SAMPLE FORMAL REPORT B

Chemical Engineering 4903 and 4905

The following sample laboratory formal report is not intended to represent the scope and depth of the projects assigned to students. It is an edited student report and contains some incorrect statements and formatting, and describes questionable experimental procedures. The report is intended to illustrate the organization and elements of an acceptable report as discussed in class, in the grading rubric, and in the lab handbook.

The comments in the margins of the report are intended to call the attention of the student to required report content. A student's report should not contain such comments in the margin.

The report is normally bound in some kind of report cover, and the cover letter is attached to the formal report by a paper clip. However, to simplify handling, bind the letter of transmittal inside the report front cover, ahead of the title page.

7400 S State Street apt 9202
Midvale, UT, 84047
December 6, 2006

Dr. Edward Trujillo
Beehive State Engineers
Salt Lake City, UT 84112

Dear Dr. Trujillo:

The average individual plate efficiency for the column at total reflux calculated experimentally is 31.64 ± 9.78 percent. The overall plate efficiency for the column at total reflux calculated theoretically is 38.46 ± 9.78 percent. According to J.D. Seader (2006) both efficiencies are lower than typical distillation column efficiencies which are around 70 percent. By Aspen simulation the optimum feed location for the isopropanol and water stream is tray 5 with a flow rate of 8 kg/min and a distillate concentration of 60.778 ± 3.062 mole percent isopropanol. The laboratory distillation column can be operated at finite reflux for 2.9 hours considering an actual reflux ratio of 8.357 and a boilup ratio of 0.4387.

The distillation column in the senior laboratory is capable of producing a 60 mole percent ethanol distillate. By applying the same assumptions for the isopropanol and water stream the resulting distillate for the ethanol/water solution will be 68.02 ± 6.51 mole percent ethanol with a recovery of 29.4 ± 4.21 percent. Higher ethanol recoveries could be achieved by evaluating reboiler and condenser duties, optimum feed location for the ethanol and water stream exclusively. The reported uncertainties are based on a 95 percent confidence level.

Sincerely,

Marcela James

Comment [T1]: Cover letter. Note that it should have no page number.

Comment [T2]: Return address.

Comment [ET3]: Note the date. Always date your letter or memo

Comment [T4]: Name and address of the project supervisor.

Comment [T5]: Salutation. This is to be written as correspondence.

Comment [T6]: This summary goes right into results. It's best to first give a little context and background, such as a quick description of the project objectives, relevant dates, and who worked on it.

Comment [ET7]: Results are shown with the uncertainty. For more information on significant digits see Jeter and Donnell, Chapter 2.16.

Comment [TASL8]: According to Jeter and Donnell: "An argument in favor of full justification is that readers do report that uneven right margins seem sloppy. This is certainly a judgment to be avoided in technical work at all costs. Since full justification does not retard the reader while it does improve the appearance, full justification is recommended, especially in reports with long letter-width lines in which the spacings are easy to adjust" (2011, p. 119).

Comment [T9]: Good one paragraph summary of the report, including principal quantitative findings and indication of uncertainty. Could cut back on the significant figures.

Comment [T10]: Conclusions & recommendations.

Comment [TASL11]: Always remember to sign your document.

Characterization of the Senior Laboratory Bubble Cap Distillation Column

by

Marcela James

Project No. 2M
Bubble Cap Distillation

Assigned: November 15, 2006
Due: December 6, 2006
Submitted: December 11, 2006

Project Team Members for Group A:
Amanda Stiff
Jared Stradley
Marcela James

Marcela James

Chemical Engineering Department
University of Utah
Salt Lake City
2006

Comment [T12]: No page number on the title page, but this would be Page i.

Comment [T13]: Title & Author(s)

Comment [T14]: Project number and category

Comment [T15]: Relevant dates

Comment [T16]: Team ID and team members

Comment [T17]: Signature and printed name

TABLE OF CONTENTS

Comment [TASL18]: “Main titles should be bolded, centered, and set off from surrounding text by blank lines above and below” (Jeter and Donnell, 2011, p. 65).

Comment [T19]: List all major sections and the page number on which they begin, with the introduction beginning on the 1st page. All headings should be identical to those found in the text.

Comment [TASL20]: Try to keep index alignment (Roman Numerals I-V) as even as possible (as is the case in Appendices A-F)

LIST OF FIGURES

Comment [TASL21]: A neat and organized example of Figures and Tables Indexes

Comment [T22]: List figure number, title and page. The titles should be sufficiently descriptive and identical to those in the text.

No Title Page

LIST OF TABLES

Comment [T23]: List table numbers, titles and pages. The titles should be sufficiently descriptive and identical to those in the text.

No Title Page

SUMMARY

Characterization of the Senior Laboratory Distillation Column,
Project 2M:
Marcela James (report author), Amanda Stiff, Jared Stradley.
Report Date: December 6, 2006.

The distillation column in the senior laboratory is capable of producing a 60 mole percent ethanol distillate. By applying the same assumptions for the isopropanol and water stream the resulting distillate for the ethanol and water solution will be 68.02 ± 6.51 mole percent ethanol with an ethanol recovery of 29.4 ± 4.21 percent. The average individual plate efficiency for the column at total reflux was 31.64 ± 9.78 percent. The overall plate efficiency for the column at total reflux was 38.46 ± 9.78 percent. Both efficiencies are lower than typical distillation column efficiencies which are around 70 percent (Seader, 2006).

By Aspen simulation the optimum feed location for the isopropanol and water stream is tray 5 with a flow rate of 8 kg/min and a distillate concentration of 60.778 ± 3.062 mole percent isopropanol. The laboratory distillation column can be operated at finite reflux for 2.9 hours considering an actual reflux ratio of 8.357 and a boilup ratio of 0.4387.

Higher ethanol recoveries could be achieved by evaluating reboiler and condenser maximum duties and optimum feed location for the ethanol and water stream exclusively. It is recommended that the major leaks in the column be fixed. It is anticipated that fixing the leaks will improve the quality of experimental data.

Comment [TASL24]: "Abstracts and executive summaries are short documents placed at the very beginning of reports. While they are almost always attached to report packages, they should be treated as separate documents, often placed on a separate sheet from the introduction sections of the reports. Abstracts and Summaries are designed to give busy readers clues about the content of your report. […] Summaries are long versions of abstracts. A summary differs from an abstract in that a summary provides a more detailed problem statement (often with motivation), it provides a longer treatment of methods, and it can give recommendations, including suggestions for future research, estimates of budget or of production schedules, and so forth. […] In your undergraduate classes, most report summaries will be between three-quarters of a page and one full page in length […] a one-page summary typically has three or four paragraphs, and these paragraphs respond to [the following] four questions:
Paragraph 1: What tasks was assigned?
Paragraph 2: What steps were performed to address task #1 and what result was reached on this task?
Paragraph 3: What steps were performed to address task #2 and what result was reached on this task?
Paragraph 4: What conclusion was reached?" (Jeter and Donnell, 2011, p. 13-14).

Comment [T25]: Summary will contain much of the same information as the Executive Summary, though it is not written as a letter to any one specific person. It is written for a more general audience.

Comment [ET26]: This summary contains almost all of the elements in the rubric- gives the results up front, mentions important discoveries, states recommendations and conclusions.

I. INTRODUCTION

According to Forbes (1948), the art of distillation dates back to at least the first century A.D. By at least the sixteenth century it was known that the extent of separation could be improved by providing multiple vapor stage contacts (Forbes, 1948). Throughout the twentieth century, multistage distillation was the most widely used industrial method for separating liquid mixtures of chemical components (Forbes, 1948).

Unfortunately, distillation is a very energy intensive technique, especially when relative volatilities of the components are similar. T.W. Mix (1978), reports that distillation in the United States in 1976 accounted for nearly 3 percent of the nation's entire energy consumption, where approximately 75 percent was consumed in petroleum refining alone. According to T.M Tham (1997), distillation processes also contribute to more than 50 percent of plant's operating costs. The best way to reduce the costs of existing and new units is by improving efficiency and operation via process optimization. To achieve this improvement a complete understanding of distillation principles and design is essential.

The primary objective of this project is to understand and characterize the bubble cap distillation column located in the senior laboratory. The purpose is to evaluate the possibility of separating an 8 mole percent ethanol in water feed into a distillate with a minimum of 60 mole percent ethanol. To accomplish this objective we will determine the number of theoretical stages by efficiency calculations based on an isopropanol and water separation. Currently used in the senior laboratory, an 8 mole percent isopropanol solution is being purified in the distillation column in question. The isopropanol and water information will be subsequently applied to the ethanol and water separation.

Comment [TASL27]: When introducing the formal report, the first few lines should relate specifically to your current research. Regarding the *Introduction* Jeter and Donnell write: "This section should describe the functional objective of the project, it should supply any *background information pertinent to the problem*, and it should raise the specific technical issues of questions that you need to address during the project" (2011, p. 64 Emphasis added). The key word here is *pertinent;* beginning an *Introduction* with an obscure reference to first- and sixteenth-century distillation techniques is not the best opening for drawing readers into the specific technical questions and/or issues that will be raised later in the report.

Comment [ET28]: Might expand on this, discussing why distillation is important to industry, as per rubric. References show you have researched the literature, but, even though distillation is old, newer texts are available.

Comment [TASL29]: While formal reports are always strengthened by supporting references, one must consider the timeliness of the reference. Considering this sample report was written in 2006, one should ask if citing a text written in 1978 (or 1948 as in the prior reference) is the best rhetorical selection that can be made? Has any other text updated or improved upon the older text? Can a more recent text be used to better illustrate your current work or current work in the field?

Comment [T30]: Establish the importance of the research. Demonstrate you have studied the background of the topic.

Comment [T31]: Short description of this project. The rest of the introduction should set the stage with relevant references, and this last paragraph should illustrate where your work fits into that setting.

Comment [TASL32]: First-person pronouns such as I, we, he/she, can be used *occasionally* in the formal report—typically only in the Summary, Introduction, Procedures, and Discussion sections—but should generally be avoided when not used for *specific rhetorical emphasis.*

II. THEORY

Comment [TASL33]: According to Jeter and Donnell: “Headings should appear alone in a line, they should be bolded, and they should be aligned in the left margin. A heading should follow a blank line, and text should begin in the next line” (2011, p. 65).

Comment [T34]: Theory is developed from fundamentals. Theory must be sound and all equations used to analyze data included. Note though that the expected formats may vary depending upon your audience.

M. T. Tram (1997) defines distillation as a process in which a liquid or vapor mixture of two or more substances is separated into its component fractions of desired purity, by the application and removal of heat. The application of distillation can roughly be divided in four groups: laboratory scale, industrial distillation, distillation of herbs for perfumery and medicinals (herbal distillate) and food processing. According to The Distillation Group, Inc (2006), industrial distillation is typically performed in large, vertical cylindrical columns known as distillation towers or distillation columns with diameters ranging from about 65 cm to 6 m and heights ranging from about 6 m to 60 m or more.

