Tray Distillation Unit (TDU)

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 Tray Distillation Unit (TDU) is a small pilot unit designed to demonstrate the process of fractionation.

2. Description of Facilities

The TDU apparatus is a modified version of the Model 9079 Scott Tray Distillation Unit, consisting primarily of a small distillation column with six (6) sieve trays, an electrically heated reboiler, and a total condenser. TDU is instrumented sufficiently to demonstrate continuous fractional distillation in the partial separation of methanol from a mixture of methanol, isopropanol, and/or water.

Apart from understanding the basic theory behind the unit operation of distillation sufficiently to study it in an experimental setting, it will be necessary to familiarize oneself with the TDU system by studying the detailed schematic in Figure 1 and then tracing out the lines, identifying the control and shut-off valves, pumps and other instruments. The Laboratory Instructor can provide a ‘tour of the unit’ to facilitate this.

An electronic power transducer on the electrically fired reboiler immersion heater indicates directly, in watts, the reboiler heat duty\(^1\). The total condenser is cooled by municipal water, measured by the rotameter mounted near the right end of the condenser. The feed and reflux streams can be preheated electrically. Feed can be directed to any tray in the column. Liquid sample points allow sampling of feed, distillate, bottoms, and individual tray liquids. An Emerson DeltaV control system provides measurement and/or control of temperature, level, flow rate, and reboiler power. Manually readable rotameters provide backup measurement capability for distillate, reflux, and feed flow rates. A dedicated Hewlett-Packard gas chromatograph allows offline analysis of feed, tray liquid, overhead and bottoms product.

The time required from a cold startup to achievement of steady state is typically 90 to 120 minutes. From that point, moving to a new set of conditions requiring compositional change may take roughly 60 minutes for column compositions to line out to steady-state values\(^2\).

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\(^1\) The voltmeter and ammeter mounted near the center of the unit panel display measurements needed to calculate any additional heat supplied by the electrically fired reboiler strip heaters. These strip heaters are normally used during heatup of TDU but may not be needed during experimental runs, depending on conditions desired. Consult with your instructor regarding their use.

\(^2\) In Fall 2009, just short of 50 lbs. of glass marbles were placed in the reboiler vessel, substantially reducing its liquid holdup and thus reducing the overall column capacity. Reduced capacity has resulted in quicker line out to steady-state conditions and faster column dynamics in general.
Figure 1 – Tray Distillation Unit (TDU)
3. Basic Operation of TDU from DeltaV Operate Run

Logging On and Bringing Up the DeltaV Operate Run Display

The DeltaV system can be accessed in virtual fashion from any of the computers in the control room. Though there are several DeltaV virtual machines that allow such access, the che-uolab-dv2 virtual machine is reserved for, and set up properly for access to the Tray Distillation Unit. Accessing this virtual machine is done through Window’s Remote Desktop Connection feature.

To access one of the DeltaV virtual machines, follow the steps outlined here:

- Logon to Windows 7 using a valid LSU ID and password
- Double-click the Remote Desktop Connection icon on the Desktop
- On the Remote Desktop Connection popup that appears, enter the name of the virtual DeltaV station desired. [For the Polymerization Reactor (POLY, use che-uolab-dv1 or che-uolab-dv4. For the Tray Distillation Unit (TDU), use che-uolab-dv2. For the Packed Bed Reactor (CAT), use che-uolab-dv3.]
- Click the Options button, and then click the Local Resources tab. Under Local devices and resources, click the More… button. Expand the Drives area if necessary. Check the apps drive box and the box for Removable Disk (if you have installed a jump drive and want it to be accessible from the DeltaV virtual machine). Click OK. Click Connect.
- At the Do you trust this remote connection? menu, click Connect.
- On the Windows Security popup that appears, click Use another account icon.
- In the two fields that appear next, type .\administrator in the Use name field and deltav in the Password field. Click OK.
- A Remote Desktop Connection warning menu appears next. Click Yes.
- A DeltaV Logon menu appears next. Type deltav in the Password: field. Click OK.
- A Flexlock menu window appears next. Click the Windows Desktop button. Minimize this menu window.

