

Distillation

Objective

A new consulting project has been assigned to our group. A client produces a 10 wt% methanol-90 wt% water mixture as a waste stream. They would like to reclaim this stream by concentrating it to 97.5 mole% methanol for use in another process. To do this, they are considering purchase of a Corning 100 liter batch distillation unit. We have an identical unit in our pilot plant and must determine the operating conditions required to achieve the desired separation.

Feedstock Preparation

Prior to coming to the laboratory for pre-lab checkout, obtain the MSDS sheet for methanol. This information is available on the web or on the back of the laboratory door. A good starting point for a web search is our safety office: <http://www.safety.utoledo.edu/safety/msds.htm>. Be sure to review this safety information prior to your checkout.

Feedstock for the column is in a container near the scale. Adjust the composition of the feed by adding methanol and/or water until you have at least 80 pounds of 10 wt% methanol. Mix the liquid well after any additions. Be sure to record the final feedstock composition as determined using the refractometer. Be careful with the units of concentration: the refractometer is calibrated to read mole % not weight % (*Question*: How can you tell which side of the peak you are on when using the refractive index graph? Two concentrations, one low and one high, can possess the same index of refraction).

You should prepare the feed solution the day before the lab. Check with the department technician for details on completing this task. Most groups start the experiment early in the morning to be able to finish before 4 PM.

Procedure for the 100 Liter Still

1. The column takes a long time (3-4 hours) to heat up and come to steady state under total reflux conditions. Familiarize yourself with the operation of the column, the refractometer and the computerized temperature logging system. A total run time of 9+ hours is not uncommon.
2. Make sure the trays have been drained and all valves are set appropriately. Charge the kettle with 80 pounds of feedstock using the feed pump.
3. Turn on the condenser water and the heating jacket nitrogen.
4. Make sure the reflux switch is off and the powerstat is set to zero.
5. Switch on the main power and the computer. If temperature data are to be recorded electronically, insert a blank, formatted 3.5" floppy disk. Run the data acquisition program.
6. Start the column heater using the powerstat. Using the highest allowable setting will speed the experiment considerably. It is important to monitor the column carefully during the startup period and reduce powerstat setting to approximately 45% before the column floods. Flooding is when the space between two of the trays in the column becomes filled with liquid. The top tray tends to flood first. If this occurs, the column will take some time to return to normal operation following a reduction of heat.

7. As the column heats up, adjust the cooling water flow rate to the condenser so that all the vapor is condensed, but with very little subcooling. *Use as little cooling water as possible while still condensing all the vapor. A visual check of the condenser will allow you to be sure all the vapor is being condensed; liquid droplets should not be visible on the coils more than 1/2 way up the condenser. A critical quantity in the heat balance for the column is the temperature rise of the water through the condenser. **Be sure that it is as high as possible.** Note that a $10^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$ rise allows about 5% error.*
8. Run at total reflux until the column reaches steady state. This will be indicated by steady temperatures and full, unflooded trays.
9. When steady state is reached, set the reflux ratio at 1.33 (15 seconds on, 20 seconds off – verify that this is correct), and begin collecting product.
10. Continue distilling under these conditions until 10% of the charge has been collected as distillate. While the distillation is progressing, periodically record the following information: (a) the tray compositions and temperatures, (b) the instantaneous distillate composition, (c) the cumulative distillate composition, (d) the condenser flow rate and temperature rise, and (e) the time of the readings.
11. When the distillation is finished, take a final set of readings and set the powerstat to zero. Exit the data acquisition software BEFORE powering off the computer. Shut off all column inputs. Let it cool until morning. Drain the kettle and each tray back into the feed container, and determine the final mass and composition of the bottoms.

Calculations

For the above separation, please determine the following information regarding the performance of the column and its suitability for the proposed task.

1. What is the minimum reflux ratio required for the desired separation? We believe that the column is capable of producing alcohol with a purity greater than 97.5 mole% methanol. Use the Fenske Equation to determine the minimum number of trays required to produce alcohol with this composition.
2. What is the maximum amount of product of the desired composition that could be obtained, based on mass balance considerations?
3. Use the method suggested in *Perry's Handbook*, 6th ed., Chapter 13, p 13-84, in the section on "Batch Rectification at Constant Reflux" to estimate the following at the final desired cumulative distillate composition:
 - the number of moles left in the kettle at completion,
 - the final bottoms composition, and
 - the time required for the distillation.
4. Perform heat and mass balances on the column. What are the losses? How do they affect the operation?
5. Use your tray temperatures and compositions to obtain the "real" operating line. Use this to estimate: (a) the average stage efficiency for the column, and (b) the heat loss for the column
6. What recommendations can you make for our clients regarding the use of this column?

