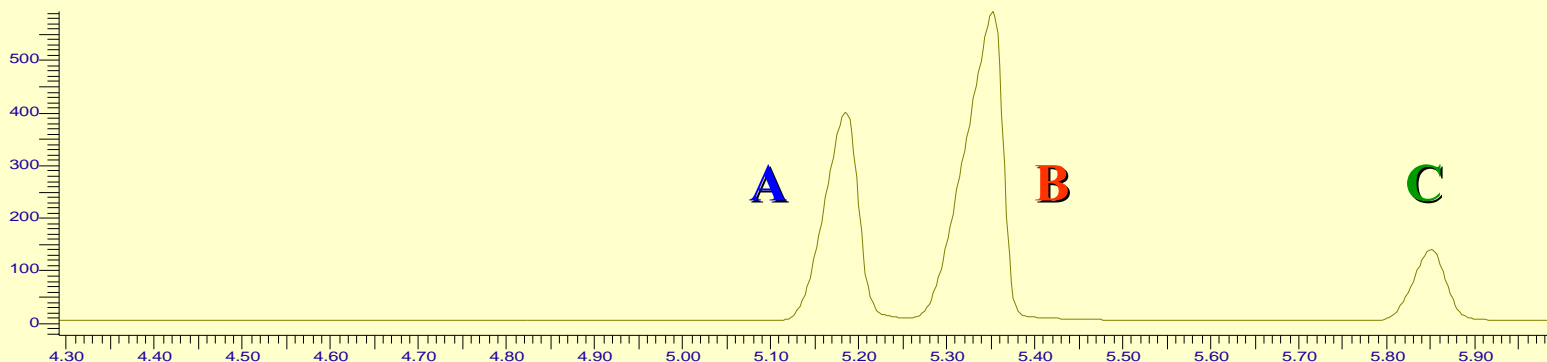


- 1) Which peak represents the compound that travels through the GC instrument in the least amount of time?
- 2) Arrange the five peaks with respect to their relative molecular weights, from heaviest to lightest.

___ > ___ > ___ > ___ > ___

Postlab 2 Assessment Question: Boiling Point & Molecular Weight

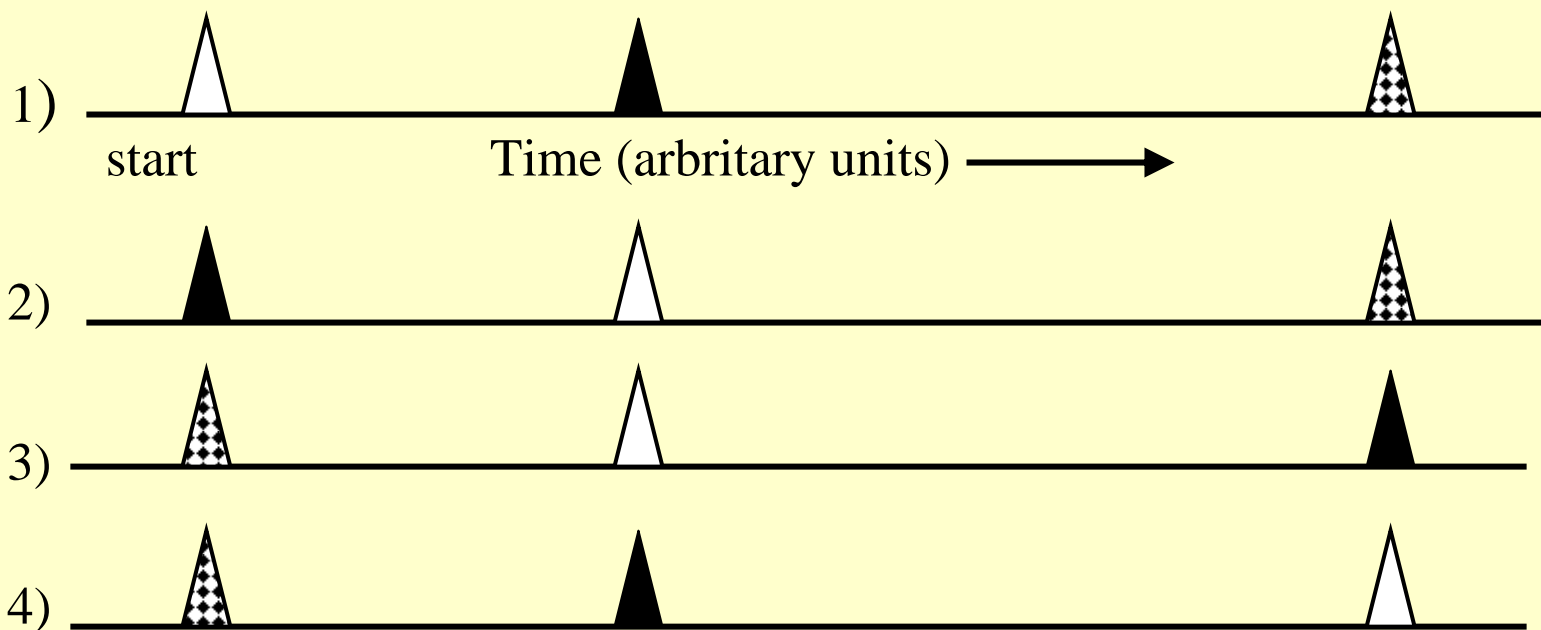
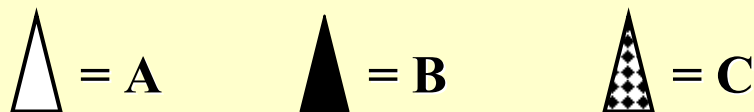
A mixture of three liquid hydrocarbons was injected into a GC at $T_{\text{oven}} = 95^{\circ}\text{C}$ (isothermal). The resulting chromatogram is below.



