An Introduction to the Gas Chromatograph: Collecting a chromatogram of clove oil

Objective

To familiarize ourselves with the operation of a gas chromatograph (GC) and to determine if there is more than one component present in clove oil isolated in lab.

Background

A gas chromatograph is an instrument that is used to separate mixtures of volatile organic compounds into their component compounds and to determine the relative amounts of the components present in the mixture, or if an internal standard is used the actual amounts of materials present in a mixture. A graphical representation of the parts of a GC can be seen in figure 1.

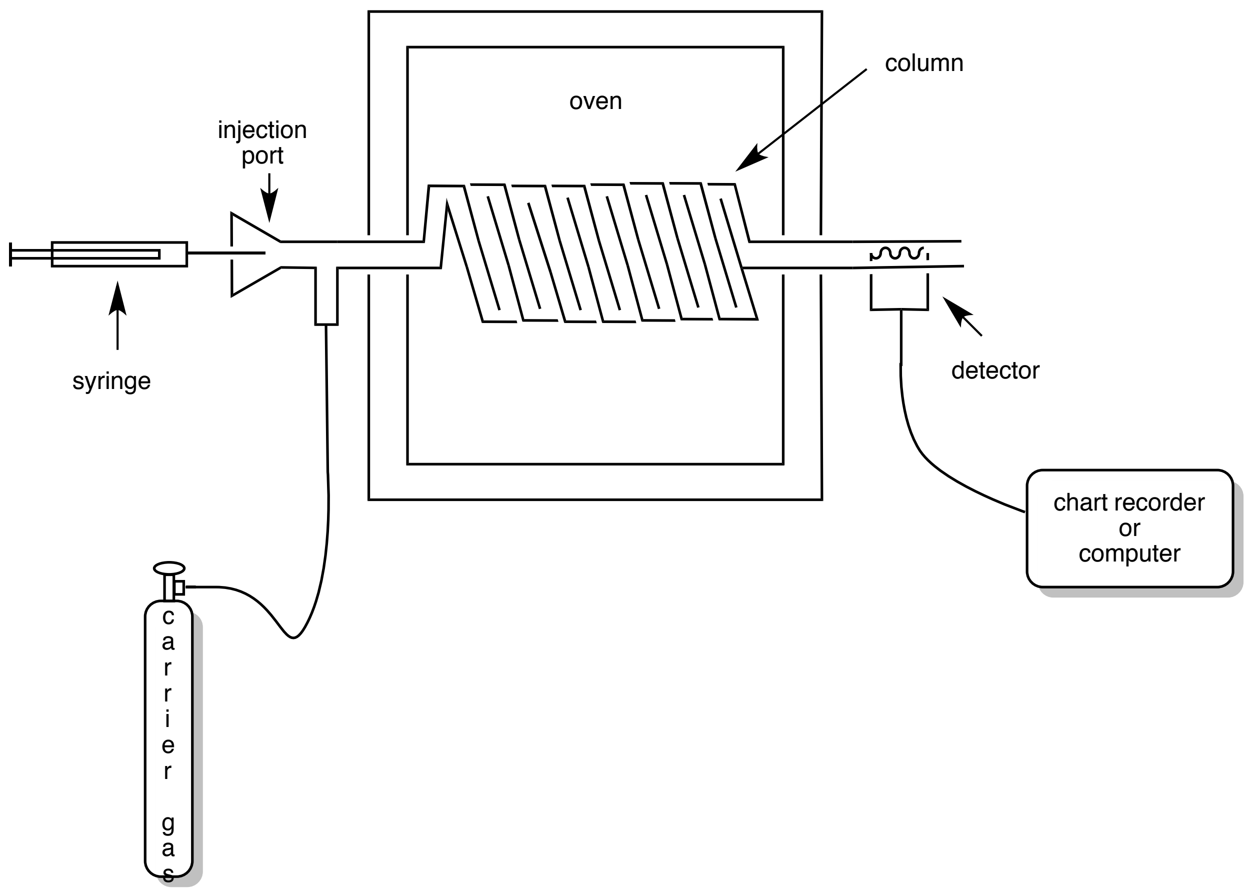
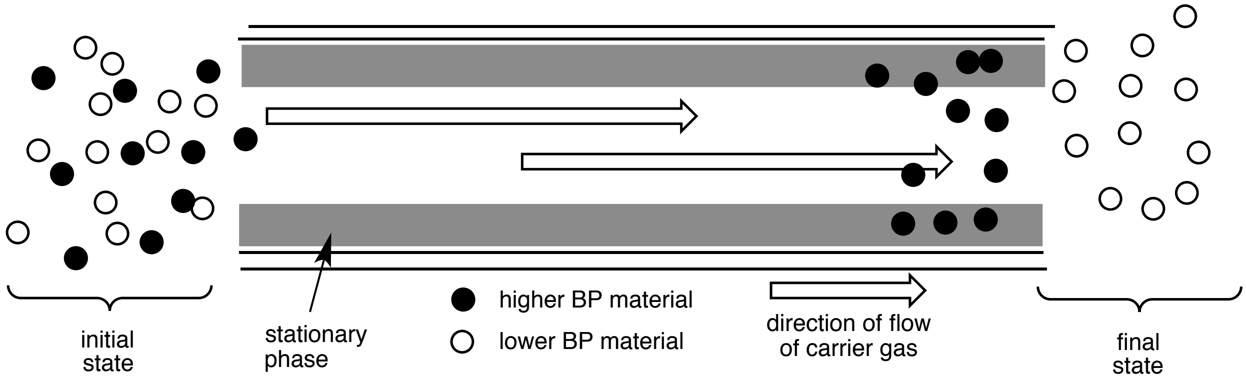


