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#13 Chromatography is a Gas: An Inquiry-Based Introduction to Gas Chromatography

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INTRODUCTION

Description

This chromatography exercise is designed to introduce students to the theory of chromatographic separation of mixtures and packed column chromatography. The students will become familiar with basic operation of the gas chromatograph and explore the relationship between temperature and retention time of individual components in a mixture. A section on instrument trouble-shooting may be included as an additional optional activity.

Student Audience

This activity was developed for college students. This activity was developed and tested in conjunction with an analytical chemistry course for students enrolled in several technology programs. The instrument trouble-shooting was included with this group. This lab, without the trouble-shooting portion, was also tested in a first-year chemistry course for chemical process operators.

Goals for the Experiment/Activity

At the completion of this activity the student will

- understand the principles of chromatographic separations;
- identify and explain the function of the parts of the gas chromatograph, including carrier gas, injector, column, detector, and output device;
- make manual injections of liquid samples into the gas chromatograph;
- make adjustments in the column temperature to change retention time;
- identify peaks by retention time when compared to standard chromatograms; and
- identify and remedy problems in instrument operation (if the optional trouble-shooting section is included).

Recommended Placement in the Curriculum

This activity is recommended for a point where the students are comfortable with general laboratory safety and techniques.

STUDENT HANDOUT

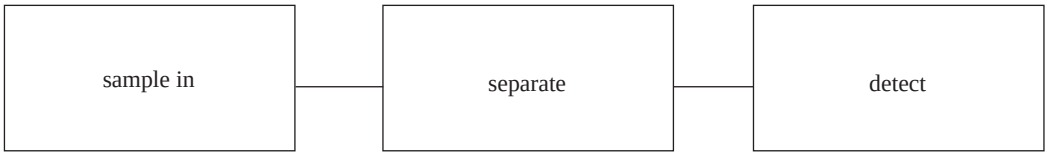
Title

Chromatography is a Gas: An Inquiry-Based Introduction to Gas Chromatography

Background Information

Chromatography is the science of separating components in a mixture. All chromatographic systems include a stationary phase and a mobile phase. The separation occurs due to differences in attraction of the analyte to the two phases. All types of chromatographic separations are based on five principles: adsorption, partition, electrostatic charge, size, and boiling point. One or more of these principles is active in each type of chromatography.

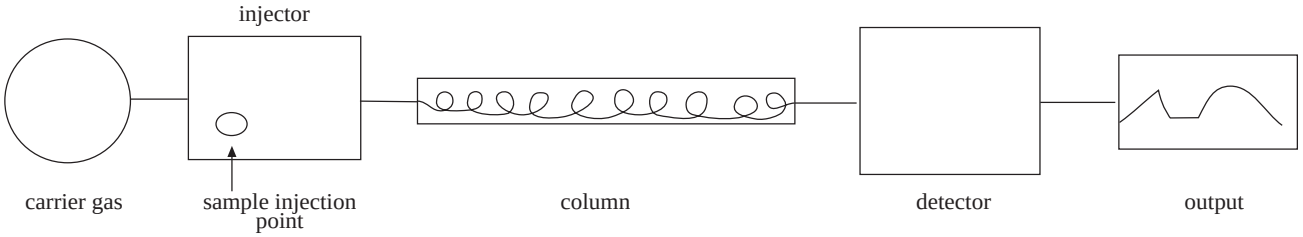
All types of chromatography can be described by the block diagram below.



Gas chromatography is suitable for analyzing samples of gases or volatile liquids. A heated injection port is used to introduce the sample into the column. The volatilized sample is swept through the column by a carrier gas. The sample then interacts with the stationary phase, which is usually a liquid that has been bonded to the capillary column or coated onto the packing material in a glass or metal column. Some types of gas chromatography use a solid packing material with no coating as the stationary phase.

The sample then enters a heated detection zone where the detector generates an electronic signal that can be digitized and fed to a computer or converted to a peak on a strip chart recorder or integrator. Different types of detectors are available for use in gas chromatography. Two of the most common are the flame ionization detector (FID), which is quite sensitive but limited to use for organic samples that burn, and the thermal conductivity detector (TCD), which is capable of detecting all types of compounds at higher concentrations.

The block diagram shown below represents a gas chromatograph:



Purpose

This exercise is designed to introduce students to gas chromatography and teach them to identify peaks in a chromatogram by retention time.

Scenario/Industrial Applications

1.
Quick-Kleen Polish Remover Corporation

To: Student Technicians
From: Consumer Relations Director
Subject: Quality Complaint
Date: 12/20/98

Enclosed you will find a 5-mL sample of a recent shipment and two retainer samples from previous shipments of our non-acetone nail polish remover. Our customer claims that this recent shipment does not remove the polish as well as previous shipments.

