Spiking Analysis of Dry Gas
Inquiry Version

PA State Standards for Science and Technology Addressed:

3.2.10.B Apply process knowledge and organize scientific and technological phenomena in varied ways.
Describe materials using precise quantitative and qualitative skills based on observations.

3.2.12.B Evaluate experimental information for appropriateness and adherence to relevant science processes.

3.4.10.A Explain concepts about the structure and properties of matter.
Apply knowledge of mixtures to appropriate separation techniques.

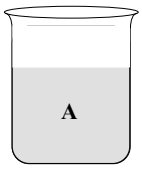
3.7.10.A Identify and safely use a variety of tools, basic machines, materials and techniques to solve problems and answer questions.
Apply advanced tool and equipment manipulation techniques to solve problems.

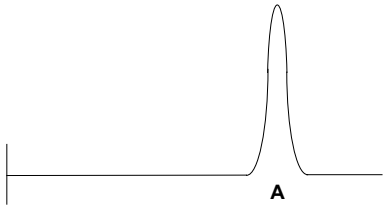
3.7.12.A Apply advanced tools, materials and techniques to answer complex questions.
Demonstrate the safe use of complex tools and machines within their specifications.

Introduction:
There are many different brands of gasoline additives on the market that use different ingredients. They all basically do the same job -- removing 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.

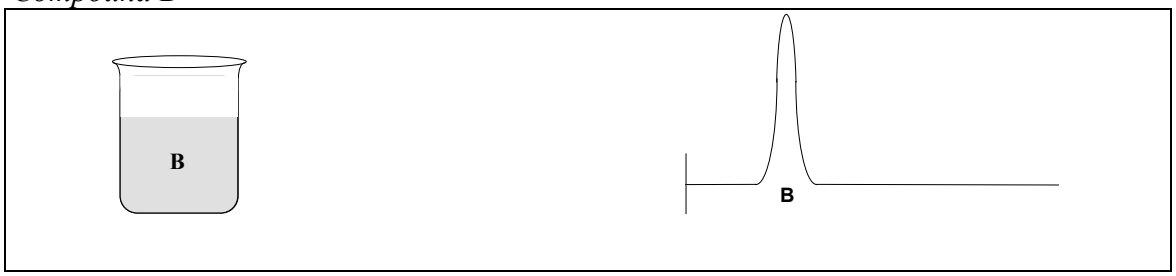
For every compound present in a sample there should be a peak recorded, and the relative peak size indicates the percent composition of each compound in the sample. Spiking involves adding a pure compound to an unknown sample to either make a solution (two or more compounds) or simply increase the volume of the gasoline additive. The following illustrations are of some samples and their corresponding chromatograms.

