Operating Instructions

Preparation for Start-up

1. If the computer system is logged off, log into the computer system.
   - Log into the operator account on the computer. Please ask your TA for
     username and password.
   - Click “DeltaV Operate” on the menu in the center of the screen

2. Check the fluid level in the reboiler using either the sight glass or the sensor data
   in the control system. The level should be around 40-70%. If the level is too high
   or too low, seek assistance from the TA/Lab Assistant.

3. Mix the contents of the reboiler:
   a. Close the bottoms valve (see Figure 1). Make sure that both the automatic and
      the manual bypass valves are closed.
   b. Open the manual recirculation valve (see Figure 1).
   c. Use the computer control system to turn on the reboiler mixing pump (see
      Figure 1). Let it mix for about 10 minutes.

Figure 1. Overview of the West Column (WC) system on the 1st floor.
4. While you are waiting:
   a. Check that the manual bypass valves on the distillate and reflux lines are closed (see Figure 3 and Figure 4).
   b. Check that the following control valves are set to the “closed” position in the control system:
      - Steam (see Figure 1).
      - Distillate (Figure 3 and Figure 4)
   c. Use the computer system to fully open the reflux control valve (Figure 3 and Figure 4).
   d. If you will be running at partial reflux conditions, check the levels in the feed and product tanks:
      - Check that the feed tank T5 (see Figure 5) has enough material.
      - Check that the product tanks T2 and T3 (see Figure 6) are empty.
      If necessary, ask the TA to help you transfer content of the product tanks into the feed tank.
5. Take a sample from the reboiler sample port (see Figure 1).
6. Use GC to determine composition of the reboiler content.

Figure 2. Front view of the WC system on the 2nd floor.
Figure 3. Back view of the WC system on the 2nd floor.

Figure 4. Side view of the WC system on the 2nd floor.
Start-up

Note:

- You will start the column under total reflux conditions on each day of the experiment. On Day 2, you will switch the column to the partial reflux mode (by introducing a feed stream and drawing off distillate and bottoms products) after it has reached a steady-state at total reflux.

- The sequence of turning on the utilities is the following:
  1. Air (no cost), required for operating pneumatic control valves
  2. Cooling water (somewhat expensive), required to prevent system from overheating.
  3. Steam (highly expensive).

The sequence will be reversed while turning the utilities off.

- Do not leave the column unattended.
• If a pneumatic valve malfunctions, use its corresponding manual bypass and notify the lab supervisor.

**Starting-up Operation in Total Reflux Regime**

1. Manually open the air valves on the 1st and 2nd floors.

2. Manually open the water valves for the steam condensate and bottoms heat exchangers on the 1st floor (see Figure 7) and the main water valve for the distillate condenser on the 2nd floor (see Figure 2).

3. Use the control system to open the steam valve. Recommended steam flow rate is about 70 lb/hr. The temperature of the liquid in the reboiler should now be slowly rising.

4. While you are waiting for the column to warm up on Day 1, perform GC calibration.

5. After about 20-30 minutes, you should see flow of the distillate from the condenser. The flow can be directly observed through a glass standpipe connected to the condenser (see Figure 2, Figure 3, and Figure 4).

6. Allow a stable liquid level to be attained in the standpipe. If the standpipe starts overfilling, use the control system to turn on the reflux pump (see Figure 4) for a few seconds to drain the excess liquid. Reduce the steam flow rate to prevent recurring overfilling of the standpipe.

7. Start taking product samples to check whether the system has reached a steady-state. It is recommended to use reflux samples for this purpose, since the reflux sample port is located on the 2nd floor (see Figure 3 and Figure 4). Take samples at regular time intervals (10 minutes is recommended). It takes about an hour to reach a steady-state.

![Figure 7. Cooling water lines on the 1st floor.](image)
Preparation for Switching to Partial Reflux (Week 2)

The following steps should be performed while waiting until the system reaches a steady-state at a total reflux.

1. Prepare to mix content of the feed tank T5 (on the 3\textsuperscript{rd} floor, see Figure 5): Ensure that ball valves on the in/out lines of the tank are in the recycle configuration so that, once the feed pump is turned on, it will pump the fluid from the bottom outlet of the tank T5 to the top inlet of the same tank.

2. Use the manual switch (see Figure 5) to turn the mixing pump on and wait for 5-10 minutes.

3. Take a sample from the feed tank.

4. Manually open a ball valve on the feed line leading to the appropriate feed tray of the column.

5. Make sure that ball valves on the in/out lines of the WC product tanks (see Figure 6) are set so that the distillate and the bottom products will flow to their corresponding tanks.

6. Make sure that the drain valves on the WC product tanks are closed.

Switching to Partial Reflux (Week 2)

After the system reached a steady state at total reflux:

1. Open the ball valve on the WC feed line (3\textsuperscript{rd} floor). Keep the recirculation line open and the feed pump on.

2. Use the bottoms, distillate, and feed control valves to set flow rates of the corresponding streams.

3. Take samples and measure the composition of the distillate and the bottoms at regular time intervals.

4. Once the system reached a steady state, record the product compositions and switch to different operating conditions.

Shutting Down

1. Use the computer system to close the steam control valve.

   \textbf{Important:} Use the \textit{manual} mode of the valve control to ensure that it is fully closed (i.e. it is 0\% open). The automatic valve controller uses steam flowrate values measured by a vortex flowmeter, which is not designed to operate at low flowrates. Therefore, the automatic controller cannot guarantee that the valve will be fully closed even if you set the desired flowrate to 0.

2. Turn off the reboiler mixing pump.

3. Return the system to the total reflux (Week 2):
   a. Open the reflux valve to 100\%.
   b. Close the feed, distillate, and bottoms control valves.
   c. Turn off the feed mixing pump on the 3\textsuperscript{rd} floor.
4. Close the manual water and air valves on the 1\textsuperscript{st} floor.

5. Wait until the liquid flow in the standpipe is negligible. Use the reflux pump to drain any standing liquid from the standpipe.

6. Close the manual water and air valves on the 2\textsuperscript{nd} floor.