

## EXPERIMENT 4

### SIMPLE DISTILLATION AND FRACTIONAL DISTILLATION GAS CHROMATOGRAPHY (GC)

This experiment has been adapted from Experiment 6 in  
Landgrebe, John A. *Theory and Practice in the Organic Laboratory*, 4<sup>th</sup> ed. Brooks/Cole, 1993.

Part 1: Distillation of the ethanol/acetone mixture

Two-student teams:

Student I uses a simple distillation apparatus

Student II uses a fractional distillation apparatus (pick one of the several fractionating columns provided)

Results are shared.

Part 2: Analysis of the result by GC

**Reading Assignment:** Pavia Sections 6.1, 6.3, 6.5-6.6, 6.9, 13.1, 14.1-14.3, + last paragraph only in 14.4, 15.1 – 15.6, 15.8, 22.1-22.10, 22.11-22.12

**Pre-lab Questions:**

- 1) Pavia chapter 14 Questions #1, #5, #6, #8.
- 2) Pavia chapter 15 Questions #1, #2, #4.

**Chemicals:** Ethanol:Acetone Solution (50 mole percent)

<b>Equipment:</b>	100 mL round-bottomed flask	thermometer adapter
	heating mantle	boiling chips
	two water-cooled condensers	4 Keck clamps
	steel wool	100 mL beaker
	distillation head	25 mL graduated cylinder
	vacuum adapter	condenser hoses
	fractionation column with steel sponge	3 three-pronged clamps
	thermometer (Hg)	

The following procedure is a modification of the experiment described in Experiment #6 of Landgrebe (p. 372). The distillations will use 14/20 standard-taper glassware.

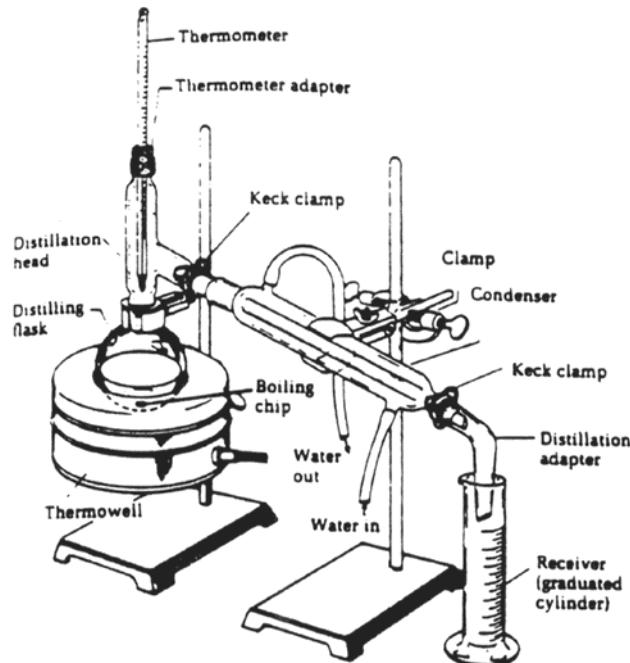
**Safety Note:**

At the beginning of any distillation, the flask should not be more than two-thirds full to allow for vigorous boiling. In all cases, great care should be taken not to distill to dryness, because superheating of the residue can result.

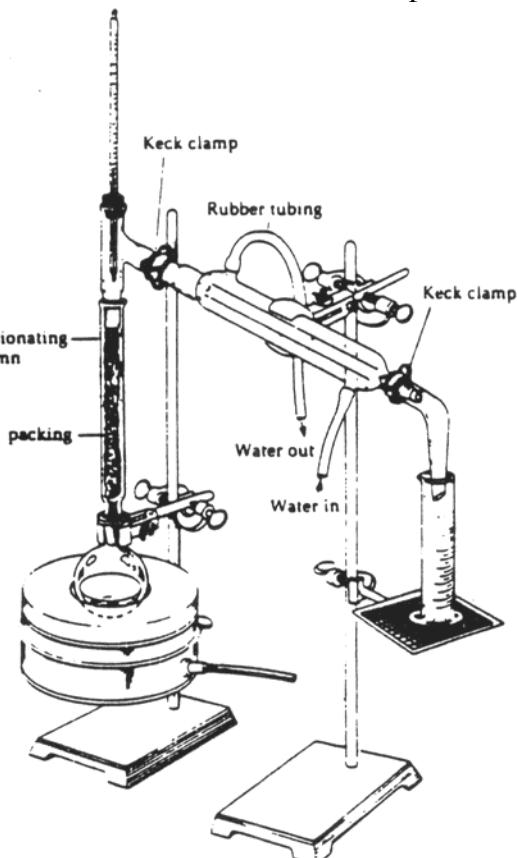
**Students I and II:** In a 100 mL round-bottomed flask place 50 mL of the ethanol/acetone mixture and a boiling chip. Assemble the appropriate (simple or fractional) distillation apparatus. Note that you should never add a boiling chip to hot liquid as it may initiate vigorous reflux and it may boil over.

**Student I: Simple Distillation Setup.**

Assemble the apparatus for *simple distillation*, starting with the 100 mL round-bottomed flask, then the support ring and electric heating mantle (or thermowell). One or two boiling stones are put in the flask to prevent bumping. Each ground-glass joint is lightly greased by putting one or two stripes of grease lengthwise around the male joint and pressing the joint into the female joint with slight turning. Make sure that none of the grease has seeped into the inner surface of your distillation apparatus as it will contaminate the product.



**Simple Distillation Setup.**



**Fractional Distillation Setup.**

Water enters the condenser at the tubulation nearest the receiver. The large heat capacity of water means that only a small stream is needed; too much water flow will cause the tubing to pop off. Use Keck clamps to hold the various glass components of the apparatus (such as the condenser and the distillation head) together.