Comment [TASL35]: Some of this information would fit more easily in the Introduction section. Typically, a Theory section only discusses the particular theory used in the experiment(s) being described in the current research project

During the steady state operation after startup, and with no external disturbance, the system is said to be in equilibrium or steady state. This means that the feed rate, composition and temperature are constant, the distillate and bottoms flow rates are constant, and the temperature and pressure on each tray in the column is constant (Distillation Group, 2006). More importantly, the great advantage of continuous distillation is that if the feed rate and feed composition are kept constant the product rate and quality will be also constant (Distillation Group, 2006).

Separation of components from liquid mixture via distillation depends on the difference in boiling points of the individual components. Also, depending on the concentrations of the components present, the liquid mixture will have different boiling point characteristics (Tram, 1997). According to J.D Seader (2006), azeotropes form in liquid mixtures that have close-boiling species of different chemical types whose liquid solutions are non-ideal. At an azeotrope, the solution contains the given component in the same proportion as the vapor, so that evaporation does not change the purity, and distillation does not effect separation. For this project both ethanol and water and isopropanol and water solutions form azeotropes. Details of these azeotropes for Salt Lake City conditions are shown in Figure 1. The ethanol and water solution at 12.46 psi (0.848 atm) forms an azeotrope around 90 mole percent ethanol. The isopropanol and water solution at 12.46 psi forms an azeotrope around 68 mole percent isopropanol.

Comment [T36]: Literature reference to source of the theory used.

Comment [ET37]: Note MS Word does not recognize the word azeotrope; make sure to check that the spelling of these technical terms is correct. You can create your own dictionary in Word. Nevertheless, check all underlined words for misspellings.

Comment [T38]: Be sure to proofread your document and pay attention to your word processor’s red flags. This should be “affect”.

Comment [T39]: Note that “Figure” is capitalized when it is used as a title for a specific figure; same goes for “Table” and “Equation”.

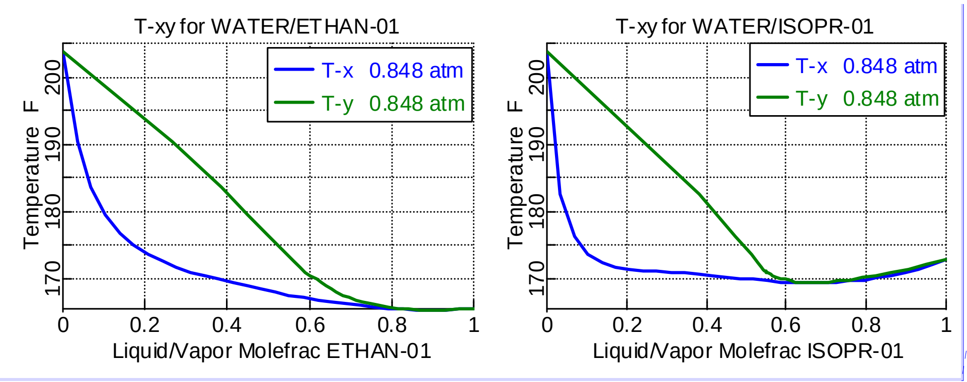


Figure 1 - Azeotropes for ethanol and isopropanol aqueous solutions.

Azeotrope for the ethanol and water solution forms around 90 mole percent ethanol. Azeotrope for the isopropanol and water solution forms around 68 mole percent isopropanol.

Equipment and Design Considerations

Distillation operations are most commonly conducted in trayed towers but packed columns are finding increasing use. Factors that influence the design or analysis of a binary distillation operation according to J.D. Seader (2006) include:

1. Feed flow rate, composition, temperature, pressure and phase condition.
2. Desired degree of separation between two components.
3. Operating pressure which must be below of the critical temperature of the mixture.
4. Vapor pressure drop, particularly for vacuum pressure operation.
5. Minimum reflux ratio and actual reflux ratio.
6. Minimum number of equilibrium stages and actual number of equilibrium stages (stage efficiency).
7. Type of condenser (total, partial or mixed).
8. Degrees of subcooling in the liquid reflux.
9. Type of reboiler (partial or total).
10. Type of contacting (tray or packing or both).
11. Height of the column.

Comment [T40]: Figures convey information efficiently, accurately, and aesthetically. Fonts are of appropriate size. Figures are titled, and captions are positioned below the figure and are sufficiently descriptive. They should be created with computer software of your preference.

It would be more appropriate to remove the titles above each graph, label these two graphs as subfigure A and B and describe each in the caption. For example:

Figure 1 - Azeotropes for ethanol and isopropanol aqueous solutions. A) Azeotrope for the ethanol and water solution forms around 90 mole percent ethanol. B) Azeotrope for the isopropanol and water solution forms around 68 mole percent isopropanol.

Comment [T41]: Figures should always include a title followed by a brief description placed directly below the inserted figure. However this student should not have put the title and description on different lines, or centered the text. See the example in my last comment.

Also note that the title and caption are at the bottom of the figure, as they should be. However, the figure title in the Table of Figures is different than the one shown here, which could potentially confuse the reader.

Comment [ET42]: Probably should be tray towers. Trayed towers is not a searchable term in Google.

12. Feed entry stage.

13. Diameter of the column.

14. Column internals.

Comment [TASL43]: While this information can appear as a list, oftentimes including it as a table, figure, or graphic is a must more effective way to present it. Always try to ask, “is there a better way to convey this information?”

McCabe-Thiele Graphical Method for Trayed Towers

Comment [T44]: Should not have figure titles at the top of the figure.

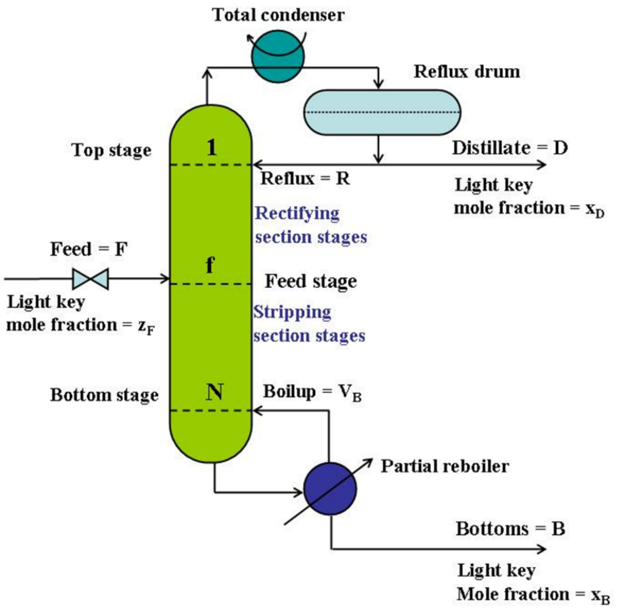


Figure 2 - Distillation Operation.

Distillation operation using a total condenser and partial reboiler adapted from Seader (2006).

Comment [ET45]: Note the reference. Anytime you take something from the literature and modify it, you need to reference where it came from. This is a very good example of describing the equipment schematically – clear, readable and not complicated. Arrows show flow patterns.

Consider the general countercurrent flow multistage binary distillation operation in Figure 2. The operation consists of a column containing N theoretical stages. A total condenser in which overhead vapor leaving the top stage is totally condensed to give a liquid distillate product and liquid reflux that is returned to the top stage. A partial reboiler in which liquid from the bottom stage is partially vaporized to give liquid bottoms product and a vapor boilup that is returned to the bottom stage, and an intermediate feed stage. The feed which contains a more volatile component the light key, and a less volatile component the heavy key, enters the column at a feed stage, f . At the feed stage pressure, the feed may be liquid, vapor, or a mixture of liquid and vapor, with its overall mole fraction composition

Comment [TASL46]: Take care to properly hyphenate and punctuate terms to avoid long strings of adjectival phrases such as this. This phrase should read: “countercurrent-flow, multistage, binary-distillation operation…”

with respect to the light component denoted by z_F. The mole fraction of the light key in the distillate is denoted by x_D, while the mole fraction of the light key in the bottoms is x_B.

The goal of distillation is to produce from the feed, a distillate rich in the light key and bottoms product rich in the heavy key. The ease or difficulty of the separation depends on the relative volatility, α of the two components, where the light key is denoted as 1 and the heavy key is denoted by 2. Thus the definition of relative volatility is given by,

$$\alpha_{1,2} = \frac{K_1}{K_2} \tag{1}$$

Comment [T47]: Equations are created in Microsoft Equation Editor or some equivalent program. Do not copy them as images into your report.

Equations are numbered and the numbers are shown at the right margin in parentheses.

The relative volatility can be expressed in terms of equilibrium vapor liquid compositions from the definition of the K value as the vapor liquid equilibrium of a binary mixture, where,

Comment [T48]: In this class symbols should be defined once in the text where they first appear, as well as in the Table of Nomenclature. In-text definition is not always necessary or required when you have a Table of Nomenclature, but it helps the reader.

Also be sure to use the same symbol and formatting in the text as in your equations. For example, “a” is not the same symbol as “*a*”. Most often students forget to italicize their symbols to match those in their equations, but that slight format change is a great way to help your reader find the symbols they are looking for in a mass of text.

$$K_i = \frac{y_i}{x_i} \tag{2}$$

Replacing in the relative volatility definition,

$$\alpha_{1,2} = \frac{y_1/x_1}{y_2/x_2} = \frac{y_1(1-x_1)}{x_1(1-y_1)} \tag{3}$$

Solving for y_1,

$$y_1 = \frac{\alpha_{1,2} \cdot x_1}{1 + x_1(\alpha_{1,2} - 1)} \tag{4}$$

In 1925 McCabe and Thiele published an approximate graphical method for combining the equilibrium curve with operating line curves to estimate for a given feed mixture and a column operating pressure the number of equilibrium stages and the amount of reflux required for a desired degree of separation of the feed (Seader, 2006). The feed flow rate is denoted as F, and the distillate and bottoms flow rate are denoted as D and B. The benefit of McCabe Thiele is that we can determine the number of stages N, and the number of the minimum number of stages $N_{\min}$ at R_∞ and the optimal stage for the feed entry.

Comment [TASL49]: Break awkward sentences such as this one, into two shorter, more cohesive sentences.

Comment [TASL50]: Always be consistent with terminology; this should read as “McCabe-Thiele method.”

Beside the equilibrium curve the McCabe Thiele method involves a 45° reference line. The

Comment [TASL51]: McCabe-Thiele

rectifying section comprehends all the stages above the feed stage. To proceed further all the compositions of all passing streams in the rectifying section must not vary from stage to stage. This is the case if:

1. The two components have equal and constant molar enthalpies of vaporization.
2. Component sensible enthalpy changes and heat of mixing are negligible compared to latent heat changes.
3. The column is well insulated so that the heat loss is negligible.
4. The pressure is uniform throughout the column, so pressure drop is negligible.