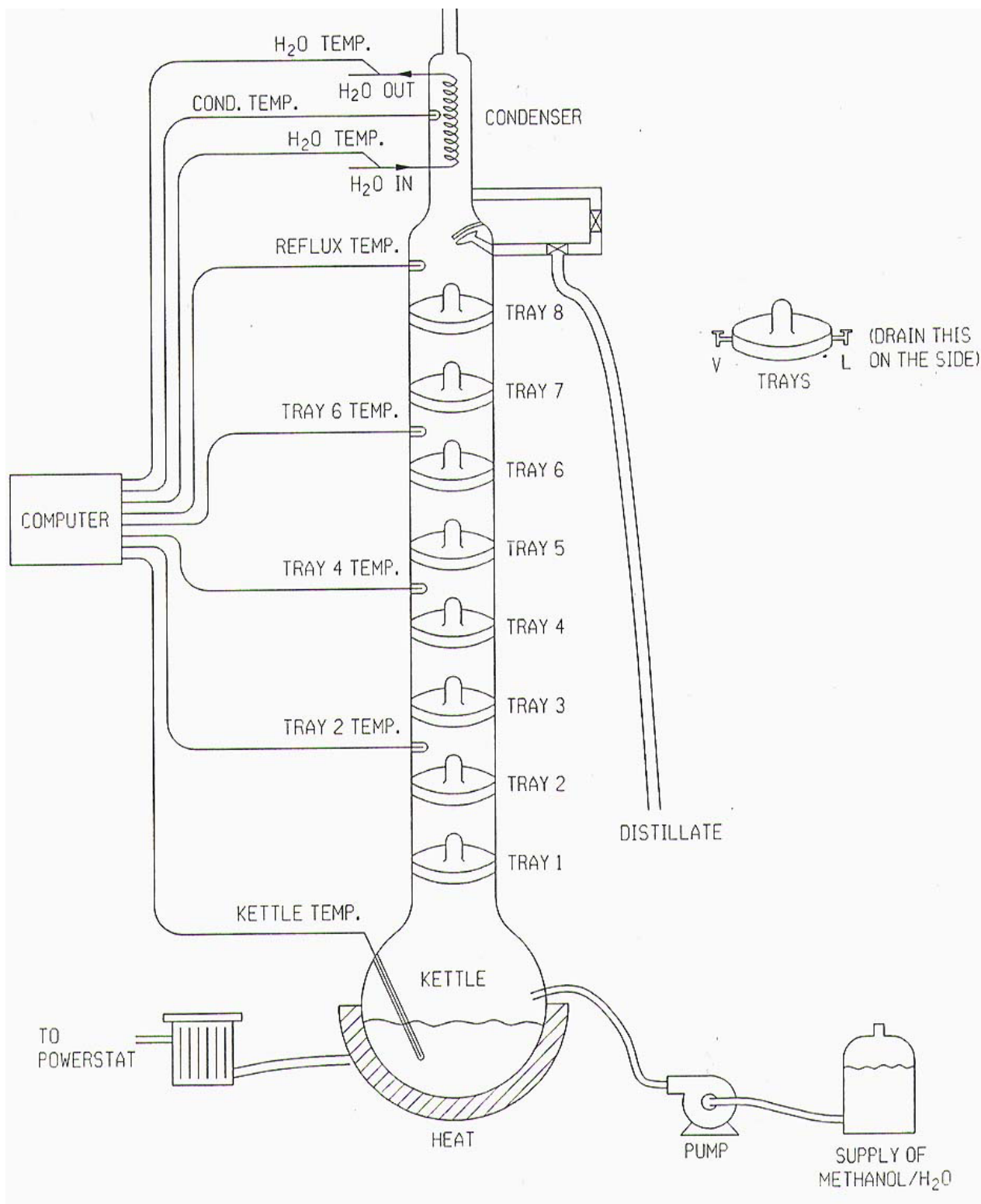
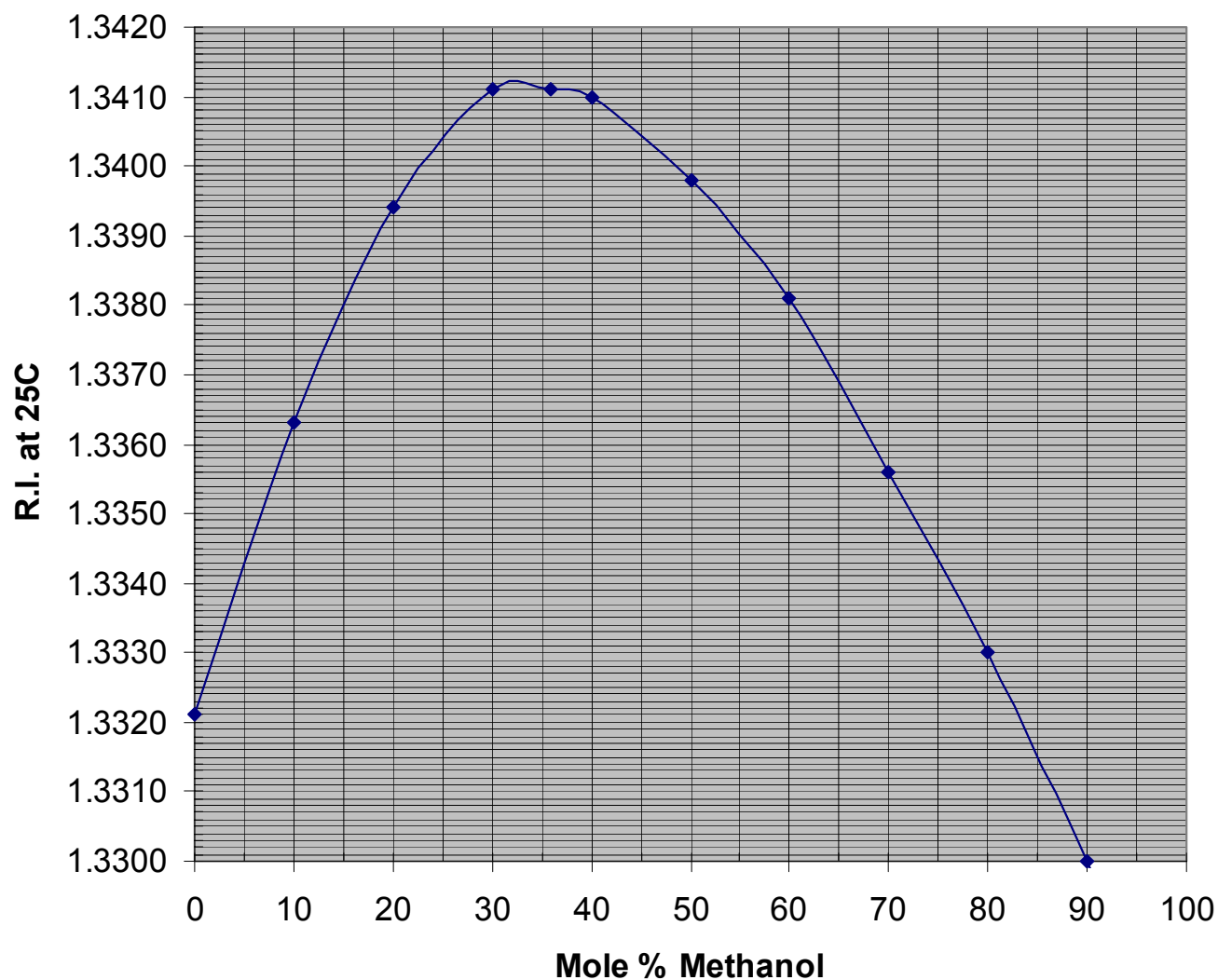


Figure 1 – Schematic of Distillation Column

Appendix: Refractive Index for Methanol-Water Solutions at 25°C

Mole % Methanol	R.I. at 25C	Mole % Methanol	R.I. at 25C
0	1.3321	70	1.3356
10	1.3363	80	1.3330
20	1.3394	90	1.3300
30	1.3411	92	1.3294
36	1.3411	94	1.3286
40	1.3410	96	1.3280
50	1.3398	100	1.3268
60	1.3381		

R.I. of Methanol/Water Solutions at 25C



R.I. of Methanol/Water Solutions at 25C

