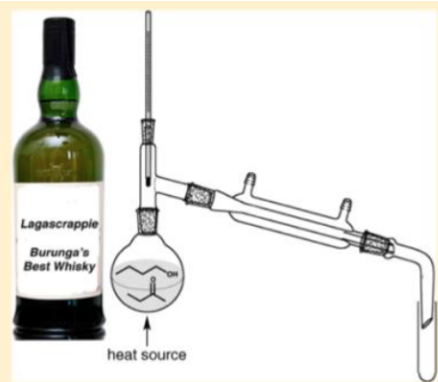


Using Whiskey-Flavoring Compounds To Teach Distillation

ABSTRACT: Laboratory techniques, such as boiling point determination, distillation, and infrared spectroscopy, are commonly taught during first-semester undergraduate organic chemistry courses. This integrated experiment teaches the usefulness of combining these different laboratory techniques for structure elucidation purposes. The idea of whiskey distillation is used as a real-world example to engage organic chemistry students and teach the importance of skills such as boiling point determination, simple and fractional distillation, structure elucidation, IR spectroscopy, and critical thinking. Students do not distill real whiskey samples but rather mixtures containing two different whiskey-flavoring compounds.



Reference: *J. Chem. Educ.* **2014**, *91*, 123–125

Note To TA's

- Each section should have 6 groups.
- Pair each group with another group (we will call the alliance a TEAM) and assign each TEAM the same unknown. Here you should have three TEAM's each TEAM is assigned a different unknown, hence 3 total unknowns and three TEAM's.
- Ensure that 1 groups from the TEAM performs simple distillation and 1 groups from the TEAM performs fractional distillation. At the end of lab each TEAM will come together to complete share worksheet data and complete the conclusion.

Before you attend lab

- Read *Making the Connection*³ pages related to distillation (143-159)
- Read the article "Using Whiskey-Flavoring Compounds to teach Distillation and IR Spectroscopy to First-Semester Organic Chemistry Students" – This article can be found in the module.
- Waste: All waste into flammable organic waste.

Objective: To demonstrate conceptual and technical understanding of simple and fractional distillation as well as boiling point analysis.