These assumptions are referred to as the McCabe Thiele assumptions leading to a condition of constant molar overflow. A material balance for the rectifying section is developed given as result,

Comment [T52]: Assumptions made in the theory are clearly stated.

$$y = \frac{L}{V}\cdot x + \frac{D}{V}\cdot x_D \tag{5}$$

Because of the constant molar overflow the reflux ratio, R, is a constant in the rectifying section, equal to L/D and $V = L + D$. So we can then define the reflux ratio in terms of the rectifying line slope,

Comment [TASL53]: This is an example of where the first-person pronoun should be avoided. As it appears, the use of "we" here conveys the idea of an omniscient narrator; in a Theory section this sentence would more simply and appropriately read: "then define the reflux ratio…"

$$\frac{L}{V} = \frac{R}{R+1} \tag{6}$$

with the intercept given by,

$$\frac{D}{V} = \frac{1}{R+1} \tag{7}$$

replacing in the rectifying line equation gives,

Comment [ET54]: Word choice

$$y = \left(\frac{R}{R+1}\right)\cdot x + \left(\frac{1}{R+1}\right)\cdot x_D \tag{8}$$

Comment [ET55]: Should be a period after this equation since it is the end of the sentence.

For the stripping section the vapor leaving the partial reboiler is assumed to be in equilibrium with the liquid bottoms product. Thus, the partial reboiler acts as an additional equilibrium stage. The vapor rate leaving it is called the boilup V_{N+1}, and its ratio to the bottoms product rate is,

$$V_B = \frac{V_{N+1}}{B} \qquad (9)$$

Where V_B is called the boilup ratio. Because of the constant molar overflow assumption V_B is constant in the stripping section. Since $L = V + B$, the operating line for the stripping section becomes,

$$y = \left(\frac{V_B + 1}{V_B}\right) \cdot x - \left(\frac{1}{V_B}\right) \cdot x_B \qquad (10)$$

To continue with further analysis the feed condition must be considered. To accomplish this task we need to consider material balances and energy balances to convert sensible enthalpy into latent enthalpy of phase change. This is done by defining the parameter q, as the ratio of the increase in molar reflux rate across the feed stage to the molar feed rate. The values of q for the different feed conditions are shown in Table 1 and graphed in Figure 3.

Comment [TASL56]: Again, pronoun usage.

Table 1- Values for q according to the feed condition.

The five feed conditions to determine q, adapted from Seader (2006).

Feed Condition	q
Subcooled liquid	>1
Bubble point liquid	1
Partially vaporized	L_F / F = 1 - molar fraction vaporized
Dew point vapor	0
Superheated vaport	<0

Comment [TASL57]: Much like inserted Figures, when Tables are inserted into the formal report they must always include a Title—signaling what data is being presented—as well as a brief account of where the data was acquired. However, unlike in Figures where the Title and description appear below, Tables always place the Title and description *above* the Table.

Comment [TASL58]: Care should always be taken when formatting figures and tables to avoided excessive amounts of white space on the page.

Comment [T59]: In industry or academia, you may often run up against a page limit for your text. Making the best use of the space you are allotted, without compromising on the look and readability of your document, is a good skill to develop.

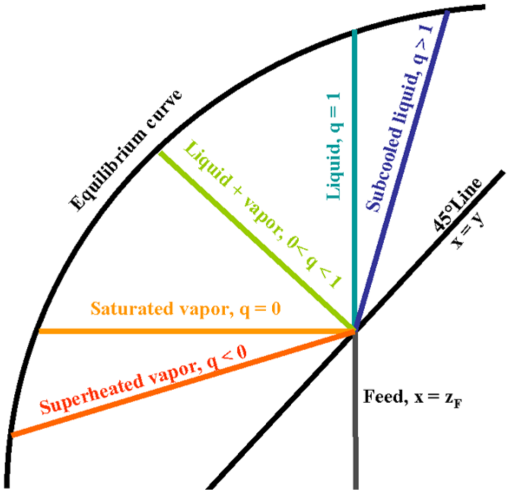


Figure 3 - Slope of q-line.

Effect of thermal condition of feed on slope of q-line adapted from Seader (2006).

Then the equation for the q line is:

$$y = \left(\frac{q}{q-1}\right) \cdot x - \left(\frac{z_F}{q-1}\right) \qquad (11)$$

Following the construction of the rectifying line, stripping line and the q line, we can then determine the number of stages. A typical set of these lines is shown in Figure 4. The stages can be stepped from the top all the way to the bottom. The stair case starts at x_D and finish at x_B .

Comment [ET60]: grammar

Comment [TASL61]: Again, excessive white space.

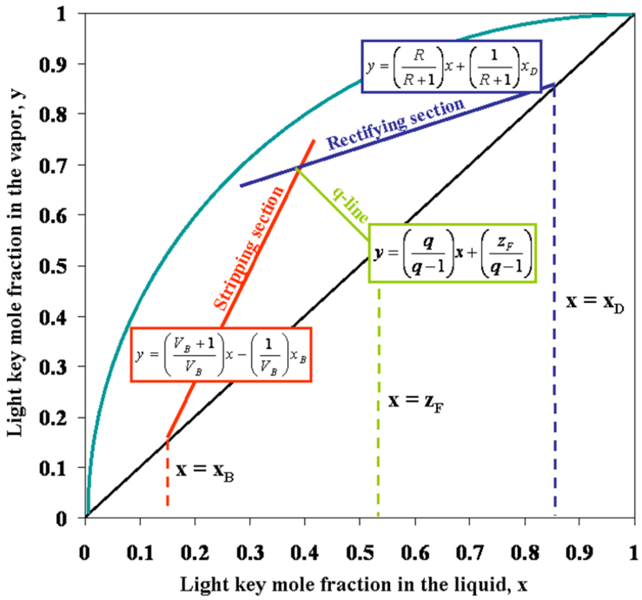


Figure 4 - Construction lines for McCabe-Thiele method.

Typical set of operating lines, assuming a partially vaporized feed, adapted from Seader (2006).

To determine the minimum number of stages the reflux ratio is increased to infinite, with a $L/V = 1$. Thus, at this limiting condition, both the rectifying and stripping operating lines coincide with the 45° line and neither the feed composition, z_F, nor the q-line influences the staircase construction. When $L = V$, $D = B = 0$ and the total condensed overhead is returned to the column as reflux, the column is functioning at total reflux. Furthermore, all liquid leaving the bottom stage is vaporized and returned as boilup to the column. Now both distillate and bottoms flow rates are zero, the feed to the column is also zero, which is consistent with the lack of influence of the feed condition.

Comment [TASL62]: Run-on sentence.

To determine the stage efficiency liquid or vapor samples are extracted from each tray at steady state. The efficiency for individual tray performance is called the Murphree plate efficiency. This efficiency can be defined on the basis of either phase and for a given component, is equal to the change in actual composition in the phase, divided by the predicted equilibrium. In industrial, countercurrent, multistage equipment the concentration changes for a given stage are usually less than predicted by equilibrium. The definition applied to the liquid phase can be expressed as,

$$E_{ML} = \frac{x_{n-1} - x_n}{x_{n-1} - x_n *} \tag{12}$$

Where E_{ML} is the Murphree liquid efficiency for stage n, where $n-1$ is the stage above and $x_n *$ is the composition in the liquid phase in equilibrium with the vapor composition leaving stage n.

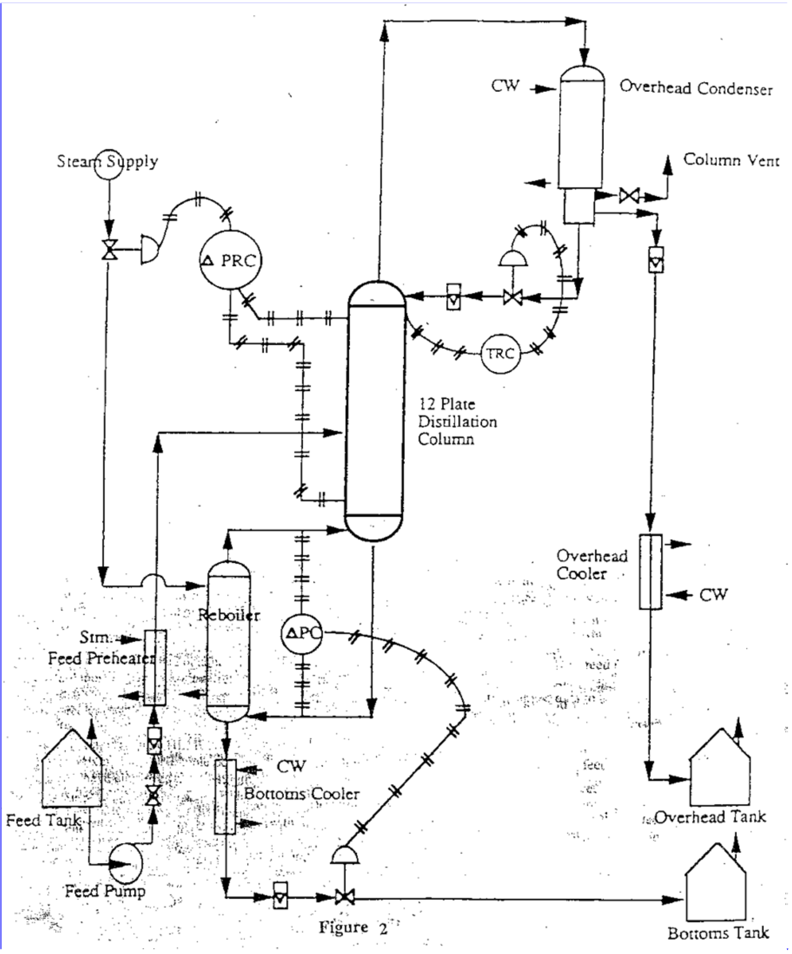
To determine the overall plate efficiency it is necessary to account for the actual number of stages of the column and the number of theoretical stages. Then the following equation can be solved to determine the overall stage efficiency,

$$E_O = \frac{N_T}{N_A} \tag{13}$$

Comment [ET63]: This is a very good example of a theory section – it generally follows the rubric and contains all the elements

III. APPARATUS AND PROCEDURE

The senior laboratory distillation column is three-stories high and is located at the left side of the laboratory entrance door. A diagram of the column with heat exchangers and major pieces of instrumentation can be found in Figure 5.



Comment [T64]: If you must copy a figure, make sure you give credit, as is done in this caption. Give credit even if the creator is your team member. You shouldn't plagiarize a block of text from anyone, and neither should you allow a reader to assume a figure in your report is your creation when it is the work of someone else.

Also it would be best for you to practice your illustration skills instead of copying figures, as illustrations are an important part of scientific communication. But if you do copy a figure, make sure you place it in your document straight and without the copy artifacts seen here. Such details may not be technically important but a lack of care could convey information to some readers that you would not want conveyed.

Figure 5 - Distillation column flow diagram.

Principal pieces of equipment of the senior laboratory distillation column copied from Ong (1952). Other pieces of equipment have been added, including a cold water bottoms cooler and a steam feed pre-heater.

The distillation column, the partial reboiler and the total condenser operate at atmospheric pressure. For Salt Lake City the atmospheric pressure is 12.46 psi or 0.848 atm. The feed is pumped from the basement and passed through a steam pre-heater before enters the column at tray number 9. Typical feed rates are between 3 and 8 kg/min for continuous feed. The senior laboratory distillation column was designed for liquid mixtures, avoiding components with high critical pressures and subsequently the use of a refrigerant. Water was used as the coolant in the two condensers. The total condenser at the top of the column is a single pass double-pipe heat exchanger for this project. The distillate removed from the column can then be cooled further with a single pass double-pipe heat exchanger. The partial reboiler is heated with steam and it vaporizes part of the bottoms functioning as an additional stage to the distillation process, adding a total of 13 stages. The distillation column has 12-plate trays and a diameter of 16-in.

