

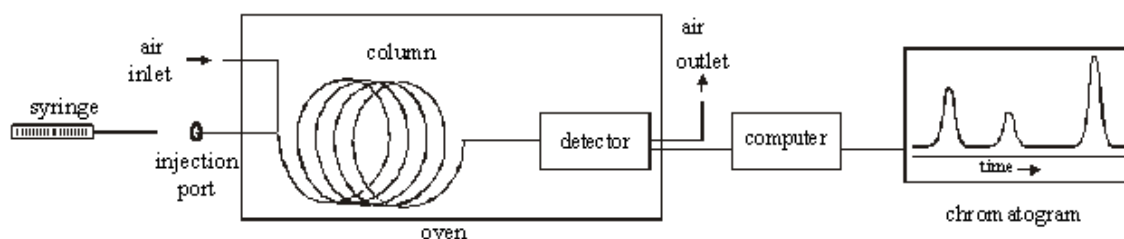
Separation of Alcohols

PA State Standards:

- 3.7.12.A Apply advanced tools, materials and techniques to answer complex questions.
Demonstrate the safe use of complex tools and machines within their specifications.
- 3.4.10.A Explain concepts about the structure and properties of matter.
Apply knowledge of mixtures to appropriate separation techniques.
- 2.1.8.D Apply ratio and proportion to mathematical problem distance, rate, time and similar triangles.
- 1.2.11.A Read and understand the central content of informational texts and documents in all academic areas.

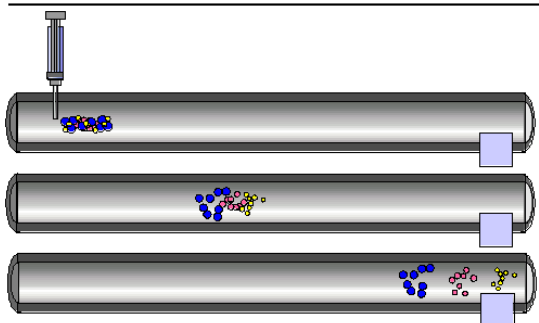
Introduction:

Chromatography is a separation technique utilizing a stationary phase and a mobile phase. In gas chromatography, the stationary phase is a solid material packed in a coiled column, and the mobile phase is a gas.



The gas chromatograph is an instrument that allows a chemist to separate a mixture of gases or volatile liquids. Separation is based on the size, structure, polarity of the molecules.

Separation by Chromatography



Retention time is used to identify the components, and the area under the curve is used to determine the relative quantities of the components. In this experiment, a HAYSEP-D column and air are used to separate 2-propanol, ethanol, and methanol.

Procedure:

1. Turn on the GC and start the Peak Sample software. A data acquisition system popup should appear with phrases like initializing, waking up, signing on, setting modes, and calibrating.
2. Be sure the time scale starts at zero and ends at 7 min. Use the arrows to adjust if necessary.
3. Clean the syringe by rinsing it 10 times with one of the standards. With the plunger fully depressed, place the needle through the septum and into the sample. Slowly draw up the plunger to obtain a sample in the syringe. Remove the syringe from the sample. Discharge this sample into the sink or onto a Kimwipe. Depress the plunger, and put the syringe needle back into the sample. Draw up a second sample and discharge it. Repeat. **Do not push on the plunger when the needle is inside a sample.**
4. Place the syringe needle back into the sample and withdraw over 1 microliter. Remove the needle from the sample bottle, then depress the plunger to the 1 microliter line. Wipe the needle with a Kimwipe.
5. Click on the Z or θ button on the run screen to zero the current.
6. Insert the needle carefully into the injection port of the GC until the entire needle is inside the instrument.
7. Inject the sample and start the data collection simultaneously by depressing the syringe plunger and pressing the space bar on the computer at the same time.
8. When the sample peak of a standard returns to baseline, the run may be stopped by pressing the end button on the computer. After pressing end, print the graph. Be sure to label the graph.
9. Repeat steps 3 -- 8 for the other 2 known alcohols and the unknown alcohol. **When running an unknown sample, be certain to let the data collect long enough to get all possible peaks.**

Data:

Standards	Retention time
2-propanol	
Ethanol	
Methanol	

Unknown 1	Retention Time (min)	Identity of Peak
Peak 1		
Peak 2		
Peak 3		

Unknown 2	Retention Time (min)	Identity of Peak
Peak 1		
Peak 2		
Peak 3		

Unknown 3	Retention Time (min)	Identity of Peak
Peak 1		
Peak 2		
Peak 3		

Unknown 4	Retention Time (min)	Identity of Peak
Peak 1		
Peak 2		
Peak 3		

Unknown 5	Retention Time (min)	Identity of Peak
Peak 1		
Peak 2		
Peak 3		

Calculations:

1. Calculate the area of each peak in the unknown. Treat each peak as a triangle to find its area. Find the total area by summing the areas of the individual peaks. (Note: if the computer is set to determine the area, that value from the print-out may be used.)
2. Use the areas of the peaks to determine the percent composition of the mixture.
3. If given the correct answers, calculate the percent error for you calculations.

Unknown 1

Component	Area	Percent Composition	Percent Error
2-propanol			
Ethanol			
Methanol			

Unknown 2

Component	Area	Percent Composition	Percent Error
2-propanol			
Ethanol			
Methanol			

Unknown 3

Component	Area	Percent Composition	Percent Error
2-propanol			
Ethanol			
Methanol			

Unknown 4

Component	Area	Percent Composition	Percent Error
2-propanol			
Ethanol			
Methanol			

Unknown 5

Component	Area	Percent Composition	Percent Error
2-propanol			
Ethanol			
Methanol			

Questions:

1. Why was it important to run known samples of possible components in the unknown mixture?
2. If a pure sample were properly injected into the gas chromatograph and several peaks were observed, what can be concluded about the sample?
3. Why is it important to clean the syringe between samples?
4. Why is it important to wipe the needle before injecting the sample into the instrument?