Manual for the distillation column

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Contents

1 Introduction

This is a manual for the continuous distillation column separating methanol and water. First a description of the column setup is presented in section 2. A control program for the column has been implemented in LabView, and the user interface to this program is presented in section 3. Then detailed start-up and operating instructions are given in section 4 and 5. A short description of the underlying source code is given in section 6. For reference, the I/O module connections are given in appendix A.

The manual does not intend to give a detailed description of the underlying control algorithms, instead the reader is referred to [1] for controller details.

2 Experimental column setup

In this section a short description of the different components in the experimental column is given, together with an overview of inputs to and outputs from the system. The different control loops involved in the automatic control system are presented.

2.1 The column

Figure 1 shows a picture of the distillation column together with a flow sheet. Table 1 gives the most important column data.

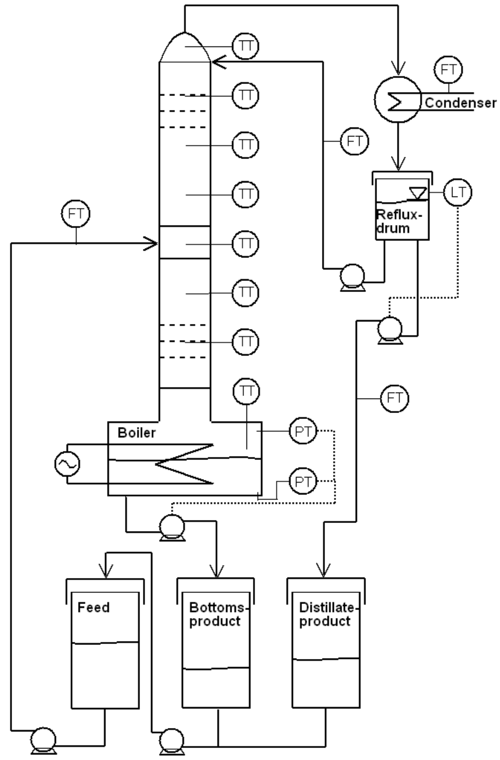
The column consists of two sections filled with 6mm Raschig-rings unstructured packing. A condenser is connected to the top of the column. The condensate flows into the reflux drum, from where a fraction is pumped back into the column as reflux to get desired composition. The distillate pumped out of the system and into the distillate product tank is used to control the level in the reflux drum.

Vapor is produced by heating coils in the boiler connected to the bottom of the column. The boiler level is controlled through the bottoms flow pumped out of the system and into the bottoms product tank. For security, the boiler is protected with a plexi-glass cover shield.

A feed mixture of methanol and water is pumped from the feed tank and into the feed section approximately in the center of the column. To avoid spill after finishing an experiment, a manually controlled recycle pump is used to transfer distillate and bottoms back into the feed product tank.

The entire system is mounted in an aluminum frame which can be moved as a single unit.

(a) Experimental setup



(b) Flow sheet with sensors and actuators

Figure 1: Distillation system

Table 1: Column data

Material:	Glass and steel
Column height:	250cm
Diameter:	50mm
Condenser:	0.3m^2 i.d.
Max boiler power:	3kW
Max reflux rate:	4ml/s
Max feed rate:	5ml/s
Boiler volume:	10l
Reflux drum volume:	5l
Product tanks volume:	25l
Frame dimensions:	$120 \times 125 \times 285$cm (l×w×h)

2.2 Instrumentation

2.2.1 Actuators

Five pumps are connected to the column system to control the different flows. The reflux and feed pumps are continuous with 4-20mA input. The distillate and bottoms pumps are on/off pumps which are pulse width modulated to allow for different pump rates.

The recycle pump is of on/off type and is turned on manually by the operator when recycling the products before a new experiment. This pump is not a part of the automatic control system.

Four heating coils generate the vapor inside the column. Two coils are 1000W each, the two other ones are 500W, giving a total of 3kW power input. The heating coils are turned on and off by relays, and are pulse width modulated.