Comment [ET65]: Probably should reference where this came from – web, literature, or actually measured in the lab – atmospheric pressure changes slightly depending on the barometric pressure

Procedure

Comment [T66]: The description of the experimental procedure should be detailed enough so that a chemical engineering student at another university could repeat the same experiments years later.

To characterize the isopropanol and water distillation process, two runs were made on November 27 and November 29, 2006. Before starting the feed tank had 160.95 gallons of a 4.77 mole percent isopropanol aqueous solution. To fulfill the project requirements we added 36.88 gallons of isopropanol for a final feed concentration of 8.3 mole percent isopropanol aqueous solution and a total of 197.83 gallons.

Comment [T67]: For all chemicals used in your work, information such as the full substance name, and vendor should be included in this section.

Comment [TASL68]: This is the correct usage of the first-person pronoun.

Comment [ET69]: Note the significant digits – the reader assumes the measurements were this accurate

The feed liquid samples were analyzed using a 2 ml pycnometer. By this method, accurate determinations were made from small samples instead of using reflective index measurements. For further discussion of why the density method was used instead of the refractive index, please refer to the results and discussion section. The density and specific gravity data for isopropanol and water solution was obtained from the seventh edition of Perry’s Chemical Engineering Handbook (1999). After approaching our target feed composition, we fed 17.4 gallons into the column and proceeded with the startup procedure for the distillation column.

Comment [T70]: For all equipment used, if you can find a manufacturer, you should include that information parenthetically after first mention of the equipment in the Methods section.

The bubble cap distillation column startup procedure based on the distillation column operation guidelines is as follows:

1. Checked in with laboratory manager.
2. Opened distillation program from desktop, and then click on Distill.
3. Entered feed data and set condenser flow at 50 percent on the Opto control computer.
4. Turned on ventilation system. Verified ventilation ducts were open for column and closed for other equipment.
5. Opened condenser water ball valve located on 3rd floor west wall behind column.
6. Verified reflux valves were set properly.
7. Set valves in basement pump room, and made sure that manual mix and feed valves were not completely shut.
8. Opened pump lubrication valve.
9. Opened main feed valve located under the feed tank.
10. Verified that both manual and Opto bottoms drain valves were closed.
11. Verified that the mix valve was open. Verified the manual and Opto controlled feed valves were both open.
12. Turned on pump and mixed tank for approximately 5 min.
13. After mixing was completed we verified the manual and Opto controlled feed valves were open then we closed the mixed valve and opened it approximately half a turn, just enough to allow flow to the feed tank. This is common practice to prevent damage of the pump.
14. Used the sight glass on the side of the feed tank to measure the amount of liquid. When the desired amount of solution was fed to the reboiler we stopped the pump and adjusted the steam valves located in the 2nd floor.
15. Verified that the steam bypass valve was closed, the main steam valve was open and the valve in line with the control valve was open but not touching the pneumatic line above it.
16. Verified product tank valves were set properly.
17. Entered control parameter into Opto and began distillation.

For total reflux operation we opened the coolant condenser valve at approximately 40

Comment [T71]: Unless you are writing a SOP, it would be best to write this procedure in a more compact, paragraph form, instead of a numbered list. Furthermore, you only need enough information so that someone else can repeat your work. For example, checking in with the lab manager is important but it is not info needed for the methods in a formal report.

Comment [TASL72]: Subject-verb agreement.

Comment [TASL73]: Write out numbers if they are ten or less.

Comment [TASL74]: As this portion of the Procedures section is written for an universal, not an individual, audience first-person pronouns should be avoided here; this sentence should read: “After mixing was completed, the manual was verified…”

percent. The steam valve was opened around 40 percent. The reflux valve was set at 100 percent. Total reflux operation speeds the attainment of steady state because there is no external disturbance. Steady state was considered to be reached when the temperatures of each tray, reboiler and condenser remained considerably stable. During the first run on November 27 of 2006 steady state was reached after 132 minutes. During second run on November 29 of 2006 steady state was reached after 90 minutes.

Liquid samples were obtained with a syringe at each tray. These were taken from the liquid sample tap that is installed on the column. The reflux and tray temperatures are measured with Iron-Constantan thermocouples attached to the outside of the trays. To calculate the tray efficiencies the liquid-vapor equilibrium data at Salt Lake City conditions were obtained from Aspen Plus software, Version 2004.1. The thermodynamic model used was NRTL.

Shutdown procedure based on the distillation column operation guidelines is as follows:

1. Notified laboratory manager when we were ready to shut down the column.
2. Verified that feed and steam valves were closed.
3. Verified all manual steam valves on the 2nd floor were closed.
4. Opened Opto controlled bottoms to 100 percent.
5. Opened manual bottoms drain valve in basement and waited until the column was drained to close the Opto controlled bottoms valve and open the manual bottoms drain valve.
6. Closed the pump lubrication valve and main feed valve under the feed tank.
7. Let the condenser water flow until column temperature is below 70 degrees.
8. Exited Opto control program.
9. Closed condenser water ball valve on west wall.
10. Shut off ventilation system.
11. Checked out with laboratory manager.

IV.RESULTS AND DISCUSSION

The bubble cap distillation column was run two times at total reflux, on 11/27/06 and 11/29/06. The raw temperature and flow data can be found in the attached CD-Rom. The specific gravity correlation data and how it was used to determine the isopropanol compositions are reported in Appendix A. The density values, mole percent concentrations and efficiencies for each run are reported in Appendix B. A complete error analysis and sample calculations are reported in Appendix C. The errors reported in this section represent a 95 percent confidence level.

Comment [TASL75]: In addition to describing "the data processing or calculation method," this section should "present the results in concise tabular and/or graphical form. [Also researchers should] compare results with literature or theory, citing the sources. If possible, explain the agreement or disagreement with comparative results. [Additionally] present any general results or overall conclusions based on this analysis" (Jeter and Donnell 2011, p.117).

Comment [TASL76]: Be consistent with dates; on page 12 the authors wrote the dates as: "November 27 and November 29, 2006."

Comment [T77]: Raw data. The reader is informed of the contents of the appendices, in case they want that extra information.

Comment [ET78]: Nice qualification.

Two sets of data were obtained in two different runs. During the first run on 11/27/06 approximately two hours were taken before staring to collect liquid tray samples. During the second run on 11/29/06 approximately an hour and a half was taken before collecting the samples, since steady state was reached faster. A strong alcohol smell was detected during the distillation operation, due to the leaks in the column. A comparison of mole percent isopropanol vs. tray number is shown in Figure 6 for both runs. The complete temperature profiles during sample collection are shown in Appendix D.

Comment [ET79]: Spelling – note it was underlined by Word

Comment [ET80]: Try to eliminate as many "the's as possible. This one is not necessary.

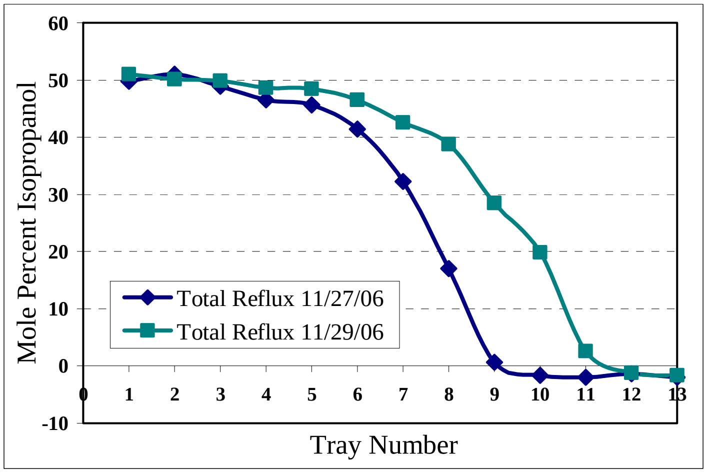


Figure 6 - Mole percent isopropanol vs. tray number.

The isopropanol concentration was determined by specific gravity and density for the two runs in the distillation column.

Comment [ET81]: Nice graph – clear and readable, but the labeling on the x-axis should be moved to the -10 line

As a consequence, the long wait in the first run reflected poor steady state behavior in the obtained results because of the complete evaporation of isopropanol in the four bottom stages. This is observable in Figure 6 where the concentration for isopropanol in stages 10 through 13 was below zero, indicating no presence of isopropanol. Also, during the first run temperatures for tray 8 and tray 9 were still varying at steady state. The second run data reflected adequate steady state behavior, in which temperatures and flow rate were basically constant. Only trays 12 and 13 had negative isopropanol concentrations. The 95 percent confidence error bars are added in Figure 6, but they are too small to be visually appreciable as will be observed in Table 2.

Comment [ET82]: This is a good paragraph, explains behavior and shows some thought. Don't just report results – explain them.

For the purposes of this project the second run on 11/29/06 will be the data considered for further predictions and calculations, because of its higher consistency. Results for the second run are shown in Table 2. For further results for each run please refer to Appendix B.

Table 2 - Results for the second run at total reflux.

All errors are based in a 95percentpercent confidence level.

Tray	Temperature ± 0.98°C	IPA Concentration mole ± 0.000417 %	Tray Efficiency %
1	78.03	50.9990	----
2	78.47	50.1325	2.056 ± 0.0163
3	78.70	49.8604	0.646 ± 0.0051
4	78.70	48.5996	3.013 ± 0.0241
5	78.89	48.4974	0.249 ± 0.0020
6	78.40	46.5472	4.947 ± 0.0420
7	79.58	42.5739	9.819 ± 0.0812
8	81.59	38.8220	9.711 ± 0.0841
9	83.94	28.5204	28.47 ± 0.2634
10	89.84	19.8549	31.37 ± 0.3803
11	92.29	2.5513	89.43 ± 1.5475
12	96.96	-1.1831	100 ± 19.2071
Reboiler	97.15	-1.6791	100 ± 11.8626

Comment [T83]: Units are given for values in tables.

The temperatures in the top trays were lower than in the bottom because they are farther away from the reboiler. The error reported in the temperatures is based on the instrument error of about 0.5°C, this is reasonable considering that Iron-Constantan (Type J)

Comment [ET84]: This shows that the student understands the equipment and its limitations. Very important to relay that to your reader.

thermocouples are used in each tray. Details of how thermocouples work are shown in Appendix F.

Comment [ET85]: This was actually an assignment in this case. Not really necessary unless your lab instructor asks you to do this.

The maximum isopropanol concentration between the two runs was 51.04 ± 0.000417 mole percent, not approaching the target concentration of 60 mole percent. A possible reason is the lack of a high enough temperature in the middle trays that would evaporate remaining isopropanol. Another possible reason is the presence of leaks on the column that permit the escape of vaporous solution. The mole percent error came primarily from the density measurements completed experimentally. Nevertheless, this error is smaller than the negative concentration values reported for trays 12 and 13 (reboiler). A reason for this is the lack of mixing at each tray developing zones where only water exists, giving as a result, negative isopropanol concentrations. If we would have used the refractive index (RI) method instead of density, the error in the concentration would be higher. This is due to the almost horizontal slope of the refractive index vs. concentration curve after 35 mole percent isopropanol where the refractive indexes are very similar. As a consequence using the refractive index method to measure concentration is not a good practice, because it adds chart reading error on the top of the experimental measuring error.

