

**Experimental verification on active vapor split control for
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Experimental verification on active vapor split control for dividing-wall columns

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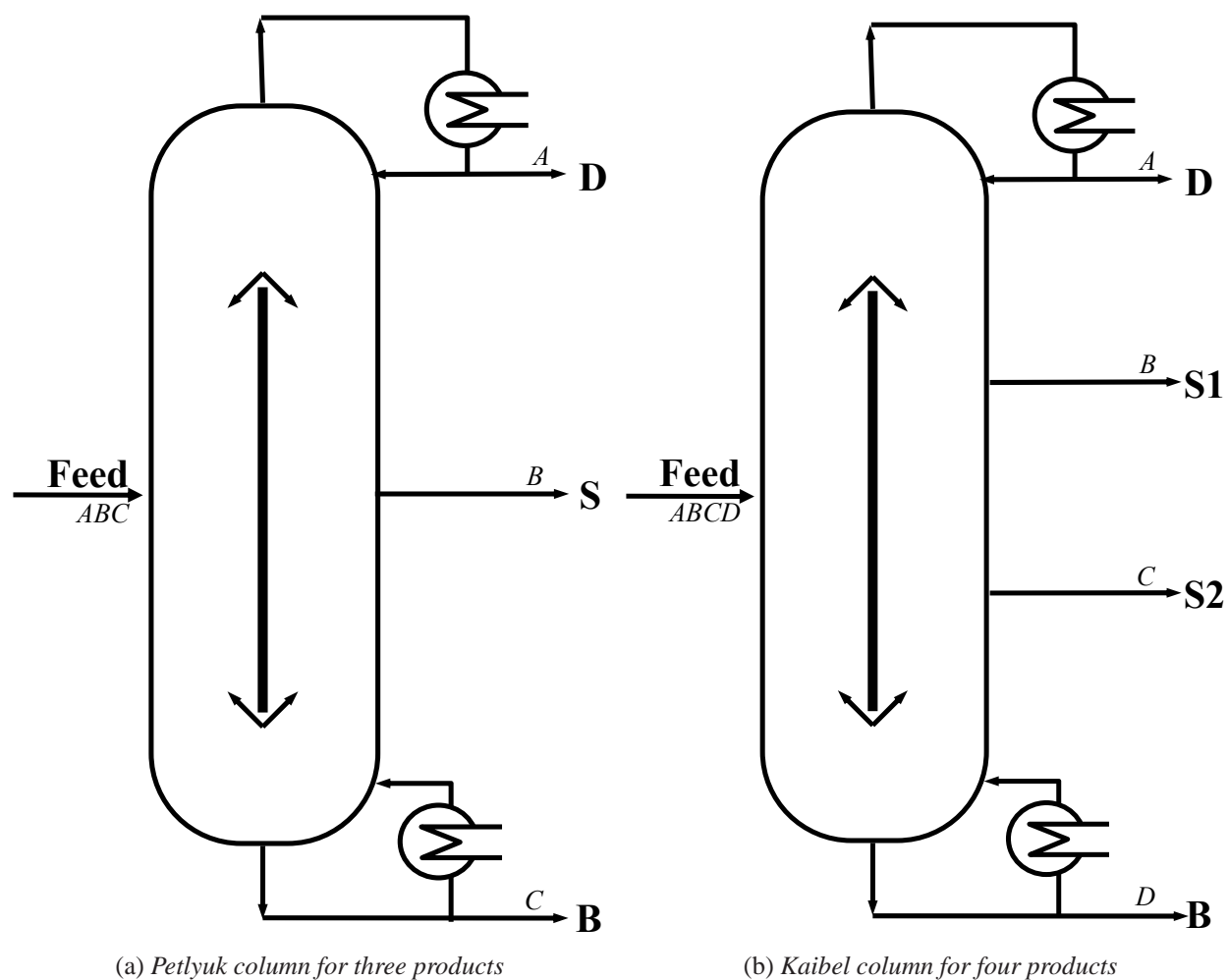
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Abstract

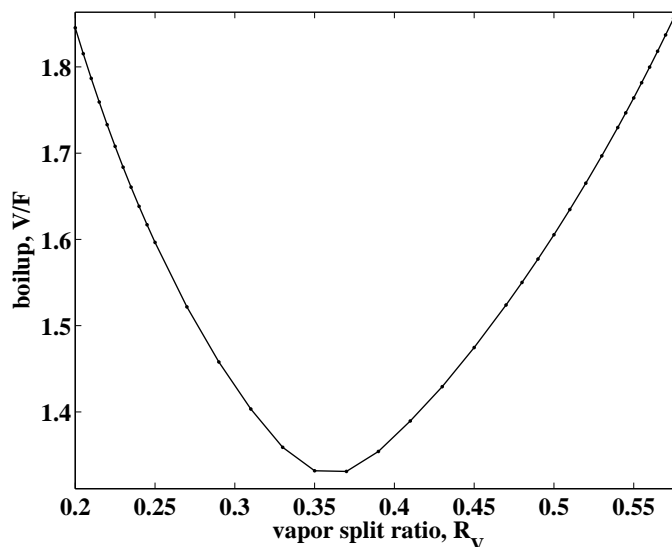
Dividing-wall distillation columns offer large energy savings, typically up to 40 % over conventional column sequences. However, the energy required for a separation depends on using an optimal vapor split. Hence, the energy saving potential may be lost if the column is operated away from its nominal point, for example, due to feed composition changes. This work demonstrates that the vapor split can be effectively used as a control degree of freedom for example, for temperature control in the prefractionator section. Together with an adjustable liquid split, the vapor split allows for optimal operation.

Keywords: Thermally-coupled columns, Dividing-wall columns, Petlyuk column, Kaibel column



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Figure 1: Dividing-wall columns with prefractionator section to the left of the dividing wall and “main” column section to the right.