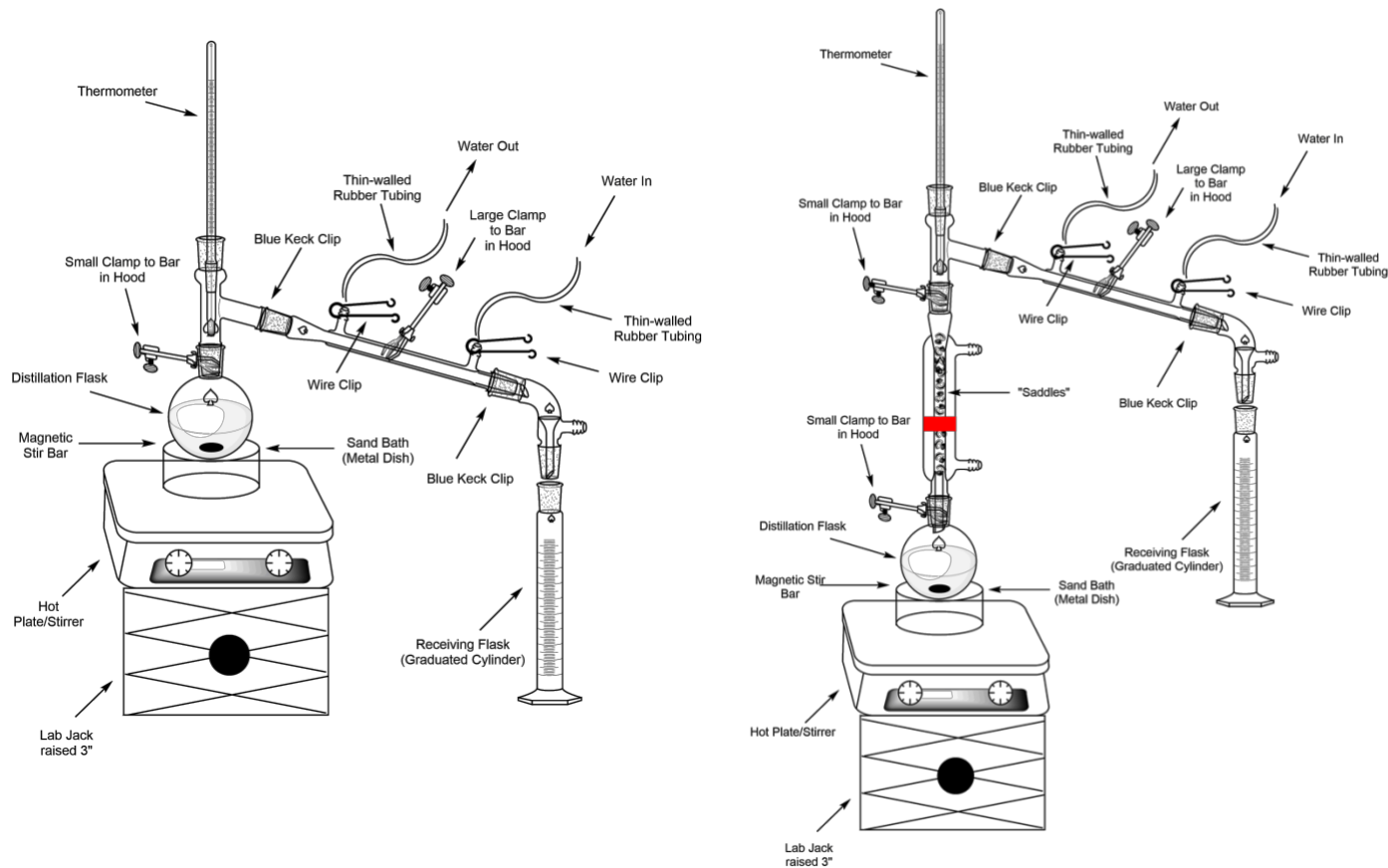
Goal of Experiment: Compare the utility of *Simple* and *Fractional Distillation* and identify under what circumstances each distillation method should be employed. Analytical analysis via boiling point determination will indicate purity of isolated distillate.

The Experiment: Teams will be assigned to perform either Simple or Fractional Distillation to isolate the low boiling point component of a two-component mixture. Once distilled the low boiling point component will be identified by Boiling Point Analysis. **Based on that information you will choose which whiskey extract, listed below, is your unknown. Boiling Points for the compounds below can be found at the end of this document.**

Whisky Name	Common Flavoring Compounds	
Gatsby Willow	2-Propanol	Ethyl Butyrate
Glen Quidditch	2-Butanone	3-Methyl-1-butanol
Lagascrappie	2-Butanone	1-Pentanol
Pedersons	Ethyl Acetate	3-Methyl-1-butanol
Tigamory	1-Pentanol	2-Pentanone

Theory:

In this experiment, you will be provided with an unknown sample containing two liquids that are organic flavoring compounds commonly found in whiskey. This sample has been obtained from a pre-extracted whiskey, in which the ethanol and water have been extracted to leave the two flavoring compounds behind. You need to distill this unknown mixture by either Simple or Fractional Distillation (your TA will assign method). The accuracy of your results is directly related to the care you take in performing the distillation. The degree of separation during distillation depends on the type of column used, the stability of the heat source, and the rate of distillation. Additionally, the packing in the fractionating column should be distributed evenly otherwise the efficiency of the column will be reduced. Fractional distillation requires patience because a slow rate of distillation is needed for good separation. You should assess the efficiency of each distillation method and then use the physical data obtained to deduce the structures of your two unknowns and determine which type of whiskey you were given. The structures of potential unknown components, their boiling points, aromas and the composition of different whiskeys are shown in the table. Please note the physical properties of these compounds as mixtures and pure compounds, note smell, color, viscosity, density, etc.