Comment [ET86]: Note the number of significant digits is wrong in this case. See Jeter and Connell, Chapter 2.16

Comment [ET87]: Good explanation – important result for reader

The average Murphree liquid efficiency or average individual plate efficiency is 31.64 ± 9.78 percent. The average individual plate efficiency error reported takes in consideration the error in both runs with a 95 percent confidence level. The individual tray efficiency error came primarily from the negative concentration values in the two bottom trays and the equilibrium concentration data. The efficiency for tray or plate number 1 was not measured because we did not collect liquid samples from the overhead condenser. The efficiency for trays 12 and 13 (reboiler) was 100 percent because a complete separation was achieved and only the presence of water was found in these trays.

Comment [T88]: An indication of uncertainty is reported.

Comment [T89]: A confidence level is made clear. A confidence interval is of no use without a confidence level.

To determine the overall efficiency a McCabe-Thiele diagram at infinite reflux is constructed for an 8 mole percent isopropanol aqueous feed and a distillate concentration of 60 mole percent isopropanol. Figure 7 shows the McCabe-Thiele diagram constructed to find the minimum of theoretical stages for the isopropanol and water solution.

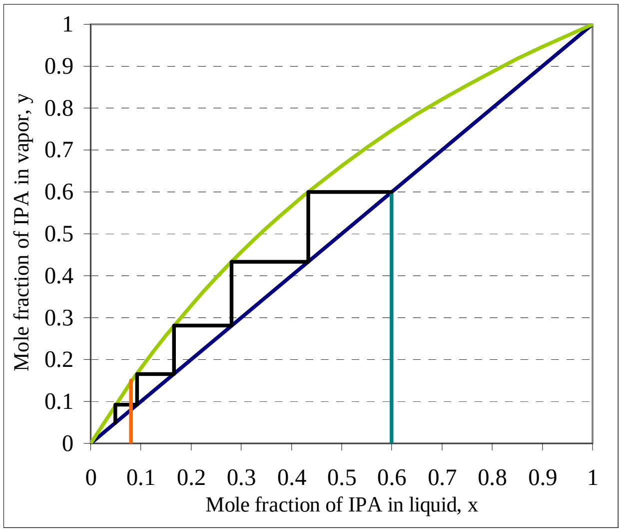


Figure 7 - McCabe-Thiele diagram for minimum stages at total reflux.

The McCabe-Thiele diagram constructed in Figure 7 used 88°C as the reference temperature, which is in between the boiling points of water (95.5°C) and isopropanol (78.23°C), at Salt Lake City conditions. To construct the equilibrium curve Equation 4 was used with a relative volatility, α of 1.96. A saturated liquid is the assumed condition for the feed, thus according to Figure 3, a straight vertical line will represent the q-line and the q value will be equal to 1. This assumption is accurate because before the feed enters the column it must pass a steam feed pre-heater that increases the temperature of the feed close to a bubble point liquid.

Comment [T90]: Specific equations from the Theory section are used and referenced in this section.

According to the McCabe-Thiele diagram at total reflux for an 8 mole percent isopropanol aqueous solution a minimum of 5 theoretical stages are necessary to achieve the separation. We then proceeded to calculate the minimum reflux ratio as a function of the slope of the rectifying line; this function is shown in Equation 6. The minimum reflux ratio, $R_{\min}$, to achieve this separation is 6.43. Furthermore, the actual reflux ratio for this case will be 8.357, which is 1.3 times the minimum reflux ratio. This assumption is based on McCabe-Thiele reflux ratio approximations reported on Seader (2006). To determine the bottoms composition, we assumed that it is located at the end of the fifth theoretical stage, representing a composition of 0.0493 mole percent isopropanol. A similar approach by the stripping section line is persuaded to calculate the boilup ratio, V_B by using Equation 9. Then the boilup ratio for this case is 0.4387.

The overall plate efficiency for the isopropanol and water solution is 38.46 ± 9.78 percent. This is calculated by Equation 13, assuming 5 theoretical stages and 13 actual stages. The error for the overall plate efficiency is the same as the average liquid tray efficiency as instructed by Dr. Trujillo. Comparing both efficiencies we can perceive that they are relatively close, however, they are lower than typical distillation column efficiencies which are around 70 percent (Seader, 2006).

To determine the optimum feed stage at a finite reflux ratio we decided to simulate the distillation operation for isopropanol and water solution into Aspen User Interface, Version 2004.1. The type of distillation column used was a RadFrac column, which considers each individual plate efficiency. The efficiency used to simulate the case was the overall plate efficiency 38.46 ± 9.78 percent. Further Aspen simulation details are shown in Appendix E. Attempts and results for the optimum feed location at finite reflux for a feed of 8 mole percent isopropanol in water solution are reported in Table 3.

Table 3 - Attempts to find the optimum feed location.

Attempt	Feed Tray	Feed rate (kg/min)	IPA in Distillate (mol %)	Condenser Duty (Btu/hr)	Reboiler Duty (Btu/hr)	Error in IPA (mol %)
1	6	3	61.925	---	---	---
2	5	3	60.778	---	---	---
3	5	5	60.779	---	---	---
4	**5**	**8**	60.778	-276259	370436	3.062
5	9	8	64.261	-276354	370613	---
6	13	8	65.282	-366946	461734	---
7	4	8	59.342	-276234	370386	---

The senior laboratory bubble cap distillation column is currently fed at tray number 9. However, the feed flow rate must be between 3 and 8 kg/min. Another aspect to consider is the water-isopropanol azeotrope formed approximately at 68 mole percent isopropanol.

Our objective is to identify the optimum feed tray and to reach more than 60 mole percent isopropanol getting as far from the azeotrope as possible. As a consequence, as soon as we reach our goal concentration we terminate our optimum feed location iteration, because there is a risk to approach the azeotrope where separation cannot be made.

To accomplish these restraints we began to run our case with the minimum feed flow rate at different stages. During this process we identified that at tray number 5 the target concentration was accomplished without getting to close to the azeotrope. We then decided to consider the effect on the increase of the feed flow rate from 3 to 8 kg/hr in the target concentration. The concentration results for the feed flow rate change where basically constant; this can be seen in attempts 2 to 4. With this in mind we decided to use a feed flow rate of 8 kg/hr, because we can get more product in a less amount of time and still be with in the distillation column specifications. We then decided to evaluate the change in the condenser and reboiler duties by varying feed tray to validate our optimum feed location. We noticed that when we increase the feed stage the reboiler and condenser duties increase substantially, this can be seen in attempts 9 and 13. However if we reduce the feed stage the condenser and reboiler duties will decrease, but our target concentration will not be met. This is the case for attempt 7. Consequently, the optimum feed location is tray number 5 with a feed flow rate of 8 kg/min and a 60.778 ± 3.062 mole percent isopropanol. The error reported is at 95 percent confidence level for this concentration and it is found by running Aspen at the same conditions and only varying the overall plate efficiency to its upper and lower limits, 48.23 percent and 28.69 percent respectively.

Comment [ET91]: Another spelling error identified by Word

Comment [ET92]: ditto

Our focus now shifts to determine how long we can operate our distillation column at finite reflux. We decided to take as reference the second run at total reflux from11/29/06, in which steady state was reached after 90 minutes. Before starting the run 197.83 gallons were present in the feed tank. A total of 17.4 gallons were used to reach steady state, leaving the feed tank with 180.43 gallons of remaining solution. We then assumed that after 90 minutes we would reach steady state, and as a result we would open the feed valve to a flow rate of 8 kg/min in conjunction with the bottoms and distillate valves. Then the total time to run the column at finite reflux would be the time to reach steady state added to the time to empty the feed tank. The total time to operate the distillation column at finite reflux is subsequently 171.6 minutes or 2.9 hours.

With the assumptions for the isopropanol and water solution inferred above, we will estimate the ability of our laboratory distillation column to process an 8 mole percent ethanol in water stream to produce a 60 mole percent ethanol distillate. To accomplish this task we simulated this process in a RadFrac distillation type column in Aspen User Interface, Version 2004.1. Results for this simulation are shown in Table 4. Further simulation details and results can be founded in Appendix E.

Table 4 - Results for the ethanol and water distillation

Limits	Feed Tray	Feed rate (kg/min)	Efficiency (%)	EtOH in Distillate (mol %)	Reflux Ratio	Boilup Ratio	Recovery (%)
Upper	5	8	48.23	70.878	8.357	0.4387	30.7
Average	5	8	38.46	68.019	8.357	0.4387	29.4
Lower	5	8	28.69	64.234	8.357	0.4387	27.7

The distillation column in the senior laboratory is capable of producing a 60 mole percent ethanol distillate; in fact the distillation column can produce a distillate with 68.02 ± 6.51 mole percent ethanol distillate if we keep the previous assumptions for the isopropanol and water distillation process. The ethanol recovery is 29.4 ± 4.21 percent. The errors reported are based on a 95 percent confidence level, and even if our composition reduces by 6.51 mol percent, we would still meet the target composition of 60 mol percent. Another advantage of the reported distillate composition is its distance with the ethanol and water azeotrope located at 90 mole percent ethanol.

Comment [ET93]: All in all this was a very good Results and Discussion section – followed the rubric for the most part.

V. CONCLUSIONS AND RECOMMENDATIONS

The average individual plate efficiency for the column at total reflux was 31.64 ± 9.78 percent. The overall plate efficiency for the column at total reflux was 38.46 ± 9.78 percent. Both efficiencies are lower than typical distillation column efficiencies which are around 70 percent (Seader, 2006). The data collected for the second run on 11/29/06 was found more reliable because it showed a very constant steady state during sample collection. The maximum isopropanol in the distillate between the two runs was 51.04 ± 0.000417 mole percent, not approaching the target concentration of 60 mole percent. A possible reason is the lack of higher temperature in the middle trays that can evaporate the isopropanol remaining.

By Aspen simulation the optimum feed location for the isopropanol and water stream is tray 5 with a flow rate of 8 kg/min and a distillate concentration of 60.778 ± 3.062 mole percent isopropanol. The laboratory distillation column can be operated at finite reflux for 2.9 hours considering an actual reflux ratio of 8.357 and a boilup ratio of 0.4387. The distillation column in the senior laboratory is capable of producing a 60 mole percent ethanol distillate. By applying the same assumptions for the isopropanol and water stream the resulting distillate for the ethanol and water solution will be 68.02 ± 6.51 mole percent ethanol with a recovery of 29.4 ± 4.21 percent. Higher ethanol recoveries could be achieved by evaluating reboiler and condenser maximum duties and optimum feed locations for the ethanol and water stream exclusively.

