

# Gas Chromatography

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## PURPOSE

The purposes of this experiment are to:

- Introduce the student to the instrument called a gas chromatograph
- Train the student to place the instrument into operation
- Train the student to interpret the data obtained
- Train the student to remove the instrument from operation

## MATERIALS

The materials needed to perform this experiment are:

- Gas chromatograph, complete with carrier gas and recorder
- Injection syringe
- Sample

## PROCEDURE

### A. Introduction

A gas chromatograph derives its name from the ability to separate the components of a mixture utilizing a gas to force the mixture through a column that is packed with an adsorbing material. As the sample travels through the column individual components will separate and exit the instrument at different times.

The following list represents a brief description of the major components of a typical gas chromatograph. The student will be required to physically identify each of the components of the gas chromatograph.

- (1) Carrier gas- the gas utilized to force the sample through the instrument. The two most commonly used carrier gases are helium and nitrogen. The carrier gas needs to be relatively inert as well as inexpensive.
- (2) Flow regulator- this device regulates the pressure coming from the carrier gas cylinder, usually very high, down to a much lower level suitable for use by the instrument. The regulator allows the pressure to be increased or decreased within limited parameters.
- (3) Injector port- this consists of a rubber septum very similar to what is found in a bottle of insulin. The sample is injected into the instrument in much the same way that an individual is given an injection of insulin.
- (4) Columns- two columns usually in the shape of a coil are found inside the oven cavity of the gas chromatograph where they bridge the gap between the injection port and the detector block. The columns are usually made from stainless steel, glass or aluminum.

- (5) Oven- a heated cavity that houses the columns and the detector block. The temperature of the oven is adjustable to provide a suitable temperature for analysis of a particular sample.
- (6) Detector block- contains the thermal conductivity detector. The carrier gas and the sample pass over the detector as they exit the column.
- (7) Recorder-provides a record of the passage of the sample over the filaments in the detector block.

## **B. Control Measures**

Most of the components of the gas chromatograph listed above also provide some degree of control over the operation of the instrument. This allows the operator to improve performance by improving separation of the sample and improving the quantitative results.

- (1) Flow rate: the rapidity with which the carrier gas forces the sample through the columns determines to a large degree the amount of time that the sample spends in contact with the packing material of the column. Generally, the longer the length of interaction the better the separation of the mixture. Reducing the flow rate will assist in the separation of peaks that are located very close to one another.
- (2) Columns: the longer the column the better the separation of the components of the mixture. Naturally, there is a limit as to how long a column can be and still fit within the confines of the oven cavity. In addition, the smaller the diameter of the column the better the resolution of the mixture.
- (3) Adsorbing material: the packing material inside a column is very important in achieving separation of the mixture. If the packing were a polar substance then it would be used for separating the polar components of a sample. If the packing were a non-polar substance then it would be more efficient in separating non-polar components.
- (4) Oven: generally, the higher the temperature the poorer the separation. However, there is a minimum temperature required to achieve instant vaporization when the sample is injected into the chromatograph. This minimum temperature is about 20 °C above the highest boiling component in the sample. The maximum temperature that the oven can be heated is dependent upon the packing material. If the oven is heated too hot the packing material will volatilize off the column and render the column useless.
- (5) Detector block: the sensitivity of the thermal conductivity detector is directly related to the amperage that is applied to the filament in the detector block. Care must be taken not to heat the filaments in the absence of the carrier gas that serves to cool the filaments. In addition, the filaments must not be heated hotter than the carrier gas is able to remove the heat. Otherwise, the filaments will burn out.
- (6) Attenuation determines the relative sensitivity of the recorder pen. At high levels of attenuation the pen is relatively unresponsive to small sample components. At lower attenuation settings the pen becomes progressively more responsive.

- (7) Recorder: the sensitivity and efficiency of the recorder is controlled by two factors: (a) the chart speed, and, (b) the pen deflection. Higher chart speeds facilitate the separation of two peaks that are located very close together. Full-scale deflection of the pen helps ensure the minor components of the sample are visible on the print out.

### C. Operation

- (1) Pre-operational checks:
- (a) Rotate filament current rheostat fully counter-clockwise to avoid damage when the power is turned on.
  - (b) Turn off the toggle switches that control the filament current.
  - (c) Rotate the oven temperature control fully counter-clockwise.
  - (d) Rotate the attenuation control clockwise to a setting of "32" or higher.
- (2) Operational procedures:
- (a) Turn on the carrier gas at the tank and adjust the pressure regulator to indicate a pressure of about 40 psi.
  - (b) Adjust the flow rate of the carrier gas until 30-60 ml/min is obtained.
  - (c) Turn on the main power switch.
  - (d) Turn on the oven and set to "50". This means the heating element of the oven is on 50 % of the time. Monitor the oven temperature until a temperature about 20 °C above the highest boiling component in the mixture is obtained.
  - (e) Turn on the recorder but disengage the drive in order to conserve paper until you are ready to inject a sample.
  - (f) Once the oven temperature has stabilized turn the attenuation down to at least an "8". Use the zero adjustments found on the recorder and the chromatograph to bring the pen of the recorder to the baseline.
  - (g) Using a 1.0 µl syringe inject a 0.5 µl sample into the chromatograph. This first injection is designed to determine how many components are in the sample and how much of each is present. If one or more peaks extend beyond the top of the chart paper and increase in attenuation is necessary. If none of the peaks reach nearly full scale then a decrease in attenuation is needed. If two or more peaks are located close to each other then an increase in chart speed or a decrease in flow rate will be required in order to achieve a separation.
- (3) Post Operational Checks:
- (a) Turn off the recorder and replace the cap on the pen.
  - (b) Increase the attenuation to "32"
  - (c) Turn off the filament current and rotate the filament rheostat fully counter-clockwise.
  - (d) Turn off the main power switch and rotate the oven rheostat counter-clockwise.
  - (e) Raise the oven door and allow the temperature to drop to 75 °C before turning off the carrier gas. The carrier gas should only be turned off by using the main valve located on the gas cylinder and not by using the needle valves.

- (f) Clean up the syringe and area around the instrument.

## RESULTS

The purpose of the chromatograph is to provide information that will allow the operator to determine how many components are in a sample and the relative concentrations of each component. The number of components in a sample is easily determined by counting the number of peaks that are recorded. Determining the relative concentration of each component is more difficult and requires some calculations.

### A. Expedient Method

The easiest method of determining the concentration of the components is to compare the areas of each peak to the total area for all of the peaks. Peak area is determined by approximating the peaks to be triangles. The area of a triangle is calculated by the equation  $A = \frac{1}{2}bh$ , where  $b$  is the base and  $h$  is the height. The area of each peak is determined and added together in order to obtain the total area. The percentage of each component in the mixture is calculated by dividing the area of each peak by the total area and multiplying by 100.

### B. Precise Method

Because the detector responds differently to various components of the mixture there exists an inherent error in the peaks produced by the recorder. This error can be eliminated through the application of a correction factor. The peak areas associated with the sample are determined in the same manner as in part "(a)" above. Then a standard sample containing a 1:1 mixture of the same components is injected into the chromatograph and peak areas determined. Each peak area in the 1:1 mixture is divided by the smallest of the peaks. The resulting correction factor is then used for correcting the areas obtained in the sample being studied.

## CONCLUSIONS

The gas chromatograph is a quantitative tool that is used to identify the relative percentage of each substance in a sample as well as how many substances are in a sample. The operator is able to determine the number of components that are in a sample as well as their relative abundance. Using the chromatograph, the operator is able to determine whether or not the composition of a solution is being maintained.

