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CH 362  
Professor Attygalle  
I pledge my honor that I have abided by the Stevens Honor System

**1) Title of Experiment:**  
Separation of Hydrocarbons and Determination of Ethanol in Alcoholic Beverage With Gas Chromatography

Date: November 7, 2020  
Name of Technique: Gas Chromatography

**2) Technique:**

Gas chromatography is a subset of chromatography, which analyzes samples by utilizing a physical method for separating mixtures. Chromatography as a whole involves two phases in contact with each other. One of these phases is called the mobile phase, that carries analytes in the system to the from the beginning to the end of the apparatus, which is usually a detector. The other phase is called the stationary phase, which causes the analytes to move slower through the apparatus. The rate at which the different analytes move through the apparatus is dependent on its chemical properties, therefore separating different chemical compounds inside a mixture.

In gas chromatography, a carrier gas is used as the mobile phase, while a lined column is used as the stationary phase. The setup for this apparatus is a heated injector, a precisely temperature-controlled oven, and a detector that responds to analytes reaching the end of the column. This setup is detailed in Figure 1. To analyze a sample, the sample is pushed into the column with the injector, which vaporizes the solution. As the carrier gas is pumped through the column with the analyte, some compounds of the mixture interact with the stationary phase and slows down inside the column. Therefore, different compounds will separate and reach the detector at different times. The detector then measures the intensity of the compound when it reaches the end of the column, and the time it took to reach there, called the retention time.

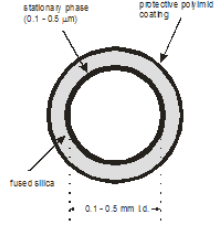
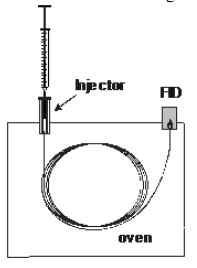


Figure : Gas Chromatograph (left) and Inside of the Column (right)

**3) Application of the Technique to my Experiment:**

For the first part of this experiment, a known hydrocarbon will be given, along with a mixture of normal hydrocarbons from n-nonane to n-hexadecane and an unknown normal hydrocarbon. Before the procedure begins, the column parameters, carrier gas flow rates, and temperature programs of the gas chromatography setup will be noted. To begin, gas chromatography will be conducted with a 1 µL sample of the known hydrocarbon sample, by injecting the sample through the heated injector and using the machine to record the chromatogram. This same process will be repeated with 1 µL of the mixture of hydrocarbons, three times, as well as the unknown sample, once, recording the chromatogram for each trial. Finally, the retention times and area of each peak will be recorded for each chromatogram.

Using the retention times for each peak, and adjusting them for the peak of the solvent, the retention indexes and the adjusted retention times in reference to the known hydrocarbon can be used to identify the peaks of each compound in the unknown, as well as the unknown compound. Furthermore, the area under each peak can be related to the amount of mixture injected into the apparatus, and a mean response per microliter can be recovered.

The second part of this experiment involves determining the unknown concentration of ethanol in an alcoholic beverage. First, five 50 mL calibration solutions of ethanol will be prepared, of 30%, 40%, 50%, 60%, and 70% v/v concentrations from ethanol and deionized water. Then, a 1.00 mL aliquot of each calibration solution will be transferred to separate 3 mL GC vials, to which 0.25 mL of 1-propanol will be added. 5 µL of each calibration solution will be injected into the GC system, and chromatograms will be recorded for each solution. Then, chromatograms for 5 µL of the alcoholic beverage will also be recorded. The alcoholic beverage trial will be repeated twice more, for a total of three times.

Then, because the intensity of the response is based on the concentration of the substance, integrating the area of the peak correlated with each ethanol calibration solution will recover an RRF value, which is known as the response factor, calculated by dividing peak area by concentration. Using this and the peak area of ethanol in the alcoholic beverage can recover the unknown concentration of alcohol in the beverage.