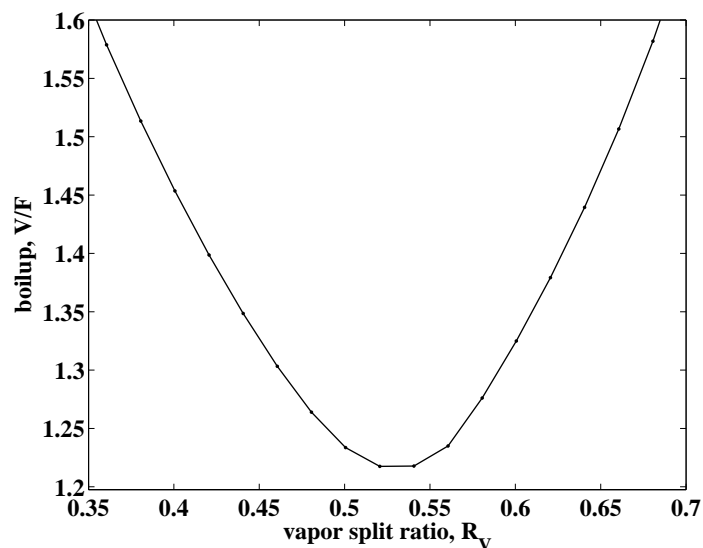


(a) *Three-product Petlyuk column: Boilup (V/F) vs Vapor Split Ratio (R_V)*

Data: Equimolar feed of methanol, ethanol and propanol with zero vapor fraction

Purities (mol %): 97.6 % (D), 97.3 % (S); 99.6 % (B)

Stages: 40 in prefractionator and 80 in main column (including top and bottom sections)



(b) *Four-product Kaibel column: Boilup (V/F) vs Vapor Split Ratio (R_V)*

Data: Equimolar feed of methanol, ethanol, propanol and *n*-butanol with 50 % vapor fraction

Purities (mol %): 98.9 % (D); 98.0 % (S1); 98.0 % (S2); 99.8 % (B)

Stages: 40 in prefractionator and 100 in main column

Figure 2: Effect of vapor split ratio (R_V) on boilup (V/F) for fixed purity specifications in dividing-wall columns. ($R_V \equiv$ fraction of vapor boilup that is sent to prefractionator from the main column)

Introduction

Dividing-wall distillation columns such as Petlyuk arrangements and the Kaibel column, shown in Figure 1 offer large capital and energy saving potentials compared to conventional schemes.¹⁻³ Their control and operations, however, remains a challenge. For three-product separation, the energy savings can be up to 30 % using a standard dividing-wall (Petlyuk) column with a single side stream. The Kaibel column with two side streams (Figure 3) can give up to 30 % energy savings for four-product separation. However, the energy saving potential can be lost if the column is operated away from the optimum vapor split ratio (see Figure 2). Thus, the flexibility in operation of such systems at minimum energy over a large range of feed conditions or product specifications, can be restricted by the absence of an active vapor split during operation.

Dividing-wall column have been successfully implemented industrially BASF.⁴ In the academic community, several works have been reported on start-up and operation of three-product Petlyuk columns (⁵⁻⁷). However, all earlier works exclude the use of vapor split as a degree of freedom. Agrawal and Fidkowski⁸ suggested as an alternative to use a vapor side draw from. Halvorsen and Skogestad⁹ showed that the feed vapor fraction affects the optimum operation of Petlyuk columns. Therefore, another alternative is to use the feed enthalpy as a degree of freedom, where the vapor fraction or degree of sub-cooling in the feed is varied to achieve optimum operation.

To motivate the need for active vapor split in dividing-wall columns, we first consider some simulation results. Halvorsen and Skogestad⁹ studied steady state optimal operation of three product Petlyuk column. They reported that there may be a narrow operating window with respect to various degrees of freedom for operation of such system at minimum energy. The control system should carefully designed to operate within this range to ensure operation at minimum energy. Further, this operating window may change in presence of various disturbances such as feed com-

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4 position and feed vapor fraction.

5 We confirm these results with a simulation study on a three-product Petlyuk column separating
6 equimolar saturated liquid feed of methanol, ethanol and propanol. The Wilson model is used for
7 the vapor-liquid equilibria and we assume constant molar overflow. For the given purity specifi-
8 cations, the boilup is minimum ($V/F=1.33$) for a vapor split ratio (R_V) of 0.37. In Figure 2a, we
9 plot the minimum boilup (V/F) required as the vapor split ratio is fixed at values different from its
10 optimum value of 0.37. By “minimum”, we mean that the liquid split (R_L) has been adjusted so
11 that the boilup is minimized for each R_V .
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20 Next we consider an example of the four-product Kaibel column separating equimolar feed of
21 methanol, ethanol, propanol and *n*-butanol with 50 % vapor fraction. Again the Wilson model is
22 used for the vapor-liquid equilibria and we assume constant molar overflow. The boilup (V/F) as
23 a function of the vapor split is shown in Figure 2b. The boilup is minimum for an optimum vapor
24 split ratio of 0.52 and again increases in both directions. Recently Ghadrddan et al.¹⁰ have reported
25 a detailed study on the need of active vapor split for four-product Kaibel columns. In summary, the
26 simulation results in Figure 2 shows that the energy usage (boil-up, V/F) is sensitive to the value of
27 R_V , and this motivates the need for introducing the vapor split (R_V) as a degree of freedom during
28 operation.
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38 In this work we demonstrate the use of direct active manipulation of the vapor split using an
39 experimental four-product Kaibel arrangement (Figure 3). The experimental column consists of
40 separate sections, but it is thermodynamically equivalent to a single-shell dividing-wall implemen-
41 tation (Figure 1b).
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49 Experimental Setup

