Distillation Column Operation Guidelines 9/2008

SAFETY GLASSES ARE REQUIRED WHEN OPERATING THE DISTILLATION COLUMN

THE COLUMN OPERATOR MUST REMAIN AT THE COMPUTER AND CONTROL CONSOLE WHILE THE CCOLUMN IS IN OPERATION.

**EQUIPMENT STARTUP PROCEDURE**

1. Check in with the lab manager.
2. Ensure control console is plugged in.
3. Turn on control console by switching on the breaker switches. The up position is “on.” The gray switch is the main power, followed by individual break switches (blue=reboiler heater, red=feed pump/reflux valve, pink=console instrumentation).
4. Ensure the main breaker is functioning by pressing the gray “Test” button. This should trip the main power switch to the “off” position. If the main power switch does not turn off, contact the lab manager. Turn the unit back on if breaker functions properly.
5. Ensure all dials on the control console for the Reflux Control, Reboiler Heater, and Feed Pump are turned to the “I/O Port” position.
6. Start the computer, login, and open the UOP3CC software (icon is on Desktop).
7. In the lower right-hand corner ensure “IFD: OK(F292)” is displayed. This indicates the computer and control consol are communicating properly.
8. Click on the “View Diagram” icon (first icon on the right of the grayed out cut/copy/paste icons). This will bring up a GUI (Graphical User Interface) of the column.

**COLUMN OPERATION PROCEDURE (Batch – Total Reflux)**

1. Ensure the following valves are CLOSED: V1, V2, V3, V4, V5, V6, V7, V8, V11, V12, V14, V15
2. Ensure the following valve is OPEN: V10 (note: there is no valve V9 or V13)
3. Open valve V5 slightly before turning on the cooling water.
4. Turn on cooling water valve at the wall (yellow). Do not fully open the valve! Only open the valve about a quarter of the way.
5. Adjust valve V5 to a cooling water flow rate of approximately 2000-3000 cc/min (2-3 L/min).
6. Operation of the reboiler, pump and reflux valve is controlled by inputting valves in the GUI interface of on the computer screen and then pressing the corresponding “On” button on the control console. MODE
7. **IF NEEEDED:** Charge reboiler with approximately **8-10 liters** of the mixture to be distilled by opening the reboiler manual filler plug on top of the reboiler and pouring in the mixture. Use a clean funnel to avoid spilling excessive amounts of the mixture.
8. Start the reboiler by entering in a value from 0-100% in the PWR box of GUI of the column.
   1. Range for PWR (Reboiler Power) is 0-100%, which corresponds to 0-2.0 kW.
   2. To reach equilibrium faster, it is advisable to start with about 85% power then reduce the power to about 25% when the temperature in the reboiler is within 5°C of the boiling point of the mixture.
9. You will begin to see vapor rise up the column and start to see bubbling on the trays. You will also see liquid flowing from the condenser into the decanter, then back to the column through the feed pipe in the rear of the column above the top tray.
10. Adjust the reboiler power as required to get a consistent amount of bubbling on the trays without flooding the column. The column is flooded or starting to flood if the liquid level in the lower tray(s) has reached the bottom of the tray above it. **TO STOP FLOODING**, turn off the power to the reboiler (set PWR=0) and wait for the flooding to subside. Restart reboiler with a power setting below (about 5%) the power level when flooding occurred.
11. The column has reached equilibrium when the temperatures in the column are stable. Samples can be taken using the procedures under “Taking Measurements”

**COLUMN OPERATION PROCEDURE (Batch – Reflux)**

1. Start column as described above and allow to reach equilibrium in batch – total reflux operation.
2. Set an appropriate Reflux Ratio.
   1. Click the PID 2 button to bring up the PID Controller window.
   2. Change the Mode of Operation to Manual.
   3. Set the Cycle Time and Initial Reflux Ratio.
      1. How Reflux operation works on this column:
         1. A cycle time is set (e.g. 10 seconds) and a reflux ratio is set (e.g. 4:1).
         2. What the column does is divert the condensate to the tops product receiver for 2 seconds and then back to the column for 8 seconds.
         3. Calculation for other ratios is as follows:
            1. Take reflux ratio and add values together (e.g. 4+1=5)
            2. Divide the cycle time by that value (e.g. 10/5=2)
            3. Multiply that result by the reflux ratio (e.g. 4:1 -> 4x2=8 seconds back to column and 1x2=2 seconds to tops product receiver)
   4. Save the settings and exit out of the PID Controller window.
3. Start reflux by pressing the Reflux Control “On” button on the Control Console to start reflux. The reflux valve can be heard clicking when it switches the direction of overhead product flow.
4. Allow the column to reach equilibrium again prior to taking samples and measurements.