Major sources of error can be attributed to the time constraint to reach complete steady state. Another source of error is the leaks present in the distillation column that allow the escape of vaporous solution and the difficulty to reach some of the trays to obtain enough liquid sample. The first recommendation is to address the major column leaks that will improve experimental data and avoid the alcohol smell during operation. It is recommended to continue evaluating the isopropanol and water concentrations by the density and specific gravity method instead of the RI method, because it works better at higher concentrations giving more accurate concentration results.

Comment [TASL94]: According to Jeter and Donnell, this section should “Briefly summarize the project, and briefly recap all the important findings. As desired or appropriate, use paragraph or list style. Recall that a list must have some introduction and closure, and the items should be indented. The items in the list must be rhetorically parallel, meaning they should be presented in similar grammatical structures such as in all noun phrases. Do not introduce any new information in the closure” (2011, p.117).

Comment [T95]: Apparently the margins were changed in this section; such formatting should remain consistent through the document.

Comment [ET96]: vapors

Comment [T97]: Insightful, sound, and useful recommendations for future work are given.

Comment [ET98]: Again, good section – see rubric

NOMENCLATURE

Comment [T99]: Every symbol that appears in the text must appear in this table and be defined. The units of each must be given. If a quantity is dimensionless, a hyphen is used to so indicate.

Though it is not done here, symbols should be listed alphabetically, with Greek letters in a separate section, to make it easier for the reader to find a symbol of interest.

Symbol	Description	Units
D	Distillate flow rate	mol/min, kg/min
x_D	Light key mole fraction in the distillate	--
B	Bottoms flow rate	mol/min, kg/min
x_B	Light key mole fraction in the bottoms	--
F	Feed flow rate	mol/min, kg/min
z_F	Light key mole fraction in the feed	--
f	Feed stage	--
N	Number of stages	--
$N_{\min}$	Minimum number of stages	--
$R_{\min}$	Minimum reflux ratio	--
R	Actual reflux ratio	--
R_∞	Infinite reflux ratio	--
V_B	Boilup reflux ratio	--
α	Relative volatility	--
K	Ratio of vapor-liquid equilibrium compositions	--
x	Liquid composition	--
y	Vapor composition	--
L	Liquid molar flow rate in the column	mol/min, kg/min
V	Vapor molar flow rate in the column	mol/min, kg/min
V_{N+1}	Vapor molar flow rate leaving the reboiler	mol/min, kg/min
q	Ratio of increase in molar reflux rate across the feed stage to the molar feed rate	--
L_F	Liquid in the feed	mol/min, kg/min
E_{ML}	Murphree liquid efficiency for stage n	%
$x_n\,*$	Composition in the liquid phase in equilibrium with the vapor composition leaving stage n	--
x_n, x_{n-1}	Liquid compositions for stage n and stage $n-1$	--
E_O	Overall plate or stage efficiency	%
N_T	Number of theoretical stages	--
N_A	Number of actual stages	--

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7. Perry, Robert H., Editor. Perry's Chemical Engineering Handbook. 7th Edition. McGraw-Hill. New York. (1999)
8. Pico Technology Limited. Thermocuples. Cambridge United Kingdom http://www.picotech.com/applications/thermocouple.html (2006)

Comment [TASL100]: Although we recognize there are numerous ways of composing a Reference section, for our purposes in this class, all References should adhere to one of the two methods described in Jeter and Donnell *Chapter 2.6: Citing and Listing Reference (*2011, p. 139-145).

Comment [T101]: Every document referred to in the text is listed in the References. They should either be listed alphabetically or in a numbered list by order of their appearance in the text. Note that this author refers in the text by the author's last name and year but then lists references in (roughly) the order of appearance, which is not correct. You should use last name and year in the text if you list references alphabetically here, and reference by number if you list them in order of appearance.

Comment [ET102]: Note Jeter and Donnell's comments on web references, pg. 143-144.

APPENDICES

A. SPECIFIC GRAVITY CORRELATION DATA

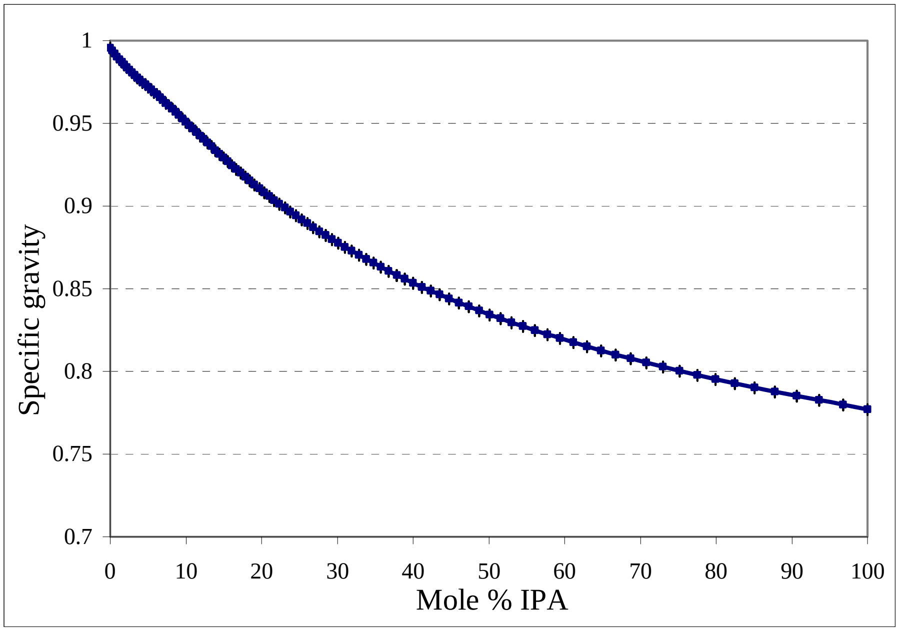


Figure 8 - Specific gravity correlation data

Correlation data for relating the specific gravity values to concentration of IPA water mixtures taken from Perry's Chemical Engineering Handbook 7th Edition (1999). All values are for 25ºC.

To determine the specific gravity of the sample we experimentally founded the density of water with a 2 ml pycnometer. The density is calculated by dividing the weight of the sample over 2 ml. Then we divided the density of the sample with the density of water to determine the specific gravity. Subsequently, from Figure 8 we read the appropriate concentration at a precise specific gravity.

B. SPECIFIC GRAVITY, CONCENTRATION AND EFFICIENCY DATA

Comment [T103]: The data collected. If you have extensive amounts of raw data, it may be more appropriate to hand in a CD or DVD with your report.

Note that units are always given along with some indication of uncertainty.

Table 5 - Total reflux data for the first run on 11/27/06

The high error at tray 9 is due to the negative IPA concentration in conjunction with the zero in the equilibrium concentration.

Tray	SG IPA ± 0.0687	Temperature ± 0.98°C	IPA Concentration mole ± 0.000417 %	Liquid equilibrium concentration, x_n* mole ± 17.081 %	Tray Efficiency %	Efficiency error %
Feed	0.9563	-	8.2926	-	-	-
1	0.8219	77.428	49.7978	-	-	-
2	0.8189	77.822	51.0416	14.471	-3.52%	0.033
3	0.8240	78.162	48.9072	10.414	5.25%	0.043
4	0.8301	78.288	46.4878	9.675	6.17%	0.053
5	0.8322	78.875	45.6430	7.545	2.17%	0.019
6	0.8434	78.862	41.4286	7.580	11.07%	0.097
7	0.8702	82.874	32.2759	3.121	23.89%	0.209
8	0.9224	88.58	17.0017	1.164	49.09%	0.528
9	0.9885	95.344	0.6624	0.018	96.21%	1.896
10	0.9988	96.975	-1.6678	0.000	100.00%	177.795
11	1.0002	96.432	-1.9824	0.000	100.00%	3.785
12	0.9975	96.81	-1.3750	0.000	100.00%	5.174
Reboiler	1.0004	96.507	-2.0160	0.000	100.00%	11.351
					Average error	16.749

Table 6 – Total reflux data for the second run on 11/29/06

The error reported in the average individual plate efficiency is an average of the efficiency error in both runs.

Tray	SG IPA ± 0.0687	Temperature ± 0.98°C	IPA Concentration mole ± 0.000417 %	Liquid equilibrium concentration, x_n* mole ± 17.081 %	Tray Efficiency %	Efficiency error %
Feed	0.9563	-	8.2926	-	-	-
1	0.8190	78.027	50.9990	-	-	-
2	0.8211	78.465	50.1325	8.8537	2.06%	0.016
3	0.8217	78.701	49.8604	8.0074	0.65%	0.005
4	0.8248	78.696	48.5996	8.0206	3.01%	0.024
5	0.8251	78.89	48.4974	7.5056	0.25%	0.002
6	0.8299	78.404	46.5472	9.08	4.95%	0.042
7	0.8403	79.576	42.5739	6.0837	9.82%	0.081
8	0.8507	81.593	38.8220	3.9369	9.71%	0.084
9	0.8822	83.942	28.5204	2.6388	28.47%	0.263
10	0.9119	89.837	19.8549	0.9002	31.37%	0.380
11	0.9803	92.294	2.5513	0.5067	89.43%	1.547
12	0.9966	96.955	-1.1831	0	100.00%	19.207
Reboiler	0.9988	97.147	-1.6791	0	100.00%	11.863
					Average error	2.793

C. SAMPLE CALCULATIONS AND ERROR ANALYSIS

Comment [T104]: Detailed sample calculations with all data sources. Explanation of how the calculation is made. This should be similar in style and detail to what appears in most textbooks.

Instead of what is done here, it would be better to show the equations with data for at least one data point.

Comment [ET105]: Note that all the equations are typed, making a neat report.

The error associated with the specific gravity measurements was ± 0.0687. This was based on the density measurements of water and the samples. To account for the error in density we accounted for the instrument error in the weight scale and the 2 ml pycnometer. The following are the estimated measured uncertainties of instrumentation:

- Weight scale reading, σ_m ± 0.00001 grams
- Volume reading, σ_V ± 0.1 ml
- Thermocouple reading, σ_T ± 0.5°C

To calculate density and specific gravity we used Equation 14 and Equation 15 respectively.

$$\rho = \frac{m}{V} \tag{14}$$

$$\delta = \frac{\rho_{sample}}{\rho_{water}} \tag{15}$$

The error in concentration of IPA is ± 0.000417 mol percent and it was found by using the definition of slope as found in Equation 14.

$$m = \frac{\Delta y}{\Delta x} \tag{16}$$

The error on any measurement can be related to the other on the opposite axis by rearrangement of this equation. The same method was used to find the error in the equilibrium concentration required in the individual plate efficiency; this value is ± 17.081 mole percent isopropanol. The error reported in the temperatures is based on the instrument error of about 0.5°C, this is reasonable considering that Iron-Constantan (Type J) thermocouples are used in each tray. We could of averaged the temperature values during steady state operation to obtain an error, but these values were considerably smaller in comparison of the thermocouple instrument error.