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52 Figure 3a shows a schematic of our experimental column which is thermodynamically equivalent
53 to the dividing-wall arrangement proposed by Kaibel² for separation of a feed into four products
54 (D, S1, S2 and B) of desired purity. The column subsections are numbered for easy reference;
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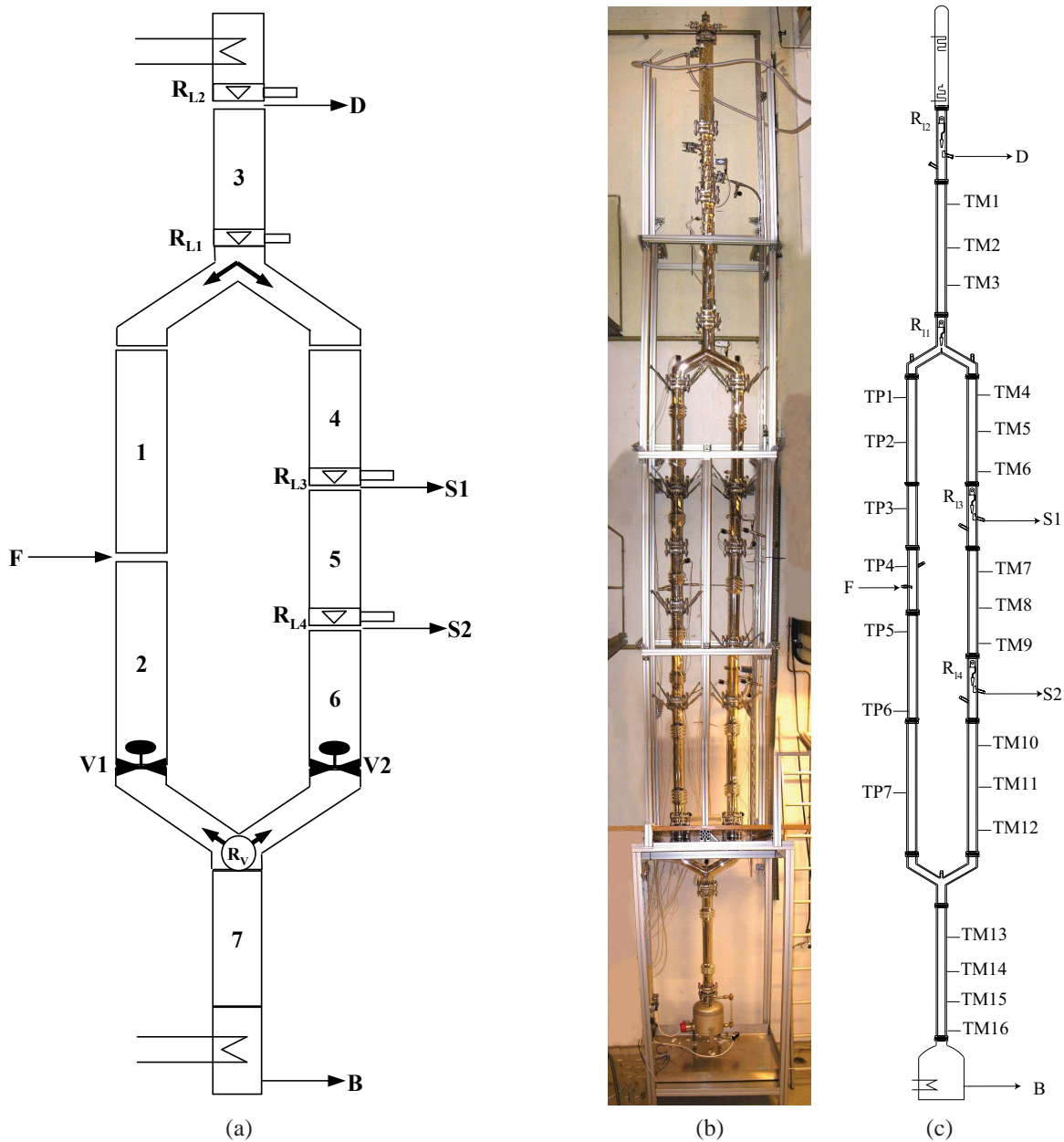
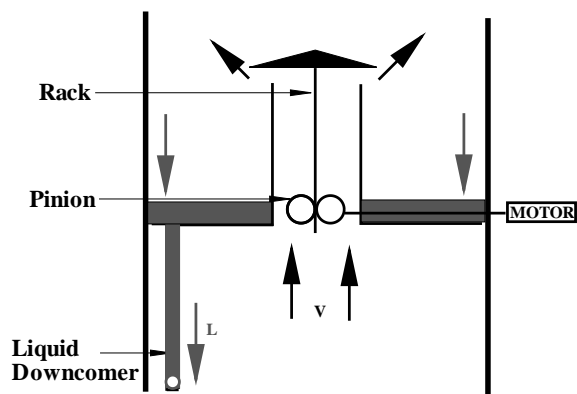


Figure 3: (a) Schematic of four-product Kaibel column with adjustable vapor split ratio (R_v)
 (b) Picture of the experimental column¹¹
 (c) Location of temperature sensors.¹¹



(a)



(b)

Figure 4: (a) Schematic and (b) picture of the two vapor split valves¹¹

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3 Sections 1 and 2 constitute the prefractionator while section 3 to 7 constitute the main column.
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5 In Figure 3b, we show a picture of the experimental column.¹¹ The height of the column is 8
6 meters and it operates under atmospheric pressure. The column subsections are packed with 6-mm
7 glass Raschig rings. The column sections have packed sections with temperature probes and their
8 locations are shown in Figure 3c.
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10 The reboiler is of the kettle type and its power is controlled by voltage to the heater elements
11 through a thyristor. The water-cooled condenser is mounted on top of the column. The condensed
12 vapor flows back to the column due to gravity; a part is take out as top product and the rest forms
13 the liquid reflux. The control setup is implemented in Lab ViewTM on a standard PC.
14

15 The liquid reflux split valve R_{L1} and the valves for the products, D, S1 and S2; R_{L2} , R_{L3} and
16 R_{L4} , respectively are all swinging funnels. These are controlled by externally placed solenoids.
17 Since these are ON/ OFF valves, a continuous output of the PI controller is implemented using
18 pulse width modulation.
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20 21 22 **Vapor Split Valves** 23

24 The two vapor split valves are made in stainless steel and are operated by externally placed electri-
25 cal motors using rack and pinion assembly. Figure 4a shows a schematic of the valves. There are
26 two such valves, one below section 2 and one below section 6 (denoted V1 and V2 in Figure 3a),
27 but they should be operated such that one of them is always fully open. The vapor flow rate through
28 the valve is manipulated by opening and closing a cap that sits on a steel valve seat. There is a liq-
29 uid downcomer which is needed to allow the liquid to flow against the pressure drop over the valve.
30 The downcomer is designed to ensure that the vapor passes only through the clearance between the
31 cap at the seat.
32