Simple Distillation.

Ensure that your distillation apparatus is clean and dry (rinse with a small amount of acetone to ensure flask is dry).

Set up the **Simple Distillation** apparatus and check it with your instructor before beginning the distillation. Using a long stem fluted funnel, add the unknown mixture (20 mL) to a round-bottom flask with one or two boiling stones. Place a clean, dry 100 mL graduated cylinder under the vacuum take-off to collect the distillate. Heat the mixture to a gentle boil and adjust the heating rate until the distillate collects at a regular rate of approximately one drop per second. **Record the temperature for every ~1 mL of distillate that comes over (This data should be graphed as shown on the worksheet).** Sometimes the heating rate has to be gradually increased to keep the distillation rate more or less uniform. **When approximately 10 mL of distillate has been collected, stop the distillation by removing and turning off the heat source.** Do not boil to dryness. Save the distillate for Boiling Point Analysis.

Fractional Distillation

Ensure that your distillation apparatus is clean and dry (rinse with a small amount of acetone to ensure flask is dry).

Set up the **Fractional Distillation** apparatus and check it with your instructor before beginning the distillation. Using a long stem fluted funnel, add the unknown mixture (20 mL) to a round-bottom flask with one or two boiling stones.

Mark five dry test tubes with a horizontal line representing 2 mL volumes.

***NOTE:** fill one test tube with 2mL of water and use a Sharpie to draw a line at the solvent level, use this as a ruler to mark all 5 dry test tubes with the Sharpie at the same level. This will allow you to collect in each test tube until the distillate has reached the line at which time you will replace with the next test tube).*

These test tubes will act as receiving vessels for 2 mL fractions. Gradually turn up the heat source until the mixture gently boils and adjust the heating rate so that the ring of condensate (vapors) rises slowly up the fractionating column. Lower the lab jack immediately if the column begins to flood with liquid. The rise should be very gradual to allow the column to acquire a uniform temperature gradient. Apply more heat only when you are sure that the ring of condensate has stopped rising, then increase the heat gradually. Adjust the heating rate so that no more than 20 drops (~ 1 mL) distills per minute. **Record the temperature when the first drop of liquid is collected, and when every 2 mL fraction is collected.** Keep this distillation rate constant by using slight increases in heating rate as required. Stop the distillation when approximately 10 mL are left in the distillation flask or when five fractions of 2 mL's have been collected.

Do not boil to dryness. Save the distillate for Boiling Point Analysis.

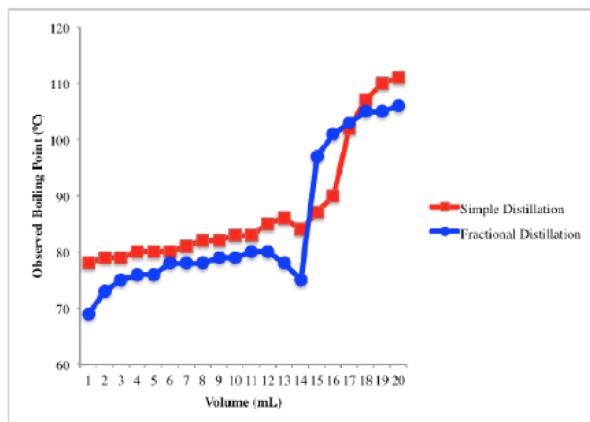
Decide which two fractions contain your distilled pure component representing the lower boiling point component from the mixture and determine its boiling point. Use the information you obtained from the boiling point to deduce the identity of the components in your mixture and hence determine the type of whisky you were given.

Sample of Graph: Represents temperature as a function of volume during a distillation. Please construct a graph plotting your groups experimental results of both simple and fractional distillation. The graph will be required on the Distillation Worksheet.

