

## Separation of Unknown Compounds by Distillation

*prepared by* Hayley Wan\*, Nada Djokic, Brenna Arlyce Brown, and Yonghoon Kwon, University of Alberta  
*modified by* Youngho Jang, Seulchan Lee and Sunkyu Han\*, KAIST

### PURPOSE OF THE EXPERIMENT

Separation of Unknown Compounds by Distillation

### BACKGROUND INFORMATION<sup>†</sup>

Laboratory techniques, such as boiling point determination, distillation, and Nuclear Magnetic Resonance Spectroscopy, are commonly taught during 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, Nuclear Magnetic Resonance Spectroscopy, and critical thinking. Students do not distill real whiskey samples but rather mixtures containing two different whiskey-flavoring compounds.

(Reference: Wan, H. et al. *J. Chem. Educ.* **2014**, *91*, 123.)

### Distillation

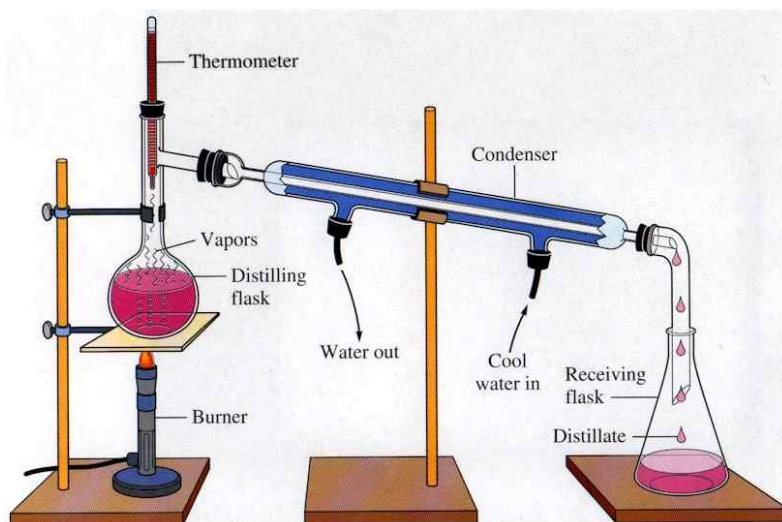
Whiskey is a common alcoholic drink consumed throughout the world. Different whiskies from different countries possess their own unique aromas and tastes according to the barrel aging and distillation processes. Chemically speaking, whiskies are very complex drinks that contain a vast number of organic compounds responsible for their flavoring and unique taste. A variety of aldehydes, ketones, lactones, phenols, alcohols, and esters are found in whiskies, and each produces different flavors.

For example,  $\gamma$ -nonalactone produces an apricot or peach-type flavor, whereas 4-hydroxy-3-methoxybenzaldehyde produces a vanilla-type flavor.

This experiment is based on the fact that different whiskies from around the world have different tastes and, therefore, contain different flavoring compounds. During this integrated experiment, students learn how to separate a mixture (containing two unknown whiskey flavoring compounds) using

simple and fractional distillation. Then, they use boiling point data and Nuclear Magnetic Resonance Spectroscopy of the pure fractions to determine the identity of the unknown liquids. Specific combinations of the two flavoring compounds correspond to fictitious whiskies that are detailed in the student handout.

Figure 1. Separating Liquids by Distillation



**EXPERIMENT A      Separation of Liquid mixture by distillation  
& characterization of isolated compounds**

**Reagents and Properties**

<i>substance</i>	<i>quantity</i>	<i>molar mass (g/mol)</i>	<i>mmol*</i>	<i>mp (°C)</i>	<i>bp (°C)</i>	<i>density (g/mL)</i>
Propionaldehyde	10.0 g				46-50	
Butyraldehyde	10.0 g				74-76	
Ethyl Acetate	10.0 g				75-77	
2-Butanone	10.0 g				79-81	
2-Propanol	10.0 g				81-83	
Ethyl Acrylate	10.0 g				98-100	
2-Pentanone	10.0 g				101-105	
1,1-Diethoxyethane	10.0 g				101-103	
2-Methyl-1-propanol	10.0 g				107-110	
1-Penten-3-ol	10.0 g				114-116	
2-Pentanol	10.0 g				118-120	
Ethyl Butyrate	10.0 g				119-121	
3-Methyl-1-butanol	10.0 g				129-131	
1-Pentanol	10.0 g				136-138	
Heptanal	10.0 g				152-154	

**PROCEDURE**

**Caution:** Wear lab coats and safety goggles at all times while in the lab. Many chemicals are potentially harmful. Prevent contact with your eyes, skin, and clothing. Wearing contact lens is strictly prohibited.