At this point, the DeltaV virtualized station of choice is up and operating. From here you can access the DeltaV Operate Run program and the Process History View program (and Control Studio from che-uolab-dv1) from the Windows Start menu. You should also be able to access the apps drive and a connected jump drive (if installed) from Windows Explorer on the DeltaV virtual machine.

To start up the control schematic navigate to Start > DeltaV > Operator > DeltaV Operate Run. The UOLAB_Overview display should come up. Click on
the hot-linked photo of the Tray Distillation Unit. Doing this should automatically open the **TDU_Main** display. If a dialog box appears indicating an error, click **Skip All** on the dialog box.

**Turning on Pumps, Flows, and Heaters**

The reflux, feed and bottoms pumps are turned to and from the on (red) and off (green) position by clicking the appropriate s/s (startup/shutdown) button in the **PUMPS** section of the display.

Similarly, the feed, reflux and reboiler heaters are turned to and from the on (red) or off (green) position by clicking the appropriate o/o (on/off) button in the **(PRE)HEATER** section of the picture.

The condenser cooling water is turned to and from the on or off position by clicking the **ON/OFF** button in the **COOLING WATER** section of the display. The button shows **green** in the off position and **red** in the on position.

The use of **green** and **red** here are helpful. When these devices are all in their non-running state, they show **green**; in the running state they show **red**.

**Using Controller Interlocks**

All the pumps and heaters have **interlocks** that will turn them off if certain conditions are not met. For example, the feed and reflux heaters can be turned on only when there is some feed or reflux flowing, the reboiler heater can be turned on only when the reboiler temperature is below 120°C, the reflux pump can be turned on only if there is enough level in the reflux drum, the feed pump can be turned on only if there is some room in the reboiler, and the bottoms pump can be turned on only while there is some minimum level in the reboiler. Each of these has an **override** button with the heading **ovr** over it, for use in case of some emergency or other unique operating situation. Checking the **override** box effectively disables the **interlocks**. Overriding these **interlocks** may require justification to the Laboratory Instructor.

**Changing Controller Parameters from Controller Faceplates**

On the larger operations schematic, the appearance of a small icon – identifiable by a gray box with three vertical lines – signals the presence of an automatic controller. To the left of each controller icon are the **process value** (PV, the measurement) in yellow, the **setpoint** (SP) in white, and the **output** (OP) in cyan. To change a parameter on a controller, click on this icon to bring up a faceplate. From the faceplate, controller mode can be changed by clicking on the desired mode button on the left side of the faceplate.

Manually changing the controller **output** (OP) is only possible in **MANual** mode. To
change the controller output, click on the MANual value field at the top of the faceplate and enter a new value. Click-dragging the large cyan pointer – present only when the controller is in MANual mode – to a new position also changes the output of the controller. The smaller cyan pointers are output limit indicators and cannot be changed from the controller faceplate.

When the mode is not MAN, the controller uses the process value (PV), setpoint (SP) and tuning constants to calculate a new output (OP) every processing pass.

Manually changing the controller setpoint (SP) is only possible in AUTO mode. To change the controller setpoint, click on the setpoint value field on the right side of the faceplate and enter a new value. Click-dragging the large white pointer – present ONLY when the controller is in AUTO – to a new position also changes the setpoint of the controller. The smaller white pointers setpoint limit indicators and cannot be changed from the controller faceplate.

**Using Cascade Control**

Sometimes controllers are stand-alone (e.g. the feed flow rate controller), and sometimes they are in a cascade structure (e.g. the reflux level controller is cascaded to the reflux flow rate controller). Cascade connections are indicated by dotted lines on the schematic in Figure 1. The upper controller in a cascade is called the PRIMARY and the lower controller 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 cascade structure fully on control). In Emerson’s DeltaV System, this mode is called CAS. When the mode of the primary is AUTO and the mode of the secondary is CAS, the cascade is closed and the primary sends its OP to the secondary controller’s SP. When the mode of the secondary is AUTO, the human operator is responsible for changing the SP to control the process.