33 The circular pinion of each valve is powered by a step motor. The full span of the valve is
34 divided into 150 small steps. In the current setting, the free cross section in the valve is somewhat
35 too large, which results in very small required movements. As will be shown in the section below,
36 the valve can affect the flows only in the first 10 steps. Whilst the performance of the valve could be
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3 significantly improved, having such a poor resolution provides an excellent case for demonstrating
4 the effect of feedback, which we document below.
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9 10 **Experiment**

11 12 13 **Vapor Split valve behavior**

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16 The first experiment was designed to test the behavior of the vapor split valves. This was done
17 under total reflux conditions (no feed or products) and with constant liquid split (R_{L1}) using only
18 two chemical components, namely methanol and ethanol. After charging the reboiler, the heating
19 was started with a fixed duty of 1.9 kW.
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25 After reaching steady state operation, step changes were made to vapor valve V1 while valve
26 V2 was fully open. The results are shown in Figure 5, where we show the effect of these changes
27 on one prefractionator temperature ($T_2 \equiv TP5$) and one main column temperature ($T_5 \equiv TM7$).
28 Any change in the vapor flow rate resulting from changes by the vapor split valve should lead to
29 changes in these two temperatures. The output of the liquid split valve is manually fixed during
30 this run.
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38 When we close valve V1 from 15 steps to 10 steps at around 3 minutes, temperature T_2 starts
39 decreasing gradually while T_5 starts increasing. This indicates, as expected, that less vapor is being
40 sent to the prefractionator, while more vapor is being directed to section 6. At around 7 minutes,
41 V1 is further closed by 5 steps. This gives a more noticeable change in the vapor flows and is
42 clearly indicated by about 1 K drop in T_2 and about 0.6 K temperature increase in T_5 . This change
43 is reversed when valve V1 is opened from 5 steps to 15 at about 13 minutes. A series of changes
44 between 10 steps to 15 steps shows insignificant changes in the two temperatures. At around 33
45 minutes, V1 is closed from 8 steps to 3 steps. This leads to sharp changes in temperatures T_2 and
46 T_5 . At 37 minutes, the valve V1 is opened from 3 steps to 50 steps. Since the vapor dynamics
47 are very fast, the initial response on the temperatures is very quick, but the steady-state is restored
48 more slowly .
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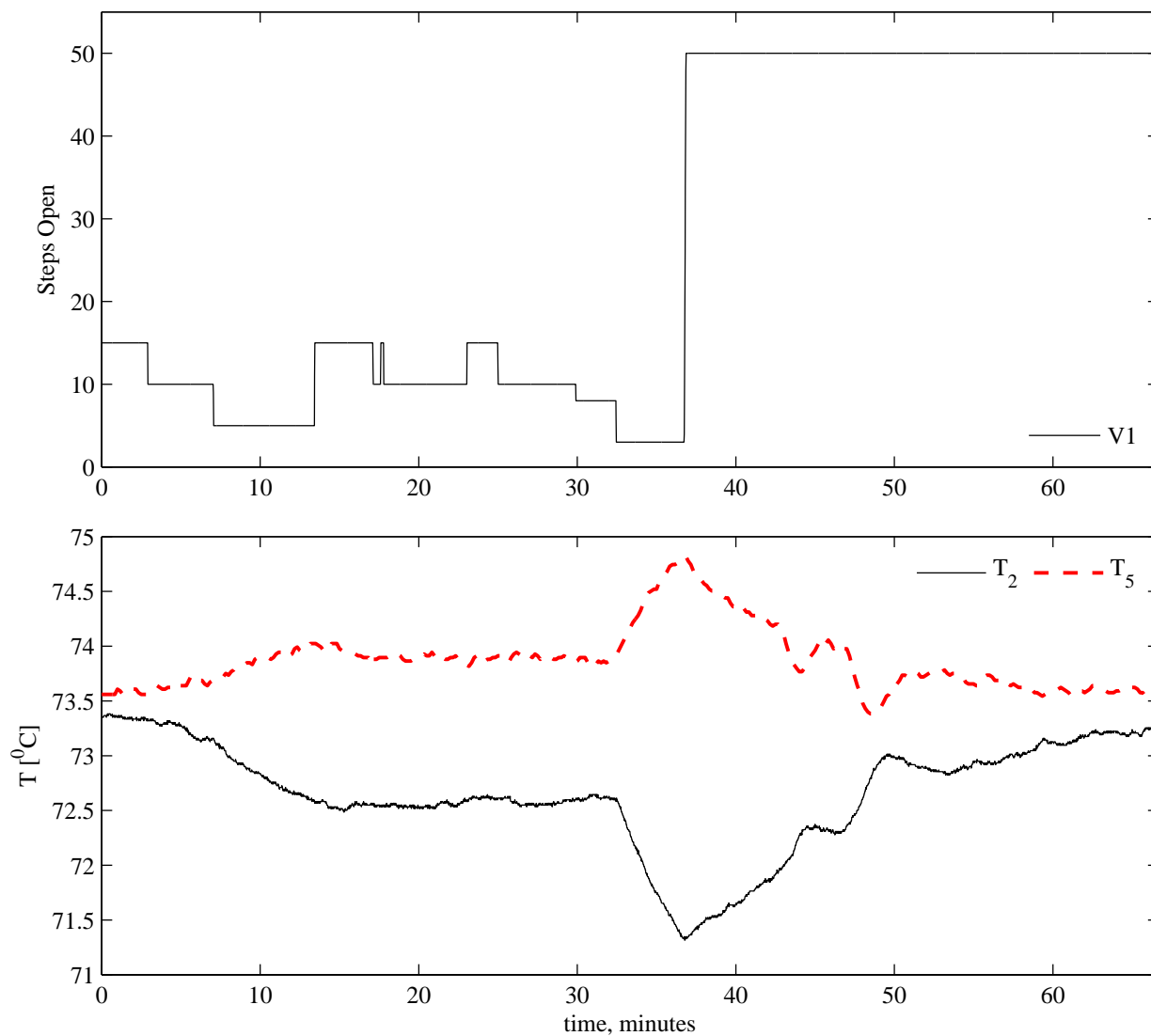


Figure 5: Experimental Run: Effect of changing the prefractionator vapor split valve, V1 with valve V2 fully open on prefractionator (T_2) and main column (T_2) temperatures.

