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Kaibel Distillation Column

Analysis of Behaviour based on Simulations and Lab Column Experiments

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PREFACE

First of all, I’d like to thank my parents for giving me the opportunity to do this internship abroad, in Norway.

Next, I’d like to thank Professor Sigurd Skogestad of the Department of Chemical Enigneering from the NTNU for his permit to do my internship in his Process Control group. People from this group I would like to thank are Deeptanshu Dwivedi, with whom I collaborated during these four months and Dr. Ivar Halvorsen from SINTEF who helped my in understading the basics of the Kaibel distillation column and who was always around to help me with problems. I also like to thank Mehdi Panahi, with whom Deeptanshu and I shared the office.

I also want to thank Guy Van Baelen as my supervisor in Belgium for reading and correcting my paper. I like to thank Barbara De Weerdt as well for checking my paper at spelling and grammar errors.

Of course, I cannot forget Kalle Lambrechts, my classmate who also did his internship in Trondheim. Together we have done and seen many great things.

Last but not least, I thank my flatmates in Moholt: Alessandro, Frode and Massimiliano; together with all the people I met in Trondheim for having a great time with them.

SUMMARY

The purpose of this thesis is to analyze the behaviour of the Kaibel distillation column. This is done by simulations and experimental runs in the lab. This research contributes to the composition of a guide for such columns, in which problems and their possible solutions are described.

The first part of this thesis is the theoretical part. In this part, the distillation and necessary diagrams are discussed, as well as some theory about controllers. The second part covers the development and theoretical operation of a Kaibel distillation column. The part is followed by a large practical part, in which different experiments and simulations are carried out. The experiments deal with adjusting setpoints and determining the number of trays in the column. The simulation part goes deeper in the influence of the vapour split and its influence on the behaviour of the column. This split cannot be controlled in de setup and is thus studied with the aid of simulations.

The entire research is an exchange between simulations and experimental runs. The experimental runs deliver data to adjust the existing model. With the new model, better setpoints are retrieved. These setpoints are applied in the lab and will deliver new data.

SAMENVATTING

Dit eindwerk heeft als doel de werking van de Kaibel destillatiekolom te analyseren. Dit gebeurt aan de hand van simulaties en experimenten in het labo. Aan de hand van dit onderzoek kan een soort handleiding voor dergelijke kolommen worden opgesteld, waarin mogelijke problemen worden opschreven samen met hun oplossingen.

Het eerste deel van dit eindwerk is het theoretisch gedeelte. Hierin worden de destillatie en de nodige diagramma besproken, alsook theorie over controllers. Het tweede deel beschrijft de ontwikkeling en theoretische werking van de Kaibel destillatiekolom. Dit deel wordt gevolg door een groot praktisch gedeelte, waarin verschillende experimenten en simulaties worden uitgewerkt. De experimenten gaan onder andere over het afstellen van setpoints en het bepalen van het aantal schotels in de kolom. In het simulatiegedeelte wordt dieper ingegaan op de invloed van de vapour split op de werking van de kolom. Deze is in de opstelling moeilijk controleerbaar en wordt daarom bestudeerd via simulaties.

Het volledig onderzoek is een wisselwerking tussen de simulaties en het experimenteel werk. Met de data uit de experimenten wordt het bestaande model aangepast. Met dit aangepaste model wordt vervolgens verder gezocht naar betere setpoints. Deze worden dan weer toegepast tijdens de experimenten en zo wordt telkens nieuwe data verkregen.

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LIST OF ABBREVIATIONS AND SYMBOLS

(X-U)	disturbance in controller
A	volatility
D	distillate flow
DWC	divided wall column
F	feedflow
H.E.T.P.	Height Equivalent Theoretical Plate
HPLC	High Performance Liquid Chromatography
K	number of tray
K_c	critical gain
K_d	derivative controller gain
K_i	integral controller gain
K_p	proportional controller gain
NTNU	Norges teknisk-naturvitenskapelige universitet
P	plant gain
p	pressure
R	refluxratio
R_L	liquid split ratio
R_v	vapour split ratio
S_1	sidestream 1
S_2	sidestream 2
T_d	derivative time
T_i	integral time
V	vapour flow
VLE	vapour-liquid equilibrium
W	bottoms flow
x_D	fraction of lightest component in distillate
x_i	moleconcentration
x_R	fraction of lightest component in bottoms
Y_d	steady state output value of a derivative controller
Y_i	steady state output value of a integral controller
Y_p	steady state output value of a proportional controller
α	vapour bypass
β	relative volatility

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INTRODUCTION

The distillation process is a very old process to separate liquid mixtures, based on their difference in volatility. Nowadays it is the most common separation technique used in chemical plants. But this success comes with a price; the columns consume a lot of energy to heat up and cool down the mixtures and products.