The specification for this product is 60% ethyl acetate, 14% 2-propanol, 20% water, and 6% other ingredients.

Please prepare an outline for an experimental plan for analysis of these samples so I can reassure the customer that we will be working to solve this problem. (Complete the following exercise to become familiar with gas chromatography before writing your outline.)

Safety, Handling, and Disposal

Wear goggles while performing this experiment.

Review all necessary MSDS.

The reagents used in this experiment are common laboratory reagents. Dispose of used reagents according to local ordinances.

The heated injection zones of the gas chromatograph are HOT. Do not touch the septum nut when making an injection.

The needle on the injection syringe is very sharp. Take care to avoid puncture wounds when handling the syringe to make injections.

Materials Needed

Equipment: Gas chromatograph

Supplies: ethyl acetate
propyl acetate
2-propanol
non-acetone nail polish remover
10-microliter syringes

Procedure

Your instructor will provide you with instruction sheets for each step of the experiment. Complete the indicated experiment, answer the questions, and report to your instructor.

2. Make a 1-uL injection of the ethyl acetate standard, being careful not to touch the septum nut. It is HOT. Repeat this twice.

a. How many peaks did you see?

b. What was the retention time for each peak?

3. Reduce the temperature of the column to 60∝C.

   Make a 1-uL injection of the ethyl acetate standard, being careful not to touch the septum nut. It is HOT. Repeat this twice.

   a. How many peaks did you see?

   b. What was the retention time for each peak?

4. Make a 1-uL injection of the propyl acetate standard, being careful not to touch the septum nut. It is HOT. Repeat this twice.

   a. How many peaks did you see?

   b. What was the retention time for each peak?

5. Make a 1-uL injection of the 2-propanol standard, being careful not to touch the septum nut. It is HOT. Repeat this twice.

   a. How many peaks did you see?

   b. What was the retention time for each peak?

6. If you injected a sample which contained 2-propanol, ethyl acetate, and propyl acetate (a mixed standard)...

   a. How many peaks would you see?

   b. What would the retention time be for each peak?

7. Make three injections of a mixed standard.

   a. How many peaks did you see?

   b. What was the retention time for each peak?

   c. What changes did you observe in the size of the peaks?

   d. Why did the peak size change?

8. If you added some ethyl acetate to the mixture...

   a. Would the number of peaks in the chromatogram change?

   b. Would the shape of any of the peaks in the chromatogram change?

   c. Would the retention time of any the peaks in the chromatogram change?

9. Add some ethyl acetate to the mixed standard and make three injections, recording your observations.

   a. Did the number of peaks in the chromatogram change?

   b. Did the shape of any of the peaks in the chromatogram change?

   c. Did the retention time of any the peaks in the chromatogram change?

10. Please prepare an outline for an experimental plan for analysis of these samples so I can reassure the customer that we will be working to solve this problem. (Address this outline to the Consumer Relations Director in the form of a memo.) Have your instructor approve the plan before proceeding.

11.
Quick-Kleen Polish Remover Corporation

To: Student Technicians
From: Consumer Relations Director
Subject: Quality Complaint
Date: 12/20/98

Thank you for your quick response outlining an analysis plan for the suspect polish remover sample. Please proceed with the analysis and report your findings to me in letter form.

INSTRUCTOR NOTES

Title

Chromatography is a Gas: An Inquiry -Based Introduction to Gas Chromatography

Time Required

This activity was designed for completion in a 3-hour block. The optional trouble-shooting section can be used for an additional 3-hour block.

Group Size

This activity was tested with groups of five students per gas chromatograph.

Materials Needed

Gas Chromatogaph

instrument:	Hewlett-Packard 5710 gas chromatograph equipped with TCD (or equivalent)
column :	6-inch by $^{1}/_{8}$-inch stainless steel
packing:	10% SE-52 on 80/100 Chromasorb W-HP (or other appropriate column)
injector :	150°C
detector :	250°C
column:	100°C
carrier:	He at 30 mL/min

Supplies: Each student group should be provided with 5 mL of ethyl acetate, propyl acetate, and 2- propanol for use as standards. Each group will receive two samples of non-acetone nail polish remover (labeled as retainer samples), one mixture that has been prepared with less than 60% ethyl acetate (labeled as suspect sample), and a 10-microliter syringe.

Safety, Handling, and Disposal

Wear goggles while performing this experiment.

Review all necessary MSDS.

The reagents used in this experiment are common laboratory reagents. Dispose of used reagents according to local ordinances.

The heated injection zones of the gas chromatograph are HOT. Do not touch the septum nut when making an injection.

The needle on the injection syringe is very sharp. Take care to avoid puncture wounds when handling the syringe to make injections.