- 1) Which compound is the most volatile?**
- 2) Which compound most likely has the smallest molecular weight?**
- 3) Which compound most likely has the highest boiling point?**
- 4) Which compound spends more time in the liquid phase relative to the other two?**

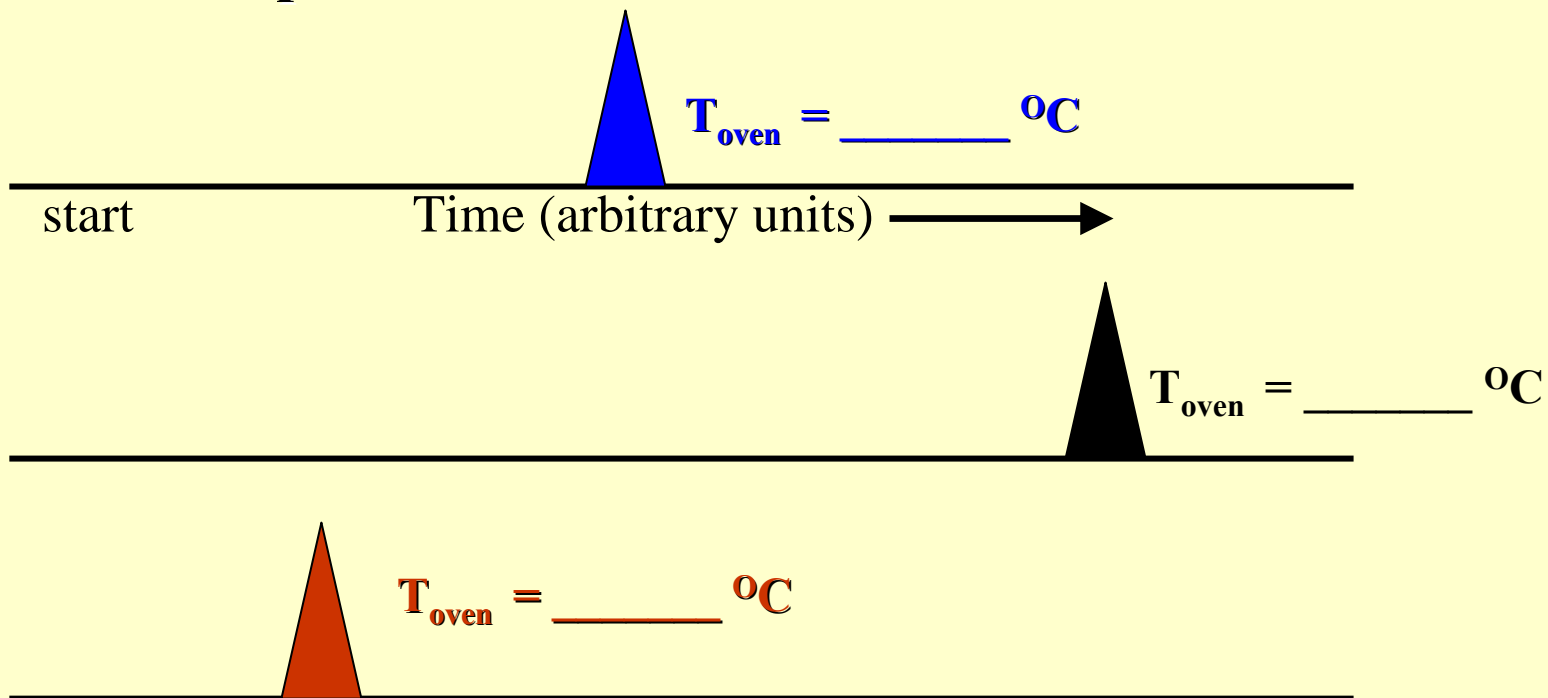
Postlab 2 Assessment Question: Boiling Point

