ChEE 326
Spring 2004
Vapor-Liquid Equilibrium Experiment

Objectives:

The goal of this experiment is to use the modified Raoult's law equation to predict vapor-liquid equilibria for the acetone-cyclohexane system using Pxy data. Your group will need to take data on:
1) the composition gas phases of several samples using a gas chromatograph (GC) with a flame ionization detector (FID)
2) the pressure of the vessel mixture using a manometer/pressure gauge
These activities should be expected to take approximately 3 hours for each group.

You will then use the Margules equation to fit your data before using the constants from that equation to estimate a Pxy diagram.

Methods and Materials:

You will determine the vapor-liquid equilibrium in a closed system consisting of several 250 ml glass bottles containing approximately 80 ml of liquid, sealed with a mini-inert valve. You have a GC with an FID that can separate and analyze the acetone and cyclohexane. The GC is equipped with a gas sampling valve that injects a sample when the knob is rotated to the "inject" position. You also have glass syringes with removable needles to obtain a gas sample from the bottle and transfer it to the injection loop.

The bottles with acetone and cyclohexane will have been prepared by Ron LeBlanc or the TA and will be in the laboratory space for your experiments. The original volume of each liquid will be noted on the bottles and these original volumes were measured accurately with volumentric pipettes. The TA will have the GC ready for you to begin your experiment. You should inject only the vapor into the GC - NO LIQUIDS!

The mini-inert valves are always sealed with a rubber septum, and may be sampled when the Teflon sleeve around the septum is moved out of the way by pushing the green button in. Note the temperature at the time of the experiment and the atmospheric pressure with the barometer in the Green Room.

Safety Issues:
1) You will be dealing with volatile and toxic chemicals during this experiment
2) Wear safety goggles at all times
3) Wear appropriate lab attire (including shoes, not sandles; no shorts)

Experimental Procedure:
1) Samples will be ready for you in labeled vials
2) Determine the vapor phase compositions using the GC by injecting at least 1.5 ml of the gas phase into the GC. Do as many replicates of each sample as needed, but you must do at least two of each sample.
3) The chromatograms will contain:
a) the area and b) the % area information. You should use judgement to decide which information to use (or use both to compare) in order to generate data that allows you to determine vapor phase compositions.
4) Once the experiment is completed and the system is turned off (by the TA), have the TA or instructor initial your data sheet

Suggested Analysis
1) The experimental data can be used to construct an x-y diagram. Determine if this system forms an ideal or a non-ideal mixture and if it has an azeotrope at the working temperature. First construct a diagram assuming that the composition of the liquid phase is maintained constant as samples are removed for each of the samples throughout the experiment. Second, if the composition is not constant, construct a diagram with calculated values of "x" from Raoult's law. Third, construct a diagram with calculated values of "x" using the modified Raoult's law and an activitiy coefficient correlation.

2) Construct a Pxy diagram
a) use your experimental data compared to Raoult's law
b) use your experimental data compared to modified Raoult's law
c) Plot all results on the same plot and discuss the implications
d) plot your experimental data and another set of experimental data from a published reference source at a similar temperature on the same plot and discuss how well they are in agreement.

3) Determine if an azeotrope for this sytem exists at the working temperature. If it does, determine the pressure and composition of both phases and the overall composition at the azeotrope.

4) Discuss the limitations and assumptions of both Raoult's law and the modified Raoult's law.

Report

Each group should submit <u>one</u> report. Reports are due by May 7th to 105E or 108 Harshbarger weeks after the experiment is performed. The reports should be written to a reading audience that is familiar with vapor-liquid equilibrium but not with this particular experiment. The reports should contain the following sections:
1) Introduction: describe the experiment, a general background on VLE, the conditions for equilibrium, ideal vapor phases, ideal solutions, Raoult's law, and the objectives of the experiment
2) Experimental: describe the experimental set up, discuss relevant aspects of the experiment, and problems encountered during the experiment
3) Results and discussion: present in table format the experimental results. Present xy and Pxy plots, comment on the results. Calculated values (models) should be represented by solid continuous lines and experimental data as individual points.
4) Conclusions: which model fits the data best? etc.
5) References
6) Appendices: data from the experiment, background calculations, etc.