In the last decades the world became aware of global warming and its causes, mainly CO_2 emission. This has accelerated the interest and research for less energy consuming processes, especially in the industry. Since the industry emits a lot of CO_2, every decrease of CO_2 emission has its effect. The best possible way to decrease the emissions is trying to reduce the amount of energy that is needed to operate the most energy demanding processes.

Since the beginning of the 20th century, many people have tried to reduce the energy that is needed to perform distillations. The first attempts were no more than drawings of systems which have never been built or applied. In the late 80s, Gerd Kaibel optimized these developments to design the Kaibel distillation column. He was also the first person who put them in practice in the industry.

These Kaibel columns are able to save up to 35% of energy, which is a major reduction. Because of this energy reduction, also the operating costs are reduced. Also the investment costs and required space to perform the separation are reduced. The most important disadvantage of these columns is that they are less flexible in use than normal distillation columns.

This paper tries to give a better insight in the practical operation of a Kaibel column, using computer models and data which are retrieved by doing experimental tests in the lab. It is part of larger research into Kaibel distillation columns. The aim of this research is to put together a practical guide about these columns. The guide will be a collection of possible errors that can occur and how they can be recognized in temperature profiles. This enables an optimal operation of this type of column which is necessary to achieve the energy reductions.

1 DISTILLATION

Distillation is a technique to separate liquids. This separation is based on the difference in volatility between products. A product with a low volatility will evaporate quicker than a product with a high volatility. By heating a mixture, the most volatile product will evaporate and condensate. Because one evaporation and one condensation are not sufficient, the process is repeated several times in a column. On each stage vapour will condensate and provide energy to evaporate liquid on that stage. If this process is repeated many times, high purity products can be achieved.

This chapter gives a brief introduction in the theory of distillations and explains some phenomena that are used to fit the computer model to the experimental data.

1.1 Equilibrium stage concept

Central in the distillation theory stands the equilibrium stage concept. This concept assumes that there is vapour-liquid equilibrium on each stage. It is also assumed that there is equilibrium between the liquid on a stage, the liquid that is going down to the lower stage and the vapour that rises to the next stage.

1.2 Vapour-liquid equilibrium

In order to say something about the vapour liquid equilibrium (VLE), the volatility of a product is first explained.

1.2.1 Volatility

The volatility (A) of a product is the tendency of the product to vaporize. The higher the volatility, the faster it will vaporize. The volatility is calculated by dividing the partial pressure (p) of the component by its mole concentration (x):

$$A_i = \frac{p_i}{x_i} \qquad 1.1$$

The partial pressure is the fraction of the total pressure that is contributed by a component. E.g. when two gases are locked in a container, the total pressure in the container is the sum of the two partial pressures of the gasses.

Distillations are used to separate a mixture of two or more liquids by boiling. The ease with which both components separate can be expressed as the relative volatility, β. This value is calculated by dividing the volatility of the most volatile component by the volatility of the less volatility component. When β is 1, the products can't be separated.

$$\beta = \frac{A_i}{A_j}$$

1.2

The value of β will depend on the mixing ratio of the two components.

1.2.2 Vapour-liquid equilibrium

When β is known, it is possible to plot an equilibrium line. In such a chart, the concentration of the lightest fraction in the liquid (x) is plotted against the concentration of the lightest fraction in the vapour (y). The equation for this equilibrium line is:

$$y = \frac{\beta \, . \, x}{1 + (\beta - 1) \, . \, x} \qquad 1.3$$

This is the equation for ideal mixtures. Normally there will be some deviation. The figures below show an ideal VLE and a non-ideal VLE (Figure 1).