**4) Calculations:**

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Concentration of Ethanol Solution (% v/v) | Volume of solution (mL) | Volume of ethanol (mL) | Volume of water (mL) | Concentration of ethanol in GC vial (% v/v) | Concentration of 1-propanol in GC vial (% v/v) |
| 30 | 50 | 15 | 35 | 24 | 20 |
| 40 | 50 | 20 | 30 | 32 | 20 |
| 50 | 50 | 25 | 25 | 40 | 20 |
| 60 | 50 | 30 | 20 | 48 | 20 |
| 70 | 50 | 35 | 15 | 56 | 20 |

**5) References:**

1. Attygalle, A. Instrumental Analysis I Lecture and Laboratory Manual <https://sit.instructure.com/courses/38802/files/6982711?module_item_id=1042514> (accessed Nov 7, 2020).
2. Detector <https://www.shimadzu.com/an/service-support/technical-support/analysis-basics/fundamentals/detector.html#6_3_1_FID> (accessed Nov 7, 2020).
3. Difference between Gas Solid Chromatography and Gas Liquid Chromatography <https://www.easybiologyclass.com/difference-between-gas-solid-chromatography-and-gas-liquid-chromatography-comparison-table/> (accessed Nov 7, 2020).
4. Harris, D. C. *Quantitative Chemical Analysis*, 8th ed.; W.H. Freeman and Co: New York, 2010. Chapter 23.

**6) MSDS:**

**n-Nonane:**

CAS No.: 111-84-2  
Molecular Weight: 128.26  
Chemical Formula: C9H20  
Appearance: colorless liquid  
Lab Protective Equipment: Lab coat, goggles  
Flammable liquid and vapor

**Health effects:**May be fatal if swallowed and enters airways, causes skin irritation and serious eye irritation. May cause respiratory irritation.

**First Aid measures:**Eye contact: rinse cautiously with water, for several minutes. Remove contact lenses if present and easy to do. Continue rinsing, and if irritation persists, contact a doctor.  
Skin contact: wash off immediately with plenty of water. Get medical attention if skin irritation or rash occurs. Remove contaminated clothing.  
Inhalation: Move to fresh air and put in comfortable breathing position. Consult a physician if feeling unwell.  
Ingestion: Call a poison control center or doctor. Do not induce vomiting.

**Other hazards:**Fire: flammable liquid and vapor.   
Explosion: vapors may form explosive mixtures with air.

**n-Decane:**

CAS No.: 124-18-5  
Molecular Weight: 142.28  
Chemical Formula: C10H22  
Appearance: colorless liquid  
Lab Protective Equipment: Lab coat, goggles  
Flammable liquid and vapor

**Health effects:**May be fatal if swallowed and enters airways, causes skin irritation and serious eye irritation. May cause respiratory irritation.

**First Aid measures:**Eye contact: rinse immediately with water, also under eyelids, for at least 15 minutes. Get medical attention.  
Skin contact: wash off immediately with plenty of water for at least 15 minutes. Get medical attention if skin irritation or rash occurs.  
Inhalation: Move to fresh air and put in comfortable breathing position. Consult a physician if feeling unwell. Risk of serious damage to lungs. If not breathing, give artificial respiration with aid of a pocket mask with one-way valve.  
Ingestion: Call a poison control center or doctor. Do not induce vomiting.

**Other hazards:**Fire: flammable liquid and vapor.   
Explosion: vapors may form explosive mixtures with air.

**n-Undecane:**

CAS No.: 1120-21-4  
Molecular Weight: 156.31  
Chemical Formula: C11H24  
Appearance: colorless liquid  
Lab Protective Equipment: Lab coat, goggles  
Flammable liquid and vapor

**Health effects:**May be fatal if swallowed and enters airways, causes skin irritation and serious eye irritation. May cause respiratory irritation.

**First Aid measures:**Eye contact: rinse immediately with water, also under eyelids, for at least 15 minutes. Get medical attention.  
Skin contact: wash off immediately with plenty of water for at least 15 minutes. Get medical attention if skin irritation or rash occurs.  
Inhalation: Move to fresh air and put in comfortable breathing position. Consult a physician if feeling unwell. Risk of serious damage to lungs. If breathing is difficult, give oxygen.  
Ingestion: Clean mouth with water and drink plenty of water afterwards. Call a poison control center or doctor. Do not induce vomiting.

**Other hazards:**Fire: flammable liquid and vapor.   
Explosion: vapors may form explosive mixtures with air.

**n-Dodecane:**

CAS No.: 1120-21-4  
Molecular Weight: 170.34  
Chemical Formula: C12H26  
Appearance: colorless liquid  
Lab Protective Equipment: Lab coat, goggles  
Flammable liquid and vapor

**Health effects:**May be fatal if swallowed and enters airways, causes skin irritation and serious eye irritation. May cause respiratory irritation.

