Ch En 385 – Knotts

Procedure

It takes 45-90 minutes for the system to reach steady state for a given set of operating conditions (composition, temperature, pressure), so the apparatus will be turned on and operating at steady state when you arrive. You will take one sample each of the liquid and vapor for this steady-state condition. You will be given more data for analysis.

- Put on gloves to handle the samples
- Gas Sample
 - Place a vial under outlet from the Gas Sample Valve.
 - Turn the Gas Sample Valve to the Sample Collection Position as indicated in Figure 1.
 Condensed distillate will begin to drip into the vial.
 - Collect between 0.5 1 mL of sample.
 - Return the valve to Operating Position as indicated in Figure 1.
 - Cap the vial to prevent evaporation.
 - Measure the refractive index of the
 - sample using the refractometer (see below).
- Liquid Sample
 - Place a waste container under Liquid Sample Valve.
 - Turn the Liquid Sample Valve to the *Drain Position* indicated in Panel A of Figure 2. Liquid will begin to flow into the waste container.
 - Drain 15 25 mL into the waste container.
 - Turn the valve so that flow stops as pictured in Panel B of Figure 2. DO NOT return the valve to default position as this will result in contamination of the liquid sample with the condensed distillate.
 - Place a vial under the liquid sample outlet.
 - Turn the Liquid Sample Valve to the position indicated in Panel C of Figure 2. Liquid will begin to flow into the sample container.
 - Collect between 0.5 1 mL.
 - Return the valve to operating position as indicated in Panel D of Figure 2.
 - Cap the vial to prevent evaporation.
 - Dispose of the waste in the organic waste container.
 - o Measure the refractive index of the sample using the refractometer (see below).



Figure 2 Liquid valve positions. A. Drain position, B. Stop position, C. Sample collection position, D. Operating position.



Figure 1 Gas Sample Valve positions. Left: Sample collection position. Right: Operating Position.

- Measuring the Refractive Index of a Sample
 - Remove the cover from the sample well (See Figure 3).
 - Uncap the vial and use a disposable pipette to place several drops of the sample into the well. Fill the well about halfway.
 - Replace the cover on the sample well and press the "Start" button shown in Figure 4. (Press anywhere on the screen if the interface is blank to wake up the machine.)
 - Replace the cap on the sample vial.
 - Wait for the machine to display the refractive index for the sample.
 - Record the refractive index in Table 1.
 - Clean the sample well on the refractometer with a Kimwipe and replace the cover.



Figure 4 Refractometer user interface. The arrows indicated the location of the Start button and the read out.

Online Procedure

- 1. Read the Procedure above to understand what is happening.
- 2. Watch the video at http://walk-inlab.groups.et.byu.net/ChEn_385/Ebulliometer/EbulliometerTest1.mp4.
- 3. Watch the video at http://walk-inlab.groups.et.byu.net/ChEn_385/Ebulliometer/EbulliometerCloseUp.mp4.
- 4. Review the first video so that you can record the Data in Table 1. (You can get all four parts of this table from the video, which shows temperature in °C and pressure in mbar.)

Analysis

- 1. Download the file ethanol-cyclohexane-ebulliometer-data.xlsm.
- 2. Use the data in the worksheet RI Calibration to generate a function to calculate the *mole* fraction of an ethanol/cyclohexane mixture given its refractive index.
 - a. Remember that these data were generated by mixing the different *weights* listed in the columns.
 - b. You need the mole fractions to generate the activity coefficients using Equation 2 of the prelab.
- 3. Add the point you measured (in Table 1) to the data in worksheet Ebulliometer Data.
- 4. Calculate the liquid and vapor mole fractions of ethanol for the data in Ebulliometer Data. Create a T-x-y diagram for the ethanol/cyclohexane system from these data. Make the plot publication quality.
- 5. Calculate the activity coefficients of ethanol and cyclohexane from the data in worksheet Ebulliometer Data. Plot $\ln \gamma_{ethanol}$ vs $x_{ethanol}$ and $\ln \gamma_{cyclohexane}$ vs $x_{ethanol}$ on the same plot. Make the plot publication quality.
- 6. Describe the behavior of the T-x-y diagram. Does the system behave ideally?
- 7. Describe the behavior of the $\ln \gamma_i$ vs x_i diagram. Why does it look the way it does?



Figure 3 Sample well on the refractometer.

- 8. Imagine you loaded the ebulliometer with a mixture of 15% ethanol. You start the system and allow it to reach equilibrium at a pressure of 860 mbar. Would the vapor be enriched in ethanol or cyclohexane?
- 9. Imagine you loaded the ebulliometer with a mixture of 85% ethanol. You start the system and allow it to reach equilibrium at a pressure of 860 mbar. Would the vapor be enriched in ethanol or cyclohexane?
- 10. Imagine you loaded the ebulliometer with a mixture of 44% ethanol. You start the system and allow it to reach equilibrium at a pressure of 860 mbar. Would the vapor be enriched in ethanol or cyclohexane?

Table 1 Experimental Data

Temperature (°C)	Pressure (mbar)	RI Liquid	RI Vapor