We can conclude from this experiment that only the first 10 steps of the 150 steps are really effective, so the resolution is poor and the valve opening is too large. Nevertheless, we will see that the valve is acceptable for control purposes.

Closed loop experiments: Total Reflux

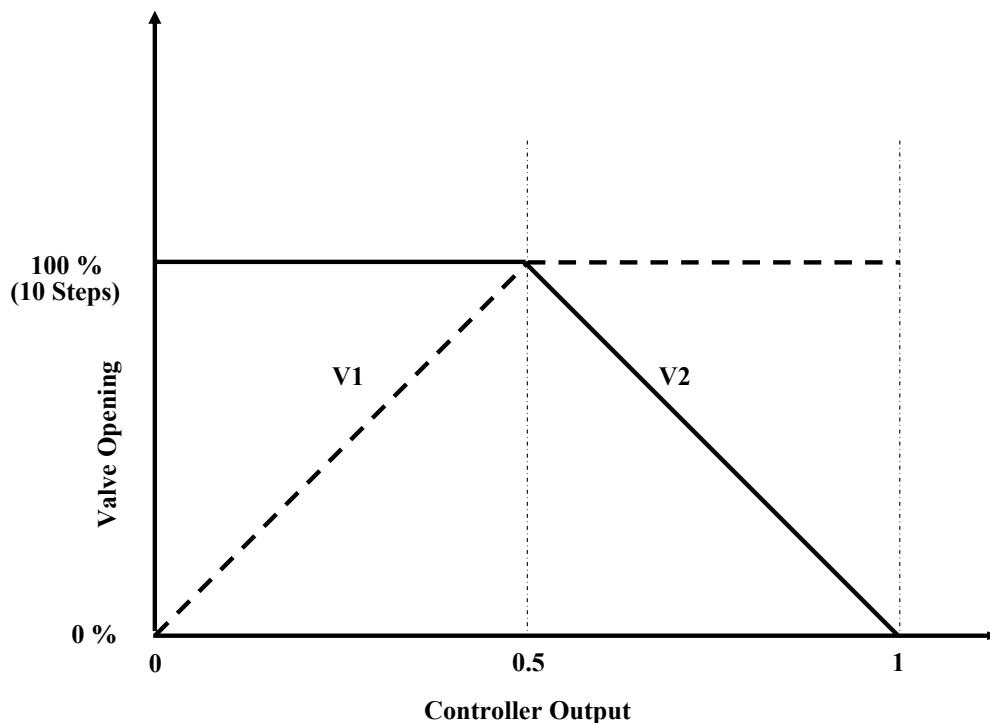


Figure 6: Split range logic (SRC) used for the vapor split controller

To study the suitability of the valve for feedback control, we performed a set of experiments under total reflux conditions using only two components, namely methanol and ethanol, with a fixed duty of 1.9 kW.

To minimize pressure drop, one of the valves should always be open. To ensure this, the valves are controlled using a split range logic as shown in Figure 6. For a controller output of 0, valve V1 is closed and valve V2 is fully open, while for a controller output of 0.5, both valves are fully open. Notice that we assume that 10 steps corresponds to a fully open valve.

The vapor split valves are used to control the temperature difference between the prefractionator and the main column, $\Delta T = T_2 - T_5$ as shown in Figure 7. The proportional-integral (PI)

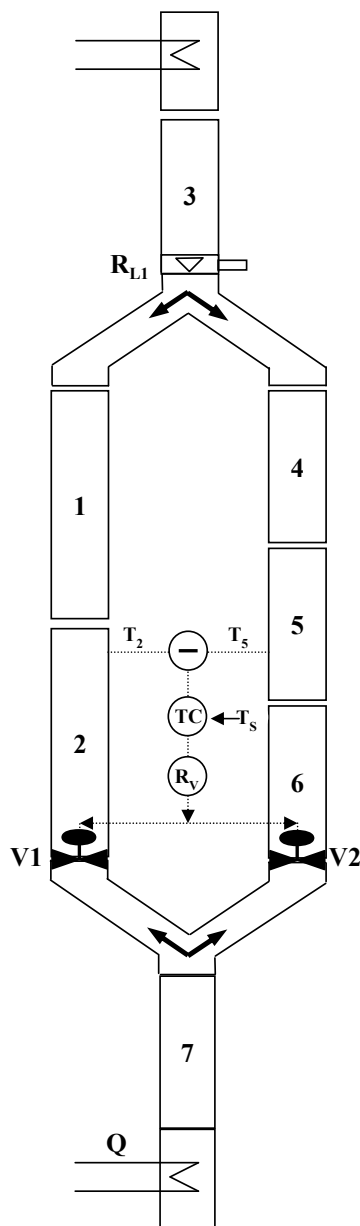


Figure 7: Control Structure used for total reflux experiments where vapor split (R_v) is used to control temperature difference between sections 2 and 5 ($T_s = T_2 - T_5$; $T_2 \equiv TP5$ and $T_5 \equiv TM8$ in Figure 3c).

controller is tuned using the SIMC rules¹² with the tuning parameter selected to be $\tau_C = 2$ minutes.

