Gas Chromatography

Gas chromatography (GC) is a very fast, repeatable and accurate analysis method for volatile organic materials. GC is most often used for analysis of very small samples of mixtures in an analogous manner to TLC. In gas chromatography the mobile phase is an inert gas called the carrier gas. The carrier gas is usually nitrogen (N_2) or Helium (He). The stationary phase is usually a high-boiling liquid coated onto an inert support.

This technique is useful for quantitative analysis (identifying how much of a compound is present) and qualitative analysis (determining the identities of compounds present). GC is widely used in industrial, research and analytical laboratories. GC instruments are reasonably priced and flexible in their analysis methodologies. It is not uncommon for an analytical testing center to have 20 or more GC machines operating simultaneously.

GC can also be used on a small scale to separate mixtures into their components and recover them in a way analogous to column chromatography. This time intensive procedure is called preparative scale GC.

PRE-EXPERIMENT ASSIGNMENT

For GC, study this chapter, the lecture notes on the Chemistry Department web site, and the introduction to chromatography on page 53 of this manual and on pages 184-186 in Williamson

A student who has prepared for the gas chromatography portions of this lab should be able to:

1. Define and explain: chromatography, gas chromatography (GC), stationary phase, mobile phase, carrier gas, retention time. Explain the differences between GC and the chromatographic methods you used earlier.
2. Draw a diagram of a gas chromatography apparatus, and label the components of the apparatus in a drawing.
3. Identify and explain the reasons chemists use gas chromatography (to ID compounds in a mixture using retention times, to separate components of small samples, and to determine the amount of given compounds present, for mixtures of volatile compounds only).
4. Draw the structure given the name, or give the name from the structure, of the possible unknowns used in the GC portion of the day's experiment.
5. Carry out the experiment in a safe manner.

Quizzes given after the experiment has been performed may also include:

8. Explain and predict the effects of experimental variables such as flow rate, column temperature, and column length have on retention time and resolution of peaks.

Safety Precautions

Syringes are sharp and can easily prink skin. If a syringe is left too long in the injection port without depressing plunger, the liquid will volatilize and expand, shooting the plunger out at an unsuspecting student. The injection port is hot and can easily burn skin. Be sure to wear goggles while operating GC.

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The dehydration product may contain a mixture of methylene cyclohexane, 3-methylcyclohexene, and 1-methylcyclohexene. If the temperature of the Hickman still was allowed to rise above 150oC, the product may also contain starting 2-methylcyclohexanol. If the product was not properly dried, the sample may also contain water. A dry clean sample should only contain the three alkenes.

The percentage of each component will be determined by GC.

Be very careful not to bend or break the GC syringe. These are fragile and expensive. Record the instrument number and instrument operating conditions (column temperature, injector temperature, detector temperature, column type, flow rate, and sample size) in your laboratory notebook.

Components elute on carbowax GC columns primarily based on boiling point. Secondarily materials elute based on polarity. Tentative identity of each component can be made based on boiling point. The peak identification should be verified by comparing to a standard sample. If two injections are made of the same compound on the same GC machine under the same operating parameters, the two resulting peaks will have the same retention time within experimental error. The amount of each component present is determined either automatically by the integrator connected to the GC or manually following the procedure outlined in the notes.

GOW-MAC GC Operating procedure

1. Carefully rise syringe 2-3 times with sample by pulling up approximately 5 μl of sample and dispensing on kim wipe.

2. Carefully pull up approximately 4 μl of sample into syringe.

3. Being careful to keep syringe level and perpendicular, insert needle into desired port (A). Do not bend needle. Insert syringe until glass barrel almost touches metal port ring. Depress plunger then immediately pull syringe out and hit "START" on integrator.

4. Wait until all desired peaks have eluted.(The next person can clean and load syringe while previous sample is coming off column.)

5. When all peaks have eluted, press "STOP" on integrator. Write name by chromatograph. After everyone has gone, the chromatograms will be cut up and distributed.
(Do not pull or tear paper. This causes the integrator to jam).

6. The next person can inject their sample.

7. The last person to inject sample, be sure to rinse the syringe 2-3 times with acetone before returning to styrofoam tray.

POST-EXPERIMENT ASSIGNMENT

Turn in the lab report at the beginning of the next lab period. Prepare for the upcoming quiz on Gas Chromatography.