Packed Distillation Unit (PDU)

Operating Manual
1. Background

Continuous distillation, or more precisely continuous fractional distillation, is one of the most important unit operations. Billions of dollars are invested in equipment to perform this process and it has been estimated that some 80% of the capital investment in an oil refinery is expended on distillation equipment. The Packed Distillation Unit (PDU) is a small pilot unit designed to demonstrate the process of continuous fractional distillation.

2. Description of Facilities

The PDU distillation tower (or column) is 3” in diameter and constructed of Type 304 stainless steel. It contains two packed sections, each of which is 3’ 0” high and contains 2’ 8” of PROPAK\(^1\) 0.24” protruded stainless steel packing (a sample of the packing is available on the unit).

The unit is equipped with a total condenser, a partial reboiler, and a pump-back reflux system. Feed is introduced between the two packed sections and reflux enters at the top of the tower. An ethylene glycol heating system is used to provide heat to a thermosiphon reboiler. The feed and product storage tanks as well as the glycol heating facilities are located outside the building with piping connections passing through the building wall. Temperature, level, and flow rate controls are provided by the Honeywell Experion PKS control system.

Note that the time required to start up a cold unit and reach the initial steady state is 2-3 hours because the entire unit must be brought up to operating temperature. Shorter time intervals are required to move from one set of operating conditions to a second set, taking perhaps 30-45 minutes to reach steady state.

Some operating changes should be made slowly (incrementally) to avoid upsetting the unit. (See Ramping Setpoint Changes below.)

3. Basic Operation of PDU from the PKS Schematic

Understanding Experion PKS Control System Terminology

In Experion, each process variable is represented by an entity called a Control Module (CM). Each CM is a collection of Function Blocks (FB). And each FB consists of many values called parameters. Within a CM (and sometimes between CM’s), the FB’s are “wired” together in various ways to monitor and control the process. Desired values of many parameters may be entered via the computer keyboard. The purpose of the next few paragraphs is to explain how to use Experion to run this equipment.

4. Logging in to Honeywell

Access to the Honeywell Experion DCS application is through a virtual machine using VMware Horizon View software. After logging into one of the computers in the UO Control Room, among the icons on the desktop shown is the VMware Horizon View Client icon.

\(^1\) Manufacturer’s literature can be found at https://www.cannoninstrument.com/en/Image/GetDocument/425
Double-clicking this icon should bring up the following popup with the `che-view.lsu.edu` icon showing. If that icon does not appear, then click **New Server**; complete the requested information to add it. The following should show:

![Image of VMware Horizon View Client with New Server option](image1)

Double-click the `che-view.lsu.edu` icon. If an additional login menu appears, login using your LSU ID and password. At this point the following view should appear:

![Image of VMware Horizon View Client with two virtual desktops](image2)
Virtual machines are available for the microscope in the UO Analytical Lab (shown as Microscope) and for access to the Honeywell DCS (shown as Uolab). Double-click the Uolab icon. A splash screen for the virtual machine should appear with an OK button. Click OK. The VMware software should start up a virtual machine and show you the desktop of that machine.

Open the Honeywell Station software either by double-clicking the Station icon if visible or by navigating using the following pathway:

Start>All Programs>Honeywell Experion PKS>Client Software>Station

If login credentials are proper, the Station program will appear and – exercise patience here – the following default splash screen within it:

The Honeywell login process is now complete.

5. Overview of Operations

From the Unit item on the menu, select PDU. The PDU P&ID schematic will appear. The PDU is controlled from this schematic. There are additional schematics which show trends of the analog values associated with the unit.

On the main schematic, each measurement transmitter and continuous controller is represented by a circle containing its tagname. The first letter of the tagname indicates the type of measurement: T for temperature, P for pressure, L for level, F for flow, or C for composition. There are also six device controllers (sometimes called discrete controllers): four pumps, a cooling water solenoid valve, and a switch to allow current to flow to heat the glycol. When you click on a circle representing any measurement or continuous controller, or any of the device controllers, a faceplate will appear in the lower right corner of the schematic. It contains the tagname, engineering units, description, and PV for transmitters. For continuous and discrete controllers, additional values and controls are available (see below).

