

# Continuous Distillation Column

version 2014

## 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 – NI Labview Virtual Instrument (VI)

- 6) Turn on the computer. Open a Firefox browser and access the Labview VI [here](#).
- 7) To turn on the main power for the column choose the toggle switch labeled Main Power (it lights up when on). 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.

8) Enter the desired duty for the reboiler:

Select the reboiler duty dialogue box.

Enter the desired input of Watts.

9) Enter temperature for feed:

Select the Feed Heater station window.

Affirm that the Manual control override is in Auto mode; you should see a blue light (it toggles between Auto/Manual)

**Enter** desired feed temperature in the dialogue box.

11) Enter the desired feed rate in ml/min into the dialogue box.

==> 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 rate of the reflux pump on the VI. 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 Labview.

12) Adjust the reflux flow Select the reflux flowrate box (on left hand side of VI). Enter the flow rate and then press **Enter**.

13) Getting desired results:

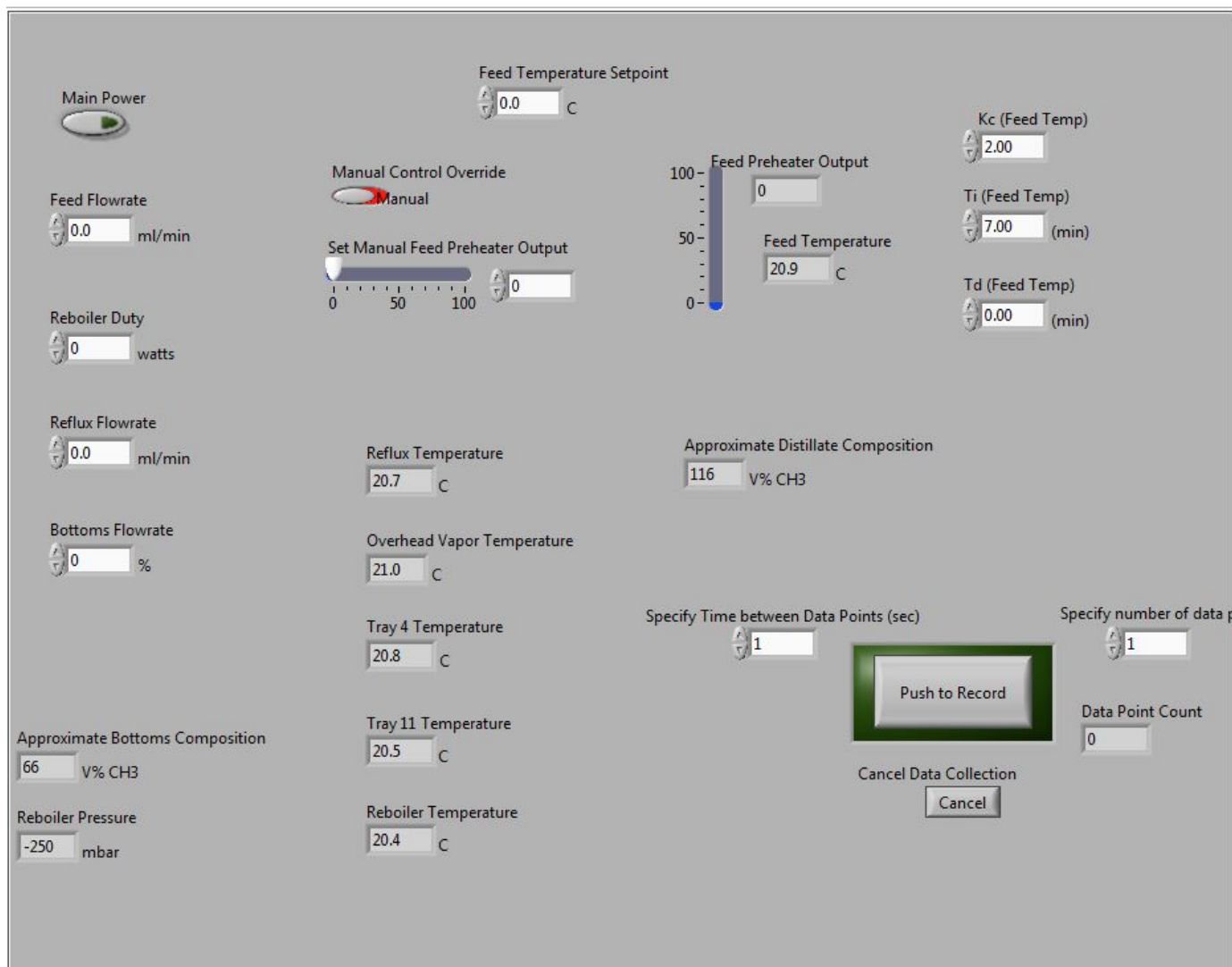
Adjust the reflux flow, feed and heat duty until the desired reflux ratio is reached. Note that with each adjustment some time will be needed for the column to reach equilibrium.

### **C. Data Acquisition**

14) Enter the number of data points to collect and how much time should elapse between data points. Click the push to record button. It will glow green. Retrieve the data in the bottom left.

15) While data is collected, samples of the bottoms and tops products can be analyzed using the refractometer.