Part 3 – Boiling Point:

You will use the apparatus below to find the boiling point of the liquid to determine its identity. This will be submerged in a flask of water on the hot plate, ensure that the temperature is increases slowly. Review the video at <https://www.youtube.com/watch?v=8b5Ha-8QGhY>. Additional instructions on finding the boiling point can be found in the lab manual. Be sure to include the boiling point correcting formula, the TA will tell you the local atmospheric pressure for the day you come to lab.

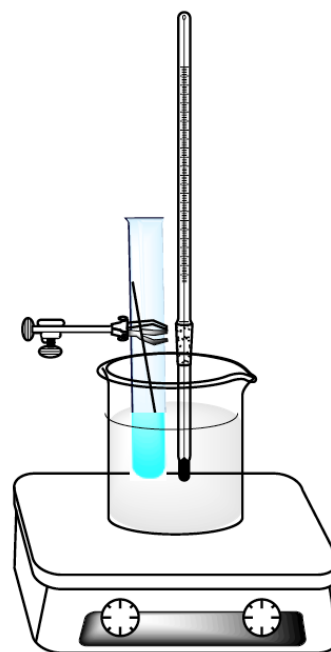
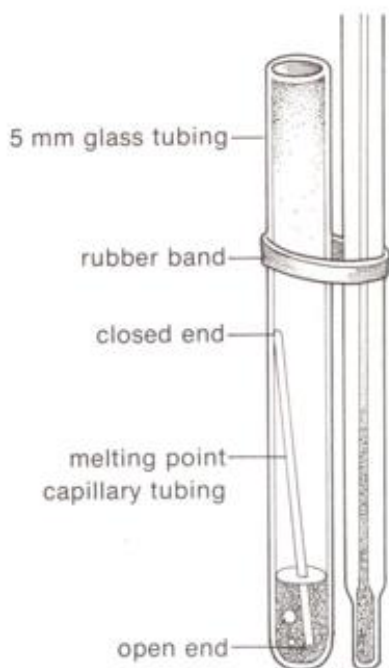
Graph Corresponding to Simple and Fractional Distillation Data



Equation for Calculating Corrected Boiling Point

$$\text{Boiling Point Correction Factor} = \left(\frac{760 \text{ torr} - \text{atmospheric pressure}}{10 \text{ torr}} \right) \times 0.5 \text{ } ^\circ\text{C}$$

$$\text{Corrected Boiling Point} = \text{Observed Boiling Point} + \text{Correction Factor}$$



Set up the apparatus shown above. Place your distillate in the test tube with a melting point capillary, open end down, placed into the distillate. Use a rubber band to attach the thermometer. Use a clamp to hold the test tube. Do not clamp the thermometer as it may crack, the thermometer will be held in place by the rubber band. Begin heating the water in the beaker with the hot plate. Once a steady stream of bubble begins to escape the capillary indicate the temperature (T_1). When the bubble stop flowing from the capillary record the ~~one~~ (T_2) once again. The experimental boiling point is the average of (T_1) and (T_2). Use the experimental boiling point in in the “Equation for Calculating Corrected Boiling Point”. Based on the calculated boiling point you will state the name of the *Unknown Whisky Sample*.

$$T_1 = 79 \quad T_2 = 83$$

LABORATORY CHEMICAL SUBMISSION SHEET - *Distillation*

GROUP No. 6
 GROUP MEMBER'S NAMES Tucker H, Tse ddiya, M, William V, _____
 EXPERIMENT TITLE & No. /DATE: _____ / _____

Unknown

Amount of Unknown: 20 mL mL

Record Observations of Aromas, Color, Viscosities, etc. of the Unknown Binary Mixture - See Aromas Below & State a Hypothesis to the Composition of Your Unknown.

Our unknown was clear, had a viscosity close to water, and smell was difficult to determine

OVERVIEW

I. SIMPLE DISTILLATION Half the groups in each section will perform Simple Distillation and Boiling Point Analysis.

II. FRACTIONAL DISTILLATION Half the groups in each section will perform Fractional Distillation and Boiling Point Analysis.