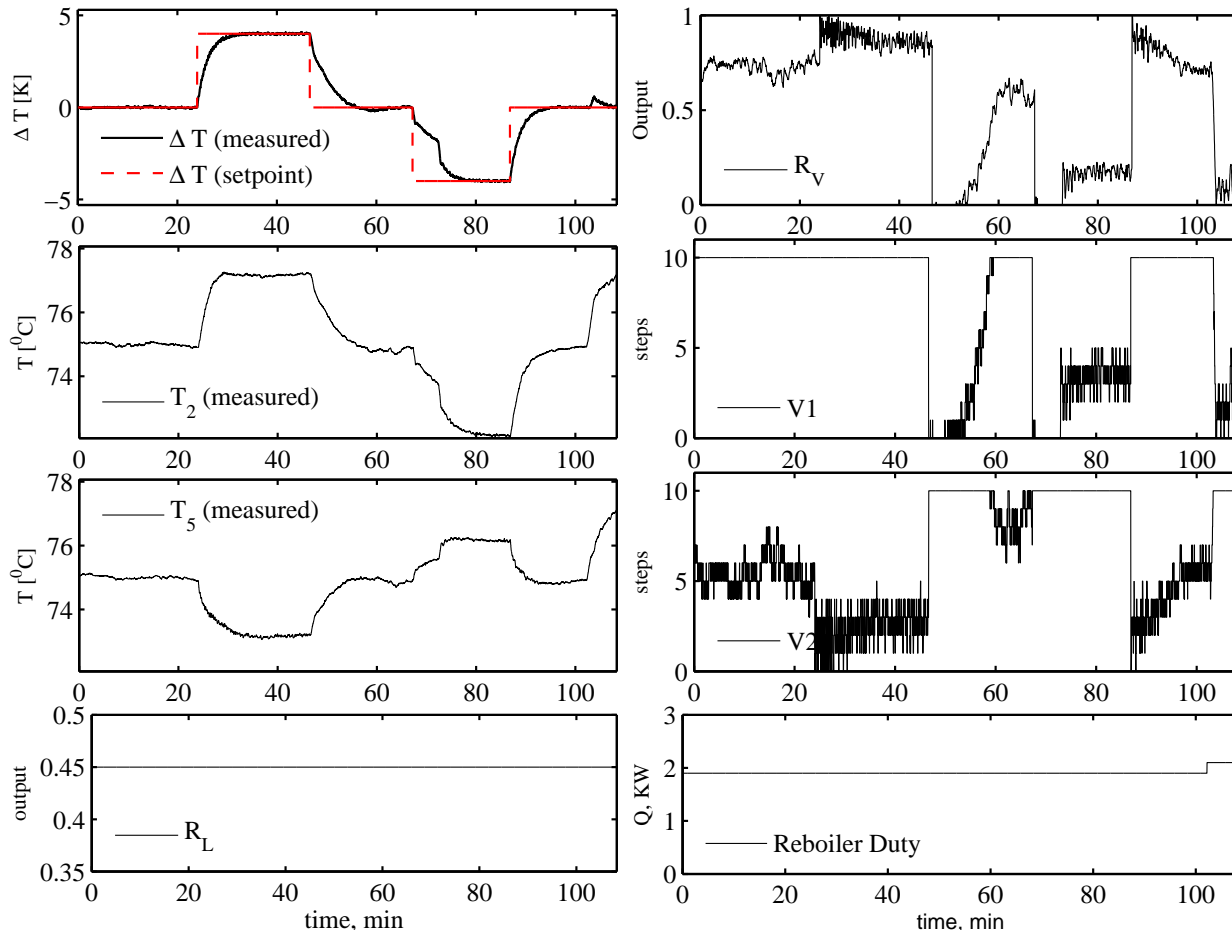


Figure 8: Closed-loop experimental run 1: total reflux conditions.

Figure 8 shows a series of setpoint changes for ΔT . We plot the controlled variable (ΔT) and the controller output (R_V in the range 0 to 1), which through the split range logic changes the valves (V_1 and V_2). The figure also shows the two individual temperatures (T_2 and T_5), the two valve opening step values (V_1 and V_2) and the values for the liquid split ratio (R_L and reboiler duty (Q)). Note that at any time at least one of the valves V_1 or V_2 is fully open.

For first 20 minutes the setpoint is unchanged at 0 K and the temperatures are steady. At 23 minutes, the setpoint for ΔT is increased to 4 K, which requires increase in the vapor flow to the prefractionator. This setpoint is reached in about 7 minutes without any overshoots. This is followed by a series of setpoint changes which can be tracked as well. At about 100 minutes, a disturbance is introduced by increasing the reboiler duty by 0.2 kW. This is shown by an increased

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difference in temperature by about 0.6 K. But the controller can bring the controlled variable back to the setpoint of 0 K. In summary, we see from Figure 8 that the vapor split valves are fully acceptable for closed-loop operation.