GC physically separates liquids based on their differences, among other factors, in boiling point (BP) temperature. Suppose there are three liquids- A, B, and C- with different boiling points: $B > A > C$. The three component mixture is injected into a GC instrument whose oven temperature is held constant, *i.e.* isothermal. Which chromatogram below (choices 1, 2, 3 or 4) best represents the order in which the three liquids exit from the GC?



Prelab 3: Oven Temperature, T_{oven}

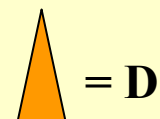
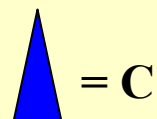
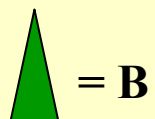
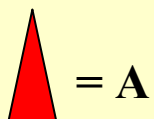
Three separate $0.2\mu\text{L}$ injections of an unknown colorless liquid were done at three different oven temperatures: 125°C , 100°C , and 75°C . The three separate chromatograms are shown below. Match each chromatogram with one of the three oven temperatures.



Postlab 3 Assessment Question:

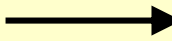
T_{oven} & Molecular Weight

Into a test tube are placed four colorless, liquid compounds labeled A through D. Their relative molecular weights are: $B > D > C > A$. On the chromatograms below arrange the four compounds with respect to how they elute from the column at $T_{oven} = 115^{\circ}\text{C}$ and 150°C . All other GC instrument conditions are the same for each analysis.



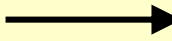
$T_{oven} = 115^{\circ}\text{C}$

start

Time (arbitrary units) 