**Accessing Additional Controller Details**

Across the bottom of the faceplate are six icons that call up other displays with more details about this controller. The two most useful ones for this experiment are the first one from the left, which provides access to controller parameters; and the second one from the right, which calls up the historical trend for this controller.

**Accessing Real-time Process History**

To start up the real-time process history view at any time, simply navigate to Start > DeltaV > Operator > Process History View, and then open TDU_Main (if it does not open automatically). Chart scales can be compressed or expanded by clicking those buttons on the menu bar. Scales can be shifted up or down by click dragging on the scale of interest.
4. Historical Data Access Using DeltaV Continuous Historian

A continuous historical record of all relevant temperatures, flows, and levels is kept by the DeltaV system on its hard disk(s). Selected portions of these data can be imported into an Excel spreadsheet for analysis. This spreadsheet must be saved to a flash drive or a personal directory if it is to be used on any other personal computer than a dedicated DeltaV workstation as files stored on the DeltaV network are not accessible from the general LSU network.

To import data from DeltaV history to Excel, Emerson has provided an Excel add-in called the DeltaV Continuous Historian. It appears under the Add-Ins menu in Excel 2007 when this program is opened on a DeltaV workstation. Any process variable that is enabled in History Collection can be imported. Most of these variables are collected every 10 seconds, 30 seconds, or 1 minute, so it doesn't make sense to try to read the data any faster.

Though the menu features of the DeltaV Continuous Historian can be used for their intended purpose, requesting an ad hoc retrieval of data for more than one or two tags is tedious and time-consuming. To avoid this labor, an Excel 2007 template file is available for TDU data retrieval. This template is preloaded to request ALL the historically trended TDU tags. The only information that the user must supply is the starting date and time, the ending date and time, and the time interval between data values.

To import data, open Excel 2010 on the DeltaV workstation and follow these steps:

1. Put Excel calculations in Manual mode before opening the template or attempting to change the starting and ending dates.

2. Open the data retrieval template file for TDU.

   It is located on the Desktop, in the DeltaV_Excel_Data_Collector_Files folder under the name TDU_Data_Retrieval.xlsx

3. Enter (or modify) the starting date and time for the data of interest as mm/dd/yyyy hh:mm into cell A4.

   For example, data starting August 5th, 2008 at 9AM would be entered as 8/5/2008 9:00

4. Enter (or modify) the ending date and time for the data of interest as mm/dd/yyyy hh:mm into cell A6.

   For example, data ending August 5th, 2008 at 3PM would be entered as
5. Enter (or modify) the desired time interval between data values.

For example, to request data values every 10 seconds, enter “10 seconds” into cell A8. For data values every 2 minutes, enter “2 minutes” into cell A8. Any value of seconds, minutes, or hours may be used, but some values make more sense than others. Using values faster than the fastest data originally collected makes no sense. So, values below 10 seconds only make sense if data collection on some variables has been set to time intervals below 10 seconds. Data will be interpolated where there are no values.

A maximum of 2161 readings (2160 intervals) are available in any one data retrieval operation. This will allow the retrieval of 6 hours of 10 second data, 36 hours of 1 minute data, or any other combination that results in 2160 (or fewer) intervals. If more data are required, two separate requests must be made and concatenated manually. Of course, if only a few tags are needed, one can bypass the use of the template and retrieve data ad hoc using DeltaV features.

6. After entering the last of these user inputs, click the Add-Ins menu tab, click the DeltaV drop-down tab, roll over the Continuous Historian option, and select Refresh. All values in the spreadsheet should update, signaling successful retrieval of data.

7. To save these results, these data must be turned into static values, as follows:
   Select the entire worksheet (by clicking in the upper left hand corner of the worksheet adjacent to the A1 cell. Copy this selection (e.g., ctrl v). Select the 2nd worksheet tab. Using Paste Values, paste the values copied from the Data Collection tab into the 2nd worksheet. These values are now static and will be available even when the spreadsheet is relocated to another computer.
   The values in the first tab can be deleted at this point IF the file will not be used to retrieve additional data from the DeltaV.

8. Save this file under a different name onto the desktop of the DeltaV virtual machine. To get this file to your apps drive, for example, simply right-click the file and click Copy. Then, minimize the RDC machine, access the desired destination from the original client desktop and Save to the appropriate folder/location. (Remember that calculation mode for that file is Manual.)