**First Aid measures:**Eye contact: rinse immediately with water, also under eyelids, for at least 15 minutes. Get medical attention.  
Skin contact: wash off immediately with plenty of water for at least 15 minutes. Get medical attention if skin irritation or rash occurs.  
Inhalation: Move to fresh air and put in comfortable breathing position. Consult a physician if feeling unwell. Risk of serious damage to lungs. If not breathing, give artificial respiration with aid of a pocket mask with one-way valve.  
Ingestion: Aspiration hazard. Call a poison control center or doctor. Do not induce vomiting.

**Other hazards:**Fire: flammable liquid and vapor.   
Explosion: vapors may form explosive mixtures with air.

**n-Tridecane:**

CAS No.: 629-50-5  
Molecular Weight: 184.36  
Chemical Formula: C13H28  
Appearance: colorless liquid  
Lab Protective Equipment: Lab coat, goggles  
Flammable liquid and vapor

**Health effects:**May be fatal if swallowed and enters airways, causes skin irritation and serious eye irritation. May cause respiratory irritation.

**First Aid measures:**Eye contact: rinse immediately with water, also under eyelids, for at least 15 minutes. Get medical attention.  
Skin contact: wash off immediately with plenty of water for at least 15 minutes. Get medical attention if skin irritation or rash occurs.  
Inhalation: Move to fresh air and put in comfortable breathing position. Consult a physician if feeling unwell. Risk of serious damage to lungs. If not breathing, give artificial respiration.  
Ingestion: Clean mouth with water and drink plenty of water afterwards. Call a poison control center or doctor. Do not induce vomiting.

**Other hazards:**Fire: flammable liquid and vapor.   
Explosion: vapors may form explosive mixtures with air.

**n-Tetradecane:**

CAS No.: 629-59-4  
Molecular Weight: 198.39  
Chemical Formula: C14H30  
Appearance: colorless liquid  
Lab Protective Equipment: Lab coat, goggles  
Flammable liquid and vapor

**Health effects:**May be fatal if swallowed and enters airways, causes skin irritation and serious eye irritation. May cause respiratory irritation.

**First Aid measures:**Eye contact: rinse immediately with water, also under eyelids, for at least 15 minutes. Get medical attention.  
Skin contact: wash off immediately with plenty of water for at least 15 minutes. Get medical attention if skin irritation or rash occurs.  
Inhalation: Move to fresh air and put in comfortable breathing position. Consult a physician if feeling unwell. Risk of serious damage to lungs. If not breathing, give artificial respiration with aid of a pocket mask with one-way valve.  
Ingestion: Aspiration hazard. Call a poison control center or doctor. Do not induce vomiting.

**Other hazards:**Fire: flammable liquid and vapor.   
Explosion: vapors may form explosive mixtures with air.

**n-Pentadecane:**

CAS No.: 629-62-9  
Molecular Weight: 212.42  
Chemical Formula: C15H32  
Appearance: colorless liquid  
Lab Protective Equipment: Lab coat, goggles  
Flammable liquid and vapor

**Health effects:**May be fatal if swallowed and enters airways, causes skin irritation and serious eye irritation. May cause respiratory irritation.

**First Aid measures:**Eye contact: rinse immediately with water, also under eyelids, for at least 15 minutes. Get medical attention.  
Skin contact: wash off immediately with plenty of water for at least 15 minutes. Get medical attention if skin irritation or rash occurs.  
Inhalation: Move to fresh air and put in comfortable breathing position. Consult a physician if feeling unwell. Risk of serious damage to lungs. If not breathing, give artificial respiration with aid of a pocket mask with one-way valve.  
Ingestion: Aspiration hazard. Call a poison control center or doctor. Do not induce vomiting.

**Other hazards:**Fire: flammable liquid and vapor.   
Explosion: vapors may form explosive mixtures with air.

**n-Hexadecane:**

CAS No.: 544-76-3  
Molecular Weight: 226.44  
Chemical Formula: C16H34  
Appearance: colorless liquid  
Lab Protective Equipment: Lab coat, goggles  
Flammable liquid and vapor

**Health effects:**May be fatal if swallowed and enters airways, causes skin irritation and serious eye irritation. May cause respiratory irritation.

**First Aid measures:**Eye contact: rinse immediately with water, also under eyelids, for at least 15 minutes. Get medical attention.  
Skin contact: wash off immediately with plenty of water for at least 15 minutes. Get medical attention if skin irritation or rash occurs.  
Inhalation: Move to fresh air and put in comfortable breathing position. Consult a physician if feeling unwell. Risk of serious damage to lungs. If not breathing, give artificial respiration.  
Ingestion: Clean mouth with water and drink plenty of water afterwards. Call a poison control center or doctor. Do not induce vomiting.