To change an analog value from a schematic or from a faceplate, click the value (if change
is allowed, its background color will change), and then enter the new desired value. To change the mode of a continuous controller, use the drop-down list labeled MD near the bottom of its faceplate (more about controller modes below). To change the output of a device controller, use the output (abbreviated OP) radio buttons on the right side of its faceplate.

At the start of a run, all controllers should be in manual mode, signified by yellow backgrounds in the circles representing the controllers. Now turn on the cooling water (click on the solenoid valve in the upper right part of the schematic, and then click on the upper OP radio button on the right side of the faceplate). Notice that the solenoid valve changed from green (representing closed) to blue (representing changing positions) to red (representing open) when you turn on the cooling water. When you turn off the cooling water, the color sequence will be from red to blue to green. You may not see the blue state because the solenoid changes very quickly.

**ALWAYS KEEP THE COOLING WATER ON WHEN RUNNING THE UNIT.**

A transmitter’s most important value is the measurement of the process variable itself, abbreviated as PV. This value is shown in cyan (light blue) near the circle.

Controllers have several additional values, the most important of which is the setpoint or SP. This is just like the speed setting on a cruise control - the controller will manipulate its output (the throttle position in this case) to move the PV to the SP and hold it there. The SP (in green) and the PV (in cyan) are shown near the circle representing the controller.

As mentioned above, a controller has an SP and an OP. The OP always has units of percent (0-100%) and the SP has the same engineering units as the PV. On PDU, flow rates are measured in gallons per hour or gallons per minute, levels are measured in percent full, temperatures are measured in degrees Fahrenheit, and pressures are measured in inches of water.

When the controller mode is MANUAL, the OP is held until the operator changes it. If you want to change the OP, simply click the OP in the faceplate, type in the new value, and press ENTER. The new OP will be held until you change it again. Note that you can change an OP only while a controller is in MAN. For practice, click the reflux flow rate controller (to call up its faceplate) and change the output to 50%, then to 100%, and back to -6% (-6% is known as “tight shutoff” in the PKS system). Notice the small bar under the control valve on the schematic – its length is proportional to the output.

When the mode is not MAN, the controller uses the PV, SP and tuning constants to calculate a new OP. When the mode is AUTOMATIC, you may enter a new SP to be used for control. Note that changing an SP affects the OP only while a controller is in AUTO.

Sometimes controllers are stand-alone (e.g. the feed flow rate controller), and sometimes they are in a cascade structure (e.g. the bottoms level controller is cascaded to the bottoms flow rate controller - cascade connections are indicated by dashed lines on the schematic). The upper controller in a cascade (the one sending its OP to another controller’s SP) is called the primary and the lower controller (the one sending its OP to a
field device) is called the **secondary**. Secondary controllers require an additional mode so the computer system will know when to “close the cascade” (i.e. put the structure fully on control). In Honeywell’s PKS, this additional mode is called **CASCADE**. When the mode of the primary is **AUTO** and the mode of the secondary is **CASCADE**, the cascade is said to be “closed” and the primary sends its **OP** to the secondary **SP**. When the mode of the secondary is **AUTO**, the operator is responsible for changing the **SP** of the secondary to control the process.

Notice that the circle representing a continuous controller is filled with a background color, which indicates the current mode of the controller. For all controllers, yellow means the mode is **MAN**. For primary controllers, cyan means the mode of the primary is **AUTO**, but the mode of the secondary is not **CASCADE** (i.e. the primary is ready to start sending new **SPs** as soon as the secondary goes to **CASCADE**). For secondary controllers, cyan means the mode is **AUTO** (i.e. any new **SP** must come from you, not from the primary). And finally, for all controllers, white means the mode is in its normal state - fully on control, which is **AUTO** for primary controllers and **CASCADE** for secondary controllers.