9. The units for the data are the same as in DeltaV. If the word shutdown appears instead, then either:
   - History collection was not enabled for that variable, or
   - No history collection because the system was shut down, or
   - No history collection because the variable did not change more than its deviation value set in history collection (i.e., nothing’s changing!)
5. Some Safety Considerations

- Never click the **ovr** (override) button for the pumps or the heaters unless certain that an override is justified. Be prepared to justify this to a Laboratory Instructor.
- All unused sample material must be returned to the feed tank.
- Check all temperatures periodically. No temperature should exceed 80°C.
- Although the bottoms pump can run dry for short periods, it should be shut off if a no flow condition is noted.
- Make sure the condenser water is flowing whenever the experiment is running. This is the only active method for removing heat from the column.

6. Startup and Operating Procedures and General Guidance

*Modes of Operation*

The two most common modes of operation for a continuous distillation column are **total reflux** mode and **finite reflux** mode.

**Total reflux** mode facilitates some aspects of column performance measurement and simpler column startup. In **total reflux** mode, all overhead material from the column is condensed and returned to the column as reflux. Therefore, **total reflux** mode is an important performance mode, since this maximum reflux condition produces the best possible component separation at otherwise comparable conditions. Additionally, there is no feed addition or product withdrawal from the column in **total reflux** mode. Without the distraction of managing these external streams, reflux flow and reflux drum level control can be more easily established.

**Finite reflux** mode is a more practical mode for operation of a continuous distillation column for the obvious reason that continuous streams of products result. Feed continuously introduced to the column is fractionated or split into (at least) two products: an overhead stream and a bottoms stream. Though **finite reflux** mode does not produce the maximum possible component separation accomplished in **total reflux** mode, it does result in a commercially viable situation with product streams.

*Ensuring the Achievement of Steady State*

As mentioned previously, the TDU column requires some time to approach steady state. An indication that steady state has been achieved will be that all temperatures, levels and flow rates in the column remain constant. However, the most conclusive confirmation that steady state has been achieved is constant product composition, determined by offline GC analysis of liquid samples.
Conditions at Startup

All controllers should be in **MANual** at the start of a run. The condenser cooling water is turned to and from the on or off position by clicking the **ON/OFF** button in the **COOLING WATER** section of the display. The button shows **green** in the off position and **red** in the on position.

**Safety Item:**
Always keep the cooling water on when running this experiment. This is the only means of removing heat from the column through condensation of overhead product.

Starting Up TDU in Total Reflux Mode

In **total reflux** mode, once the composition of the initial feed charge has been set, only one independently selectable variable remains: reboiler heat duty. The reflux rate that ultimately results from a steady state **total reflux** mode operation is forced. This inevitability suggests an obvious strategy for starting up in **total reflux** mode: fix the reboiler heat duty and adjust reflux rate to hold constant level in the reflux drum. To this end, the following method

3 is suggested for establishing steady state **total reflux** mode operation in the TDU:

- **Supply cooling water to the TDU** – Turn on the cooling water. The condenser cooling water is turned to and from the on or off position by clicking the **ON/OFF** button in the **COOLING WATER** section of the display. The button shows **green** in the off position and **red** in the on position.

(Condenser water flow rate is controlled by a rotameter on the front panel of the TDU. Unless performing experiments that have as their intent the manipulation of this rate, operate this rotameter at full-scale open.)

- **Ensure that the starting reboiler liquid level is appropriate** – Normally this level has already been established prior to each day’s run(s). Starting levels from 85 to 95% provide reasonable operational capabilities after some of this material is displaced to the column proper and the reflux drum during startup. Experimental program may also suggest the choice of level here. There is a low level alarm at 30% to ensure that the liquid level does not fall below the level of the heaters. If the level is too low prior to startup, add feed before starting experiments; if too high prior to startup, use the bottoms pump to withdraw material and return it to the feed tank. (Though it is fine to add material AFTER startup, withdrawing material AFTER startup in total reflux experiments will change the composition of the material in the unit.)