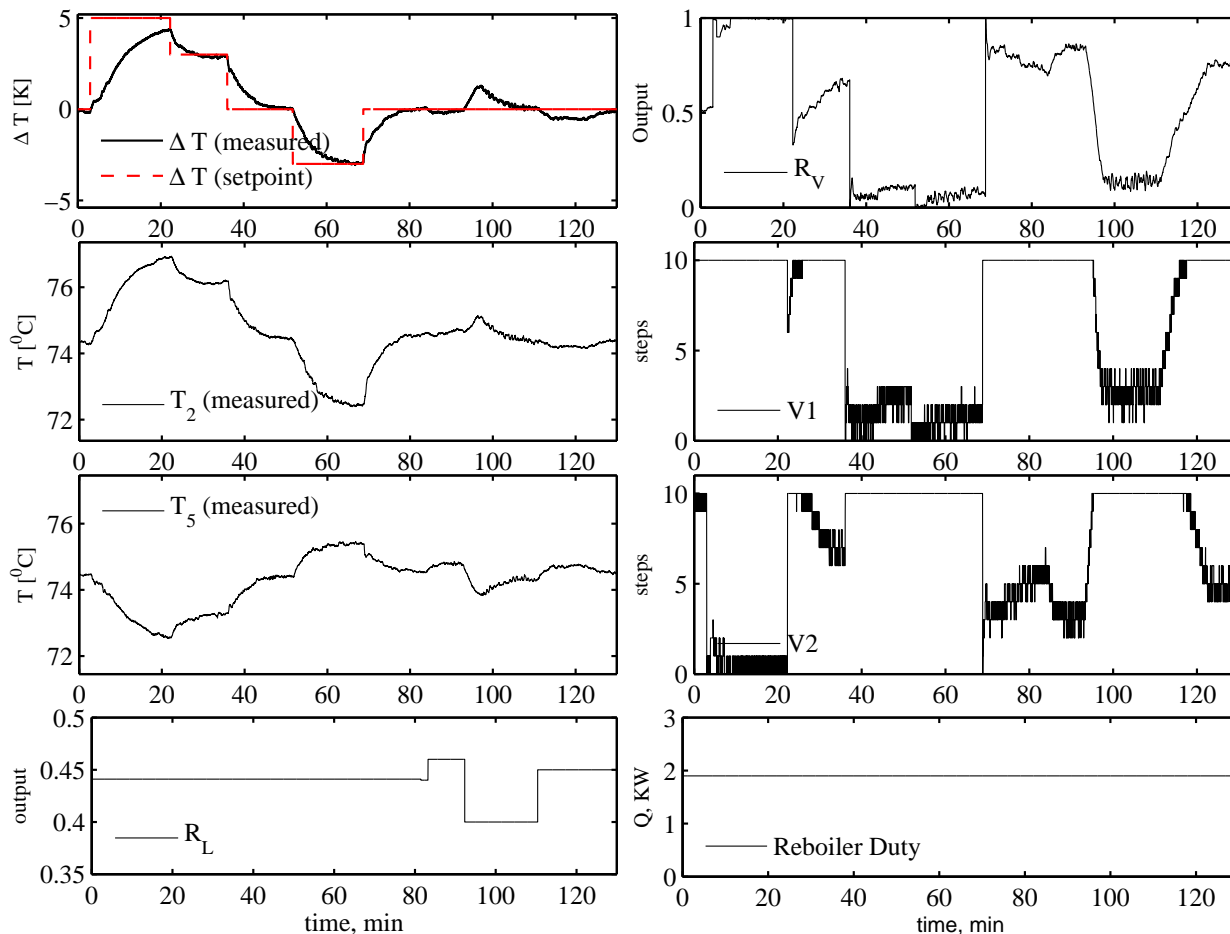


Figure 9: Closed loop experimental run 2: total reflux conditions

Figure 9 shows another experiment under more difficult conditions. With a large setpoint change for ΔT of +5 K at about 3 minutes, the output of the controller saturates and the setpoint can not be reached. The reason is probably that the valve V2 is nearly fully closed. However, when the setpoint is reduced, it can be reached. During last 30 minutes of the run, we also give disturbances by changing the output of the liquid split valve between 0.4 to 0.46. These disturbances can also be handled by the vapor split valve.

Based on these experiments, we conclude that even with rough manipulation of the vapor flow, yields good temperature control when implemented in an appropriate feedback loop.

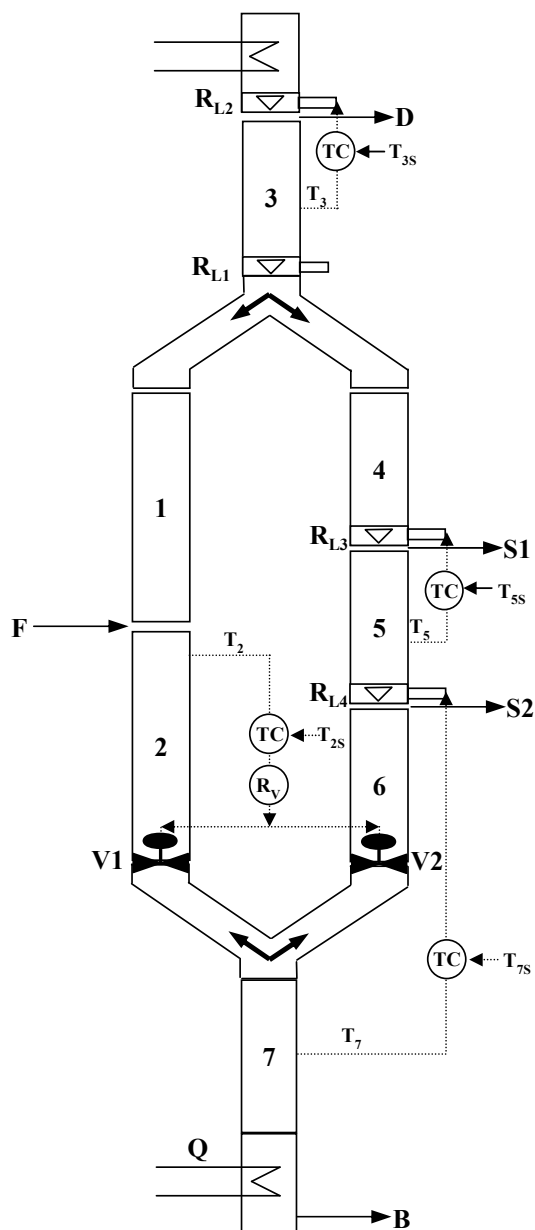


Figure 10: 4-point temperature control structure for continuous operation of Kaibel column using active vapor split (R_V) for control of prefractionator temperature (R_{L1} is kept constant, but could have been used for control for example, of a temperature in top section of prefractionator).

