

## PURDUE UNIVERSITY INSTRUMENT VAN PROJECT

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### DETERMINING THE IDENTITY OF ALCOHOLS IN UNKNOWN MIXTURES USING THE GAS CHROMATOGRAPH

(Revised: 2-6-96)

#### INTRODUCTION

You have already seen how gas chromatography is used to determine percent composition of mixtures. We can also use gas chromatography to identify substances in an unknown mixture.

You will use a process called **co-injection** or **spiking** to identify alcohols in an unknown mixture. Co-injection involves adding a known substance to the unknown. If this known substance is also present in the unknown, then the peak for that substance will be larger in the spiked sample than in the unspiked sample. If the known is not a substance present in the unknown, then an extra peak will appear in the spiked sample compared to the unspiked sample. This technique will be used with each of four known alcohols. Results will be obtained by analyzing the peak heights and the time required for peaks to appear on the chromatogram.

If you cannot decide which of the two alcohols corresponds to a peak you will rerun the unknown and two unknown samples spiked with the alcohols in question. The attenuation and chart speed will be changed to widen and separate the peaks.

#### PURPOSE

To determine the identity of the substance causing each peak in the chromatogram of an unknown mixture by spiking samples to be analyzed in the gas chromatograph.

#### SAFETY

#### **GOGGLES MUST BE WORN AT ALL TIMES!**

- Cautions:
- 1) Injection port is **very hot**.
  - 2) Syringe needle is delicate and sharp. Use extreme caution when handling.
  - 3) **Do not** adjust knobs and control buttons on the machines. They have already been set for you!

#### MATERIALS

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Gas Chromatograph

10  $\mu\text{L}$  syringe

Unknown samples A, B, C & D (one per group)

Known samples: *n*-butyl alcohol (1-butanol)  
*t*-butyl alcohol (2-methyl-2-propanol)  
*n*-propyl alcohol (1-propanol)  
isopropyl alcohol (2-propanol)

### PROCEDURE

When it is your turn, you will perform several tasks. Read through all procedures before you begin.

1. You will be assigned an unknown mixture (A, B, C or D) to analyze by spiking. Record this letter on your data table.
2. Clean the syringe:
  - a. Draw 10  $\mu\text{L}$  of your unknown mixture into the syringe and expel it onto a paper towel.
  - b. Repeat once more.
3. Gather unknown sample mixture:
  - a. Insert syringe into your unknown mixture and draw 1  $\mu\text{L}$  into the syringe.
  - b. Remove the syringe from the bottle, cap the bottle and draw 5  $\mu\text{L}$  of air into the syringe.
4. Run the sample:
  - a. Check to be sure baseline needle is set and recorder is turned on and in standby mode.
  - b. Carefully insert syringe needle into **INJECTION PORT A**. **Remember**: The port will be **very hot**. Quickly and smoothly depress the plunger and remove the syringe.
  - c. As this is being done, have a partner turn the recorder to run and record the following information on your chromatogram:
    - a. Attenuator setting
    - b. Sample size
    - c. Identity of sample
    - d. Chart speed
    - e. Group name or number
5. Record your observations on the data sheet. While chromatogram is running, have one member of your group prepare a spiked sample of your unknown. (follow steps 6 & 7).

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6. Clean the syringe:
  - a. Draw 10  $\mu\text{L}$  of your unknown mixture into the syringe and expel it onto a paper towel.
  - b. Repeat once more.
  
7. Gather unknown + known mixture:
  - a. Insert syringe into your unknown mixture and draw 1  $\mu\text{L}$  into the syringe.
  - b. Draw 1  $\mu\text{L}$  of air into the same syringe and wipe off the needle with a Kimwipe<sup>®</sup>.
  - c. Insert the needle into *n*-propyl alcohol and draw 1  $\mu\text{L}$  into the syringe (along with the other sample).
  - d. Draw 5  $\mu\text{L}$  of air into syringe and close all bottle caps.
  
8. Run the sample:
  - a. Check to be sure the last chromatograph has finished. Do not inject until it has!
  - b. Carefully insert syringe needle into **INJECTION PORT A**. Remember: port will be **very hot**. Quickly and smoothly depress the plunger and remove the syringe.
  - c. As this is being done, have a partner record the following information on your chromatogram:
    - a. Attenuator setting
    - b. Sample size
    - c. Identity of sample
    - d. Chart speed
    - e. Group name or number
  
9. Record your information on the data sheet. While this is running, prepare to run a second spiked sample of the unknown mixture. Follow the same instructions as before, this time running your unknown + isopropyl alcohol.
  
10. Repeat the entire procedure (steps 6-9) twice more using:
  - unknown + *t*-butyl alcohol
  - unknown + *n*-butyl alcohol
  
- 11) Remove the chromatogram after you are finished.

### DATA TABLE #1

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	UNKNOWN	SPIKE 1 <i>n</i> -butyl alcohol	SPIKE 2 <i>t</i> -butyl alcohol	SPIKE 3 <i>n</i> -propyl alcohol	SPIKE 4 isopropyl alcohol
unknown sample letter					
amount used					
number of peaks observed					
attenuation					
chart speed					

### CONCLUSIONS

At this point, you should have a good idea about the identity of the alcohols in your unknown mixture by comparing chromatograms of the unknown mixture with spiked samples. Identify the alcohols if you are able to do so!

If you are unable to decide which of the two alcohols are responsible for a peak, you will need to analyze three more samples (unknown, 2 unknown samples each spiked with one of the alcohols in question) at different instrument settings. Before running the three samples, please notify your teacher that you will be doing the following:

- a. Increase the attenuation from 8 to **16**.
- b. Increase the chart speed from 2 cm/min to **3 cm/min**.

Identify the alcohols in the unknown mixture that corresponds to each peak in the chromatogram of the unknown mixture.

### DATA TABLE #2

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	identity of substance	identity of substance	identity of substance
Peak 1			
Peak 2			
Peak 3			