Separation Processes 1: Continuous Assessment

Laboratory: Distillation Column

2014

In this lab you will start-up, operate and shut-down a distillation column in order to rate the process equipment (determine its effectiveness). The distillation column you will operate is the CE 600 Continuous Rectification column produced by Gunt-Hamburg. The manual for this equipment is also available on the MyAberdeen site for the course.
Your objectives for this lab session and report are to:

- To become familiar with columns (in particular distillation columns) and their associated ancillary equipment (storage, pumps, condensers, etc.).
- To gain a better understanding of the difficulties of controlling a distillation column.
- To rate the column using experiments performed under total reflux.
- To be able to estimate the overall efficiency **AND** the Murphree tray efficiencies of an existing column.
- Perform an energy balance over the column to estimate the energy lost from the column.

If there are any unusual circumstances or difficulties during your experiments these should be noted in your lab book and discussed in your report.

1 Requirements

To participate in this experiment, you will need

- To have read the student safety handbook in advance.
- A printed copy of this document, and to have read these instructions in advance.
- Your lab coat and safety glasses.
- Your lab book and a pen.

2 Regulations

If you have missed any of the above requirements, you will not be allowed to enter the laboratory so please ensure you are prepared. While in the lab, please act in a calm and safe manner and respond promptly to the instructions of the lab coordinators and demonstrators. If you miss your allocated laboratory session without good cause, you will receive a CAS 0 for the assignment and will be issued a C6 for the course. These laboratory sessions can take the full 3 hours to complete so you must arrive on time for the start of the laboratory session.

3 Experimental Procedure

1. Before entering the lab, you must have read the student safety handbook and be wearing your lab coat and safety glasses.

2. Ensure the column has electrical power, is plumbed into the drainage system and is supplied with cooling water.

3. Switch on the column control unit and the column control computer. Use the thermocouple selection dials on the top of the control unit to check that the column is at a safe temperature before touching any part of the equipment.

4. Ensure that **all** valves are closed on the column. Ensure you know what a closed valve looks like before turning any valves. If you are unsure, ask the demonstrators in the lab for assistance. Closed valves have a handle perpendicular to the flow direction.

5. Familiarise yourself with the column while it is inactive and cool. Locate the condenser (III), the phase separator (IV), the reboiler (II), the column pressure gauge (P2), the column pressure-drop transducer (P1), and the condenser cooling-water rotameter (XVI) (see Fig. 1).

6. Locate and open the column vent valve underneath the column pressure gauge (P2). This will ensure the distillation is carried out at atmospheric pressure.

7. Locate and open the two valves on either side of the column pressure-drop transducer (P1). This will allow you to determine the pressure drop across the column, an important control and design variable.

8. Record the reported column pressure drop (while the column is inactive) in your lab book. You will need to use this value to calibrate your pressure drop readings.

9. Start the flow of cooling water to the condenser. An acceptable flow-rate is 100-200 l/hr of cooling water (record the value you use). Check this flow rate with the condenser cooling-water rotameter (XVI).

10. The re-boiler at the base of the column has already been charged with a 5% v/v isopropanol-water mixture. You will need to take a sample of the mixture in the reboiler to confirm this concentration before starting the experiment.

11. Log into the control computer and start the Gunt-Hamburg control software. Ensure that you have sensible readings for all sensors and that the PC control light is extinguished on the column control. You cannot operate the column without the control computer connected.

12. Start the logging of the column readings to a file located on your home directory. You will need to open the charts section of the software, switch to the file and sample selection page to save the data in and click the continuous record button. When the software is logging data the green bar at the top of the chart page will periodically “tick”. Ensure you have set the number of samples to a large amount so that data collection is not stopped early (¿10000).

13. The reflux ratio control should remain off on the control panel. When the reflux control is off it will cause the column to operate under total reflux.

14. Using the control panel, ensure the heater is set to computer control and switch it on. Use the control software to set the heater to 100% to begin heating the mixture.

15. Once the temperature in the reboiler (T3) reaches around $80°C$ the mixture will begin to boil. Write down the vapour temperature (T12) at which condensate is first seen entering the phase separator (IV).
16. When you see condensate first entering the phase separator (IV) **AND** being returned to the column, lower the heater power to 30%. This should result in a column pressure drop of around 15 mbar.
17. You will now need to wait and allow the column to come to steady state, you can track this using the graph page available in the Gunt-Hamberg software.
18. Once the column is at steady state, periodically sample the distillate and reboiler concentrations (3 samples spaced at least 10 minutes apart). These will be analysed later either using Raman spectroscopy or a Gas Chromatograph (GC).
19. Ensure you record the time at which the samples are taken to correlate these to the logged data.
20. If the level of liquid begins to rise above the weir in the phase separator, temporarily lower the reboiler power (as low as 20% if needed) and allow sufficient time for the excess liquid to drain out of the column (this effect is known as flooding).

