DEMYSTIFICATION ACTIVITY FOR THE GAS CHROMATOGRAPH

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INTRODUCTION

Gas chromatography (GC) is a very powerful tool for separating components of a mixture. The GC utilizes the physical and chemical properties of the components to achieve the separation.

A mixture of compounds, the **sample**, is injected and vaporizes in a stream of **carrier gas**. The carrier gas is used to force the sample through the system, much as water forces a boat down a river. The vaporized sample and the carrier gas make up the **mobile phase** of the GC. All of the components of the mobile phase move through the system at the same rate until they reach the **stationary phase**. The stationary phase is designed to resist the flow of the mobile phase. Each component of the mobile phase is slowed a different amount as it passes through the stationary phase. Therefore, each component of the mobile phase leaves the system at a different time and produces a separation. A **detector** is placed at the end of the system to record when each of the components exits the system. Below is a schematic of a typical GC.

SAFETY CONSIDERATIONS

1)The carrier gas for this activity is methane. Do not leave the gas jet on without lighting the flame.
2)The burner tip of the GC will remain hot for a long time after the flame is extinguished.
3)Take care when bending glass in a burner flame. It will cool slowly.
4)Halogenated hydrocarbons tend to be flammable and somewhat toxic. This activity should be carried out in a well ventilated room. Keep the samples away from open flames.
5)The samples should be kept in sealed containers to prevent them from evaporating. These compounds are hazardous to the ozone layer and should be contained as much as possible.

Preparation of the GC

MATERIALS

1 x 20 cm glass tubing
1 x 60 cm rubber/ tygon tubing
2 x 2 cm rubber/ tygon tubing
1 x 4 cm rubber/ tygon tubing
1 glass Pasteur pipet
1 cotton ball
1 x 30 cm fine copper wire
1 box of matches
2 clothes pins (utility clamps)
l small cup of activated TIDE® (dried in an oven overnight)
1 glass/ plastic T
1 pinch clamp
1 plastic syringe without the needle

PROCEDURE

PART A - PREPARATION OF THE GC COLUMN

1.Put a small cotton plug into one end of the glass tube.

2.Attach a funnel to the open end of the tube with a small piece of rubber tubing.
3.Fill the glass tube with activated Tide® by pouring it through the funnel.

4.Tap the funnel or tube to break up any clogs. DO NOT stick anything into the funnel or tube to break up blockages. This will only make matters worse.

5.After filling the tube, tap it gently on a rubber stopper to settle the Tide® in the column. If necessary add more Tide® to fill the tube to within 0.5 cm of the top.

6.Place a cotton plug into the open end of the tube.

PART B - PREPARATION OF THE BURNER ASSEMBLY

1.If your teacher has prepared your burner assembly for you, go on to the next section.

2.Obtain a glass Pasteur pipet. Place the tapered part of the pipet into a burner flame. Rotate the pipet until it starts to bend. Stop rotating the pipet and allow it to bend to a right angle.

3.Remove the pipet from the flame and allow it to cool.

Caution the glass will remain hot for several minutes!

PART C - PREPARATION OF THE DETECT0R

1.Wind the piece of copper wire around the large end of the pipet as shown below. As you wind the coils, keep your thumb on the end of the wire to prove tension.

2.Make 10 wraps around the pipet and leave a 3 cm tail of wire. Cut the remaining wire. The wire must be wrapped tightly together.

3.Slip the wire off of the pipet.

4.Bend one of the tails so that it runs down the center of the coil perpendicular to the coils.

5.Place the bent tail into the SMALL opening in the burner assembly.

6.Tuck the other tail into the coil.

PART D - ASSEMBLY OF YOUR GC

1.Push one end of the long piece of rubber tubing onto the gas jet at your lab station

2.Attach the other end of the tubing to the glass/ plastic T.

3.Attach a 4 cm piece of tubing to the T at a right angle to the first.

4.Attach a 2 cm piece of tubing to the third opening of the T.

5.Attach the Tide® column to the other end of the 2 cm piece of tubing.

6.Attach the other 2 cm piece of tubing to the other end of the Tide® column.

7.Connect the burner assembly to the 2 cm piece of tubing.

8.Adjust the burner assembly so that the detector is in a vertical position.

9.Secure the GC with the clamps provided.

Lab Activities For Your GC

PART A - AGING YOUR BEILSTEIN DETECTOR

The detector for your GC is based upon the Beilstein test for Halogenated hydrocarbons. This test identifies compounds that contain halogens by burning them in a flame with copper metal. A blue-green flame indicates the presence of a halogen. Our GC, therefore, can only be used to separate compounds that contain a halogen.

For the detector to work properly it must first be "aged." That is, it must be burned in a flame until the copper metal turns black.

1.Assemble the GC as directed above.
2.Turn on the gas jet.

CAUTION:
Do not leave the gas jet on without lighting the flame. An explosion could result due to the build up of methane gas in the room.

3.Light the flame with a match.
4.Allow the flame to burn for several minutes before you move to the next activity.

CAUTION:
You may leave the flame burning throughout the period as long as you do not leave it unattended.

PART B - MEASURING THE RETENTION TIME OF THE GC COLUMN

The retention time is the time that it takes for a substance to pass through the GC. Different substances will move through a GC at different rates depending upon their properties.

1.First we will measure the retention time for air.
2.Turn the gas on all the way and light the flame.
3.Inject 0.5 mL of air into the injection port of your GC.
4.Carefully measure the SHORT amount of time that it takes for the air to pass through the GC. The flame will dip, become smaller, when the air gets to the flame of the detector. This is caused by a change in the fuel air mixture.

Now your are ready to run samples of known compounds through your GC to measure their retention times.

5.Draw up a syringe full of VAPOR, not liquid, out of one of the sample bottles.

6.Hold the tip of the syringe near the flame of the detector and slowly inject the sample into the burner flame. Notice the color change in the flame. This is the indicator to look for when injecting samples through the GC.

7.Fill the syringe with vapor from the same sample as before. Inject the sample into the injection port of the GC and determine the retention time for the sample. Record in the DATA TABLE the amount of time that it takes for the first color change in the flame. Also record in the DATA TABLE the amount of time it takes for the flame to return to its original color.

8.Repeat the process for the other samples that are provided. Record your data and observations.

9.If time permits, try injecting a mixture of two of the samples into the GC.

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DATA TABLE

Sample	Amount of Time For First Color Change In The Flame (sec)	Amount of Time For Flame to Return to its Original Color (sec)

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