2.2.2 Sensors

There are eight PT-100 temperature sensors inside the column to measure the temperature profile. One of the sensors is in the boiler, the remaining ones are inserted via the feed section and the top, and are held in place by the column packing. A Fieldpoint RTD I/O module measures temperature sensor resistance and converts resistance to temperature.

Two pressure sensors are located at the top and at the bottom of the boiler respectively. The pressure difference between the sensors is proportional to liquid level in the boiler and is used as input to the boiler level controller. The column is open to air after the condenser in the top, hence the absolute pressure above the liquid boiler holdup gives the pressure fall over the column.

A level sensor is placed in the reflux drum to give feedback to the reflux level controller.

The cooling water is equipped with a flow meter to assist the operator when turning on cooling. The sensor output is also monitored during operation, and warnings are issued if the cooling water for some reason should stop.

Flow meters are placed on the feed, reflux and distillate flows. These sensors give visual feedback to the operator through displays on the sensors. They are not used as measurements in the control system for two reasons: The squeezing tube pumps produce pulsating flows which are below the measurement range of the flow meters in parts of the oscillation period. Hence the measurement signal is unsymmetrical and averaging or possibly a notch filter on the signal is not very suitable. The other reason why the flow meters are not used in the controller, is that especially the feed flow meter quite often gets stuck on a specific flow value. The flow meters use the angle of a small pendulum as a measurement proportional to mass flow rates. Small particles in the feed jam the pendulum in a certain position thereby making the measurement signal useless. Instead of using flow meters, pump characteristics are used to give estimates of the actual feed and reflux flows.

2.3 Control loops

As mentioned there are two level control loops in the system. A conventional LV-configuration is used where reflux and vapor flows are used for composition control, the level in the reflux drum is controlled through the distillate flow, and the boiler liquid level is controlled through the bottoms flow. Both of the level controllers are P-controllers where suitable gains were found by trial and error. Steady state level offsets are unimportant in this configuration, which is why no integral terms are included in the controllers.

If dangerous situations occur, an emergency switch is placed on the aluminum frame. Pressing the emergency switch cuts the main power supply and the column will be shut down. Dangerous situations happens very rarely, but the operator should pay attention to especially boiler and reflux drum holdup. If the boiler level gets below the heating coils, the coils may get overheated and possibly ignite the methanol gas, thereby causing an explosion. Another situation is if cooling water stops; after a while the condensator will then not be sufficiently cooled to condensate the distillate, possibly causing a gas leak.

With level loops closed, what is left to control are the compositions of the bottoms and distillate flows. The operator's job is to make sure the composition controller does this properly. Two different controllers are implemented; a conventional diagonal PI-controller where reflux is used to control distillate composition and boilup is used to control bottoms composition, and a 2×2 H_∞ controller where both of the inputs are used to control both composition outputs together.

3 LabView User Interface

Figure 2 shows a screen shot of the operator interface for the control program. The operator sees various controlled and manipulated variables in the left chart. All the temperatures are displayed in the center chart, and the temperature profile is displayed in the right chart.

In manual control mode, the operator can turn on and off the bottoms and distillate pumps by pushing the buttons in the lower left part. Reflux, heat and feed can be set by typing in numerical values in the corresponding input dialogs. These are the nominal inputs.

Nominal distillate and bottoms composition outputs (steady state outputs with a given nominal input) are typed into the the fields yd_{nom} and xb_{nom} respectively.