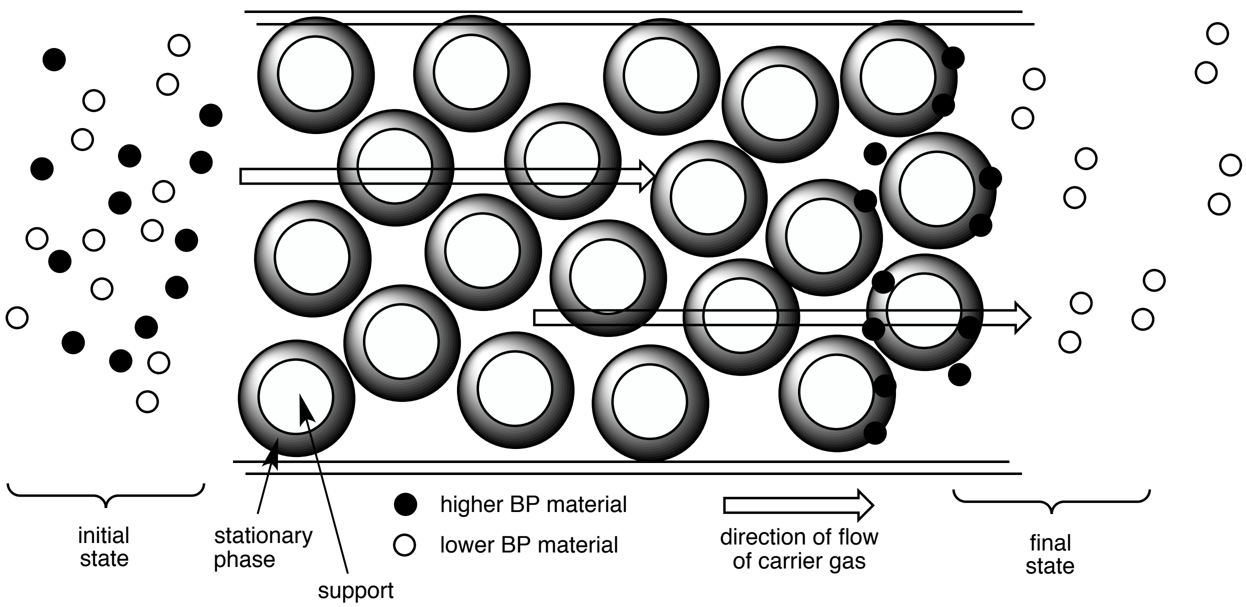
Figure 1

A syringe is used to inject a sample into the instrument though an injection port. When the sample enters the injection port, it is vaporized. The carrier gas — typically helium is used and the carrier gas is constantly traveling from the injection port, though the column, past the detector, and out the vent — carries the evaporated organic materials onto the column.