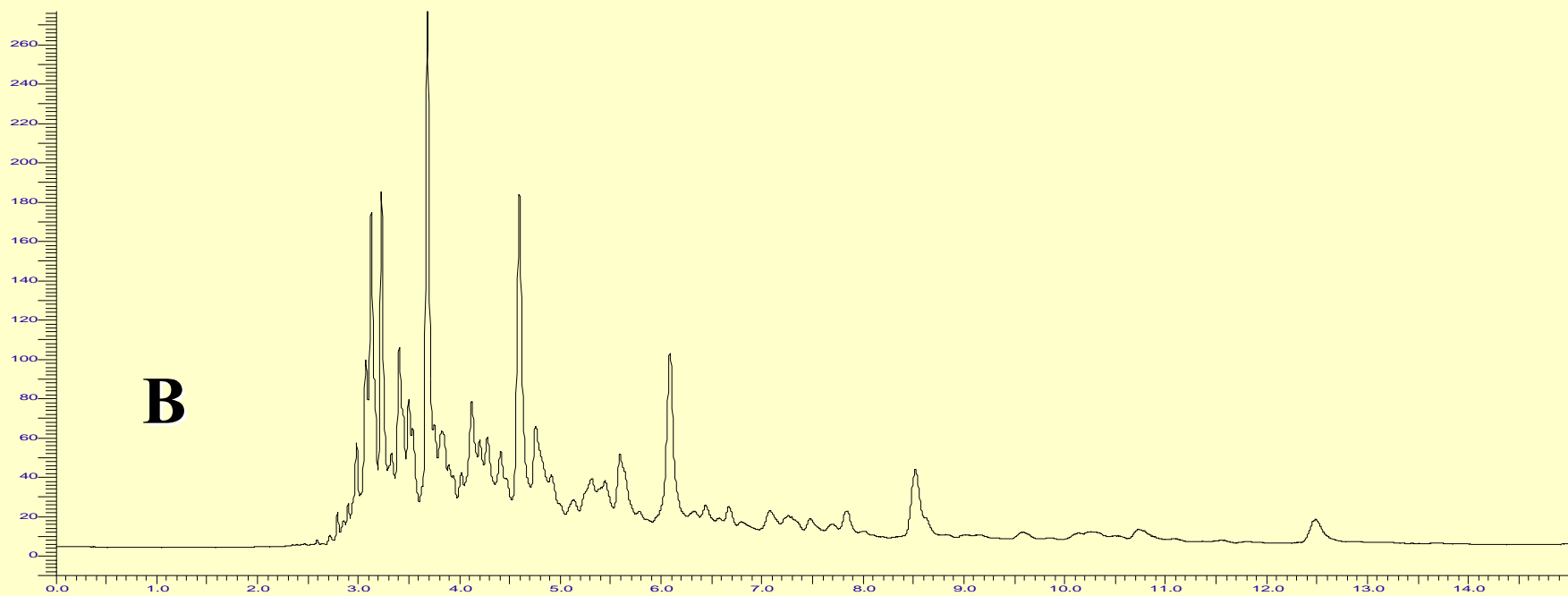
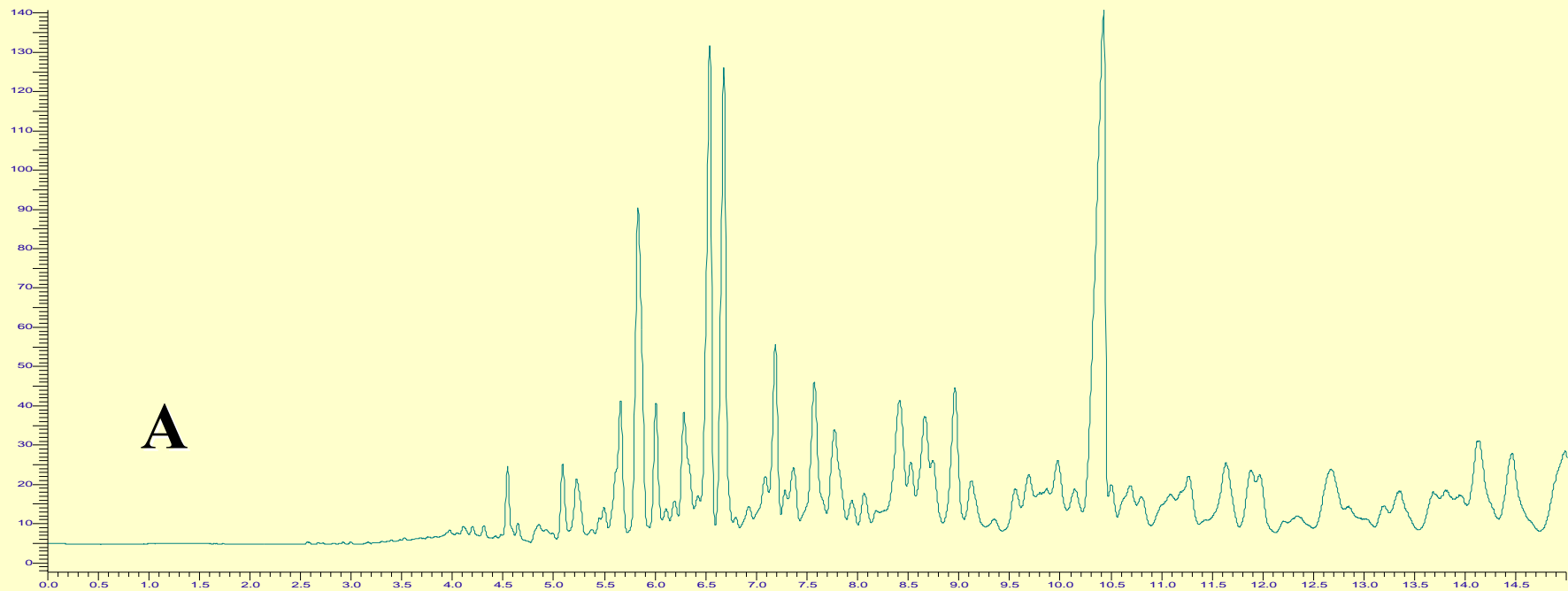
$T_{oven} = 150^{\circ}\text{C}$

start

Time (arbitrary units) 