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3 When confidence with TDU operations is obtained, one may adjust this method to speed up start-up.
• **Supply heat to the TDU by establishing the desired reboiler heat duty** – Turn on the reboiler heater (and the strip heaters via the switch at the top right of the unit), put the reboiler temperature controller (H004) in **MAN**, give it an **output (OP)** of at least 60% or other value required by the experimental program⁴, and wait for overhead material to condense and the reflux drum to start filling.

(Acceptable TDU performance is obtained with reboiler heat duty produced by reboiler heater **output (OP)** in the range from 60% or more with the use of additional heat from the reboiler strip heaters. Without the strip heaters, higher reboiler heater **output (OP)** is required for best performance. Consult with your instructor regarding this service if you have questions.)

• **Establish a low but sustainable reflux flow rate** – When the reflux drum level reaches 50%, put the reflux flow rate controller (F002) in **AUTO**, give it a **setpoint (SP)** of 20%, and turn on the reflux pump. As soon as the reflux begins to flow (signified by the yellow bar on the controller faceplate), start decreasing the **setpoint (SP)** in steps of a percent or so every few seconds until the reflux flow rate is 12-13%. At reflux rates much lower than this, the turbine meter may stop reading⁵.

• **Establish reflux preheating** – The reflux preheater power is turned on with the reboiler heater power. However, one still needs to put the reflux preheat controller (H002) in **AUTO**, give it a **setpoint** of 65°C, or other suitable value. (Use this heater unless the requirements of a specific experimental program suggest otherwise.)

• **Find and sustain the correct total reflux flow rate** – Wait for the reflux drum level to reach at least 50%. Manually adjust the reflux rate by changing the **setpoint (SP)** on the reflux flow controller as necessary to provide a constant reflux drum level of between 25 and 75% in the reflux drum. Again, experimental program requirements may suggest a more appropriate reflux drum level. Depending on the nature of the experiment, it may be possible – even advisable – to use the Reflux Drum Level / Reflux Flow Rate **cascade** control system to automatically adjust the reflux rate to keep the drum level constant.

• **Identify the existence of steady state** – When all flows, levels, temperatures and compositions are lined out, steady state has been achieved.

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⁴ Getting the unit to operating temperature more quickly can be accomplished by firing this heater (and perhaps the strip heaters) at 100% of output until tray temperatures have nearly peaked, at which point heater output can be set to the level required by the experimental program. Indeed, it may be possible to fire this heater at 100% of output until the reflux drum level has approached the midway point. In any case, ultimate lineout to steady state will be necessary prior to collection of quality data.

⁵ Successful operations in manual mode have been observed with the reflux flow rate controller setpoint as low as 8 to 9%. No guarantees, however.
Conducting Operations in Finite Reflux Mode

Establishing finite reflux mode operations in TDU is more complicated than establishing total reflux mode operations. Three external flows must be established: column feed, distillate product, and bottoms product. In finite reflux mode, after the composition, rate, and entry point of the feed stream have been set, two independently selectable variables remain: one bottoms-related variable and one overhead-related variable.

This premise suggests a strategy for establishing finite reflux mode⁶: establish total reflux mode operation at the desired reboiler heater duty – bottoms-related variable; begin feed addition; adjust the bottoms product rate to maintain reboiler constant; adjust the reflux and distillate rates to meet the overhead-related variable requirements and to maintain constant reflux drum level; attain steady state.

To move from total reflux mode to finite reflux mode:

- **Establish total reflux operation** – Start the column up in total reflux mode at the reboiler duty desired for the finite reflux operations, establishing a steady reflux drum level, but do not wait for the column to come to steady state otherwise.

- **Select desired feed tray** – Move the flexible feed line to the feed input point of choice. One can choose any of the six sieve trays or the reboiler as a feed input point. To move the flexible feed line from one location to another, simply pull back on the red-colored locking ring and then pull the feed line out from the current location, letting the locking ring slide forward. At this point, simply push the feed line into the new location. If the new location is a sieve tray, use your gloved hand to support the back of the glass column when inserting the flexible feed line.

