Experiment 10: Gas Chromatography and Identification of an Unknown

Chemistry 113 Lab Preparation Form

Name_____ Lab Section_____

Purpose of Experiment:

Objectives:

Key Terms:

Chromatography -

Chromatogram -

Moving Phase -

Stationary Phase -

Gas Chromatography -

Retention Time -

Calculations:

Safety Warnings:

Procedure Notes:

Questions before starting experiment?

Comments from Briefing:

Experiment 10: Gas Chromatography and Identification of an Unknown

Prelab Exercise

Name_____ Lab Section_____

1. From a mixture, on what basis can the substances be separated using the technique of chromatography?

Experiment 10: Gas Chromatography and Identification of an Unknown

Introduction:

Chromatography is a technique used to separate and identify small quantities of mixtures into their component parts. This technique was so named due to the highly colored (Gk., chroma; colored) components separated from chloroplast extracts when this technique was first used in 1906 by M. Tswett..

All forms of chromatography employ two phases; a *moving phase* and a *stationary phase*. The moving phase is a solvent, or mixture of solvents, that flows over the other material, the stationary phase. In chromatography, mixtures can be separated because each substance in the mixture will have different affinities for the stationary and moving phases. When the correct stationary and moving phases are selected the mixture separates because:

each substance in the mixture is adsorbed on the stationary phase with a different degree of tenacity; and

each substance in the mixture has a different affinity for the moving phase.

A compound with a high affinity for the moving phase and a low affinity for the stationary phase will move through the *stationary phase* rapidly. A compound with a high affinity for the stationary phase and a low affinity for the moving phase will move through the *stationary phase* slowly.

Gas chromatography utilizes a gas as the moving phase. The carrier gas for the GC's in lab this week is Helium. There is a column containing a stationary phase that the carrier gas passes through. A mixture is injected with a syringe and is carried to the stationary phase by the carrier gas. The closer a component in a mixture is to the stationary phase, the more time it will spend on the stationary phase this means it will take longer for the substance to exit the column and be detected.

This time required for the mobile phase to move a solute from its injection point, through the stationary phase and to the detector is the *retention time*. Retention time can be used to identify components of a mixture by comparing a solute's retention time with that of a known compound. If the retention times are different, the compounds are different. If the retention times are the same, there is evidence that the compounds are the same.

When trying to positively identify a component of a mixture more information is needed. This can be obtained in several ways. One, several different types of stationary

phase/ mobile phase combinations can be used. Usually three different combinations offer sufficiently different conditions to be able to determine the identity of the unknown. It is extremely unlikely that two different chemicals will respond the same under the three different conditions. Two, extra information can be obtained. For example, the detector can be a mass spectrometer or an infrared spectrometer that can provide extra information about the identity of the component. To keep this experiment as simple as possible, we will not be using these extra techniques.

Experimental (work in groups):

The following should be located at the back of the laboratory room.

- syringe
- 3 GC's labeled GC#1, GC#2, and GC#3
- 1 computer with data acquisition software
- 1 printer
- 1 sample vial with 1-propanol
- 1 sample vial with 2-propanol
- 1 sample vial with iso-butanol (also known as 2-methyl-1-propanol)
- 1 sample vial with a mixture of 1-propanol, 2-propanol, iso-butanol
- 1 sample vial with your unknown (note: your unknown is some combination of one or more of 1-propanol, 2-propanol, iso-butanol)

The class will be broken into 3 groups. Each group will be assigned to a specific GC. When using the computer, it will reference your GC by its number. For example, GC#1 will be referenced as GC 1 or Channel 1.

Your lab instructor will demonstrate how to use the computer to acquire data from the GC, how to make an injection into the GC and how to print out the results of your trials. Each trial requires 8 minutes to complete. You will need to make efficient use of your time. When finished with each trial, print out enough chromatograms for each member of your group to have one for their laboratory report. Make sure each chromatogram is labeled with exactly what is injected.

Trial 1: Inject 2 μL of 1-propanol into the GC and start data collection for that GC at the same time. Print out the chromatograms.

Trial 2: Inject 2 μL of 2-propanol into the GC and start data collection for that GC at the same time. Print out the chromatograms.

Trial 3: Inject 2 μL of iso-butanol into the GC and start data collection for that GC at the same time. Print out the chromatograms.

Trial 4: Inject 4 μL of the mixture of 1-propanol, 2-propanol, iso-butanol into the GC and start data collection for that GC at the same time. Print out the chromatograms. You should be able to identify each of the components of the mixture by its retention time.

Trial 5: Inject 4 μL of your unknown into the GC and start data collection for that GC at the same time. Print out the chromatograms. Identify the unknown based upon the retention time(s)

You will have to turn in a copy of the chromatograms.

Experiment 10: Gas Chromatography and Identification of an Unknown

Data Sheet-1

Name_____ Lab Section_____

Lab Instructor_____ Lab Partner(s)_____

Unknown ID: _____

Boiling Point Data(use reference material like the Merk index or the CRC Handbook)

- 1-propanol _____°C
- 2-propanol _____°C
- iso-butanol _____°C

Reference used:

Retention time: (include units)

- 1-propanol _____
- 2-propanol _____
- iso-butanol _____
- mixture:
    - 1-propanol _____
    - 2-propanol _____
    - iso-butanol _____
- Unknown _____
  _____
  _____

Experiment 10: Gas Chromatography and

Identification of an Unknown

Postlab Exercise

Name_____ Lab Section_____

1. What is your unknown identity number?

2. What is the identity of the component(s) in your unknown?(5 points)

3. Explain how you were able to determine the identity of the component(s) in your unknown? (10 points)