

SPIKING ANALYSIS OF DRY GAS

LAB GC 3

From Juniata College SIM

INTRODUCTION

There are many different brands of gasoline additives, sometimes referred to as dry gas, on the market and at least as many different formulas (ingredients). They all do basically the same job; remove water and dirt from fuel lines. Many laboratories test these formulas to determine the list of components by utilizing a gas chromatography technique called spiking.

Gas chromatography is an analytical method that can separate gases or volatile liquids and allow determination of the relative quantities of the components of the sample. A gas chromatograph, GC, takes advantage of a component's unique interaction with the stationary phase and the mobile phase. The stationary phase is the material found in the coiled column of the instrument. Helium gas serves as the mobile phase. For every compound present in a sample there should be a peak recorded and the relative peak size indicates the percent composition of each compound in the sample. Spiking involves adding a pure compound to an unknown sample to either make a solution (two or more compounds) or simply increase the volume of the gasoline additive if both the spiking compound and the gasoline additive are the same.

The following figures illustrate chromatograms for a variety of samples.

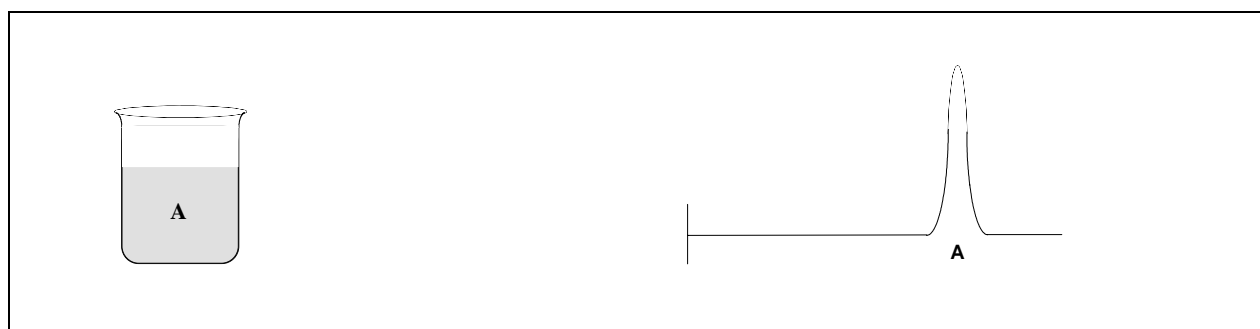


Figure 1. Gas chromatogram of compound A.

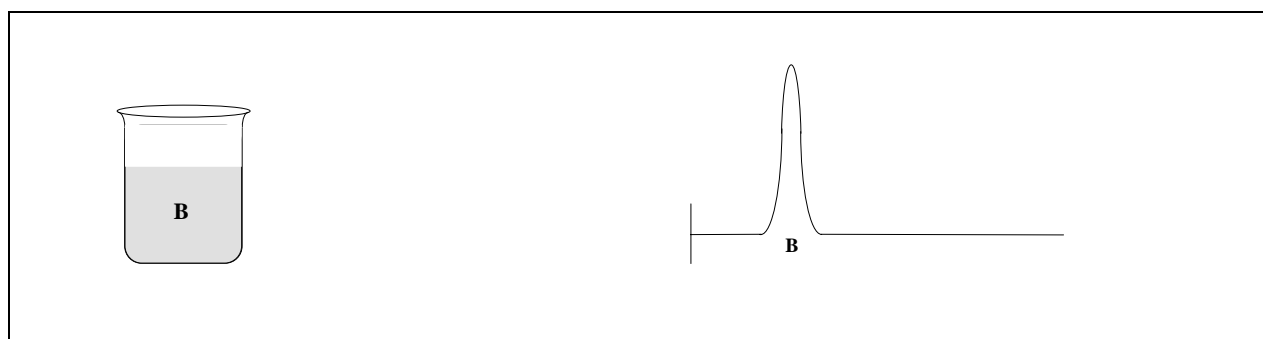


Figure 2. Gas chromatogram of compound B.

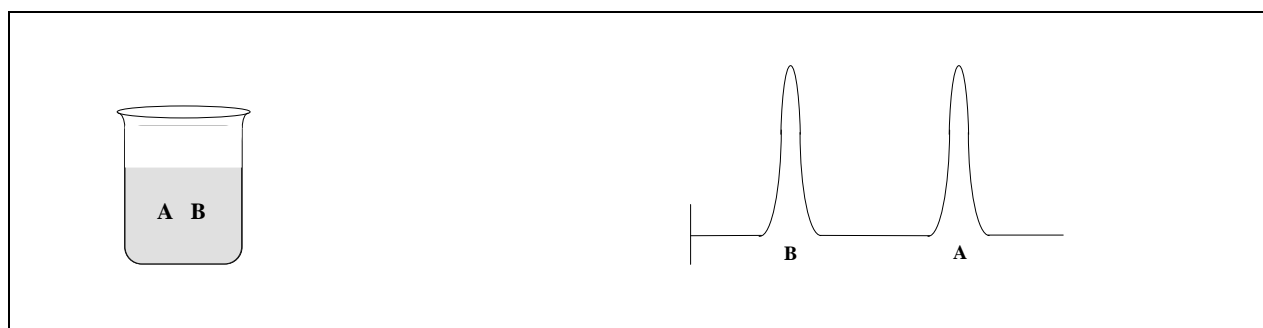


Figure 3. Gas chromatogram of a mixture of compounds A and B.