For practice, set both the bottoms level and bottoms flow rate controllers to **AUTO**. Note that the background of both controllers changed from yellow to cyan. Now set the flow rate controller to **CASCADE**. Note the change in background colors of both the primary and secondary controllers. Now put both controllers back into **MAN** and set the flow rate **OP** back to -6%.

### 6. Display Navigation

When you logged into Flex Station, you used an item from the menu bar to call up the main PDU schematic. There are several additional ways to go from one display to another. For example, you can enter the tagname of a CM in the Command field at the top of the screen and press **F12** to call up the detail display. Try it with your bottoms level controller (L330). For continuous and discrete controllers, the detail display has 7 tabs, and for a measurement, only three.

Most of the toolbar buttons are used for navigation – some require a name or number to be entered, and some go directly to the display. Most of the same functions are on the function keys. For example, to return to the previous display, click **K**, or press **F8**. To return to the display before that, do it again.

From most displays (both system displays and custom schematics such as PDU), double clicking any value associated with a CM will take you to its detail display. From a detail display, click **T** or press **F2** to return to the main PDU schematic. On most custom schematics there may also be buttons to quickly get you from one display to another.

### 7. Understanding Trend Schematics

There are several buttons on the main PDU schematic to call up trends. The top button displays the **PVs** of the four pairs of cascaded controllers. The next displays the tower temperature profile, pressure drop and flows into the tower. The third displays other miscellaneous temperatures, etc. Click the **Trend Cascades** button.
At the bottom of the trend is the legend with all the tag.block.parameters associated with the traces. The checkboxes in the Pen column indicate which traces are currently on the trend. Click in the chart area of the trend and a white hairline cursor appears on the chart and the values at the hairline cursor appear in the Reference Value column of the legend. Along the bottom of the chart area is a horizontal scroll bar which allows you to scroll the chart area back and forth. Along the left axis you’ll find the low and high range of the selected trace. These allow you to change the range of the trace for the selected parameter. Practice by changing the range of one of the level controllers to 20-80.

Immediately above the left side of the chart area is a drop-down list which allows you to select one of the traces (you may also click anywhere on the line for this trace in the legend area). When you select an active trace, it is highlighted (i.e., becomes thicker) in the chart area. Above the right side of the chart area is the Period drop-down list. This drop-down list allows you to select the period of time for which you want data to be displayed in the chart area. To the right of that is the Interval drop-down list. This drop-down list allows you to select the interval between points in the chart area. Practice changing to a different period and interval. Leave the period set to 1 day and the interval at 1 minute for now.

For practice, scroll back until some variation in some of the traces appears. Notice that the timestamps below the chart area change as you scroll. Find some local max or min in one of the traces and click or drag the hairline to it. Now change the period back to one hour and notice that the cursor is centered on (or at least near) the local max or min. If necessary, move the hairline so it is exactly on the peak or valley and notice that the values, as well as the date and time, are shown in the Reference Value column in the legend. Now return the trend to the current time by clicking 🔄.

All changes you make to the trend can be saved by clicking the familiar Windows Save icon just above the right end of the chart area next to the word (Modified).

8. Ramping Setpoint Changes

As mentioned above, changing from one set of operating conditions to another should be done slowly to avoid upsetting the unit. SP changes can be ramped – changed gradually in a linear fashion – from the present setting to a new setting by using the system’s setpoint ramping feature.

For practice, click the feed preheat controller (T350), put it into AUTO and give it a SP of 150°F. Double click the faceplate to call up the detail display and then click the Setpoint tab (the setpoint ramping values are at the lower left).

- Make sure the Set Point Ramping Enabled box is checked.
- If necessary, change the Requested Ramp Rate (EU/min).
- Enter the desired final SP value in the Set Point Target Value (SPTV).
- Change the Set Point Ramping State to RUN.

The SP will start moving linearly to the desired value.

If you make a mistake, change the Set Point Ramping State back to PRESET, enter
a new Requested Ramp Rate if necessary, enter a new SPTV, then change the Set Point Ramping State back to RUN. When the ramp finishes, put the controller back into MAN and give it an OP of −6%.