Note that the bulb of the thermometer must be below the opening into the side arm of the distillation head to obtain a steady and reliable temperature measurement

**Student II: Fractional Distillation Setup.** Assemble the apparatus for *fractional distillation*, starting with the 100 mL round-bottomed flask, then the support ring and electric heating mantle (or thermowell). The fractionation column is already packed with a metal sponge. The column should be vertical. It can be insulated with glass wool covered with aluminum foil, however insulation is omitted for this experiment in order to observe what is taking place within the column. Follow the directions for assembling the simple distillation glassware, above, including greasing joints and attaching tubing.

**Students I and II:** The stillhead delivers into a short condenser fitted with a bent adapter leading to a 10 mL graduated cylinder. Gradually turn up the heat to the heating mantle until the mixture just begins to boil. You will find that the heating mantle responds slowly. As soon as the boiling starts, turn down the power a bit. Heat slowly at first. A ring of condensate will rise slowly through the column; if you cannot at first see this ring, locate it by cautiously touching the column with the fingers. The rise should be gradual so that the column can acquire a uniform temperature gradient. Do not apply more heat until the ring of condensate has stopped rising; then increase the heat gradually. In a properly conducted fractional distillation, the vapor-condensate mixture reaches the top of the column only after several minutes.

Once distillation has commenced, it should continue steadily, without any drop in the head temperature, at a rate of about 1 drop every 1-2 seconds, and not greater than 1 mL in 1.5-2 min. Observe the rate of distillation and keep it steady by slowly increasing the heat, as required. Protect the column from drafts if necessary with aluminum foil. Record the temperature as each milliliter of distillate collects, and make more frequent readings when the temperature starts to rise abruptly.

After the temperature has stabilized and after discarding the initial forerun, collect the first 5 mL of the distillate in a tightly capped, labeled vial for later GC analysis. Each time the graduated cylinder fills, quickly empty it into a series of labeled 25 mL Erlenmeyer flasks. For the simple distillation experiment, save a late higher-boiling 5 mL fraction for GC analysis in a labeled vial. With the fractionating column, collect a 5 mL "center cut" of the higher boiling fraction for GC analysis after a second constant temperature is reached.

Stop the distillation when most of the liquid in the distillation pot is gone. Generate a distillation curve by plotting boiling point temperature versus volume on a graph paper and record what you observed inside the column in the course of the fractionation. Combine the fractions that you think are pure and turn in the product in a tared bottle labeled with your name, your student number, the product name, the bp range, and the tare weight and the weight of the contents.

**Students I and II: GC Analysis:** Carry out a GC analysis of the starting acetone/ethanol mixture. Record all of the relevant GC conditions (i.e., column temperature, injection sample size, chart speed, etc.) for each run. Calculate the retention time for all peaks. Decide which column type achieves better separation (resolution) of the sample components and calculate the response factor for each compound using the data from that run.

Perform a GC analysis of the initial 5 mL fractions from both the simple and the fractional distillations. To be certain which peak is ethyl alcohol, and which peak is acetone, inject pure acetone or pure ethanol and compare the retention times.

The relative areas under the peaks for acetone and ethyl alcohol will not accurately reflect the molar amounts of each, because acetone and ethanol both have a different response to the detector. In order to obtain relative areas that accurately reflect the number of moles of each of the components, you will have to calculate a response factor from a chromatotron of a 1:1 acetone:ethyl alcohol mixture. Use these calculated response factors to correct the measured areas of your GC's. Use these corrected areas to calculate the mole fractions of acetone and ethanol in the two distillation fractions, as well as the percent acetone and ethanol present in the first 5 mL collected for both types of distillations. From the data on the initial 5ml fraction of the simple distillation, calculate the enrichment factor,  $\alpha$ . Because the mole ratio of acetone to ethanol in the starting mixture was 1, the value of  $\alpha$  is the mole ratio of acetone to ethyl alcohol in the initial distillate from the simple distillation. Use your calculated enrichment factor and the mole ratio of acetone to ethanol in the initial distillate from the fractional distillation to calculate the number of theoretical plates and the HETP for the packed column used in the fractional distillation. Show all of your calculations and summarize the results in clearly labeled tables.

The team members will share their data. Make copies of the GC traces for each other as well as any data from the distillation. These should be included in the lab writeup. Make sure to write the full name of your lab partner in your notebook and in your report.

#### **Topics to be included in your lab report conclusion:**

- a) Identify which GC peak corresponds to which compound, based on retention times.
- b) Compare the two types of distillation in terms of:
  - the degree of separation achieved between the two components
  - the boiling point *versus* volume plots.

#### **Post-lab Questions:**

- 1) Why is the better separation of two liquids achieved by slow rather than fast distillation?
- 2) Explain why a packed fractionating column is more efficient than an unpacked one.
- 3) Pavia chapter 22 Question #4

- 4) A 1:1 molar mixture of **a** and **b** separates on a GC column to give peaks with areas 315 and 540 units. Assume the molar response factor for compound **b** is 1.0. What is response factor for **a**? If a mixture of unknown composition gives an **a** peak with an area of 540 and a **b** peak with an area of 1200, what is the mole % of **a** and **b** in the mixture?
- 5) A solution consists of 10g of liquid **X** (MW:50) and 9g of liquid **Y** (MW:30). The vapor pressures of pure **X** and **Y** are 40 and 70 mmHg, respectively, at 20°C. What are  $N_x$  and  $N_y$  ( $N$  = mole fraction)? What is the partial pressure of **X** over the solution at 20°C?