**COLUMN OPERATION PROCEDURE (Continuous – Reflux)**

1. Start column as described above and allow to reach equilibrium in batch – total reflux operation.
2. Charge a feed tank with an appropriate mixture that would match the liquid composition in the 4th tray (feed location).
3. Set an appropriate Reflux Ratio as described in Step 21 above. (Do not press the Reflux control “On” button yet)
4. Install the flexible, black feed tubing into the peristaltic pump by lifting the front cover and placing the tubing over the rotors.
5. Input an appropriate feed rate into the GUI of the column in the F1 box.
   1. Range for F1 (Pump Speed) is 0-300 RPM, which corresponds to feed rates of approximately 0-300 mL/min (recommended feed rates are 30-60 mL/min).
6. Start the feed pump by pressing the Feed Pump “On” button on the control console. The pump will begin to rotate and the liquid can be seen flowing to the column.
7. Once the liquid reaches the column, turn start reflux by pressing the Reflux Control “On” button on the control console and open valve V1 to start flow from the reboiler to the Bottoms product receiver.
8. Allow the column to reach equilibrium again prior to taking samples and measurements.

**TAKING MEASUREMENTS**

1. **Column pressure drop** is measured by the water manometer on the left of the column. VALVE OPENING AND CLOSING MUST BE DONE IN THE ORDER BELOW TO AVOID GETTING VAPOR IN THE MONOMETER.
   1. **Open** valve **V6**.
   2. **Open** valve **V7**.
   3. Take a reading.
   4. **Close** valve **V7**.
   5. **Close** valve **V6**.
2. **Boil up rate** is measured by performing a timed collection of the condensate returning to the column. This is a 2-3 person operation.
   1. **Partially open** valve **V3** and drain the condensate into a 250mL beaker or similar. **Do not** completely open the valve and **do not** completely drain the condensate feed tube that returns the condensate back to the column.
   2. Adjust valve V3 so that the liquid level in the condensate feed tube is consistent (level).
   3. Perform a timed collection using a stopwatch and a graduated cylinder.
3. **Overhead composition** is measured using a similar procedure to measuring the boil up rate.
   1. **Partially open** valve **V3** and drain the condensate into a 250mL beaker or similar. **Do not** completely open the valve and **do not** completely drain the condensate feed tube that returns the condensate back to the column.
   2. Adjust valve V3 so that the liquid level in the condensate feed tube is consistent (level).
   3. Collect approximately 5mL of tops product in a sample vial or similar container.
   4. Measure the refractive index of the sample using a refractometer to obtain the composition of the tops product.
4. **Bottom composition** is measured by taking a sample directly from the reboiler. It is preferred that this sample is taken at the same time as the overhead composition. **CAUTION:** THIS SAMPLE IS AT THE TEMPERTURE OF THE REBOILER AND THERFORE WILL BE HOT.
   1. **Partially open** valve **V2** and drain the some liquid into a 250mL beaker or similar. **Do not** completely open the valve, open enough only to obtain enough flow to obtain an adequate sample.
   2. Collect approximately 5mL of reboiler liquid in a sample vial or similar container.
   3. IMMEDIATELY close the vial or cover container to avoid having vapor escape and causing the composition to change. Cool the sample prior to opening to avoid haing vapor escape.
   4. Measure the refractive index of the sample using a refractometer to obtain the composition of the bottoms product.
5. **Column Operating Parameters** (temperatures, etc) are taken from the GUI of the software.
   1. Type in the flowrate (L/min) and column pressure drop (mm H2O) by clicking on their respective buttons on the right hand side of the GUI of the column.
   2. On the bottom left, should be listed a “Sample Mode.” It should be on “Auto – [Time Period].” The time period may be adjusted by configuring the sample parameters in the “Sample” drop down menu of the menu bar.
   3. To begin recording data, press the green “GO” button of the GUI (above the condenser on the GUI of the column).
   4. Press stop when a sufficient amount of sampling intervals has passed.
   5. The data can be viewed in a spreadsheet by clicking the “View Spreadsheet” button to the right of the “View Diagram” button.
   6. From the File drop down menu, choose “Export” and export the file to a Microsoft Excel file. The file is saved to the Windows desktop as “Results.”
   7. Save the results file on your own disk, thumb drive, etc. and delete from the Windows desktop.