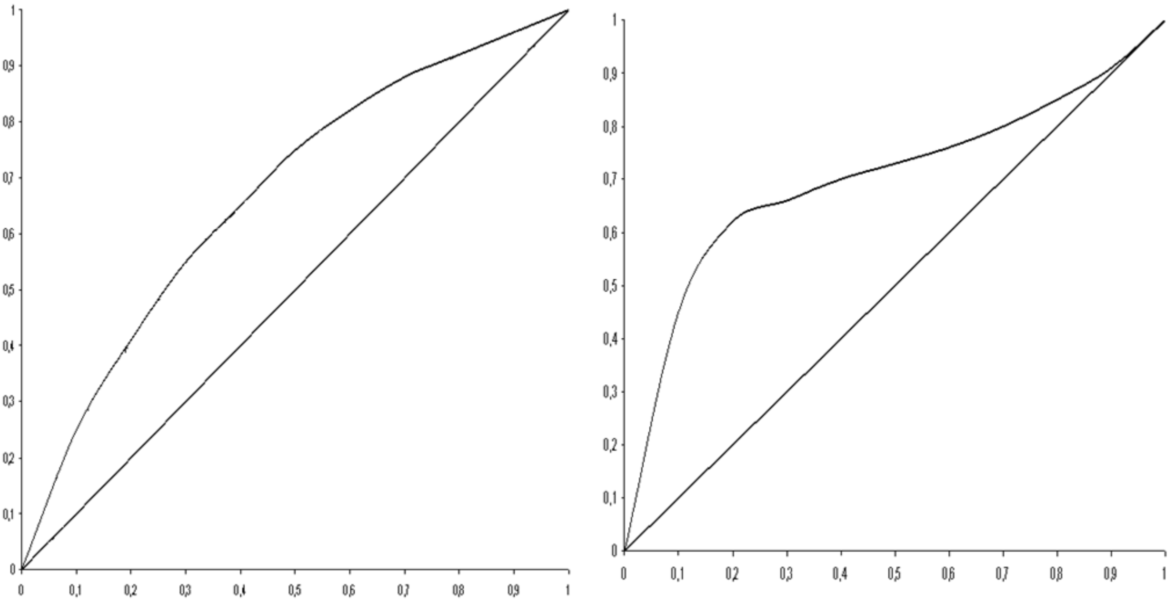


Figure 1: Vapour liquid equilibrium

1.3 Material balance on a stage

The material balance gives the relation between the amount of product on a stage, the amount that leaves the stage and the amount that enters a stage. These relations can be placed on an equilibrium diagram and are known as the operating lines. Usually two operating lines are drawn; one above the feed and one below the feed. The place where both line cross is the ideal feed stage. The equation for the upper operating line is given below. The Mc Cabe-Thiele method uses these lines together with the VLE to determine the theoretical number of trays. The equation for the upper operating line is given below:

$$y = \frac{1}{(1+R)} \, . \, x_D + \frac{R}{(1+R)} \, . \, x \qquad 1.4$$

R denotes the reflux ratio and is calculated by dividing the mass of liquid that returns to the column by the mass of liquid that is drained off from the top. x_D Is the fraction of the lightest product in the distillate and x specifies the tray.

The equation for the lower operating line is given below. V denotes the mass of vapour, L denotes the liquid that is coming down, W is the bottoms flow rate, x_r is the fraction of light product in the bottoms and k specifies the tray.

$$y = \frac{A}{V} \, . \, x \; - \; \frac{W}{V} \, . \, x_r \qquad 1.5$$

When both operating lines are plotted on the equilibrium curve, one is able to determine the number of trays which are needed to perform a separation and also the composition on a stage can be determined. This is the Mc Cabe-Thiele method.

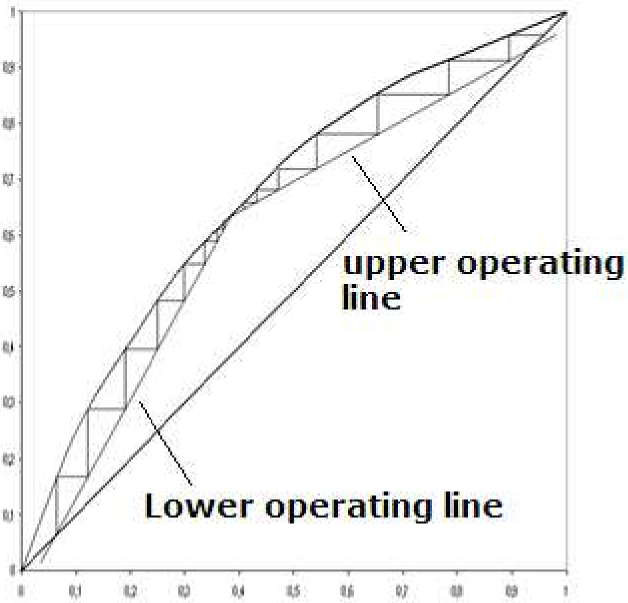


Figure 2: VLE with operating lines

1.4 Vapour bypass

Vapour bypass is a phenomenon that can be used to represent no ideal mixing on a stage. This is shown in Figure 3. The vapour bypass (α) is an efficiency parameter that describes insufficient vapour mixture at the column stages. (Kvernland, 2009) α is the fraction of vapour that will not equilibrate on the next stage, but escapes to the second stage above. Introducing this parameter keeps the number of physical stages the same, but will reduce their performance and hence lead to lower purities.