9. Unattended Operations

If experimental guidance from your technical supervisor calls for, or you believe that you will need to start up the PDU AND leave the area for a sustained period of time while the unit is operating, you may seek and may be given permission to run the unit in an Unattended Operations mode. If you acquire this permission, complete, comply with, and post the appropriate Unattended Operations Placard found at the end of this Operating Manual.

10. Saving Data into Excel

Successful completion of the objectives of this experiment will require analysis of a great deal of data. To collect this data, an Excel workbook containing a Visual Basic Add-In is provided. On the apps (X:) drive, in the CHE4162 folder, look for a folder named CHE4162-HW Excel Data Recorders

Within that folder, open the folder named

    snr

and then double click on PDURecorder.xls. (For control-only studies, the file name is PDURecorder_for_control.xls.) The workbook will open with a Start button, the experiment name, a collection frequency drop-down menu box, and a Stop button on the top line. Enable the VBA add-in by clicking on the “Options...” button on the Security Warning just under the toolbars, and then click “Enable this content” in the dialog box which appears. Click on the workbook’s Start button, and it will start collecting the relevant data at the specified collection frequency. These data will be extremely useful in analyzing your results. While the workbook is collecting data, it may be scrolled, but you should not attempt to do anything else in this instance of Excel until after you click on the Stop button. If you do, the collector may stop and you may lose valuable data.

When you finish a run, click on the Stop button and cut or copy whatever data you need to your daily workbook in a separate instance of Excel.

11. Tag Descriptions with Engineering Units

<table>
<thead>
<tr>
<th>TagName</th>
<th>Description</th>
<th>EngrUnit</th>
</tr>
</thead>
<tbody>
<tr>
<td>C302</td>
<td>Bottoms Composition Control</td>
<td>Deg F</td>
</tr>
<tr>
<td>C305</td>
<td>Distillate Composition Control</td>
<td>Deg F</td>
</tr>
<tr>
<td>D300</td>
<td>Feed Pump Control</td>
<td>Run/Stop</td>
</tr>
<tr>
<td>D310</td>
<td>Reflux Pump Control</td>
<td>Run/Stop</td>
</tr>
<tr>
<td>D330</td>
<td>Bottoms Pump Control</td>
<td>Run/Stop</td>
</tr>
</tbody>
</table>
12. Gas Chromatograph Operation

An Agilent 7890 gas chromatograph (GC) is used to separate and quantify the components in the liquid samples produced by PDU. Each component will be recorded as a separate peak in the integrating and recording ChemStation software. The elution order is: air (eliminate and renormalize if small; rerun if not), water, methanol, isopropanol (not all peaks may register on every analysis).

The following procedure (prototype) should be used to analyze samples:

- Log on to the Agilent-dedicated PC in the Analytical Lab using your ID and password.
- Open the Agilent ChemStation software from the Start menu by clicking on Programs, then Agilent ChemStation, then PDU-GC Online.
If not already selected, select the Method and Run Control window by clicking on that button on the lower left hand portion of the screen.

On the icon-filled menu bar, use the left-most drop down menu to select Method and pick the START.M method. You should see the message Method START.M loaded! at the bottom of the screen.

Go to the front panel of the GC. Press the Front Inlet button. Use the up and down arrows to select Pressure on the display, then use the ON/OFF button to set ON, if it is not already in that mode.

When the pressure reaches the target value, a green READY message is displayed on the ChemStation screen, signifying that you may now inject a sample.

(There are occasions when the GC pressure NEVER reaches its target value and the green READY message will NEVER appear. If the pressure does not reach the programmed value within a minute or two, the programmed value must be changed to the current value to obtain READY status. Ask the Lab Coordinator or your instructor to assist in this.)

Inject a 0.1 microliter sample (same volume each time) and press the Start button on the GC front panel.

Immediately the ChemStation will report the status of the GC as Run in Progress / Data Acquisition in blue, with the elapsed time counting up.