Postlab 3 Assessment Question: T_{oven}

As a new Environmental Science hire you're given an assignment involving GC. Your boss doesn't have a clue about GC so gives you the two chromatograms. Both represent the same sample. The chromatograms were submitted to the Arizona Department of Environmental Quality (ADEQ) but returned because of the absence of the GC operating conditions, namely T_{oven} . Rather than doing the analysis again, you use your newly acquired knowledge of GC to argue that the two chromatograms were done at different T_{oven} . Which chromatogram- A or B- represents the analysis carried out at the higher oven temperature? On the next page provide two reasons to ADEQ that support your conclusion.

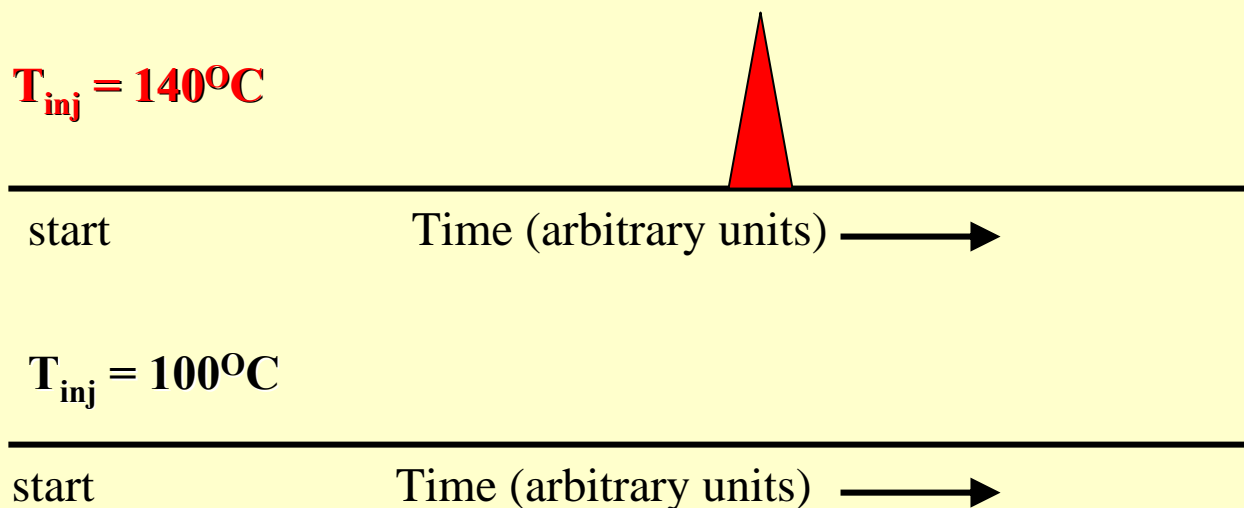


Postlab 3 Assessment Question: T_{oven}

As a new hire at a prestigious environmental consulting firm, you are asked to conduct a GC analysis of a bottle-containing a colorless, homogeneous liquid- from one of your firm's clients. The bottle's label reads only "Alcohols". Undaunted from such lack of information, you inject a small volume of the bottle's liquid content into a GC at $T_{oven} = 120^{\circ}\text{C}$ (isothermal). The resulting chromatogram shows three peaks. Your boss is incompetent when it comes to GC so asks how you are certain that the bottle does not contain instead four or five compounds, presumably all alcohols. What do you do to answer your boss' question? *Offer a brief but thorough explanation.*