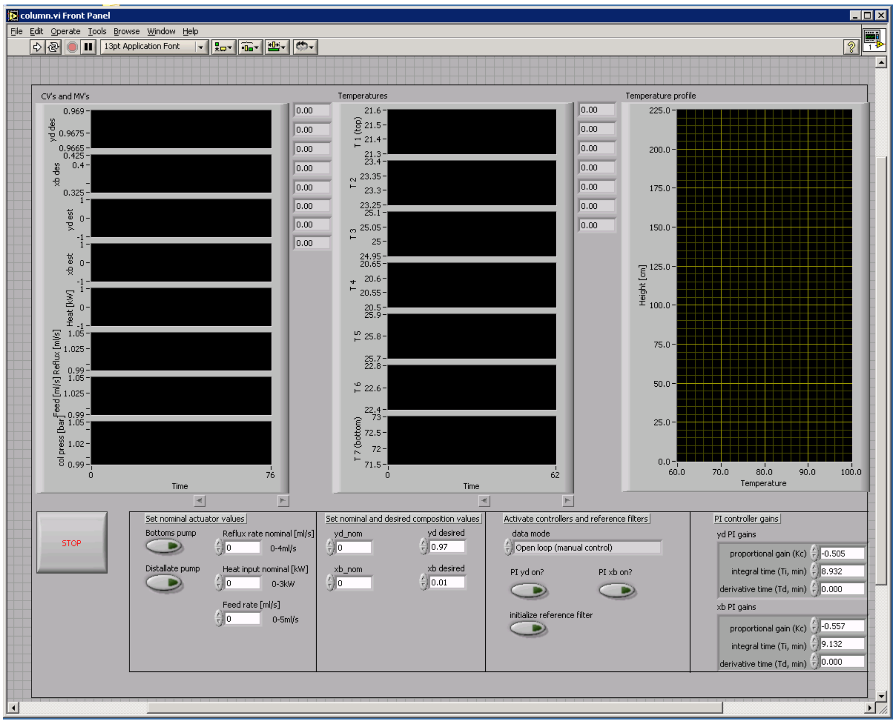


Figure 2: User interface

Desired outputs (reference values) are typed into the fields labeled yd_{desired} and xb_{desired}.

In the box marked "Activate controllers and reference filters", the operator selects what kind of controller to use. Available options are: Open loop (manual control), Level control, Composition control H_∞, and Composition control PI. In open loop, the operator decides all actuator values. With level control, the holdup in the reflux drum and in the boiler is controlled using the distillate and bottoms pump, but the operator is still in charge of reflux, heat input and feed rate. With composition control the two composition loops are closed, and the automatic controller will compensate for offsets between measured and desired compositions using the reflux and heat inputs. When using PI composition control, the two composition loops for y_d and x_b can be closed independently.

Steps in desired compositions are low-pass filtered to avoid large actuator changes. The filter can be initialized to the nominal values yd_{nom} and xb_{nom} by pushing the button labeled "initialize reference filter".

PI controller gains can be changed by the operator in the lower left dialog boxes. The default controller gains are believed to be reasonably good.

4 Operator instructions

Before you start, make sure the boiler is filled with water up to the black line, the reflux drum filled with methanol to just above where the glass begins and that there is feed in the feed tank. If the boiler level is low, pump some feed into the column. If the reflux drum level is low, you will have to run the column with little reflux for a while. Pump all the distillate and bottoms one at a time back into the feed tank using the recycle pump. Open the valve underneath the product tanks and turn on the recycle pump pushing the green button on the door of the I/O module enclosure. Close the valves and stir in the feed tank when finished. The column is now ready to be used. If this is the first time the column is used, and all product tanks are empty you can first fill the feed tank with 6 liters of water which you pump directly into the column to fill the boiler. Then pour 10 liters of methanol and 10 liters of water into the feed tank, and use this as feed.

Open the LabView program `column.vi` located in the folder `C:\Documents and Settings\Administrator\My Documents\destillasjonskollonne\column control final`. User name and password for the computer is Administrator/etanol.

Turn on cooling water, adjust the rate to 100-150liters/hour. Turn the boiler on full power (3kW). Wait until it boils and the temperature starts rising in the feed section, indicating that the vapor has reached the middle of the column. Then turn on the feed pump, and set feed rate F=1ml/s. When the vapor reaches the top temperature sensor, reduce boiler power to 1.5kW and turn on reflux. Try a reflux rate of L=2ml/s. This takes around 15 minutes. Turn on level control.