Compound A

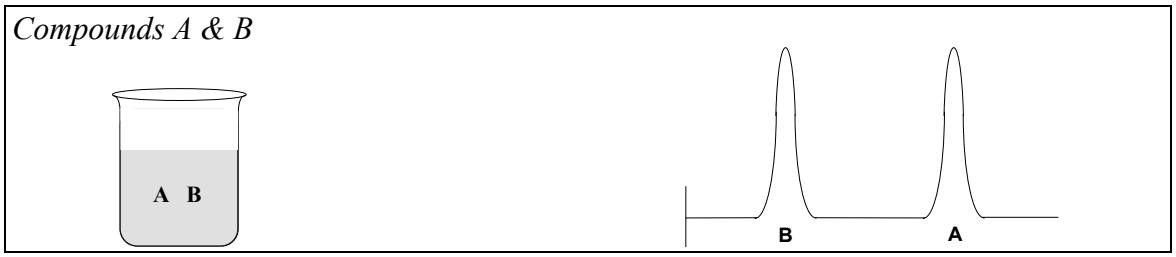




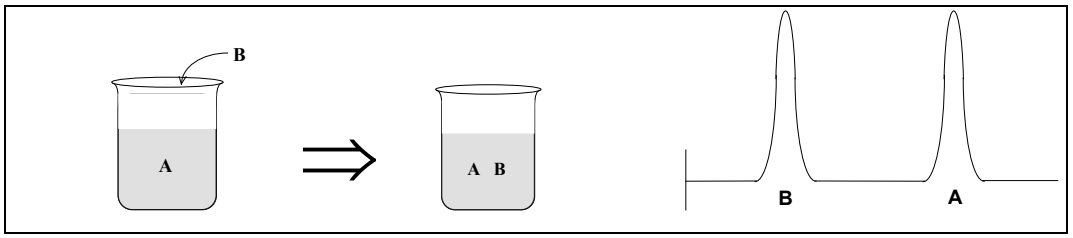
Compound B



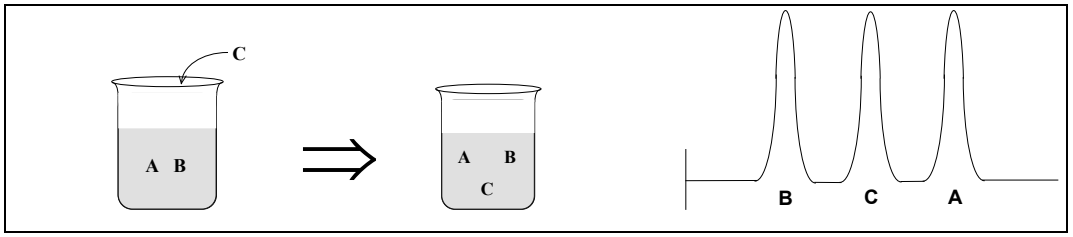
Compounds A & B



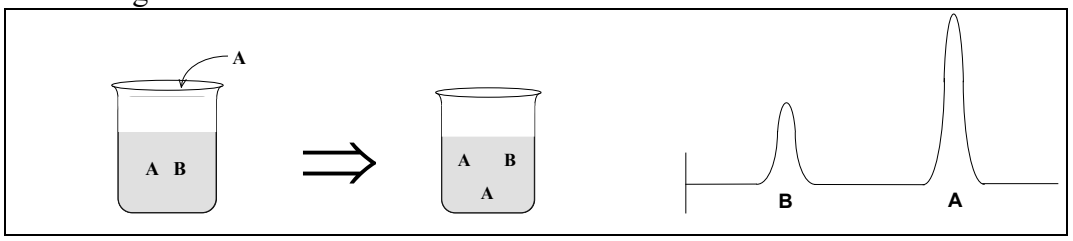
Suppose sample A is spiked with sample B. How many peaks would be recorded?



Suppose the solution of A and B is spiked with a third compound 'C.' How many peaks should be recorded?



Suppose the solution of A and B is spiked with pure compound A. How will the chromatogram look now?



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

Equipment / Materials:

air GC	several gasoline additives	methanol
computer	micro syringe	acetone
printer	1mL micro pipets and tips	2-propanol
small sample vials	ethanol	Kimwipes

Safety:
- Wear goggles at all times; eyes are particularly sensitive to these organic compounds.
- Use extreme caution in handling the GC syringes.
- Do not touch the injection port, its temperature is over 100ºC.

Procedure: Set-up and Use of the GC

1. Turn on the GC and start the Peak Sample software. A data acquisition system popup should appear with phrases like initializing, waking up, signing on, setting modes, and calibrating. If you get a popup saying can't wake – check power and cable, try one of the following options.
    a. Check to make sure that the cable between the GC and computer is properly attached and that the GC is on.
    b. Close the program. Unplug the USB cord from the computer, then plug it in again. Restart the program. If it still won't wake, try the next option.
    c. Click on the Edit Menu, then select Overall. In the COM Port/USB device number box, try a number from 1 – 5. Keep trying until the data acquisition system begins to function.

2. Click on edit menu, then select channels. Under Channel 1, pick Temperature. Click on the numbers to highlight them, then click Change. Change the numbers as follows:
    Start 150°C
    Hold 10 minutes
    Ramp 0 degree / min
    Until the temperature is 150°C

3. Be sure the time scale starts at zero and ends at 10 min. Use the arrows to adjust.

4. Clean the syringe by rinsing it 10 -- 15 times with the desired sample. With the plunger fully depressed, place the needle into the sample. Slowly draw up the plunger to obtain a sample in the syringe. Remove the syringe from the sample. Discharge this sample into the sink or onto a Kimwipe. Depress the plunger, and put the syringe needle back into the sample. Draw up a second sample and discharge it. Repeat. **Do not push on the plunger when the needle is inside a sample.**

5. Place the syringe needle back into the sample and withdraw over 1 microliter. Remove the needle from the sample bottle, then depress the plunger to the 1 microliter line. Wipe the needle with a Kimwipe.

6. Click on the Z or θ button on the run screen to zero the current.

7. Insert the needle carefully into the injection port of the GC until the entire needle is inside the instrument.

8. Inject the sample and start the data collection simultaneously by depressing the syringe plunger and pressing the space bar on the computer at the same time.

9. The run may be stopped by pressing the end button on the computer. After pressing end, print the graph. Be sure to label the graph.

Experiment:
Test pure and spiked samples of gasoline additives. Determine the active ingredient in as many samples as instructed.

Questions:

1. If there is only one peak, does this always indicate that the sample was pure? Explain why or why not.

2. If the same number of peaks is present before and after spiking, what can be inferred?

3. Suppose the sample showed two peaks before spiking (e.g. acetone and methanol). What will happen to the relative peak size if it is spiked with more acetone?

4. If an additional peak is present after spiking the sample, what can be inferred?

5. How can this technique be used to identify pollutants such as pesticides or drugs?

6. If more than one extra peak is present after spiking with a pure compound, what probably happened in the solution?