Some GC's use direct, on-column injection, and in that case, the liquid material is placed directly on the column. Separation of the components in the mixture is then accomplished based on the amount of time each component spends on the stationary phase and the the amount of time the component spends in the mobile phase, the carrier gas.



Capillary or Megabore Column Based on Diameter



Packed Column

Figure 2

The stationary phase can be a thin waxy or liquid coating along the inside lining of the column or a thin waxy or liquid layer suspended on beads inside the GC column. In general, though not always, the stationary phase is similar to the materials that are to be separated. For example, if nonpolar materials are to be separated, a nonpolar stationary phase is used. Likewise, if one wishes to separate polar substances, polar stationary phases are typically used. When the stationary phase and the compounds to be separated are similar, the components of the mixture are separated based on their boiling points.

In the column of a GC, materials with high boiling points will tend to stay stuck[1] to the stationary phase. Materials with higher boiling points don't evaporate as easily, so they spend more time sorbed[2] to the stationary phase. Materials with lower boiling points will tend to desorb and leave the stationary phase more quickly. When materials leave the stationary phase, they move into the mobile phase, the carrier gas, and the materials are moved along or "down" the column. The more time a component spends on the stationary phase the more slowly it moves down the column. Similarly, the more time a component spends in the mobile phase, the faster it moves down the column. Thus, if the appropriate conditions are chosen, the materials can be separated because one component will move down the column faster than the others (see figure 2).

A variety of detectors are available on GC's, and our GC's use thermal conductivity detectors (TCD). A TCD relies on the observation that different materials conduct heat differently. For example, aluminum is an excellent conductor of heat, whereas glass is a much less effective conductor of heat. In any event, the carrier gas that is flowing over a filament of the TCD constantly cools the wire. When a component of the mixture passes over the filament, the thermal conductivity of the gas changes. Because the thermal conductivity of the gas changes, the temperature and the electrical properties of the wire change. This change is recorded by a chart recorder or a computer. The benefit to using a TCD is that they are sturdy and simple to operate. The drawback is that they are not particularly sensitive, and they may not be able to identify comments that are present at low concentrations.

Procedure

Prepare your Clove Oil Sample

Only 1 µL of solution is needed for injection into the gas chromatograph, so keep that in mind when preparing the clove oil sample.

1. Making certain that there are no solids present in the clove oil collected by co-distillation, dilute the sample of clove oil with methanol in a 10:1 methanol:clove oil ratio. For example, 0.4000 g of methanol combined with 0.0400 would be more than enough solution for analysis. It is easiest to measure out a small amount of clove oil first, and add the appropriate amount of methanol to the oil.

2. Cap the vial that contains the methanol-clove oil to prevent the methanol from evaporating.

Collect a Chromatogram

1. Under the supervision of the instructor, inject 1.0 µL of the methanol clove oil mixture into the gas chromatograph..

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[1] The analyte may be adsorbed onto or absorbed into the stationary phase.

[2] "Sorbed" means that the material could be absorbed or adsorbed. Whether the material is adsorbed or adsorbed depends on the conditions, the stationary phase, and the material.

Determine the Number of Components Identified and Their Relative Yield[3]
To simplify our calculation, we will assume that the response rate for each component is the same; that is, we are going to assume that the TCD responds identically to each component. This assumption means that if the sample were a 50/50 mixture of two components, then the areas under each of the two peaks would be equal, and thus, half of the total area of all of the component peaks.

1. Find the peaks in the chromatogram that correspond to the components of the analyte. The first peak in the chromatogram will be the solvent, methanol.

2. Excluding the methanol peak, find the total area reported by the computer under all of the component peaks. Using the areas listed for each of component peaks, determine the percentage of each component present in the sample.

Report

An "experimental" describing the procedure that you followed is not required for this activity. For this activity a typed report that contains the following information is required:

- Report the amount of ground cloves used,
- Report the amount of clove oil collected
- Report the percent yield for the oil. In this calculation you are comparing the mass of the oil collected to the mass of the ground cloves used
- Report the appearance of the clove oil
- Report the % yield of each of the clove oil components
- Attach a copy of the chromatogram and a copy of the infrared spectrum

[3] It should be noted that due to the limitations of our method and the thermal conductivity detector we will not be able to detect all of the components of clove oil. See *Food Chemistry*, **101** (2007) 1558–1564 for more information on the composition of clove oil.