**Caution:**

Safety glasses and lab coats are required. Acetone, ethyl acetate, propionaldehyde, ethyl acrylate, 1,1-diethoxyethane, 2-propanol, 1-pentanol, butyraldehyde, 3-methyl-1-butanol, ethylbutyrate, 2-butanone, 2-pentanol, 1-penten-3-ol, heptanal, 2-pentanone, and 2-methyl-1-propanol are flammable and irritants. Heptanal is harmful to the environment and corrosive. Propionaldehyde, butyraldehyde, 3-methyl-1-butanol, and heptanal possess strong odors. Students are told not to consume or taste any of the samples or distillates.

**General procedure:**

Here we have an example of a typical distillation apparatus that we would use in a laboratory. We can use this setup to demonstrate the principle of distillation that would, in fact, be used on a commercial scale to produce our spirits. So in a typical distillation setup (Figure 1.), we have a heating source water or oil bath, because we need to be able to raise

the temperature of the substance that we're going to distill to its required boiling point. We have an adapter, because we need a thermometer to measure the temperature at which our distillation is occurring. Our adapter then links into this piece of apparatus here (Figure 1.), which is known as a condenser. We use tubing to basically run cold water around the outside of the condenser. And that's going to cool the vapour that we've produced by boiling and allow it to condense into our pure liquid here that we're going to collect in this receiving flask. When we reach the boiling point of **unknown compound 1**, only the **unknown compound 1** will start to boil. Our **unknown compound 2** isn't hot enough to boil. Therefore, by boiling and condensing, we can remove our **unknown compound 1** in RBF from the liquid mixture.

So we're going to turn the temperature on now (**higher than unknown compound 1 boiling point**), leave it, and start watching for bubbles to form within our liquid, because that's going to tell us when our distillation process is beginning. When we reach the boiling point of **unknown compound 2**, collect fraction in cylinder.

The number of compounds in the liquid mixture will be announced (**unknown compound 1, 2 and 3**). In this section, we are going to separate total 3 unknown compounds. Following the aforementioned protocol, **unknown compound 1** and **2** will be separated and collected in cylinder. The last compound, which has the highest boiling point among the unknown compounds, will remain in the round bottom flask. Therefore, three unknown compounds are separated by distillation. Then, by using the NMR, separated compounds will be characterized.

### **1. Setting-up Distillation Apparatus**

Based on the basic set-up of distillation (Figure. 1), set-up the distillation apparatus. **The gap between glasswares should be sealed by Teflon-tape.** Position of thermometer is important to measure the temperature of vapour. Do not contact any wires and tubes to the hot surface. This may leads to accidents.

[Heat-plate, RBF, stirring bar, supporting-jack, condenser 1 (air cooling), condenser 2 (water cooling), rubber tube, thermometer, oil bath]

### **2. Isolating Product**

Fill the 60 mL RBF with liquid mixture (20 mL of each liquid), the flask should be no more than two thirds full because there needs to be sufficient clearance above the surface of the liquid so that when boiling commences the liquid is not propelled into the condenser, compromising the

purity of the distillate. Stirring bar or boiling chips should be replaced in the RBF for some reasons (Pre-lab question 3).

In this experiment, **three unknown compounds with different boiling points are in the liquid mixture.** The boiling point of **unknown compound 1** will be announced by TA to set-up the initial temperature.

Heat the RBF slowly until the liquid begins to boil. Vapour will begin to rise through the neck of the distillation flask. As the vapor passes through the condenser, they will condense and drip into the collection receiver. Record the temperature at every 1 mL of distillate and the final volume of each distillate obtained.

As the distillate begins to drop from the condenser, the temperature observed on the thermometer should be changing steadily. When the temperature stabilizes, use a new test tube to collect all drops that form over 2~5 degree range of temperature. Collect about 2~3 mL fractions in premarked test tubes. As the temperature begins to rise again, switch to a new test tube to collect the distillate. This process should be repeated.

After collecting the unknown compounds, remove the heat source from the RBF. And clean-up the bench and wash the used apparatus by acetone and water. **Keep the used apparatus on your bench, not the front. Your apparatus will be checked by your TAs and this will be reflected in the score.**

### **3. Characterizing unknown compounds**

Obtain the NMR spectrum as directed by your TA. Based on the boiling point of each compounds recorded during the experiment, NMR spectrum, and above table, characterize the **unknown compounds 1, 2 and 3.**

#### **Pre-Laboratory Questions**

1. Summarize all MSDS's of chemicals used in this experiment.
2. What is the role of given apparatus in the distillation set-up? (thermometer, condenser 1, condenser 2)
3. Why are boiling chips added to the liquid mixture prior to the start of the distillation?
4. Explain about "azeotropic effect".

#### **Post-Laboratory Questions**

1. Assign peaks in  $^1\text{H}$  NMR spectrum to confirm the product.
2. Identify unknown compounds
3. Use a temperature-composition diagram to explain why

fractional distillation of two components is more easily accomplished if the components have boiling points that differ by 20 degree or more.

4. What happens if a fractional distillation is done very quickly by rapidly raising the temperature of the heat source?
5. If a fractional distillation failed to separate two components efficiently, what alternatives would one consider to effect clean separation by distillation?