4 Shutdown Procedure

1. Once the samples are collected, set the reboiler to 100% power. Observe the column pressure and the phase separator. What happens and why?
2. Set the the reboiler to 0% power. Observe the column pressure and the glass tray. What happens and why?
3. The column is now cooling down, stop the recording on the GUNT software and log out of the control computer.
4. Leave the control panel active so the temperature in the column may be monitored.
5. Lower the condenser flow-rate to 50 l/hr to condense any vapour produced while the column cools.

5 Report Requirements

Your report should include:

- A discussion on the calculation of the VLE properties of isopropanol-water mixtures and any remarks concerning the distillation carried out today.
- An estimate of the overall efficiency of the column, describing how this was performed and detailing the calculations and data sources.
- An estimate of the Murphree tray efficiencies of the column (you will need to use the tray temperatures to estimate the tray concentrations).
- A calculation of the heat lost from the column and recommendations to reduce this heat loss.
- A discussion on the importance of and the behaviour of the column pressure drop during the experiment.

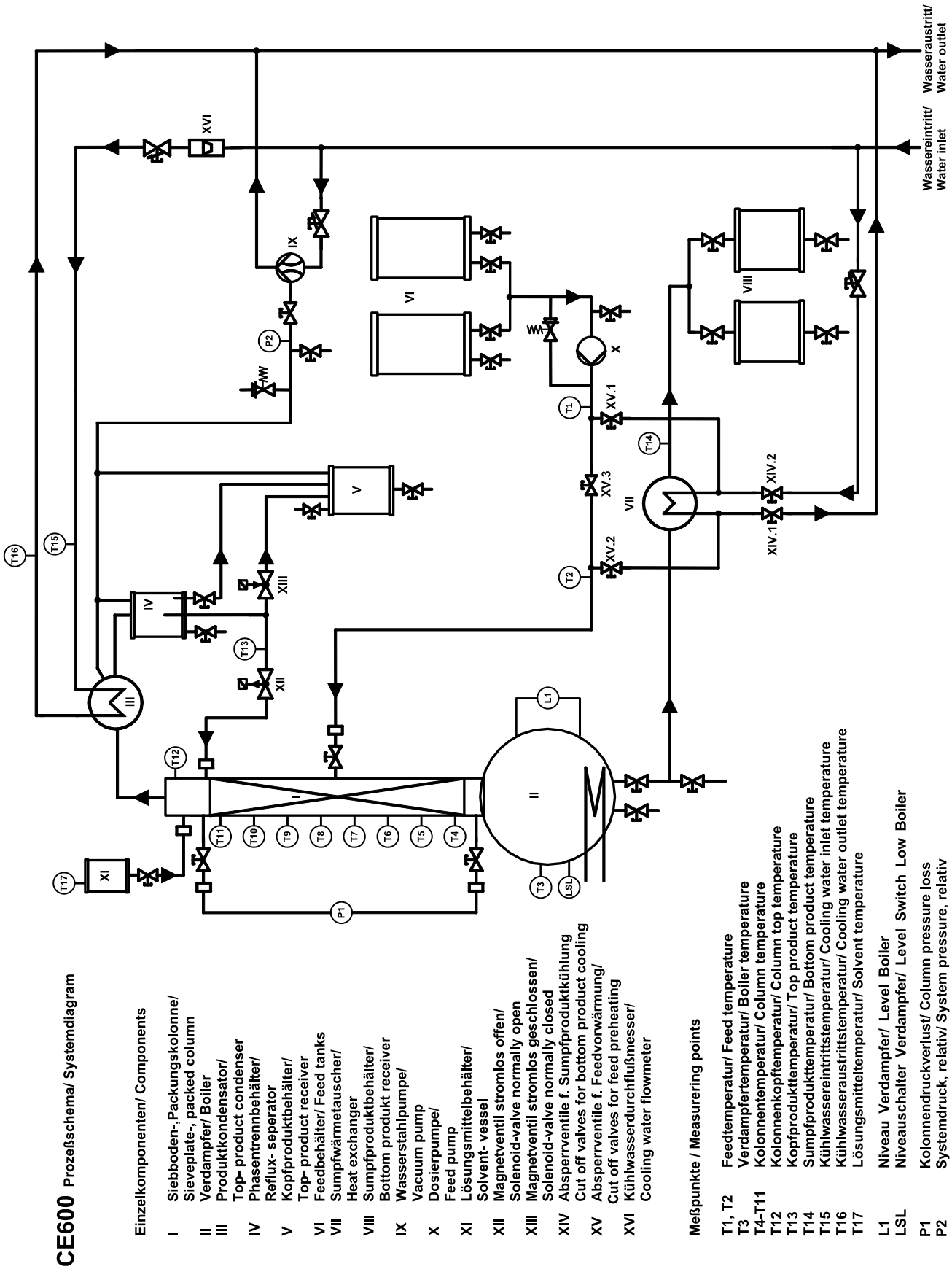

Figure 1: The process flowsheet for the CE600 unit.

6 Equipment Notes

- The equipment is prone to flooding if the evaporator/reboiler is too full. There must be at least 6 litres charged in the evaporator for the bottom product syphon (where the samples are taken from), so somewhere above this level but below 9 litres is acceptable. The reboiler will cease to operate somewhere around a filling level of 3–4 litres for safety.