Results should appear in a report on the ChemStation screen in roughly 6 minutes. There will be a chart with the GC peaks on it. Scroll down the report to see the Norm % results. Values are in weight percent. However, sometimes one or more component values will not show up under the Norm % values. To determine the correct values in those cases, you will need to perform the following calculation, using the proper response factors for each component (these can be found in the calibrations area of the ChemStation program) and the area values given in the report:

\[
\text{Weight fraction, } i^{th} \text{ component} = \frac{\sum_{i=1}^{3} \text{Area}_i \cdot RF_i}{\sum_{i=1}^{3} \text{Area}_i \cdot RF_i}
\]

If you will not be using the GC for an hour or two but will return during lab to make runs, place the GC in STANDBY.M mode; this will save carrier gas. You can return it to START.M when you are ready.

At the end of the day, place the GC in overnight mode by loading the COOL.M method in much the same manner as the START.M or STANDBY.M methods were loaded. If the ChemStation presents a menu inquiring whether you want to save changes to the START.M method, say No. Do NOT power down the GC and do NOT shut off the helium cylinder.
If you’d like, your instructor will demonstrate how to operate the GC. It should be in the **STANDBY.M** (or **COOL.M**) method when you arrive. If not, ask the UO Lab Coordinator or one of your instructors for help.

Return all unneeded samples to one of the gallon bottles on the unit at the end of the day and replace the empty sample vials, caps and septa in the tray in the PDU-labeled lab drawer.

If there are any samples that must be saved for GC analysis at a later time, cover the cap of these bottles with Parafilm®, place them in a beaker in the laboratory refrigerator. Make sure the beaker is labeled with your name(s) and the chemical nature of the samples.

### 13. Troubleshooting Flow Rate Problems

During operations, it may happen that one of the major flows is unable to reach the desired rate. For example, an experimental run may require a feed flow rate of 4.0 GPH but the flow only reaches 3.2 GPH, even with the respective flow controller output at or near 100%. Typically, this problem is due to a plugged (or plugging) filter upstream of the associated pump, a filter designed to protect the pump from particulate wear. These filters are installed as a pair: one in service and one available for service. The following photos show these filter pairs for the feed, bottoms, and reflux-distillate pumps, respectively.

If a plugged (or plugging) filter is the source of the reduced flow, the problem can be remedied by switching from the in-service to the available-for-service filter. The procedure for doing this is simple: a) open both the block valves to put the available-for-service filter into the flow path, b) close both block valves to take the filter that is plugged out of the flow path, and c) notify the Lab Coordinator regarding the service and the action taken.

If a plugged filter was the issue, the flow rate should show an increase to the desired flow rate. If not, then the problem is more circumspect, requiring direct involvement of the Lab Coordinator.

### 14. Some Safety Considerations
• All unused sample material must be returned to the sample jug.
• Keep all sample containers closed as much as possible to minimize evaporation.
• Check the temperatures periodically. All tower temperatures should be below 185°F.
• Always open the downstream valve before turning on a pump.
• Make sure the reboiler contains liquid before turning on the glycol heater.
• Make sure the condenser water is flowing whenever the experiment is running.

15. General Operating Plan

Ordinarily, an experimental program will require the operation of PDU in both total reflux mode and finite reflux mode. The total reflux condition requires no feed entering the unit and no bottoms or overhead product leaving. While this mode has little practical application for distillation columns designed to produce finite product streams, it is an important test condition, since at total reflux the unit provides an indication of the maximum separation capability of the equipment. Finite reflux runs require the addition of feed and the withdrawal of bottoms and overhead product. Inasmuch as three additional streams must be managed during finite reflux operations, a simple startup strategy consists of establishing reflux flow prior to introducing feed and withdrawing products.

For all runs, bear in mind that the unit will require some time to reach steady state. Observing that all temperatures, levels and flow rates are 'constant' is an indication that steady state has been achieved. However, satisfying the steady state requirement can be confirmed by running GC analysis of time-spaced distillate samples taken from the condenser to determine when the composition remains 'constant'.

