Brooklyn College
Department of Chemistry

Chemistry 41 ***Gas Chromatography***

Analysis of a Mixture

A gas chromatograph (GC) is used in this experiment to gain familiarity with the basic techniques of gas-liquid chromatography. Chromatographs for a select group of compounds are obtained on a polar and a non-polar column.. A table of retention times is prepared. A three-component mixture containing three compounds from this group is analyzed qualitatively using this table.

Chapter 28 in your text provides a general introduction to chromatography and Chapter 29 describes gas chromatography, in particular. Section 29A (pp. 687-690) describes the basic components of an instrument for gas chromatography and should be read before starting this lab.

The Gow-Mac GC employs dual columns with a thermal conductivity detector. The retention characteristics of the liquid stationary phase provides a mechanism for separation, and the use of two columns with different stationary phases aids in uniquely identifying the components of an unknown.

Instrument set-up.

The instrument will be turned on before the laboratory begins to allow for thermal equilibrium. The instructor will assist you in checking the following (approximate) settings:

- Column temperature: 100 ^{o}C
- Injection port & detector temperature: 10-15 ^{o}C higher than column temperature.
- Carrier gas (helium) flow rate: 60 mL/min
- Detector power switch ON and CURRENT CONTROL to 150 mA.
- ATTENUATOR to X4 (this is adjusted as necessary to bring peaks back on scale).
- POLARITY SWITCH to the left column (Carbowax - a polar polyglycol compound). *Remember to change this switch when the right column (DC-200, a non-polar silicone oil) is to be used.*
- Recorder sensitivity to 10 mV full-scale and recorder chart speed to 2"/min.

Qualitative Analysis

- Determine the air peak time. This is a measure of the time taken by an unretained sample to pass through the system. Retention times for retained solutes are measured relative to the air peak (adjusted retention time). It is important to develop a consistent sample-injection technique.

  The 10 μl syringe is fragile and expensive; use care in handling to avoid bending the needle.

  Using a sample size of 5 μl of air and the left column, follow this 3-step technique: insert the needle through the septum (use two hands - one for support), depress the plunger of the syringe and withdraw the syringe smoothly. Your partner should start the recorder chart drive at the same time. An air peak should be observed in approximately 30 seconds. The

actual time depends strongly on carrier gas flow rate and column temperature. Approximately 5 μl of air should be injected in all of your runs to obtain this reference peak. Only a small (1-2 cm high) peak is needed for this purpose.

- Obtain a sample of the alkane mixture (pentane, hexane, heptane, and octane). Use the 5 mL plastic containers that are provided for this purpose. Keep these capped when not in use to minimize organic vapors in the work area and concentration changes in the mixtures. Rinse the syringe several times with the sample; use a small beaker to contain the wastes. Draw a 1 μl sample into the syringe and then draw about 5 μl of air. Inject the sample onto the Carbowax (left) column and obtain a chromatogram. With a homologous series you can safely assume that the solutes will elute in order of increasing molar mass (or, equivalently, carbon number or boiling point).
  Run a chromatogram of the alkane mix on the DC-200 (right) column. Remember to change the POLARITY SWITCH.
- Now obtain chromatograms of the following compounds on both columns: propanol, toluene, cyclohexane, ethylbenzene. Prepare a table summarizing the adjusted retention times for the nine compounds studied on both columns. Distances in chart paper blocks are sufficient at this point in the experiment. Later, these can be converted to seconds for your report.
- Obtain a chromatogram of the unknown mixture on both columns. Identify the three components using your table of retention times. It is important that all data be obtained under the same operating conditions, in effect, during the same lab period. One way to confirm an uncertain assignment is to inject a sample of the unknown spiked with a small amount of the suspected compound. It is also important to avoid evaporation losses of this sample.
- When you finish, turn off the detector, and the oven controls. Open the oven door to aid the cool-down process. The carrier gas is left on until the column temperature drops below 40 ^{o}C. Remove and cap the recorder pen. Turn off the recorder. Discard your samples in the organic waste container and leave the plastic cups open to air dry.

Report.

Prepare a spreadsheet analysis of your data; it should include a table of retention times for the standard samples and your unknown. Based on retention times determine all three components of your unknown mixture