ChE 434 – Spring 2009 – Multicomponent Distillation Experiment

To: ChE 434 Lab Squads 1-4
Cc: Dr. David Drown, Lab coordinator, Dr. David MacPherson, Lab associate
From: Eric Aston, Distillation lab instructor, aston@uidaho.edu

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 pressure, reflux, and feed temperature (within allowed physical limits) as well as stage thermocouples that may be read on-line and saved to data files for trouble-shooting and post-analysis.

Experimental Plan

Two groups (squads) will work together on the same day of their experiments. The first (morning) group (Team 1) will characterize the column at total reflux and at their targeted conditions (based on prelab results). Sampling top and bottom liquids at steady state provides composition data (from GC analysis) to determine how closely targets have been met. By this time, the feed tank may be nearly exhausted, and the first group will hand over operations to the afternoon group (Team 2). Ideally, if time permits, the two teams should work together to hand-off column operations and complete sampling of all stages under this production scenario before the feed tank empties.

Before Team 2 target conditions can be set, the products must be pumped back into the feed tank and remixed. The two squads should discuss conditions for improving operations to meet afternoon targets. Team 2 will continue with a new steady-state production, sampling at all points of the column as feasible. At ~ 8 minutes/sample for GC analysis, a complete set of data may take a few hours to generate. Collect and label samples for cold storage and return at a later time to complete GC analysis 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 two persons must attend the running column at all times, especially when climbing the platform or making operational changes. Each squad must schedule (in advance) and meet with Prof. Aston 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 to be used in *individual* reports and presentations.

Objective 1: Separate methanol and IPA 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*.

Group (Squad) Targets:

Squad 1: recover 90% methanol, free of butanol and minimizing IPA
Squad 2: recover 95% IPA in the bottoms with minimum methanol
Squad 3: recover 90% methanol and 80% IPA
Squad 4: recover 85% for both methanol and IPA

Objective 2: Compare column operating conditions at your target settings with theoretical predictions (that is, “fit your data”), both ideal estimates and realistic models; that is, fit your data including feed rate, reflux and boil-up ratios, actual overall column efficiency and individual plate efficiencies, temperature & composition profiles, recoveries, etc., for your operating conditions. You may use ChemSep for both and other spreadsheet calculations or estimation methods as needed.

Preliminary Report:

Each group (squad!) must predict and turn in a *short written report* ***at least 48 hours*** *before lab* containing the following information based on Fenske-Underwood-Gilliland (FUG) procedures, Excel calculations, ChemSep models, and anything else deemed necessary. ChemSep provides the FUG analysis, which may also be accomplished by hand. Your preliminary team reports should be very brief in memo style.

You must provide:

1. Minimum reflux and boil-up ratios for your predicted compositions.
2. Minimum number of ideal stages: actual plate efficiency range is ~70-80%
3. Optimum feed plate location: compare Kirkbride eq. from FUG with full ChemSep simulations.
4. 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—how changing these operating conditions affects the steady-state column? Remember to be concise.
    a. Reflux and boil-up ratios (zero to total reflux)
    b. Feed rate and temperature (how are these limited?)
    c. Steam pressure (assume saturated)
    d. Product flow rates and compositions
    e. Recoveries
5. Outline corrective actions during column operation to reach target recoveries, related to #4.
6. Determine the effect of non-ideal solution behavior, recommending an appropriate vapor-liquid equilibrium model for this system based on simulation results.
7. Safety report: required equipment, possible emergencies and appropriate responses.

Tour the distillation facilities to familiarize yourselves with the equipment and flow lines. I recommend one or more team meetings beyond the "common training" session. Consider how reflux is operated; how to fill and drain the column; what to do if the reboiler level falls below the coils; 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.

NOTE: ChemSep or Excel data printouts, graphs, and tables by themselves are not acceptable for prelab or final reports and presentations, nor are raw data printouts of any kind. 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

The first squad of the day (Team 1) should take the initiative in starting up the column, with limited assistance from supervisors. Be prepared for quick calculations in lab to aid in your operating corrections; this will require on-the-fly volumetric flow rate conversions to molar flow rates, and vice versa, among other things. Also, being able to convert GC data quickly into compositions while you work will aid you in the post-lab analysis and report preparation.

1. (Team 1) Take the necessary data at total reflux to determine the number of equivalent ideal stages for the same separation. Observe and record the temperature profile to determine when the column has reached steady state. This typically takes roughly 30 minutes from startup.
2. (Team 1 & 2) Operate the column at your target conditions for production. Feed at ~100 mL/min. 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 μL of a liquid sample. [To determine recovery, you also need flow rates and mixture densities.] If time allows, Team 1 can make adjustments to optimize for their targets. You must have 30 minutes or more before hand-off to Team 2 to gather samples from at least half of the column.
3. (Team 2) Trade off with Team 1 at collecting and/or analyzing samples from the rest of the column. Then, change the operating conditions in order to reach the desired (Team 2) recoveries and wait for steady state. Your computer predictions should guide you in determining the direction and magnitude of operating condition changes (e.g., reflux ratio, feed temperature, steam pressure). Once the recovery is reached, analyze (GC) vapor and liquid samples taken from each stage (or as possible) and record temperature profiles.
4. If you see you are not meeting the targets 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. Why or why not? Discuss what you could have done better or differently.
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 separation model, implications for the future, etc.
7. Executive Summaries may contain appendices, which can be exact duplicates of the Formal Report's supporting documentation. In theory, it should be unnecessary for the Executive Summary to have any appendices, as it is acceptable to refer to the Formal Report, presuming it in fact contains the referenced materials. Either way, organization and clarity are as important as content.