Points to Cover in the Pre-Lab Discussion

Safety: Thermal burns are the most common injury I have experienced with this exercise. Caution the students not to grab the septum nut while making injections. (They seem to do this because the needle is so flimsy and they are trying not to bend the needle.) Show the students how to hold the needle so that they do not puncture themselves while making an injection.

Discuss the principles and operation of the GC.

Procedural Tips and Suggestions

Be sure to have several syringes on hand. The needles will bend and break with beginners.

Have the students draw a small amount of air into the syringe before they inject. This will prevent them from losing their sample if they have trouble injecting and need to make more than one try. It will also show a small peak on the chart indicating the start of the chromatogram.

Stress the need for reproducible technique when making injections.

Sample Results

1.
Quick-Kleen Polish Remover Corporation

To: Student Technicians
From: Consumer Relations Director
Subject: Quality Complaint
Date: 12/20/98

Enclosed you will find a 5-mL sample of a recent shipment and two retainer samples from previous shipments of our non-acetone nail polish remover. Our customer claims that this recent shipment does not remove the polish as well as previous shipments. The specification for this product is 60% ethyl acetate, 14% 2-propanol, 20% water, and 6% other ingredients.

Please prepare an outline for an experimental plan for analysis of these samples so I can reassure the customer that we will be working to solve this problem. (Complete the following exercise to become familiar with gas chromatography before writing your outline.)

(The students should outline a plan to analyze the good and bad samples and compare the chromatograms.)

(Students should be provided with a good sample of commercial polish remover and a bad sample, which is a synthetic polish remover sample. The bad sample can have less ethyl acetate or no ethyl acetate.)

2. Make a 1-uL injection of the ethyl acetate standard being careful not to touch the septum nut. It is HOT. Repeat this twice.

   a. How many peaks did you see?

      (one peak each time)

   b. What was the retention time for each peak?

      (Record retention time here, it should be the same each time.)

3. Reduce the temperature of the column to 60°C .

   Make a 1-uL injection of the ethyl acetate standard, being careful not to touch the septum nut. It is HOT. Repeat this twice.

   a. How many peaks did you see?

      (one peak each time)

b. What was the retention time for each peak?

(The retention time is later than at 100°C, the same each time.)

4. Make a 1-uL injection of the propyl acetate standard, being careful not to touch the septum nut. It is HOT. Repeat this twice.

a. How many peaks did you see?

(one peak each time)

b. What was the retention time for each peak?

(The retention time is later than the ethyl acetate.)

5. Make a 1-uL injection of the 2-propanol standard, being careful not to touch the septum nut. It is HOT. Repeat this twice.

a. How many peaks did you see?

(one peak each time)

b. What was the retention time for each peak?

(The retention time is earlier than the ethyl acetate.)

6. If you injected a sample which contained 2-propanol, ethyl acetate, and propyl acetate...

a. How many peaks would you see?

(3 peaks)

b. What would the retention time be for each peak?

(The three peaks should have retention times very close to the retention times for the standards injected individually.)

7. Make three injections of a mixed standard.

a. How many peaks did you see?

(3 peaks)

b. What was the retention time for each peak?

(The three peaks should have retention times very close to the retention times for the standards injected individually.)

c. What changes did you observe in the size of the peaks?

(Each of the peaks was smaller than when injected alone.)

d. Why did the peak size change?

(In the individual standards the peak was 100% of the sample; the mixed standard was approximately 33% of each.)

8. If you added some ethyl acetate to the mixture...

a. Would the number of peaks in the chromatogram change?

(no)

b. Would the shape of any of the peaks in the chromatogram change?

(The second peak would get larger.)

c. Would the retention time of any the peaks in the chromatogram change?

(They should not, but may see a slight shift in retention time due to size of peak.)

9. Add some ethyl acetate to the mixed standard and make three injections, recording your observations.

a. Did the number of peaks in the chromatogram change?

(no)

b. Did the shape of any of the peaks in the chromatogram change?

(The second peak became larger.)

c. Did the retention time of any the peaks in the chromatogram change?

(They should not, but may see a slight shift in retention time due to size of peak.)

10. Please prepare an outline for an experimental plan for analysis of these samples so I can reassure the customer that we will be working to solve this problem. Have your instructor approve the plan before proceeding.

11.
Quick-Kleen Polish Remover Corporation

To: Student Technicians
From: Consumer Relations Director
Subject: Quality Complaint
Date: 12/20/98

Thank you for your quick response outlining an analysis plan for the suspect polish remover sample. Please proceed with the analysis and report your findings to me in letter form.

(The students should respond with information which indicates that the suspect sample does not have the same concentration of ethyl acetate in the remover sample.)

Plausible Answers to Student Questions

Does the volume of the sample injected have to be exactly the same each time?