When you are satisfied that steady state has been reached, allow the recording of a sufficient number of process data readings and collect all the necessary samples for analysis (Note: The instructor will show you where the sample points are; use personal discretion on the number of samples to take.). These analyses and averages of the recorded data can then be used to perform the process calculations needed to meet the experimental program objectives.

Monitor tower loading by careful observation of the column pressure drop (or \( \Delta P \)) at all times, and the bottoms product rate when in finite reflux mode. As the vapor flow up the column increases, column pressure drop rises. Excessive vapor flow entrains liquid and restricts its downward flow, causing a condition known as flooding. If the \( \Delta P \) exceeds 2 inches of water per foot of packing (this is just a guideline), the tower may be flooding or flooded. In this event, the bottoms product rate would decrease sharply, perhaps to zero. If this happens, you can dump the tower (stop the flooding) by reducing reboiler heat input.

The UO Lab Coordinator will prepare and monitor the feed composition near 50 wt% MeOH, 30 wt% IPA, and 20 wt% water (or other suitable specification). But in any case, as stated above, you will be checking the feed composition by GC analysis during each finite reflux run and before each total reflux run.

Ternary vapor-liquid equilibrium data for this system have been made available with
assignment documents, along with any necessary ChemSep and HYSYS template files for simulations.

16. Some Total Reflux Details

If operating strategy suggests that it would be desirable to control the reflux drum level using the reflux flow rate controller in cascade mode, the button labeled **Switch to Reflux Control of Reflux Drum Level** rearranges the connections between the various overhead controllers. Pick the mode which connects the reflux drum level to the reflux flow controller. This mode allows for cascade control of level by reflux flow rate, disables level control by the distillate flow rate, and disables inferential composition control using column temperature.

The difference between the top and bottom tower temperatures should be in the neighborhood of 20-to-25°F – and it will likely take upwards of an hour or more after boil-up to reach this degree of separation.

17. Some Final Reflux Details

If operating strategy suggests that it would be desirable to control the reflux drum level using the distillate flow rate controller in cascade mode, the button labeled **Switch to Distillate Control of Reflux Drum Level** rearranges the connections between the various overhead controllers.

An acceptable time to begin adding feed is as the reflux drum level reaches 20% (or other justified final set point). Start feed into the column slowly (e.g., 0.5 gph). After establishing this rate, put the feed preheater controller (T350) into AUTO and give it a setpoint in the range of 140-to-160°F, or other value suitable to the experimental program. Start raising the feed rate (use the Ramp function) to that defined by the experimental program or determined by other means.

As the feed rate is increasing and over a 15-to-20 minute period, ramp the reflux flow rate to roughly 60% of the typical rate at total reflux. This should be a good ballpark value to help establish finite reflux operations.

Start withdrawing distillate product to maintain a level of 20% (or other justified final set point) in the reflux drum. You can accomplish this by putting the Reflux Drum Level controller (L310) into AUTO, giving it the proper setpoint, and putting the Distillate Flow Rate controller (F310) into CAS.

Start withdrawing bottoms product to maintain a level of 50% in the bottom of the tower. You can accomplish this by putting the Tower Bottom Level controller (L330) into AUTO, giving it a setpoint of 50%, putting the Bottoms Flow Rate controller (F330) into CAS, and turning on the bottoms pump.

For best material and energy balance results, one should attempt to maintain constant bottoms and distillate stream temperatures at the orifice meter measurement points. Do this by establishing cooling water flow to the process stream coolers for each of these streams. Trace out the water lines to determine how this is done.

Performing these operational details will establish finite reflux operations.
18. **Cascade Controller Schemes**

The following cascade controller schemes are available to simplify column operations in various experimental modes:

<table>
<thead>
<tr>
<th>Variable of Primary Interest</th>
<th>Primary Controller</th>
<th>Secondary Controller</th>
</tr>
</thead>
<tbody>
<tr>
<td>Overhead Temperature (and, in effect, Composition)</td>
<td>Overhead Temperature controller, C305</td>
<td>Reflux Flow Rate controller, F310</td>
</tr>
<tr>
<td>Bottoms Temperature (and, in effect, Composition)</td>
<td>Bottoms Temperature controller, C302</td>
<td>Hot Oil Temperature controller, T340</td>
</tr>
<tr>
<td>Reflux Drum Level (Finite Reflux)</td>
<td>Reflux Drum Level controller, L310</td>
<td>Distillate Flow Rate controller, F320</td>
</tr>
<tr>
<td>Reflux Drum Level (Total Reflux)</td>
<td>Reflux Drum Level controller, L310</td>
<td>Reflux Flow Rate controller, F310</td>
</tr>
<tr>
<td>Reboiler Level</td>
<td>Bottoms Level controller, L330</td>
<td>Bottoms Flow Rate controller, F330</td>
</tr>
</tbody>
</table>

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2 For many experimental programs, the use of the overhead or bottoms temperature control cascade systems may be counterproductive for at least two reasons: 1) primary controller tuning has not been verified, and 2) short-term experiments may not justify the time needed to achieve steady control of the primary variables. Unless the experimental program in question specifically calls to the use of these loops or the program objectives are particularly benefited by it, avoid their use.
19. Unit Startup (in Total Reflux Mode)

- Turn on the cooling water solenoid (in upper right corner of the screen); place the cooling water temperature controller (T363) in AUTO with an SP of 10°F, if not already set there.

**ALWAYS KEEP THE COOLING WATER ON WHEN RUNNING THE UNIT.**

- Start circulating the hot oil
  - Put the hot oil flow rate controller (F342) into AUTO
  - Give it a SP of 4 GPM
  - Turn on the hot oil pump

- Start heating the hot oil
  - Turn on the switch on the hot oil heater
  - Put the hot oil temperature controller (T340) into AUTO
  - Give it a SP of 230°F
  - Wait for boil-up (overhead vapor will start condensing and filling the reflux drum in about 30 minutes)

- Start refluxing the overhead condensate as soon as reflux drum level exceeds 15%
  - Put the reflux flow rate controller (F310) into AUTO
  - Give it a SP of 1.0 gph
  - Turn on the reflux pump (Interlock on this device prevents operation if reflux drum level is below 10%)

- Decrease the reboiler duty when the tower bottoms level reads below 100%
  - Lower the SP of the hot oil temperature controller to 210°F

- Adjust reflux flow rate as necessary to hold a “constant” reflux drum level
  - (20.0 ± 0.5%, 50.0 ± 0.5%, or other justified drum level)
  - Change the SP of the reflux flow rate controller as necessary

- Adjust the tower bottoms level as necessary to bring the level to 50.0 ± 1%
  - If too low, add some feed
    - Give the feed flow rate controller (F300) an OP of 20%
    - Turn on the feed pump
    - Wait for the level to rise to 49%
    - Turn off the feed pump
    - Give the feed flow rate controller an OP of -6%
  - If too high, withdraw some bottoms
    - Give the bottoms flow rate controller (F330) an OP of 20%
    - Turn on the bottoms pump
    - Wait for the level to drop to 51%
    - Turn off the bottoms pump
    - Give the bottoms flow rate controller an OP of -6%
20. **Unit Shutdown (from Finite Reflux Mode)**

When leaving for the day, use the following procedure to shut down the unit (takes about 30 minutes):

- **Stop heating the hot oil**
  - Turn off the switch on the hot oil heater
  - Put the bottoms composition controller (C302) into **MAN**
  - Put the hot oil temperature controller (T340) into **MAN**; give it an **OP** of -6%
  - Turn off the hot oil pump
  - Put the hot oil flow rate controller (F342) into **MAN**; give it an **OP** of -6% and an **SP** of 0 GPH

- **Stop preheating the feed**
  - Put the feed preheat controller (T350) into **MAN**; give it an **OP** of -6%

- **Stop feed to the unit**
  - Turn off the feed pump
  - Put the feed flow rate controller (F300) into **MAN**; give it an **OP** of -6% and an **SP** of 0 GPH

- **Stop withdrawing distillate product**
  - Put the reflux drum level controller (L310) into **MAN**
  - Put the distillate flow rate controller (F320) into **MAN**; give it an **OP** of -6%