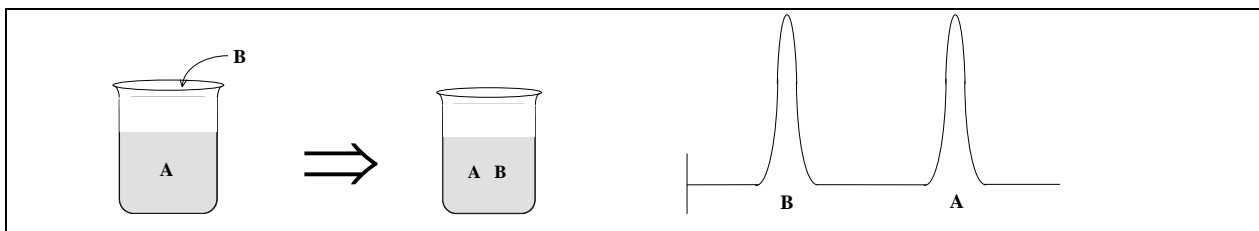


Figure 4. Gas chromatogram of compound A spiked with compound B.

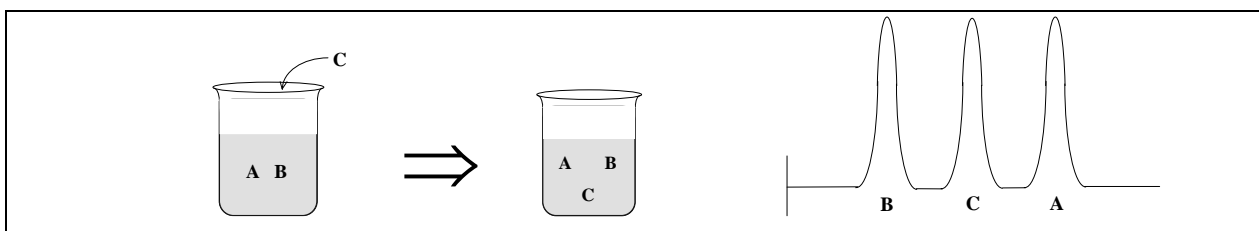


Figure 5. Gas chromatogram of compounds A and B spiked with compound C.

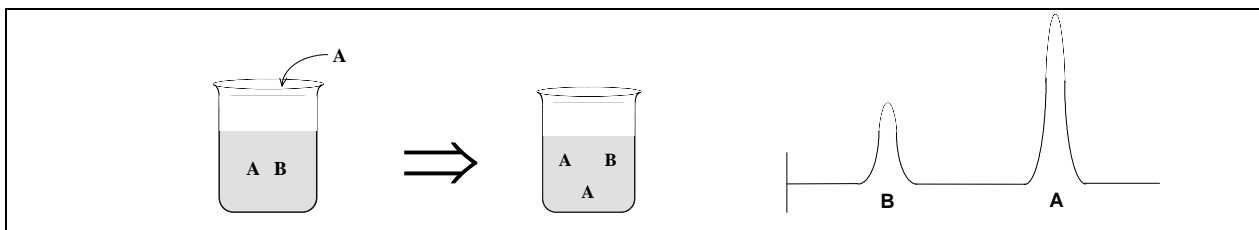


Figure 6. Gas chromatogram of compounds A and B spiked with additional A.

PURPOSE

The purpose of this experiment is to use the spiking technique to determine the ingredients of an unknown gasoline additive.

MATERIALS

gas chromatograph	laptop computer with PeakSimple for GC
He carrier gas	micro syringe
small sample vials	clean micro pipets
several gasoline additives	ethanol
Methanol	acetone
2-propanol	Kimwipes
graduated cylinder	

SAFETY

- Wear an apron and goggles at all times; eyes are particularly sensitive to these organic compounds.
- DO NOT misuse the syringes.
- The gas chromatograph is like an oven, so the ports and top of the instrument may be hot. Use with caution.
- Good laboratory procedure should be followed at all times.

PROCEDURE

1. The gas chromatograph needs time to warm up before it is used. Make sure the instrument is running and proper settings have been made before the laboratory begins. When more than one GC is present in the classroom, complete your portion of the experiment on a single instrument.
2. Obtain a microliter syringe and a vial of an unknown gasoline additive. Clean the syringe by rinsing it 3 times with this sample. Be careful not to inject any of the rinse back into the vial as this will contaminate the sample. Pull 2 microliter, μL , of gasoline additive into the syringe. Wipe the needle with a Kimwipe.
3. Carefully insert the needle of the syringe into the injection port. *At exactly the same time*, one member of the group injects the sample and a second group member presses the **Space-Bar** on the computer.
4. After the chromatograph is recorded, press the **End** key on the computer. Record the number of peaks.
5. Obtain one of the standard ingredients and add it to the gasoline additive by placing 1 mL of each into a small clean vial. This is the spiking step.

6. Make an injection of the spiked sample and determine if the standard ingredient is any part of the original gasoline additive. (Did the gasoline additive contain any of that standard compound before it was spiked?)
7. Repeat the steps with the other standards. Make sure the gasoline additive is spiked with only one standard at a time.

DATA SHEET

Name _____

Name _____

Period _____ Class _____

Date _____

SPIKING ANALYSIS OF DRY GAS

DATA TABLES

Gasoline Additive	Number of Peaks
1	
2	
3	

Gasoline Additive	Standard Ingredients Present (Y/N)			
	Acetone	2-propanol	Methanol	Ethanol
1				
2				
3				

QUESTIONS

1. If the same number of peaks are present before and after spiking what can be inferred?
2. Suppose before spiking the sample it showed two peaks (e.g. acetone and methanol), what would happen to the relative peak size if it were spiked with more acetone?
3. If peaks are present, after spiking the sample, that were not present before, what can be inferred?
4. How can this technique be used to identify pollutants such as pesticides or drugs?
5. If more than one extra peak is present after spiking with a pure compound, what probably happened in the solution?