Let the column settle for a few minutes and adjust the nominal boiler and reflux inputs to get as close as possible to the desired operating region. Increasing reflux increases top composition, increasing boiler input increases bottoms composition. When you are satisfied, use the displayed composition estimates as nominal outputs. Set the desired composition outputs. There should not be a larger difference than 0.01 between nominal and desired composition. The composition estimates is not accurate outside $0.94 < y_d < 0.99, 0.005 < x_b < 0.015$, so select a desired composition between these values and try to keep the system within these bounds before turning on automatic control. Finally, turn on the composition controller. Select either the H_∞ controller or the PI controller. If using PI controller, you have the option to close one loop at a time by activating each controller independently with the corresponding on/off buttons. This is sometimes easier if the column is relatively far away from the desired setpoints.

Problems may occur when switching from manual to automatic control. If actual compositions are too far away from the desired compositions, or if there are large temperature oscillations when turning on the controller, output saturation is likely to occur. If the output saturates only for a short period of time (less than 15sec) this may not be a problem, the controller will go into oscillations but

Table 2: Start-up procedure for distillation column

Condition	Operation to be done
Time = 0	Boiler input = 3kW
	Cooling water > 100l/h
	Feed flow = 0ml/s
	Reflux flow = 0ml/s
Temp4> 40°	Feed flow = 1ml/s
Temp1> 40°	Boiler input = 1.5kW
	Reflux flow = 2ml/s
	Level control on
Temp. settled	Nominal compositions := actual compositions
	Desired compositions := something not too far from nominal values
	Composition control on

may be able to recover by itself. Wait and see if the time of saturation decreases, if it does the transition from manual to automatic control will most likely be successful. If on the other hand, saturation persists, eventually flooding will occur or the reflux drum will run empty. If this happens, turn of the controller, wait for the column to settle with only nominal inputs and restart the controller, this time with desired composition equal to actual composition. It may be necessary to restart the program (push stop, then play) to initialize the controllers, more specifically to nullify the integrated error for integral action.

5 Data logging

Inputs to and outputs from the column during operation are logged to file `data\columndata_x.dat` with x being an appended running number. This log file may be processed using the matlab script `readData.m`, placed in the same folder, which reads the log file and presents composition outputs and column inputs in plots. The file may easily be changed to display other variables.

6 Source code

The program is divided into two parallel loops. One loop runs every 0.1 seconds and writes actuator values to the column. This loop pulse width modulates the actuator values to the boiler and the distillate and bottoms pump. The second parallel loop is the main loop and runs every 2 seconds. This loop reads sensor data, does the necessary scalings, calculates actuator values, presents signals to the

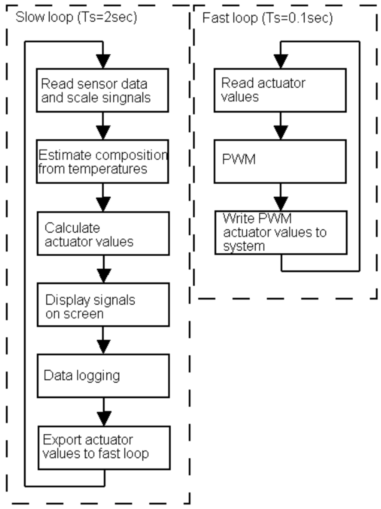


Figure 3: Control program flow sheet

operator and is responsible for logging data. A flow sheet for the control program is presented in figure 3. The LabView block diagram showing the two loops is shown in figure 4. The upper timed loop is the fast loop, while the lower one is the slow loop. The slow loop is in the read sensor values state in the shown figure.