III. BOILING POINT – DETERMINE THE EXPERIMENTAL BOILING POINT (CHOOSES PUREST FRACTION OF DISTILLATE TO PERFORM THE BOILING POINT MEASUREMENT)

IV. BOILING POINT CORRECTION FACTOR CALCULATION

$$V. \quad \left(\frac{760 \text{ torr} - \text{Current Barometric Reading}}{10 \text{ torr}} \right) \times 0.5 \text{ } ^\circ\text{C} = \text{Boiling Point Correction Factor}$$

i. TO FIND THE CURRENT BAROMETRIC READ: WEATHER.COM

VI. **CORRECTED BOILING POINT = BOILING POINT CORRECTION FACTOR + EXPERIMENTAL BOILING POINT**

VII. IDENTIFY THE UNKNOWN

Whisky Name	Common Flavoring Compounds	
Gatsby Willow	2-Propanol	Ethyl Butyrate
Glen Quidditch	2-Butanone	3-Methyl-1-butanol
Lagascrappie	2-Butanone	1-Pentanol
Pedersons	Ethyl Acetate	3-Methyl-1-butanol
Tigamory	1-Pentanol	2-Pentanone



VIIIa: IDENTIFY THE UNKNOWN - Simple Distillation Group

Simple Distillation Team: _____ (indicate team)

1. Boiling Point Range of the Lowest Boiling Point Substance in the Binary Mixture:
T₁ = 79 T₂ = 83 81 BP
2. State the identity of the Lowest Boiling Point Substance in the Binary Mixture:
2-Butanone
3. State the identity of the Highest Boiling Point Substance in the Binary Mixture:
1-Pentanol
4. State the name of your Unknown Whiskey Extracts: Lagavulin

VIIIb: IDENTIFY THE UNKNOWN - Fractional Distillation Group

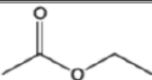
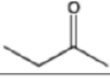

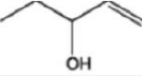
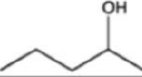
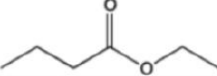

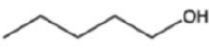
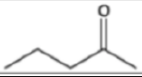
Fractional Distillation Team: _____ (indicate team)

1. Boiling Point Range of the Lowest Boiling Point Substance in the Binary Mixture:

2. State the identity of the Lowest Boiling Point Substance in the Binary Mixture:

3. State the identity of the Highest Boiling Point Substance in the Binary Mixture:

4. State the name of your Unknown Whiskey Extracts: _____

Ethyl Acetate		Fruit, Brandy	75-77
2-Butanone		Ethereal	79-81
2-Propanol		Butter	81-83
1-Penten-3-ol		Butter, Horseradish	114-116
2-Pentanol		Oily	118-120
Ethyl Butyrate		Banana, Pineapple	119-121
3-Methyl-1-butanol		Whisky	129-131
1-Pentanol		Sweet, Vanilla	136-138
2-Pentanone		Apple	101-105