- **Stop withdrawing bottoms product**
  - Turn off the bottoms pump
  - Put the tower bottoms level controller (L330) into **MAN**
  - Put the bottoms flow rate controller (F330) into **MAN**; give it an **OP** of -6%

- **Cool the tower with reflux as much as possible**
  - Put the overhead composition controller (C305) into **MAN**
  - Put the reflux flow rate controller (F310) into **MAN**; give it an **OP** to 18%
  - When the reflux pump stops due to reflux drum level below to 10%, give the reflux flow rate controller an **OP** of -6%

- **Continue to cool the tower until all condensation stops**
  - Wait for the top tower temperature to drop below 148°F
  - Turn off the cooling water by closing the solenoid; leave (T363) controller in **AUTO** with **SP** of 10°F
  - If on, manually turn off the cooling water to the distillate and bottoms coolers

- **Save any necessary data and log off the Flex Stations.**
21. Some Instrument Conversions / Calibrations

The following conversions / calibrations obtain:

<table>
<thead>
<tr>
<th>Item</th>
<th>Conversion / Calibration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed tank ( \Delta % ) to ( \Delta ) weight</td>
<td>( 2.76 \pm 0.02 ) ( (95%CI) ) lb/%</td>
</tr>
<tr>
<td>Distillate tank ( \Delta % ) to ( \Delta ) weight</td>
<td>( 2.74 \pm 0.08 ) ( (95%CI) ) lb/%</td>
</tr>
<tr>
<td>Bottoms tank ( \Delta % ) to ( \Delta ) weight</td>
<td>( 2.66 \pm 0.02 ) ( (95%CI) ) lb/%</td>
</tr>
</tbody>
</table>
PDU Unattended Total Reflux Operation

Unattended Use\(^1\) by: ______________ between _______ and\(^2\) _______ on __________

- ☐ Cooling Water ON.
- ☐ Hot Oil Pump ON.
- ☐ Hot Oil Flow Rate on control with SP = __________ GPM, PV = __________ GPM.
- ☐ Hot Oil Temperature on control with SP = __________ \(^\circ\)F, PV = __________ \(^\circ\)F.
- ☐ Reflux Pump ON.
- ☐ Reflux Drum Level cascaded to Reflux Flow Rate.
- ☐ Reflux Drum Level on control with SP = __________\%, PV = __________\%.
- ☐ Reflux Flow Rate PV = __________ GPH.
- ☐ Bottoms Level between 48 and 52%.
- ☐ Notified Lab Coordinator by email (iramir1@lsu.edu) at: ________ AM/PM

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\(^1\) Hang this form on the thermosiphon reboiler after completing accurately with needed information.

\(^2\) Here, post the times between which the unit will be unattended by you, and the date.
PDU Unattended Finite Reflux Operation

Unattended Use\(^1\) by: ______________ between _______ and\(^2\) _______ on __________

- Cooling Water ON.
- Hot Oil Pump ON.
- Hot Oil Flow Rate on control with SP = __________ GPM, PV = __________ GPM.
- Hot Oil Temperature on control with SP = __________ °F, PV = __________ °F.
- Reflux Pump ON.
- Reflux Drum Level cascaded to Distillate Flow Rate.
- Reflux Drum Level on control with SP = __________ %, PV = __________ %.
- Distillate Flow Rate PV = __________ GPH.
- Distillate Composition cascaded to Reflux Flow Rate.
- Distillate Composition on control with SP = __________ °F, PV = __________ °F
- Feed Pump ON.
- Feed Flow Rate on control with SP = __________ GPH, PV = __________ GPH.
- Bottoms Level on control with SP = 50% and PV = __________ %.
- Bottoms Flow Rate PV = __________ GPH.
- Notified Lab Coordinator by email (iramir1@lsu.edu) at: __________ AM/PM

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\(^1\) Hang this form on the thermosiphon reboiler after completing accurately with needed information.

\(^2\) Here, post the times between which the unit will be unattended by you, and the date.