

Continuous Distillation Column

version 97.5

A. Hardware

The following steps should be taken before the Continuous Distillation Experiment is started.

- 1) **Important:** Verify that the cooling water is on before powering up the experiment. The valve marked "CW" on the east wall next to the refractometer should be fully opened.
- 2) Plug the yellow-ended cord into the wall outlet.
- 3) Make sure that the thermapulse flowmeters are turned OFF. (These flowmeters should not be turned on until distillate has started flowing through the flowmeter cells).
- 4) Verify that the feed-tank valve is open and that there is feed in the tank.
- 5) The bottoms pump must be turned over to manual control by setting the switch in the back. To do this, make sure that the switch located at the rear top left corner of the pump is facing the east wall. The pump's other settings should be as follows: Front top black knob - ZERO at start up. Front bottom switch - OFF.

NOTE: 1 lab partner should stay with the column from this point on. ==> Signifies steps taken by the lab partner watching the column.

Do not leave the column unattended.

B. Computer Control -- Bailey

- 6) Turn on the computer. Press **escape** when the node number appears on the screen. Press **N** to boot from the network. When string of indicators stop and it prompts you to login, press **Control** and **Alt** and **6** simultaneously. You are now on the Bailey Network.
- 7) Choose the graphic displays by pressing **F7**. Then follow the prompts to get the display for the continuous distillation column. To change a setting in one of the displayed windows, type the number (sometimes a number and letter) of the window. Then follow the prompting to change the settings. To display an item that is not in a window look at the flow diagram. Near the pumps and heaters are red labels. These are window numbers. Type in one of these labels and one of the windows on the screen will be replaced with the requested one. To exit a window once it has been selected press **F10**.
- 8) To turn of the main power for the column choose the appropriate window. Notice the menu that appears on the bottom of the screen (F7 set = on and F9 reset = off). Press **F7** to turn on the power. **Warning:** do not turn on main power until the cooling water is on; this should have been done in step 1 above.

==> Make sure the pumps are not running. The pumps should not be sucking air through the column. Make sure that the Thermapulse flowmeters are turned OFF.

9) Enter the desired duty for the reboiler:

Select the reboiler heat window.

Enter the desired input of Watts.

Press **F10** to deselect window.

10)..Enter temperature for feed:

Select the Feed Heater station window.

Affirm that the station is in Auto mode; you should see a green "Auto" in the lower left hand corner of the station window

(use **F6** to toggle between Auto/Manual).

Press **F1** to set the feed temperature set-point.

Enter desired feed temperature and press **Enter**.

Press **F10** to deselect window.

11) Enter feed rate:

Select the feed pump window.

Enter the desired feed rate in ml/min and press **Enter**.

==> Vapor will begin to collect in the reflux splitting vessel (the tall thin glass vessel with condensate outlet tube clamped to the green frame below the thermapulse flowmeters). Bottoms product will begin to collect in a collecting vessel below the reboiler. Turn on the pump below this bottoms vessel and adjust the top black knob on pump to ensure that the collecting vessel does not get too full or completely empty. Maintain a constant level in the reflux splitting vessel by adjusting the flow % of the reflux pump on the Bailey. Continue to adjust this until a thermal steady state condition is reached. Once a constant flow has been established for overhead condensate and the reflux, turn **ON** the 2 thermapulse flowmeters. The thermapulse flowmeters and the bottoms pump are turned on and off by hand. The reflux pump is controlled through the bailey.

12) Adjust the reflux flow

Select the reflux pump window.

Choose manual control.

Press **F2** and enter a percentage of pump flow capacity and then press **Enter**.

13) Getting desired results:

Adjust the reflux flow, feed and heat duty until the desired reflux ratio is reached. Note that with each adjustment some time will be needed for the column to reach equilibrium.

C. Data Acquisition

14) Exit the graphic display for the column and return to Bailey's main menu by pressing F10 several times.

Push **F4** from the main menu.

Push **F9** for new data acquisition.

With Group Highlighted press **Return** (pushing **G** is equivalent).

With Setup highlighted press **Return**.

With the letter corresponding to your experiment highlighted press **Return**.

Press **Q** to quit the second menu.

Press **O** to get the Output menu.

Press **S** to setup your output files.

Insert a **DOS** disk (do not use the hard drive, it won't be what you want).

Carefully read the prompts (in naming your file you must specify the drive followed by ":/").

Make sure you are ready to record data.

Arrow over to Go and press **Return**.

Type **Q** to quit data acquisition when you are finished.

15) While the Bailey is recording data such as the reflux and temperatures samples of the bottoms and tops products can be analyzed using the refractometer:

Turn on the refractometer.

Make sure the knob on the front is turned to na.

Make sure the sample slides are clean.

Put the sample on the slide and close it.

Bring the light up to the sample slide.

Look through the eye piece and line up the dark horizontal to the center of the X.

Press the reading button and record the refractive index.

Press and hold the temperature button to get the temperature reading.

D. Shutdown

The following steps should be taken when powering down the Continuous Distillation Experiment.

- 1) **Important:** When powering down the experiment, leave the cooling water on until the reboiler has cooled off (boiling has stopped).

- 2) Make sure that the following equipment are turned off (or values set to zero where applicable):
 - reboiler
 - feed preheater
 - feed pump
 - flowmeters
 - main power.

- 3) Unplug the yellow ended power cord.