LABORATORY CHEMICAL SUBMISSION SHEET - *Distillation*

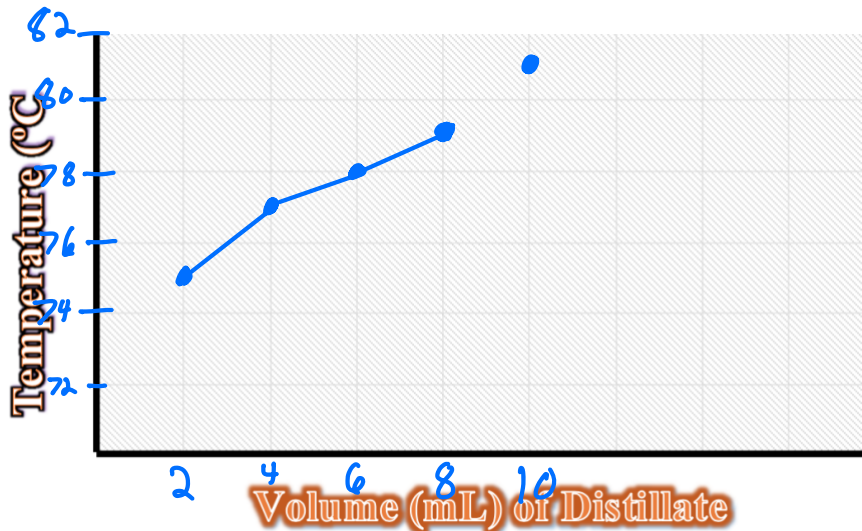
I.

SIMPLE DISTILLATION

DISTILLATE FROM FRACTIONAL DISTILLATION IS ANALYZED VIA BOILING POINT.

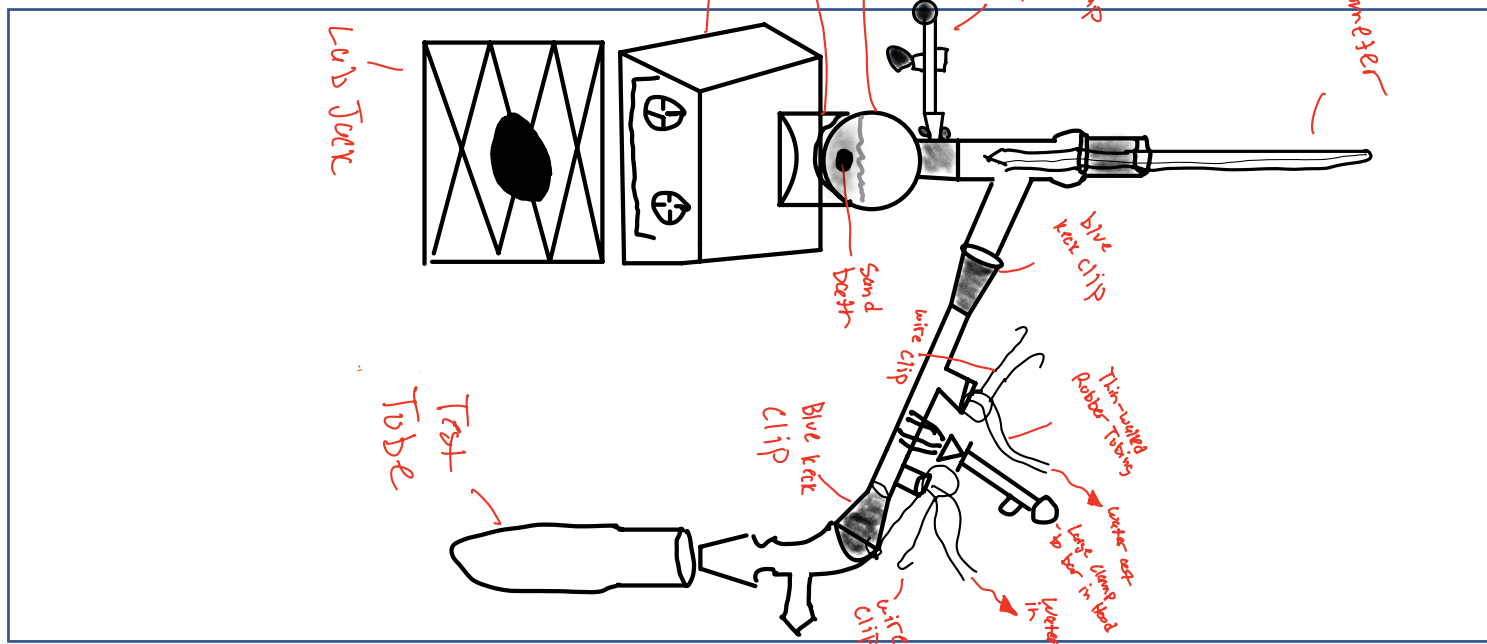
Start: 74

Simple Distillation	
Volume (mL)	Observed Boiling Point (°C)
2	75
4	77
6	78
8	79
10	81



DRAW A SIMPLE DISTILLATION SET-UP.