To determine the error in the individual plate efficiency we used Equation 17, which is the partial derivation of each variable with its respective the error propagation. The original individual plate efficiency expression is defined in Equation 13.

$$\sigma_E^2 = \left(\frac{1}{x_n{*}-x_{n-1}}\right)^2 + \left[\left(\frac{x_{n-1}-(x_n{*}-x_{n-1})}{(x_n{*}-x_{n-1})^2}\right)\cdot\sigma_{x_{n-1}} + \left(\frac{x_n}{(x_n{*}-x_{n-1})^2}\right)\sigma_{x_{n-1}}\right]^2 + \left[\left(\frac{-x_n}{x_n{*}-x_{n-1}} + \frac{x_{n-1}}{(x_n{*}-x_{n-1})^2}\right)\sigma_{x_n{*}}\right]^2$$

D. TEMPERATURE PROFILES AT TOTAL REFLUX

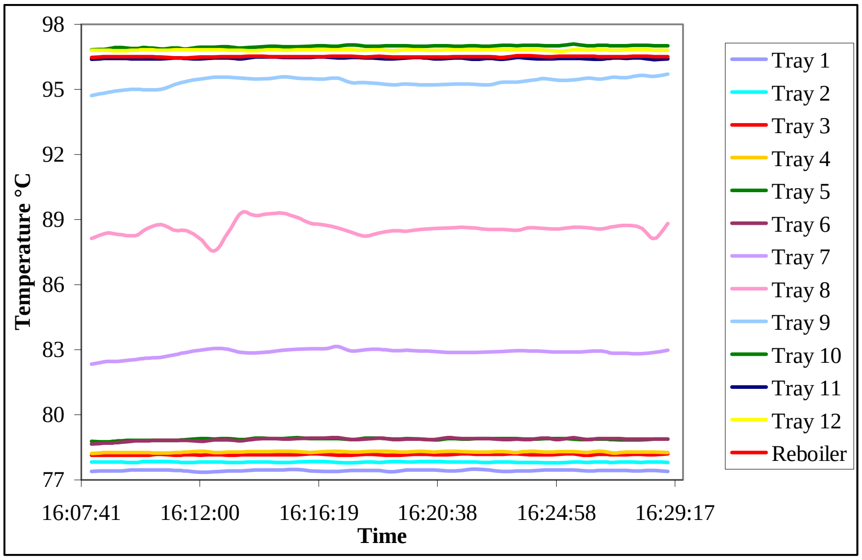


Figure 9 - Temperature profiles during sample collection for first run on 11/27/06.

As indicated before the tray 8 and tray 9 temperatures were still unstable during sample collection, affecting the data collected.

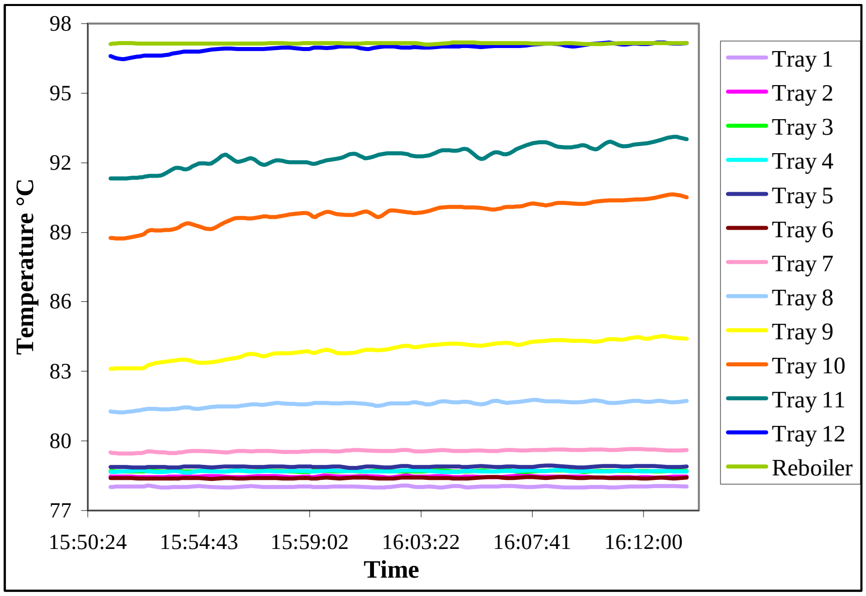


Figure 10 - Temperature profiles during sample collection for second run on 11/29/06.

Data seems more stable and there is a more appreciable temperature difference between the top trays.

E. ASPEN SIMULATION DATA

Comment [ET106]: If you use a commercial software package be sure to include all the important parameters used in your simulation, as done here. The reader needs to be able to duplicate your run. Probably should indicate what version was used.

Table 7 - Inputs for the Aspen distillation simulation

The same values are used for the isopropanol and ethanol simulation.

Feed		**Distillation column**		
Pressure	Flow	No of Stages	Condenser type	Condenser pressure
0.848 atm	8 kg/min	13	total	0.848 atm
Mole fraction		Reflux ratio	Boilup ratio	Reboiler pressure
IPA/EtOH	0.08	8.357	0.4387	0.848 atm
Water	0.92	Column type	Feed location	
		RadFrac	Tray 5	

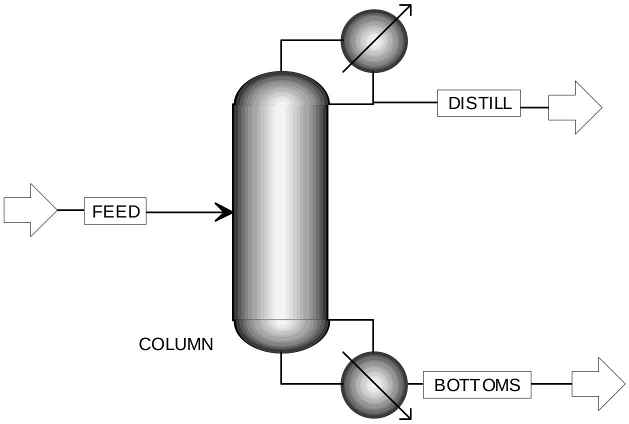
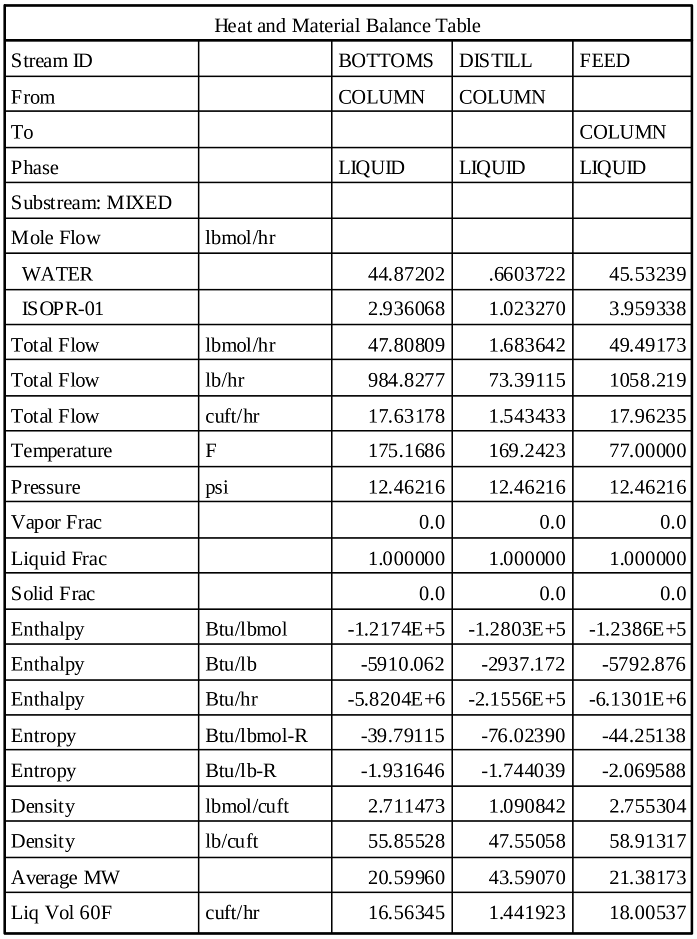


Figure 11 - Schematic of the streams for the Aspen simulation.

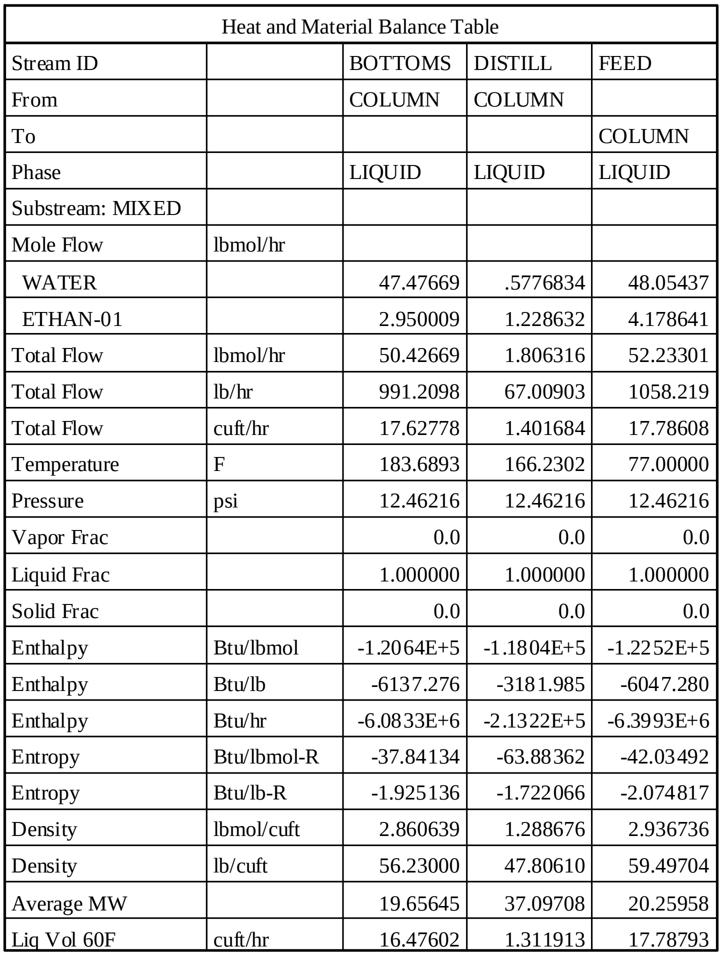
As mentioned before the column type used was RadFrac.



Heat and Material Balance Table				
Stream ID		BOTTOMS	DISTILL	FEED
From		COLUMN	COLUMN	
To				COLUMN
Phase		LIQUID	LIQUID	LIQUID
Substream: MIXED				
Mole Flow	lbmol/hr			
WATER		44.87202	.6603722	45.53239
ISOPR-01		2.936068	1.023270	3.959338
Total Flow	lbmol/hr	47.80809	1.683642	49.49173
Total Flow	lb/hr	984.8277	73.39115	1058.219
Total Flow	cuft/hr	17.63178	1.543433	17.96235
Temperature	F	175.1686	169.2423	77.00000
Pressure	psi	12.46216	12.46216	12.46216
Vapor Frac		0.0	0.0	0.0
Liquid Frac		1.000000	1.000000	1.000000
Solid Frac		0.0	0.0	0.0
Enthalpy	Btu/lbmol	-1.2174E+5	-1.2803E+5	-1.2386E+5
Enthalpy	Btu/lb	-5910.062	-2937.172	-5792.876
Enthalpy	Btu/hr	-5.8204E+6	-2.1556E+5	-6.1301E+6
Entropy	Btu/lbmol-R	-39.79115	-76.02390	-44.25138
Entropy	Btu/lb-R	-1.931646	-1.744039	-2.069588
Density	lbmol/cuft	2.711473	1.090842	2.755304
Density	lb/cuft	55.85528	47.55058	58.91317
Average MW		20.59960	43.59070	21.38173
Liq Vol 60F	cuft/hr	16.56345	1.441923	18.00537

Figure 12 - Aspen stream table for the isopropanol and water simulation.

