ChE 434 – Spring 2008 – Multicomponent Distillation Experiment

To: ChE 434 Lab Teams 1-4
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Background

This experiment is very much like a realistic engineering application you might encounter on the job. The feedstock is a molar mixture of ~30% methanol, ~40% isopropanol (IPA), ~25% 2-butanol, and ~5% water. (These may change from lab to lab!). The distillation column has a fixed feed plate location (check it out), preheated feed (F = 100-150 cm^3/min), a total condenser, a partial reboiler, computer-controlled steam, reflux, and feed temperature (within allowed physical limits) as well as stage thermocouple readings that may be acquired on-line and saved to data files for trouble-shooting and post-analysis.

The experiment will be run by two groups (one team). The first group will run the column at total reflux, sampling at top and bottom to characterize total reflux. Then the first group will set reflux and feed conditions determined in the prelab calculations to achieve the team targets, sampling top and bottom at steady-state to determine how closely targets have been met. By this time the feed tank will be nearly exhausted, and the first group will hand over operations to the second, afternoon group. The second group will pump products back to the feed tank and mix the contents. The two groups should discuss conditions for a new and improved run that should more closely meet targets. The second group will run under these operating conditions sampling at steady-state from column at all sampling points. Sample analysis may take a few hours longer, averaging 8 minutes per GC sample. Collect and label samples for cold storage and return to complete GC analysis for every stage when possible; only top and bottom compositions and the temperature profiles need to be monitored during the experiment to determine steady state. Thorough preparation is beneficial and expected. At least one person must attend the running column at all times, preferably two for safety, especially when climbing the platform or making operating changes.

*Each team must schedule to meet with Prof. Korus for approval of prelab materials ***at least 24 hours*** before beginning their experiments. The two groups should work together on preliminary calculations and should combine all data for reports and presentations.

Objectives:

1. Separate methanol and isopropanol as products from an alcohol mixture using a staged distillation column. The maximum separation between top and bottom will first be determined (experimentally and theoretically) from total reflux conditions to see if the recovery constraints are feasible.
   **In the event that steady state cannot be reached within 3 hours for your predicted optimum operating conditions, collect enough data (temperature and compositions) to complete a post-lab analysis of what you have achieved*.

   <u>Team targets:</u>
   Team 1: recover 90% methanol, free of butanol with minimum IPA
   Team 2: recover 95% IPA in the bottoms with minimum methanol
   Team 3: recover 90% methanol and 80% IPA
   Team 4: recover 85% for both methanol and IPA

2. Compare column operating conditions at your target settings with theoretical predictions, both ideal estimates and realistic models. You may use Excel or ChemSep for both. You may use spreadsheet calculations and other estimation methods for convenience.

Determine:

A. Actual overall column efficiency, feed rate, temperature & composition profiles, reflux ratio, recovery, etc., for your operating condition(s).
B. Rerun Excel or ChemSep to fit your experimental results, including stage-by-stage calculations.

Preliminary Report:

Each group must predict and turn in a ***short written report* <u>at least 48 hours</u> *before lab*** containing the following information based on Fenske-Underwood-Gilliland (FUG) procedures, Excel or ChemSep models, and anything else deemed necessary; ChemSep also provides the FUG analysis, so Excel is not needed. While there are many items you must supply before running the lab, your responses should be precise and concise. The preliminary report should be a very brief memo style.

You must provide:

1. Minimum reflux ratio & minimum number of stages at total reflux.
2. Optimum, ideal feed plate location (Kirkbride equation): actual efficiency range 70-80%.
3. Recommended operating conditions: provide numerical answers where possible for the following parameters and describe in short how you will measure and/or control these parameters.
    a. Reflux ratio (zero to total reflux)
    b. Feed rate and temperature (how are these limited?)
    c. Steam pressure (assume saturated)
    d. Product flow rates
    e. Recoveries
4. Demonstrate how changing these operating conditions affects the steady-state column. Be concise!
5. Outline corrective actions during column operation to reach target recoveries. Be brief!
6. Determine the effect of non-ideal mixtures. Describe and recommend the an appropriate vapor-liquid equilibrium model for this system.
7. Safety report: required equipment, possible emergencies and appropriate responses. Be brief!