  This operation is best done before starting up the unit, but can be done safely as described above even if liquid is in the column and even after feed if flowing through the feed line. The quick disconnect locking ring assembly prevents liquid from flowing out of the feed line when disconnected, even if the feed pump is on.

- **Establish feed to the column** – Put the feed flow rate controller (F001) in AUTO, give it a **setpoint (SP)** corresponding to the desired feed rate, turn on the feed pump, turn on the feed preheater, put the feed preheat controller (H001) in AUTO, and give it a **setpoint (SP)** of 65°C or other temperature consistent with the experimental program.

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⁶ When confidence with TDU operations is obtained, one can modify this procedure to speed up start-up.
(Use this heater unless the experimental programs suggests otherwise.)

- **Establish a trial reflux rate** – Put the reflux flow rate controller in **AUTO** and give it a **setpoint** (SP) of 80% of the **total reflux** case as a starting point\(^7\).

- Make any operational changes needed to achieve desired operating conditions.

- Start withdrawing distillate product to maintain a level of 25-75% in the reflux drum. Put the distillate flow rate (F003) in **AUTO** and adjust its **setpoint** (SP) unless other instructions specifically call for **MANual** control.

- Start withdrawing bottoms product to maintain a constant level of 60 to 80% in the reboiler. Put the bottoms flow rate (F004) in **AUTO**, turn on the bottoms pump, and adjust its **setpoint** (SP) unless other instructions specifically call for **MANual** control.

- **Identify the existence of steady state** – When all flows, levels, temperatures and compositions are lined out, steady state has been achieved.

**Collecting and Saving/Disposing of Liquid Samples**

Sampling needs differ depending on experimental program requirements. The TDU is equipped with the following sample points: distillate/reflux, bottoms, feed, and individual trays.

**Distillate/Reflux**

A small sample valve is located just below the total condenser. A sample taken via this valve is representative of the current composition of the overhead product. There is a small amount of stagnant holdup in the line and valve that must be discarded to get a fresh, representative sample.

**Bottoms**

A small sample valve is located at the bottom right of the reboiler. A sample taken via this valve is representative of the current composition of the reboiler contents and hence bottoms product. There is a small amount of stagnant holdup in the line and valve that must be discarded to get a fresh, representative sample.

**Feed**

In total reflux mode, the only feed used by the column is that which has been

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\(^7\) This is only a suggested value, consistent with previous studies. Obviously, this rate would be lower than its total reflux counterpart inasmuch as distillate withdrawal will be part of any finite reflux program.
charged to the reboiler prior to operations. Thus, a representative sample of feed for total reflux operations would be taken from the reboiler bottoms sampling valve prior to start of operations.

In finite reflux mode, the feed source to the column is the feed tank in the rear of the unit. A small pipette inserted into the lift-top opening of this vessel can be used to retrieve a sample of this material. If for some reason this material is not also representative of the contents of the reboiler, then reconciling column performance may be problematic.

**Individual Trays**

Measurement of individual tray performance requires knowledge of tray composition. Each TDU sieve tray is equipped with a septum port for retrieving a representative sample of liquid from that tray. The use of a curved-needle sampling syringe is essential to collect such a sample. The curved needle permits locating the point of the syringe needle at the desired liquid location on the tray while not distorting the septum port.

**Caution:** Sampling syringe needle is very sharp. Handle with care.

**Saving/Disposing of Liquid Samples**

Return all unneeded samples to the feed tank at the end of the day and replace the empty sample vials, caps and septa in the tray in the TDU-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.

**Column Flooding**

In the course of all runs, monitor column loading by careful observation of the trays. If a tray fills completely with froth, the column may become [flooded](#). If this occurs during finite reflux operations, the flow of bottoms product would decrease sharply, probably to zero, or the reboiler level may show a strong, steady drop. To correct this condition, try dumping the tower by reducing reboiler heat input.

In an experimental program that explores column flooding, reaching such conditions can be an explicitly desired part of unit operations.