**Other hazards:**Fire: flammable liquid and vapor.   
Explosion: vapors may form explosive mixtures with air.

**1-propanol:**

CAS No.: 71-23-8  
Molecular Weight: 60.10  
Chemical Formula: C3H8O  
Appearance: colorless liquid  
Lab Protective Equipment: Lab coat, goggles  
Flammable liquid and vapor

**Health effects:**May be fatal if swallowed and enters airways, causes skin irritation and serious eye irritation. May cause respiratory irritation.

**First Aid measures:**Eye contact: rinse immediately with water, also under eyelids, for at least 15 minutes. Get medical attention.  
Skin contact: wash off immediately with plenty of water for at least 15 minutes. Get medical attention if skin irritation or rash occurs.  
Inhalation: Move to fresh air and put in comfortable breathing position. Consult a physician if feeling unwell. If not breathing, give artificial respiration.  
Ingestion: Give victim 2-4 cupfuls of water or milk. Never give anything by mouth to an unconscious person. Call a poison control center or doctor. Do not induce vomiting.

**Other hazards:**Fire: flammable liquid and vapor.   
Explosion: vapors may form explosive mixtures with air.

**Ethanol:**

CAS No.: 64-17-5  
Molecular Weight: 46.0414  
Chemical Formula: C2H5OH  
Appearance: colorless liquid  
Lab Protective Equipment: Lab coat, goggles  
Flammable liquid and vapor

**Health effects:**May be fatal if swallowed and enters airways, causes skin irritation and serious eye irritation. May cause respiratory irritation.

**First Aid measures:**Eye contact: rinse immediately with water, also under eyelids, for at least 15 minutes. Get medical attention.  
Skin contact: wash off immediately with plenty of water for at least 15 minutes. Get medical attention if skin irritation or rash occurs.  
Inhalation: Move to fresh air and put in comfortable breathing position. Consult a physician if feeling unwell. If not breathing, give artificial respiration.  
Ingestion: Give victim 2-4 cupfuls of water or milk. Never give anything by mouth to an unconscious person. Call a poison control center or doctor. Do not induce vomiting.

**Other hazards:**Fire: flammable liquid and vapor.   
Explosion: vapors may form explosive mixtures with air.

**7) Pre-lab questions:**

1. 1. Chromatography is the practice of separating a mixture by passing it through a medium at which components move at different rates.
   2. A chromatograph is the apparatus or setup used to carry out chromatography. In gas chromatography, for example, the chromatograph involves the injector, the column, the oven, and the detector.
   3. A chromatogram is the diagram showing the result of the chromatography procedure that details the intensity of the peaks recorded by the detector over time.
   4. A chromatographer is a person who is skilled at chromatography or the person who is carrying out chromatography by operating a chromatograph.
2. Gas-liquid chromatography (GLC) is when the stationary phase is a liquid coating the column, while gas-solid chromatography (GSC) is when the stationary phase is a solid, where the liquid coating the column is absent. While in both versions, a carrier gas is used as the mobile phase and both are used for the separation of volatile compounds and mixtures. The differences include that GSC is an adsorption technique which relies on interaction between the analyte and the stationary phase, while GLC is a partition technique which relies on the partitioning between the analyte and the stationary phase, as they are both liquids. Retention times in GLC are generally shorter than they are in GSC, and GLC requires lower temperatures in the oven to keep the analyte in liquid phase compared to GSC. GSC also uses packed columns, while GLC uses capillary columns, which are two differing types of columns used to pipe the analyte between the injector and the detector. Packed columns are wider and shorter than capillary columns, and are packed with adsorbent material compared to capillary columns, which are lined with liquid stationary phase.
3. Flame ionization detection relies on the flammable properties of the analyte to detect when they reach the detector at different times. When the flammable compound, usually hydrocarbons and other organic compounds, reach the detector, they are aspirated into the flame, which causes an ionization reaction, which is detected a collector electrode into an electrostatic field, which is measured by a computer and recorded. Thermal conductivity detectors take advantage of the different thermal conductivities of gases, as carrier gases like helium and hydrogen have high thermal conductivities, while other samples might have lower conductivities. When those gases pass through a filament, the temperature decreases, which causes a change in voltage across the filament, which is measured by a computer. This technique can be used to detect inflammable compounds not compatible with flame ionization detection.
4. Concentration of 1-propanol and ethanol are found in the calculations table.