The size of the peak is proportional to the volume and concentration of the sample. If the volume is changed, the peak size will change and will affect quantitation.

Why does the peak come out faster at a higher temperature?

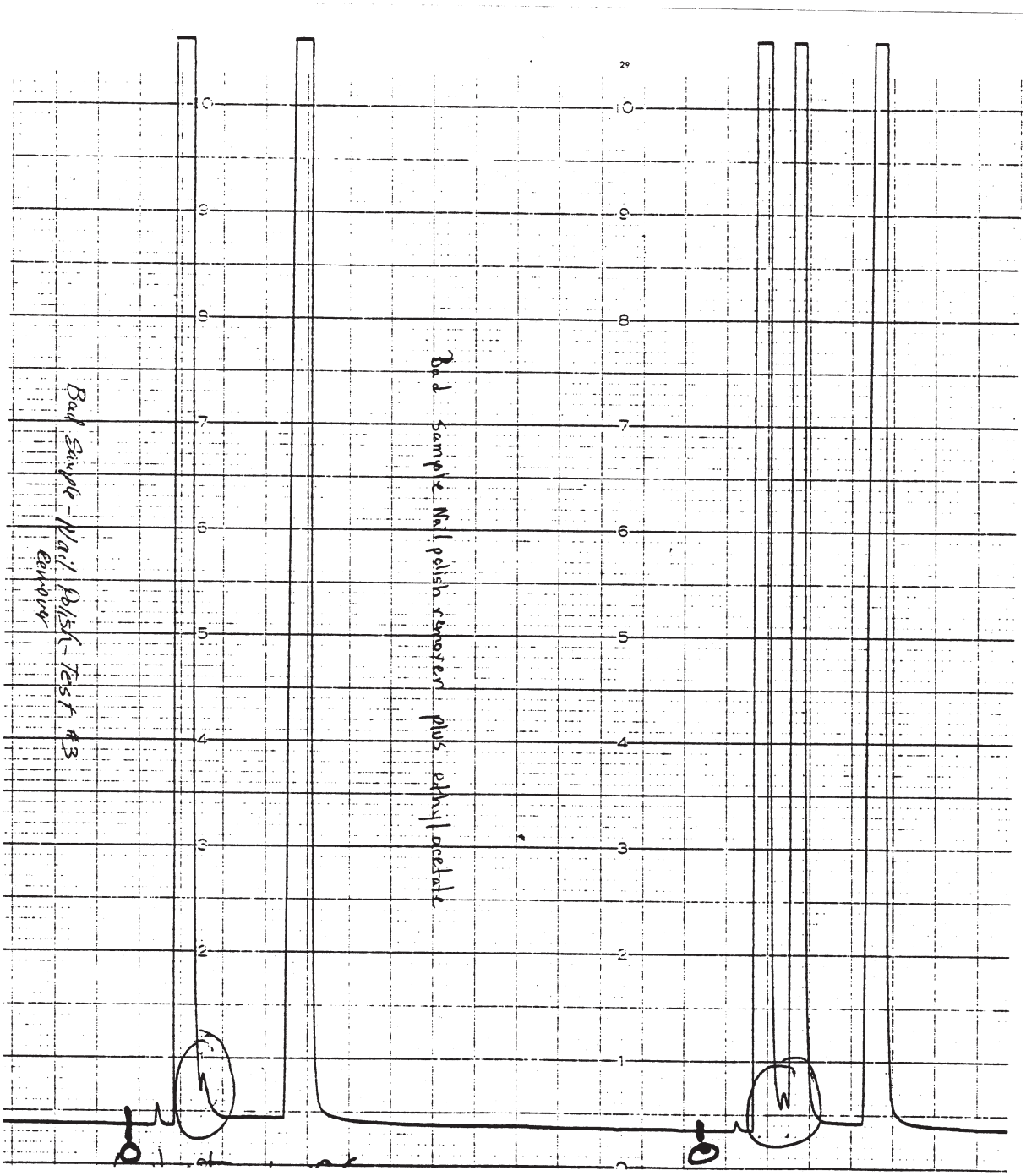
The molecular velocity increases with an increase in temperature.

Extensions and Variations

Troubleshooting: After the students have collected some data, sabotage the instrument by doing things like loosening the signal cable, turning off the power to the detector, changing the column temperature, changing the detector temperature, or unplugging the recorder. (Do not do anything that would cause permanent damage to the column.)

Quantitation: If an integrator is available, have the students measure the peak areas and perform an external standard analysis on the standards and suspect samples. An alternate quantitative method is to cut out and weigh the individual peaks or measure peak heights.

SAMPLE CHROMATOGRAM—BAD POLISH SAMPLE



SAMPLE CHROMATOGRAM—MIXED STANDARDS CHROMATOGRAM