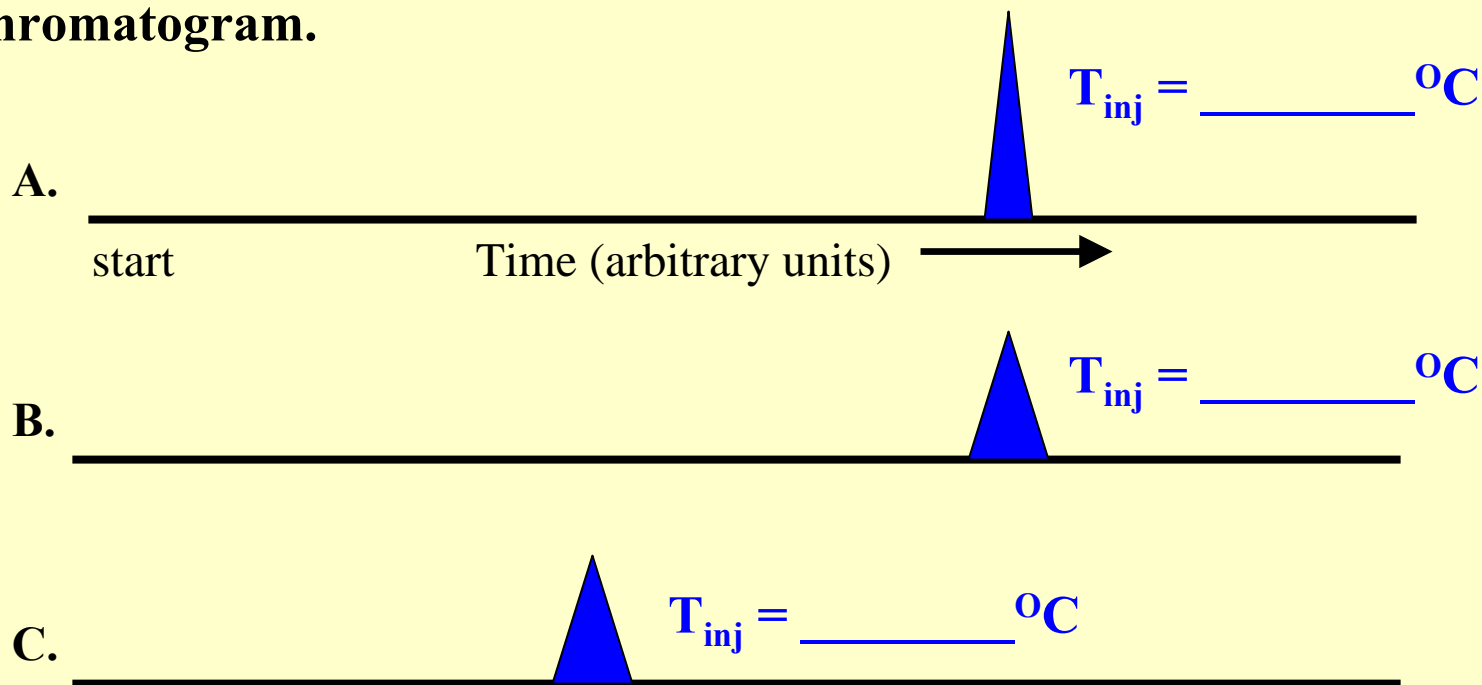
Prelab 4: Injector Temperature, T_{inj}

2-Pentanol's boiling temperature is 120°C . The red triangle below represents 2-pentanol when $T_{inj} = 140^{\circ}\text{C}$. Suppose the same volume of 2-pentanol was injected into the GC but with $T_{inj} = 100^{\circ}\text{C}$. Using the bottom blank axis, draw the chromatogram for 2-pentanol when $T_{inj} = 100^{\circ}\text{C}$. Use a black triangle to represent 2-pentanol.