LABEL ALL CLAMPS, GLASSWARE, AND EQUIPMENT.



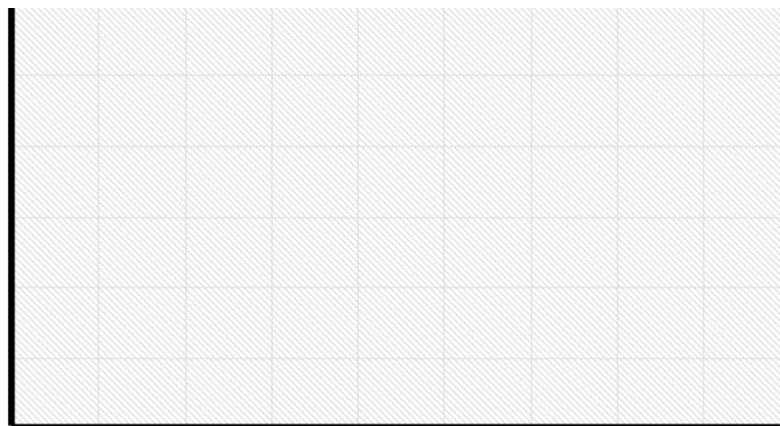
LABORATORY CHEMICAL SUBMISSION SHEET - *Distillation*

II. FRACTIONAL DISTILLATION

DISTILLATE FROM FRACTIONAL DISTILLATION IS ANALYZED VIA BOILING POINT.

Fractional Distillation	
Volume (mL)	Observed Boiling Point (°C)


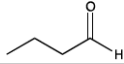
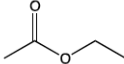


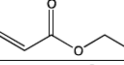
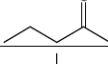
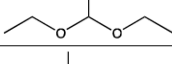
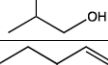
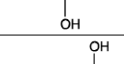
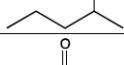
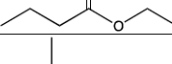

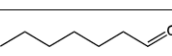
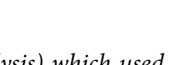
Temperature (°C)



Volume (mL) of Distillate

DRAW A FRACTIONAL DISTILLATION SET-UP.
LABEL ALL CLAMPS, GLASSWARE, AND EQUIPMENT.

Appendix: Boiling Point Values

Compound	Structure	Aroma	b.p. (°C)
Propionaldehyde		Almond, Apple, Banana	46-50
Butyraldehyde		Apple, Chocolate	74-76
Ethyl Acetate		Fruit, Brandy	75-77
2-Butanone		Ethereal	79-81
2-Propanol		Butter	81-83
Ethyl Acrylate		Fruity	98-100
2-Pentanone		Apple	101-105
1,1-Diethoxyethane		Honey, Floral	101-103
2-Methyl-1-propanol		Fruity	107-110
1-Penten-3-ol		Butter, Horseradish	114-116
2-Pentanol		Oily	118-120
Ethyl Butyrate		Banana, Pineapple	119-121
3-Methyl-1-butanol		Whisky	129-131
1-Pentanol		Sweet, Vanilla	136-138
Heptanal		Nutty, Fruity, Woody	152-154

Conclusion:

Groups should pair with another group (six total groups equals 3 teams for data analysis) which used a different distillation method. Groups should share fractional and simple distillation data to complete the work-sheet. In the conclusion below discuss what is distillation, what distillation is used for, the similarities and differences of fractional and simple distillation, when each method (Fractional or Simple) is appropriate to use, and finally discuss which method provided the most accurate results for today's lab.

Please note which group you collaborated with here: Group 1