## Continuous Distillation Column



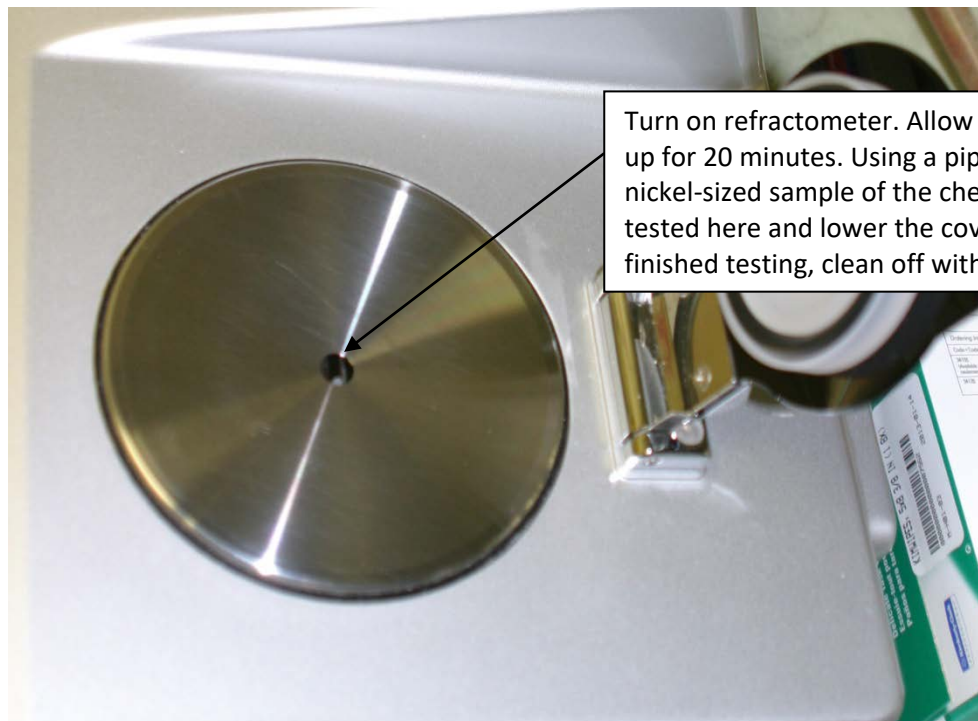
### D. Shutdown

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

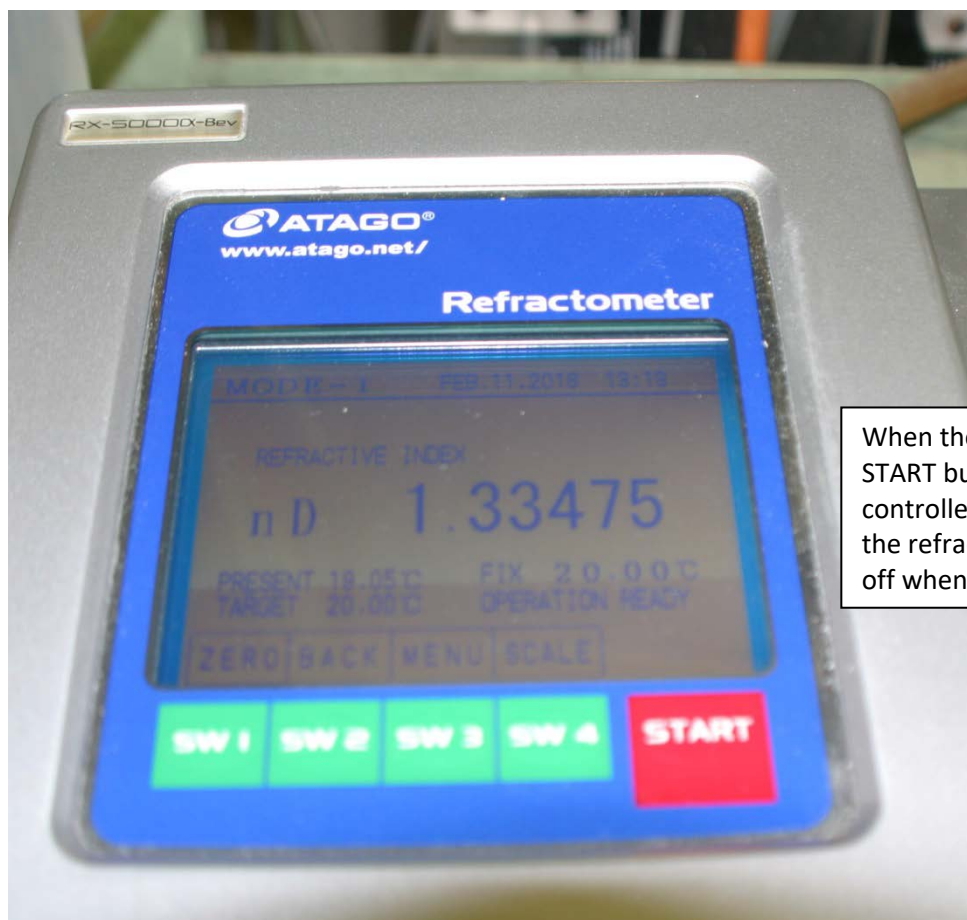
- 1) **Important:** When powering down the experiment, leave the cooling water on until the reboiler has cooled off (boiling has stopped).
- 2) Make sure that the following equipment are turned off (or values set to zero where applicable):
  - reboiler
  - feed preheater
  - feed pump
  - flowmeters
  - main power.

3) Unplug the yellow ended power cord.

# Refractometer instructions



Turn on refractometer. Allow it to warm up for 20 minutes. Using a pipette, place a nickel-sized sample of the chemical to be tested here and lower the cover. When finished testing, clean off with a Kim wipe.



When the sample is placed, press the red START button. The temperature will be controlled. The number listed after nD is the refractive index for the sample. Turn off when done using for the day.