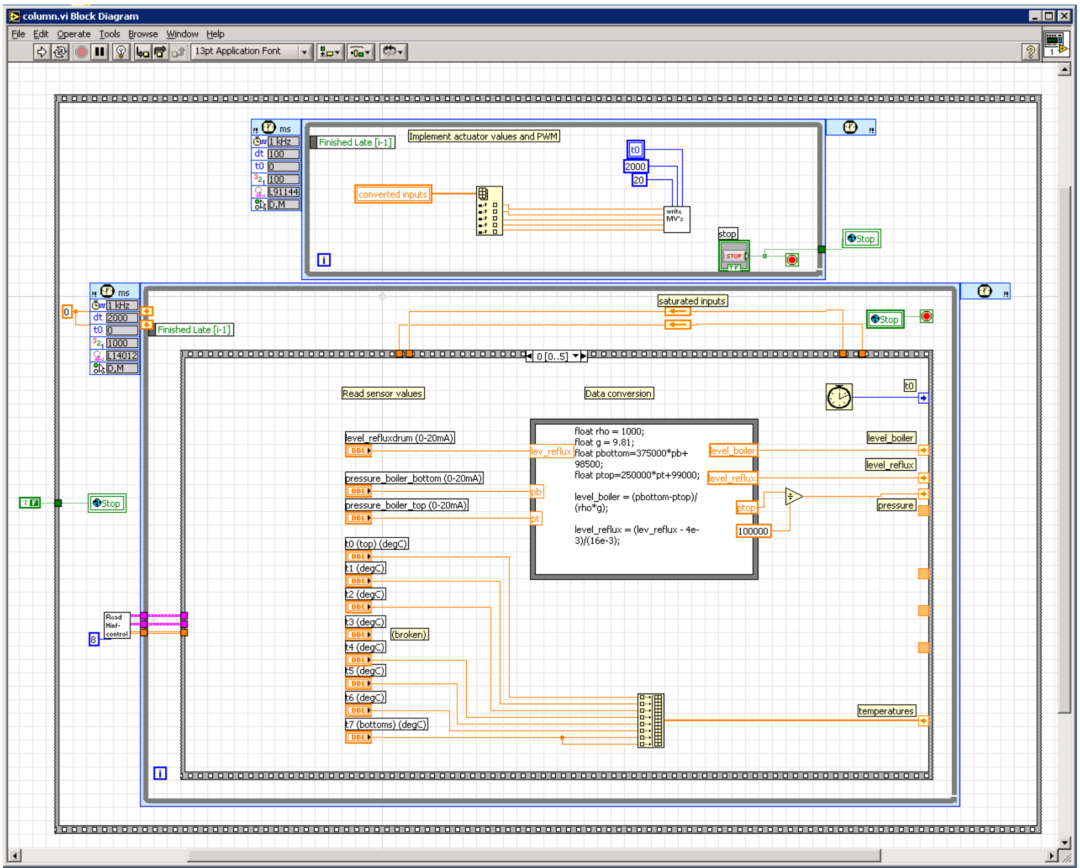


Figure 4: User interface

A I/O connections

Below follows a summary of equipment connected to the NI FieldPoint I/O modules.

Table 3: FieldPoint I/O connections

FP-AO-200	
Channel 0	Heating coils, relay 1
Channel 1	Heating coils, relay 2
Channel 2	Heating coils, relay 3
Channel 3	Pump Distillate, relay
Channel 4	Pump Bottoms, relay
Channel 5	Pump Reflux
Channel 6	Pump Feed
Channel 7	
FP-AI-100	
Channel 0	Flow meter, Reflux
Channel 1	Flow meter, Distillate
Channel 2	Flow meter, Feed
Channel 3	Level sensor, reflux drum
Channel 4	
Channel 5	Boiler pressure sensor, bottom
Channel 6	Boiler pressure sensor, top
Channel 7	
FP-RTD-124	
Channel 0	Temperature 0 (top)
Channel 1	Temperature 1
Channel 2	Temperature 2
Channel 3	Temperature 3 (broken)
Channel 4	Temperature 4
Channel 5	Temperature 5
Channel 6	Temperature 6
Channel 7	Temperature 7 (boiler)

References

[1] Jørgen K. Johnsen. *Robust Distillaton Control - Application of H-infinity Loop Shaping*, Master Thesis, NTNU, 2005