**Feed Composition**

Traditionally, TDU feed consists nominally of methanol (~50 wt.%), isopropanol (~30 wt.%), and water (~20 wt.%). However, particular experimental objectives may call
for different compositions Rely on the Laboratory Coordinator, Mr. Bob Perkins, to prepare, monitor, and adjust if necessary, the feed composition – unless otherwise dictated by the experimental program objectives. The Lab Coordinator also ordinarily transfers feed from the feed tank into the reboiler in preparation for operations. Inasmuch as column performance is highly dependent on feed composition, assuming nominal compositional values is likely inappropriate.

Ternary vapor-liquid equilibrium data for this system has been included as an attachment to this Operating Manual.

7. **Shutdown Procedure**

When operations are complete, complete the following steps to shutdown TDU safety:

1. Turn off all three heaters, the bottoms pump and the feed pump.
2. Put all controllers except the reflux flow rate in **MAN** and set their **outputs** to 0%.
3. Put the reflux flow rate controller in **MAN** and set its **output (OP)** to 20%.
4. Turn on the reflux pump **override** and leave the reflux pump on until the reflux drum is empty.
5. As soon as the reflux drum is empty, take off the reflux pump **override**, turn off the reflux pump, put the reflux flow rate controller in **MAN**, and set its **output (OP)** to 0%.
6. Leave the cooling water on until all (six) tray temperatures are below 62 °C, and then turn it off.
7. Close the **DeltaV Operate Run** program and logoff the workstation from the **Start** menu.

8. **Gas Chromatograph Operation**

An Agilent 7890 gas chromatograph (GC) is used to separate and quantify the components in the liquid samples produced by TDU. 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 should be used to analyze samples:

- Using the thin-client terminal labeled CHE-UOLAB-27 dedicated for the TDU
  **Connect to: che-uolab-gc2.lsu.edu**. A login screen appears. Log in using your user name and password, on the LSU domain.
• Click on the icon **CHE-7890a-02 Online**. The Agilent ChemStation program opens.

• 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. The pressure should be set to 4.5 psi. It may take a minute to get there, or it may never get EXACTLY there. You can still proceed as long as it’s reasonably close.

• If (or when) the pressure reaches the target value, a green **READY** message is displayed on the ChemStation screen.

• Inject a 0.1 microliter sample (same volume each time) and press the **Start** button on the GC front panel. Your instructor or the lab coordinator will demonstrate proper injection technique.

• 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 3 minutes. There will be a chart with the GC peaks on it. Scroll down the report to see the **Area%** results. Area% is related to, but not equal to, wt%. Note: To see your results at a later date, select “Data Analysis” (lower left hand of screen), then “File” (upper left hand), then “Load Signal”, and browse to your old data file.

• To obtain wt% from area%, perform the following calculation, using the proper response factors for each component. You can find these by selecting “Data Analysis” (lower left hand of screen), then “Calibration”. The response factors are given in a table. The equation to use is:

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

• When you are finished all work on the CHE-7890a-02 virtual desktop, click the Start icon and then select the **Log off** option. This will log you out of this virtual desktop.

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 TDU-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.

9. Additional Operating Data and Considerations

*Flow Meter Calibrations*

Current calibrations of all flow meters are as follows (where $F = \text{mL/min}$, $R = \text{device reading}$, and $G = \text{gal/min}$):

<table>
<thead>
<tr>
<th>Service</th>
<th>Instrument</th>
<th>Calibration Equation</th>
<th>Calibration Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reflux</td>
<td>Rotameter</td>
<td>$F = 5.69^*R - 8.58$</td>
<td>$(14 \leq R \leq 32)$</td>
</tr>
<tr>
<td></td>
<td>Turbine meter</td>
<td>$F = 5.83^*R - 5.74$</td>
<td>$(13 \leq R \leq 31)$</td>
</tr>
<tr>
<td>Feed</td>
<td>Rotameter</td>
<td>$F = 6.05^*R - 11.0$</td>
<td>$(11 \leq R \leq 30)$</td>
</tr>
<tr>
<td></td>
<td>Orifice meter</td>
<td>$F = 3.73^*R + 18.0$</td>
<td>$(10 \leq R \leq 40)$</td>
</tr>
<tr>
<td>Distillate</td>
<td>Rotameter</td>
<td>$F = 1.085^*R - 0.22$</td>
<td>$(12 \leq R \leq 71)$</td>
</tr>
<tr>
<td></td>
<td>Orifice meter</td>
<td>$F = 1.719^*R - 5.25$</td>
<td>$(4 \leq R \leq 42)$</td>
</tr>
<tr>
<td>Bottoms</td>
<td>Orifice meter</td>
<td>$F = 1.765^*R + 4.74$</td>
<td>$(20 \leq R \leq 60)$</td>
</tr>
<tr>
<td>Condenser water</td>
<td>Rotameter</td>
<td>$G = 1.189^*R$</td>
<td>Full range</td>
</tr>
</tbody>
</table>