*Tour the distillation facilities to familiarize yourselves with the equipment and flow lines. I recommend a team meeting. Consider how the reflux is returned, controlled, adjusted; how to fill and drain the column; what to do if the reboiler level falls too low, how and in what direction the pumps work (BEFORE you turn them on); where they pull from; what valve(s) must be opened or closed, etc. After this, brief and well thought out questions regarding the design and operation are welcomed before your lab period.

** Take the initiative in starting up the column. Be prepared for quick calculations in lab to adjust reflux, steam pressure, or feed temperature quickly and correctly; this will require on-the-fly volumetric flow rate conversions to molar flow rates, and vice versa. Also, being able to convert GC data quickly into compositions while you work will aid you in the post-lab analysis and report preparation.

***ChemSep or Excel data printouts, graphs, and tables by themselves are not acceptable for prelab or final reports/presentations, neither will "raw data" printouts of spreadsheets and the like. These may be included in appendices for calculation examples and referencing. All useful and necessary data must be plotted or tabulated in a clear and accessible manner for reports and presentations with clear labels, as appropriate.

Experimental Procedure

1. To determine maximum column separation performance, operate at total reflux. It may take up to an hour to reach steady state. Take the necessary data to determine the number of equivalent ideal stages.
2. Feed at <150 mL/min. Initially, the distillation column will be operated at conditions that your preliminary calculations indicate should meet or exceed the required recovery. Observe and record the temperature profile to determine when the column has reached steady state. At steady state, collect and analyze samples of the distillate and bottoms by gas chromatography (GC) to determine mol% of each component and to see if you have obtained the required recoveries. The GC requires the injection of ~1 microL of a liquid sample. To determine recovery, you need flow rates and mixture densities.
3. Change the operating conditions, if necessary, in order to reach the desired recoveries and wait for steady state. Your computer predictions beforehand should guide you in determining the direction and magnitude of operating condition changes (e.g., reflux ratio, feed temperature, steam pressure). You should also be set up for quick in-lab calculations for unforeseen issues. Once the recovery is reached, analyze (GC) vapor and liquid samples taken from each stage and record temperature profiles.
4. If you see you are not meeting the target recovery of Objective 1, you can still collect enough meaningful data for making an excellent analysis and report.

Final Report or Presentation

Follow Dr. Drown's Engineering Reports Manual and include the following in the order and detail relevant for the report/presentation medium.

1. Give a brief overview of distillation (applications, etc.) with length and detail as appropriate to the presentation format: formal, informal, written or oral.
2. Summarize objectives of the preliminary work, experiment, and final model(s) used. This may basically be a repeat of the Abstract or Summary section that should precede a Formal Report's Introduction or an Oral Presentation's Background section.
3. Show fundamental equations used and summarize applicability, restrictions, assumptions, etc.
4. Describe equipment and operating procedures. Pictures and diagrams are very important.
5. Present the results: plots or tables, predictions & accuracies thereof, did you attain the goal and objectives, etc.
6. Summarize your findings, state important conclusions drawn from them, and make recommendations from your results, e.g., new operating conditions for desired recoveries, best model separation, implications for the future, etc.
7. Executive Summaries may also be followed by appendices, which could be exact duplicates of the Formal Report supporting documentation. However, in theory, it is unnecessary for the Executive Summary to have any appendices, as it is acceptable to simply refer to the Formal Report, presuming it in fact contains the referenced materials. In either case, organization and clarity are just as important as content.

Team and Experimental Run Schedule

1. Monday, Feb. 25th Groups 2 = morning and 1 = afternoon.

2. Tuesday, Feb. 26th Groups 3 = morning and 4 = afternoon.

3. Wednesday, Feb. 27th Groups 5 = morning and 8 = afternoon.

4. Thursday, Feb. 28th Groups 6 = morning and 7 = afternoon.

The morning group will start up the column beginning at 8:00 AM each morning. The afternoon group must be present for the 'handover' discussion at 12:00 noon