Closed loop results: 4 Product Kaibel Column

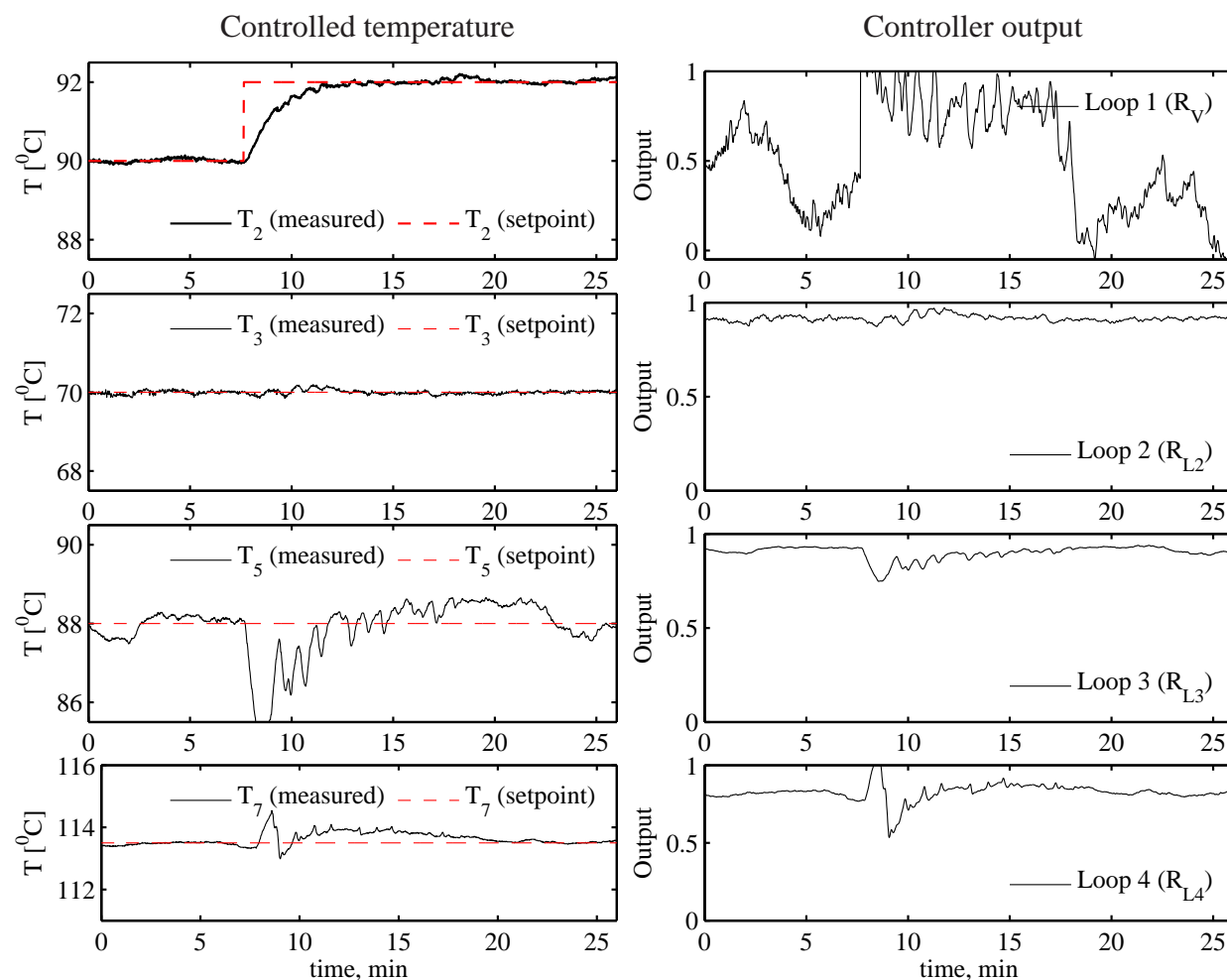


Figure 11: Closed-loop experimental run: Continuous operation of Kaibel column using 4-point temperature control with active vapor split (R_V).

Strandberg and Skogestad¹³ found in a simulation study that a 4-point temperature control scheme with one temperature controlled in the prefractionator can stabilize the column and as well as prevent “drift” of the composition profiles during operation. Correspondingly, in our previous experimental work,¹⁴ we used the liquid split (R_{L1}) to control a temperature in prefractionator (with a constant vapor split R_V).

Here, we show that the temperature in prefractionator can be controlled equally well using the vapor split R_V (with a constant liquid split, R_{L1}). Figure 10 shows the control structure where a sensitive temperature in prefractionator section 2 (T_2) is controlled using the vapor split valve. In

addition, one temperature in each of sections 3, 5 and 7 are controlled by the distillate split valve (R_{L2}), upper side product split valve (R_{L3}) and lower side product split valve (R_{L4}), respectively. The details of the loop pairing is given in Table 1. The additional degree of freedom, i.e., the liquid split is not used in this stabilizing layer and is available for optimizing objective such as to reduce energy consumption for a required purity specification.

Table 1: Four-point temperature regulatory control structure for Kaibel column ^{a,b,c,d}

Control loop	Manipulated Variable	Controlled Variable
Loop 1	Vapor split valve (R_V)	temperature in section 2 (T_2)
Loop 2	Distillate split valve (R_{L2})	temperature in section 3 (T_3)
Loop 3	Upper side product split valve (R_{L3})	temperature in section 5 (T_5)
Loop 4	Lower side product split valve (R_{L4})	temperature in section 7 (T_7)

^a The ratio R_{L1} is fixed and is not used in the control structure.

^b Controlled variables are temperatures as shown in figures Figure 3c: $T_2 = TP5$, $T_3 = TM3$, $T_5 = TM8$ and $T_7 = TM14$.

^c Definitions of swinging funnel ratios:

$$R_{L1} = \frac{L_1}{L_3}, R_{L2} = \frac{L_3}{L_3+D}, R_{L3} = \frac{L_5}{L_5+S1}, R_{L4} = \frac{L_6}{L_6+S2}$$

where, L_1 , L_3 , L_5 and L_6 are liquid flows in sections 1, 3, 5 and 6, respectively. $S1$ and $S2$ are side product flow rates (see Figure 3).

$$R_V = \frac{V_2}{V_7} = \frac{V_2}{V_2+V_6}$$

where, V_2 , V_6 and V_7 are vapor flows in sections 2, 6 and 7, respectively (see Figure 3).

An experimental run is shown in Figure 11. At about 8 minutes, the setpoint for the temperature T_2 controlled by the vapor split valve (Loop 1) is changed from 90°C to 92°C. This setpoint change can be handled well and the temperature settles in less that 5 minutes. The other temperature loops show some deviation due to interactions, however, all the temperatures are brought back to their setpoints in about 20 minutes.

There is a large scope for improving the vapor split valve and suggesting alternative designs. Nevertheless, even with our prototype valve with poor resolution, experimental results show that the vapor split can be manipulated effectively in feedback mode to achieve more energy efficient operation of dividing-wall columns.

Discussion

Feedback implementation of vapor split

We here argue in favor of feedback control using vapor split valves to set “optimum vapor split” between prefractionator and the main column in dividing-wall columns. There are two advantages of using the vapor split valve for using vapor split valve in feedback loop. First, the vapor split valve is a very fast handle since the vapor dynamics are much faster than the liquid. Further, there is no need to precisely measure the vapor split, the feedback action can “drive” the vapor split to its optimum value by tracking some controlled-variable like a composition or a temperature (Figure 10).

The additional degree of freedom, i.e., the more precise liquid split can be used for optimizing objective such as to reduce energy consumption for a required purity specification.

Conclusions

The experimental results show that the vapor split can be used as a degree of freedom during operation of integrated columns, such as, Petlyuk, Kaibel and dividing-wall columns. In particular, it is useful for closed-loop temperature or composition control, where deficiencies and inaccuracy in the vapor valves are corrected for by use of the feedback.

Acknowledgement

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