[The feed, distillate and bottoms meters were calibrated using **water** at 20°C. The reflux

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8 Inasmuch as instrument calibrations, especially electronic instruments, can change with time, it is wise practice to take backup manual readings from the rotameters (where available) to cover those situations where the electronic counterpart is more suspect.
meters were calibrated using water at 17°C. The condenser cooling water rotameter was calibrated using water at supply temperature (~90°F) and reading the bottom edge of float wide-spot. Use relevant meter theory to determine actual flows. Reflux and feed rotameter floats are glass (SG = 2.53); distillate float is stainless steel (SG = 8.02).

Instrumentation Issues

Turbine and Orifice Flow Meters

The reflux turbine meter sometimes does not provide a proper signal to the DeltaV system. This typically occurs at flow rates too low to impart sufficient momentum to the turbine. If this occurs, rely on the rotameter to measure the flow for that service. To minimize the occurrence of this problem, briefly open the flow control valve completely while in MANual mode in order to get the turbines spinning; then cut back to the desired flow rate. Of course, because the distillate and bottoms flow rates vary a lot but are key to good material balances, their accurate measurement is important. Averaging them over a steady-state time span of interest provides the best data. (Note that negative readings on some of the flow meters can have physical meaning.)

Level ΔP Meters

The reflux drum ΔP level meter works well. The reboiler ΔP sometimes does not. This is largely due to liquid loss from the top pressure leg, which is supposed to operate full of liquid. When the ΔP reading stops changing, but the visual level is changing, this problem is occurring. When the ΔP meter works correctly, it is possible to control the reboiler level directly by closing the cascade from the Bottoms Level Controller to the Bottoms Flow Rate Controller. Use a level setpoint consistent with the marks. When the reboiler level ΔP meter doesn’t work, switch the Bottoms Flow Rate Controller to AUTO and adjust its setpoint to keep the reboiler level approximately constant. This should not be hard to accomplish, because the bottoms flow rate controller responds rapidly.

10. Distillation Theory References


9 Though using cascade to control reboiler level is an option, cascade control will likely result in more variation in reboiler bottoms flow rate than may be desirable for effective material balanced experimental runs. Consider this option only when this concern is not relevant.
11. Combinations of Finite Reflux Specifications Known to Work in ChemSep6:

<table>
<thead>
<tr>
<th>Top Specification</th>
<th>Bottom Specification</th>
<th>Solution Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reflux Ratio</td>
<td>Mole Fraction MeOH</td>
<td>Either</td>
</tr>
<tr>
<td></td>
<td>Reboiler Temperature</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Reboiler Heat Duty (Enter as negative value)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Bottoms Flow Rate</td>
<td>Continuation Only</td>
</tr>
<tr>
<td>Reflux Flow Rate</td>
<td>Mole Fraction MeOH</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Bottoms Flow Rate</td>
<td>Continuation Only</td>
</tr>
<tr>
<td></td>
<td>Reboiler Temperature</td>
<td></td>
</tr>
<tr>
<td>Distillate Flow Rate</td>
<td>Reboiler Heat Duty (Enter as negative value)</td>
<td>Either</td>
</tr>
<tr>
<td>Condenser Heat Duty</td>
<td>Mole Fraction MeOH</td>
<td>Either</td>
</tr>
<tr>
<td></td>
<td>(Enter as positive value)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Reboiler Temperature</td>
<td></td>
</tr>
</tbody>
</table>

Necessary ChemSep templates files have been included with this Operating Manual.