These are the results to determine the optimum feed location for the isopropanol and water distillation process with an overall plate efficiency of 38.46 ± 9.7 percent.



Heat and Material Balance Table				
Stream ID		BOTTOMS	DISTILL	FEED
From		COLUMN	COLUMN	
To				COLUMN
Phase		LIQUID	LIQUID	LIQUID
Substream: MIXED				
Mole Flow	lbmol/hr			
WATER		47.47669	.5776834	48.05437
ETHAN-01		2.950009	1.228632	4.178641
Total Flow	lbmol/hr	50.42669	1.806316	52.23301
Total Flow	lb/hr	991.2098	67.00903	1058.219
Total Flow	cuft/hr	17.62778	1.401684	17.78608
Temperature	F	183.6893	166.2302	77.00000
Pressure	psi	12.46216	12.46216	12.46216
Vapor Frac		0.0	0.0	0.0
Liquid Frac		1.000000	1.000000	1.000000
Solid Frac		0.0	0.0	0.0
Enthalpy	Btu/lbmol	-1.2064E+5	-1.1804E+5	-1.2252E+5
Enthalpy	Btu/lb	-6137.276	-3181.985	-6047.280
Enthalpy	Btu/hr	-6.0833E+6	-2.1322E+5	-6.3993E+6
Entropy	Btu/lbmol-R	-37.84134	-63.88362	-42.03492
Entropy	Btu/lb-R	-1.925136	-1.722066	-2.074817
Density	lbmol/cuft	2.860639	1.288676	2.936736
Density	lb/cuft	56.23000	47.80610	59.49704
Average MW		19.65645	37.09708	20.25958
Liq Vol 60F	cuft/hr	16.47602	1.311913	17.78793

Figure 13 - Aspen stream table for the ethanol and water simulation.

These are the results to determine the feasibility of the ethanol and water separation with an overall plate efficiency of 38.46 ± 9.7 percent.

F. THERMOCOUPLES

Comment [T107]: This information was specifically asked for by the lab instructor, but would not be in one of your typical formal reports.

This report, though, does need an appendix of major items of equipment.

Sufficient detail should be given so that another researcher could obtain the equipment and repeat the same experiments.

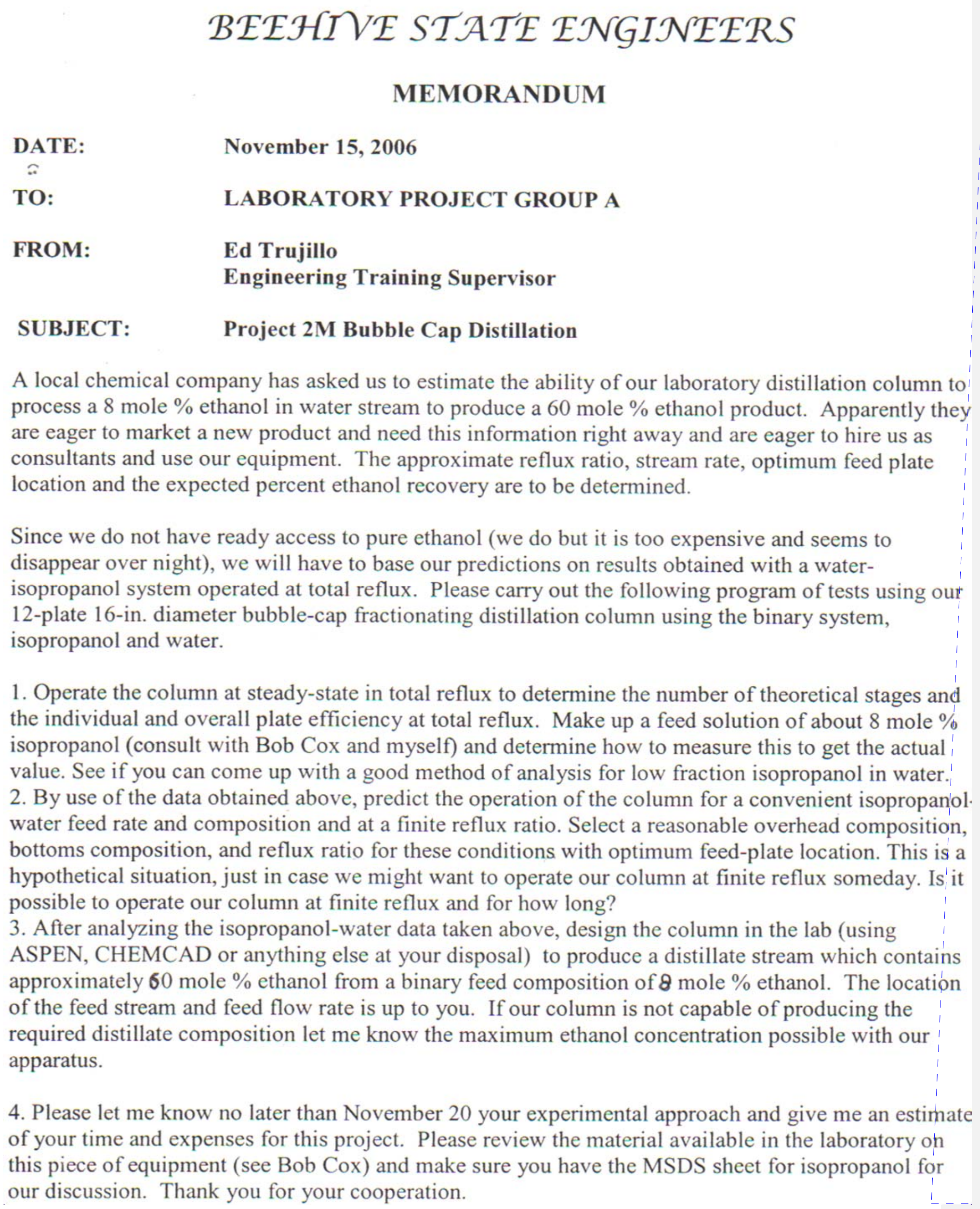
If chemicals were used in this project, they should be listed in that appendix with their manufacturing information (manufacturer, purity, lot number…).

Thermocouples take advantage of the junction between two metals because they generate voltage in proximity with one another. Conveniently voltage is a function of temperature. Thermocouples rely on this Seebeck effect. Although almost any two types of metal can be used to make a thermocouple, a number of standard types are used because they possess predictable output voltages and large temperature gradients. The diagram below shows a K type thermocouple, which is the most popular (Pico Tech, 2006).

To relate voltage and temperature measurement there is a technique known as cold junction compensation (CJC).When the thermocouple wires are connected to an instrument; two more thermocouple junctions are created, because the terminals are made of a different material than the thermocouple wires. These "extra" junctions, (called cold junctions), create their own Voltage, which alters the Voltage generated by the actual thermocouple. Cold junction compensation negates the voltage created by these cold junctions, allowing only the Voltage created by the thermocouple to be sensed by the instrument and determine the correct temperature measurement. (Pico Tech, 2006). Principal types of thermocouples include:

- Type K (Chromel / Alumel)**,** ‘general purpose' thermocouple. Available in the -200 °C to +1200 °C range. Sensitivity is approximately 41 µV/°C.
- Type E (Chromel / Constantan)**,** has a high output (68 µV/°C) which makes it well suited to low temperature (cryogenic) use.
- Type J (Iron / Constantan)**,** limited range (-40 to +750 °C). The main application is with old equipment that can not accept 'modern' thermocouples. These are the thermocouples used in the senior laboratory.
- Type N (Nicrosil / Nisil)**,** high stability and resistance to high temperature oxidation makes type N suitable for high temperature measurements without the cost of platinum (B,R,S) types.
- Type B (Platinum / Rhodium)**,** suited for high temperature measurements up to 1800 °C.
- Type R (Platinum / Rhodium)
  Suited for high temperature measurements up to 1600 °C. Low sensitivity (10 µV/°C) and high cost makes them unsuitable for general purpose use.
- Type S (Platinum / Rhodium)
  Suited for high temperature measurements up to 1600 °C. Low sensitivity (10 µV/°C) and high cost makes them unsuitable for general purpose use.

Comment [T108]: You should include the project statement at the end, so that we have it for future reference. However, this would not typically be part of a formal report in the workplace.



BEEHIVE STATE ENGINEERS

MEMORANDUM

DATE: **November 15, 2006**

TO: **LABORATORY PROJECT GROUP A**

FROM: **Ed Trujillo**
Engineering Training Supervisor

SUBJECT: **Project 2M Bubble Cap Distillation**

A local chemical company has asked us to estimate the ability of our laboratory distillation column to process a 8 mole % ethanol in water stream to produce a 60 mole % ethanol product. Apparently they are eager to market a new product and need this information right away and are eager to hire us as consultants and use our equipment. The approximate reflux ratio, stream rate, optimum feed plate location and the expected percent ethanol recovery are to be determined.

Since we do not have ready access to pure ethanol (we do but it is too expensive and seems to disappear over night), we will have to base our predictions on results obtained with a water-isopropanol system operated at total reflux. Please carry out the following program of tests using our 12-plate 16-in. diameter bubble-cap fractionating distillation column using the binary system, isopropanol and water.

1. Operate the column at steady-state in total reflux to determine the number of theoretical stages and the individual and overall plate efficiency at total reflux. Make up a feed solution of about 8 mole % isopropanol (consult with Bob Cox and myself) and determine how to measure this to get the actual value. See if you can come up with a good method of analysis for low fraction isopropanol in water.
2. By use of the data obtained above, predict the operation of the column for a convenient isopropanol-water feed rate and composition and at a finite reflux ratio. Select a reasonable overhead composition, bottoms composition, and reflux ratio for these conditions with optimum feed-plate location. This is a hypothetical situation, just in case we might want to operate our column at finite reflux someday. Is it possible to operate our column at finite reflux and for how long?
3. After analyzing the isopropanol-water data taken above, design the column in the lab (using ASPEN, CHEMCAD or anything else at your disposal) to produce a distillate stream which contains approximately 60 mole % ethanol from a binary feed composition of 8 mole % ethanol. The location of the feed stream and feed flow rate is up to you. If our column is not capable of producing the required distillate composition let me know the maximum ethanol concentration possible with our apparatus.

4. Please let me know no later than November 20 your experimental approach and give me an estimate of your time and expenses for this project. Please review the material available in the laboratory on this piece of equipment (see Bob Cox) and make sure you have the MSDS sheet for isopropanol for our discussion. Thank you for your cooperation.