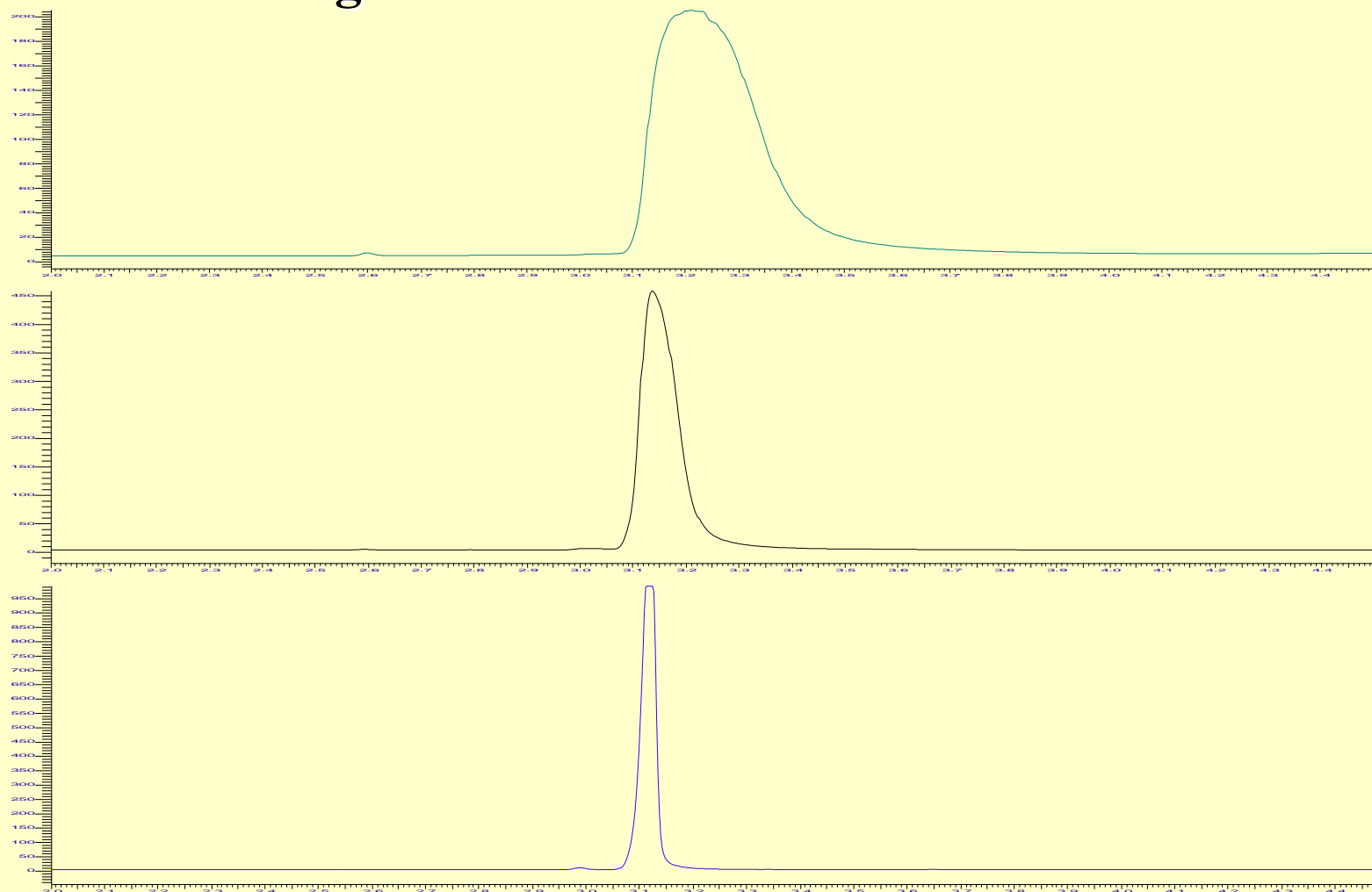
Postlab 4 Assessment Question: T_{inj}

Two separate 0.2 μ L injections of para-xylene (bp = 135-137 $^{\circ}$ C) were done under identical isothermal oven temperature, carrier gas flow rate, and split ratio conditions but at different injector temperatures, T_{inj} : 100 $^{\circ}$ C and 170 $^{\circ}$ C. Three chromatograms are shown below but only two are realistic. Match a chromatogram with either T_{inj} . Which chromatogram represents T_{inj} 100 $^{\circ}$ C and which T_{inj} 170 $^{\circ}$ C? Give a reason for not choosing the remaining chromatogram.



Postlab 4 Assessment Question: T_{inj}

Below are a series of chromatograms of 2-octanol taken in order from top to bottom. Offer an explanation for why the chromatograms differ.



Student Comments

Most of the time all we learn in school is very theoretical. We don't apply it or use equipment. I've always wanted to at least handle some lab equipment in my life before it becomes a necessity.

Being able to use instrumentation definitely demystifies certain elements of science. It's easier for me to assimilate knowledge through doing things I'm learning about.

Wanted to let you know that I have seriously considered going into research. A big part of this was my introduction to the use of instrumentation in your class.

I enjoyed using the expensive machinery [in order] to understand what we were learning in class.

Acknowledgements

1) National Science Foundation (NSF)

**•Course, Curriculum, &
Laboratory Improvement
(CCLI) grant (DUE- 0310264)**

2) All my students