SEPARATIONS LABORATORY

EBULLIOMETER TA INSTRUCTIONS

Ch En 445

Overview

- Students will have about 1 hour in the lab. Part of this will be doing experiments, but make sure they are answering the analysis questions during waiting periods.
- The apparatus will require about 1.5 hours to approach the desired equilibrium. Coordinate with the lab manager if you are unable to come in that far ahead of time to start the apparatus.
- Before operating the equipment alone, you should have passed it off with either your instructor, Will Davis, or Mike Beliveau.

General Instructions

1. Do the following to prepare for students.
    a. Learn how to run the equipment and pass off your understanding with either the instructor, Will Davis or Mike Beliveau.
    b. Perform the experiments that the students will do.
2. You should have set up specific times for students to perform the experiments. You will be in the lab during these times.
3. You will setup the apparatus before each lab hour following the instructions below.
4. After students are finished with the lab, you will shut down the apparatus following the procedures below.
5. You can grade assignments when in the lab and not answering student questions; however, you should be closely monitoring the students.
    a. Keep them on task.
    b. Ask questions to deepen understanding.

Apparatus

Figure 1 is a picture of the apparatus. Notice the following.

- The main power switch
- Heater On button
- Heater Off button
- Liquid fill cap
- Default valve position
- Temperature and Pressure displays

Figure 5 is a picture of the refractometer. This will be used to measure sample composition.

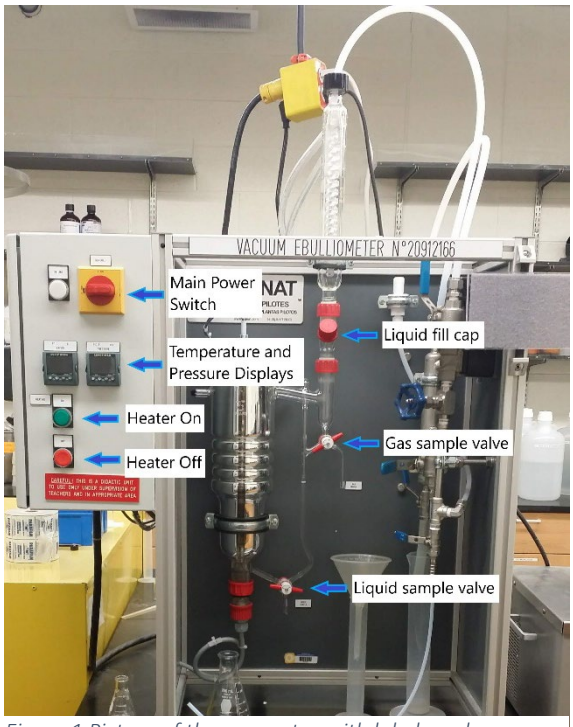

Figure 1 Picture of the apparatus with labels on key components

Set Up of the Starting Configuration

Do the following, **before students arrive**, if not already done.

- Turn on the cooling water refrigeration bath about 1 hour before turning on the heater.
- Check the fluid level in the ebulliometer. If the fluid level is close to or below the white mark on the distillate return line, refill the ebulliometer through opening the red liquid fill cap. Never operate the ebulliometer at a liquid level below the white line as this will expose the heating element, leading to possible damage. The liquid level must stay below the Y junction below the gas sample valve or the return flow of distillate will be impeded, which could lead to errors in composition readings.
- (NOTE: It takes about 60 ml to raise the liquid level 1 inch. At the white line, the ebulliometer contains about 250 ml and at the maximum liquid level, the ebulliometer contains about 380 ml. The nominal operating volume is about 350 ml.)

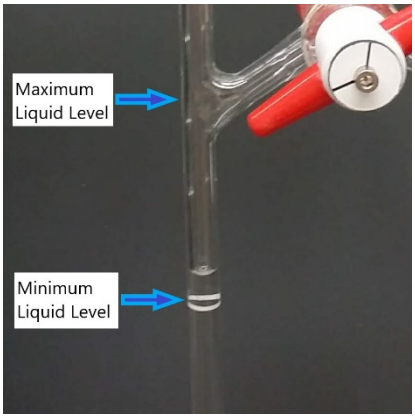

Figure 2 Ebulliometer liquid level reading

- Turn on the Main Power switch
- Make sure the valves are in the operating positions, shown in Figures 3 and 4. The black lines on the front of the valve indicate the flow path through the valve.
- Once the temperature and pressure displays show digits, turn on the heater by pressing the green button.
- The ebulliometer requires 30 minutes to reach equilibrium before students begin taking samples.
- Bumping (the forceful vaporization of superheated liquid when nucleation is hindered) may occur during heating, while this is a normal for the ebulliometer, it may pose a hazard if the fill cap is open when it occurs. To prevent this hazard, wait at least 30 minutes after boiling has stopped to open the fill cap.

Lab Operation Procedures

During the lab time, students will have only enough time to run one starting concentration, taking two sample sets, so they will be provided with a set of data points in their assignment to complete their analysis. Below is an overview of what the students will be doing during operation of the ebulliometer, more detailed instructions may be found in the Student Manual.

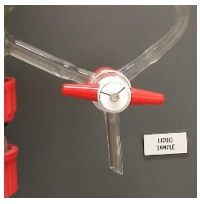

Figure 4 Liquid Sample Valve

Figure 3 Gas Sample Valve

- Sample collection
  - Students will collect samples from the sample valves into vials for testing
  - Vials will be provided by lab staff to collect samples for testing

- Sample testing
    - The samples will be tested for refractive index using a refractometer on the lab counter next to the cooling bath
    - Samples are loaded into the sample well using the provided pipettes
    - The refractometer is run, which will regulate the temperature and produce the measured refractive index

Figure 5 Refractometer

Shutdown Procedures

1. Turn off the heater by pressing the red "Stop" button.
2. Wait about 30 minutes for the liquid to cool.
3. While waiting for the apparatus to cool, make sure the pipettes and vials have been emptied into a waste vessel, empty waste liquid into the organic waste container in the flammables cabinet next to the counter and place used vessels inside the fume-hood to allow remaining liquid to evaporate.
4. Once the cooling water is back to 10^{o}C and the ebulliometer is approaching room temperature, turn off main power and shut off cooling water.