When α is introduced, the vapour flow for component i can be written as:

$$V_k \,.\, y_{i,k} = (1-\alpha)V_{k-1} \,.\, y_{i,k-1} + \alpha V_{k-2} \,.\, y_{k-2} \qquad 1.6$$

When all components i are summated, it becomes clear that the vapour is distributed to the two stages above, or that vapour from two lower stages reach stage k:

$$V_k = (1-\alpha)V_{k-1} + \alpha V_{k-2} \qquad 1.7$$

When there is no vapour bypass, α equals zero. When α differs from zero, some vapour will skip steps on its way up and this will lead to more impurity in the products.

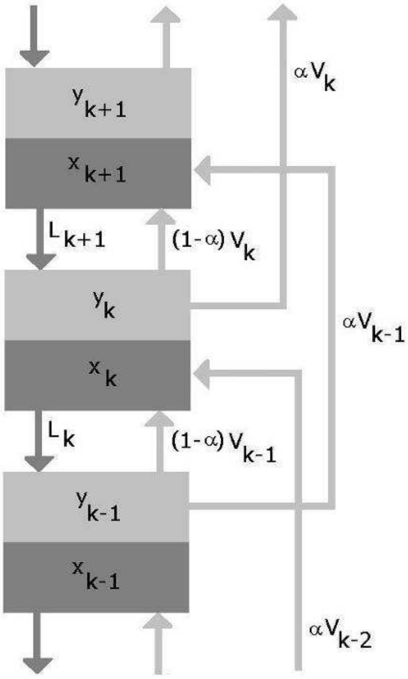


Figure 3: Vapour bypass

1.5 Minimum energy diagram

This part handles about the minimum energy which is an important factor in obtaining the suggested energy savings.

1.5.1 Definition

For a given separation, the reflux can be reduced if the number of stages is increased. When the number of stages approaches infinity, a pitch zone will occur in the column and the reflux can't be reduced any further. This is a minimum reflux that is required to perform the separation. As long as the energy is higher than needed with this minimum reflux, the separation can be performed (Halvorsen, 2003).

1.5.2 Assumptions

Minimum energy can be shown graphically on a diagram. In order to create a minimum energy diagram, some assumptions need to be made.

The vapour flow rate (V) is in this diagram used as the energy measurement. Therefore, it is necessary that the vapour flow is considered as the vapour flow generated from all reboilers. In this case, there is only one reboiler.

The assumptions are:

1. infinite number of stages;
2. constant relative volatility;
3. constant molar flow;
4. constant pressure;
5. no internal heat exchange.

The first assumption is really necessary to create the diagram; the others help to make the diagram linear.

1.5.3 Column with a three-component feed

A minimum energy diagram, or V_{min} diagram, is shown in Figure 4.

All the values in the chart are chosen randomly.

A normal distillation column has two degrees of freedom which can be represented as D/F and V/F. These two degrees are respectively the x- and the y-axis on a minimum energy diagram. D/F represents the composition of the feed and the y-axis (V/F) represents the energy, measured as vapour flow.

In order to create the diagram, the feed composition is plotted on the x-axis, beginning with the fraction of the lightest component. The composition of feed in this example is 33% A, 42% B and 25% C. The x-coordinate of point K is the fraction of A in the feed, the x-coordinate of M is the sum of fractions A and B. When fraction C is added, the sum of the fractions is 1.

Points K, L and M represent the possible separations of the 3-component mixture. Point K is the sharp separation of ABC into A and BC. The y-coordinate is representative for the energy that is needed to perform this separation. Point M represents the sharp separation of ABC in AB and C, the y-coordinate represents also the needed energy. Point L is the preferred split of ABC in AB and BC. It is not a sharp split because B is not equally divided. These points form a Minimum Energy Mountain when they are connected.

Inside this mountain, three zones can be distinguished in which no sharp separations are performed. In zone 1, the feed will be separated in AB and ABC. In zone 2, the feed is separated in ABC and ABC. In the third zone (3), the feed is separated in ABC and BC. The separations under the mountain are not sufficient since the energy is lower than the minimum energy.

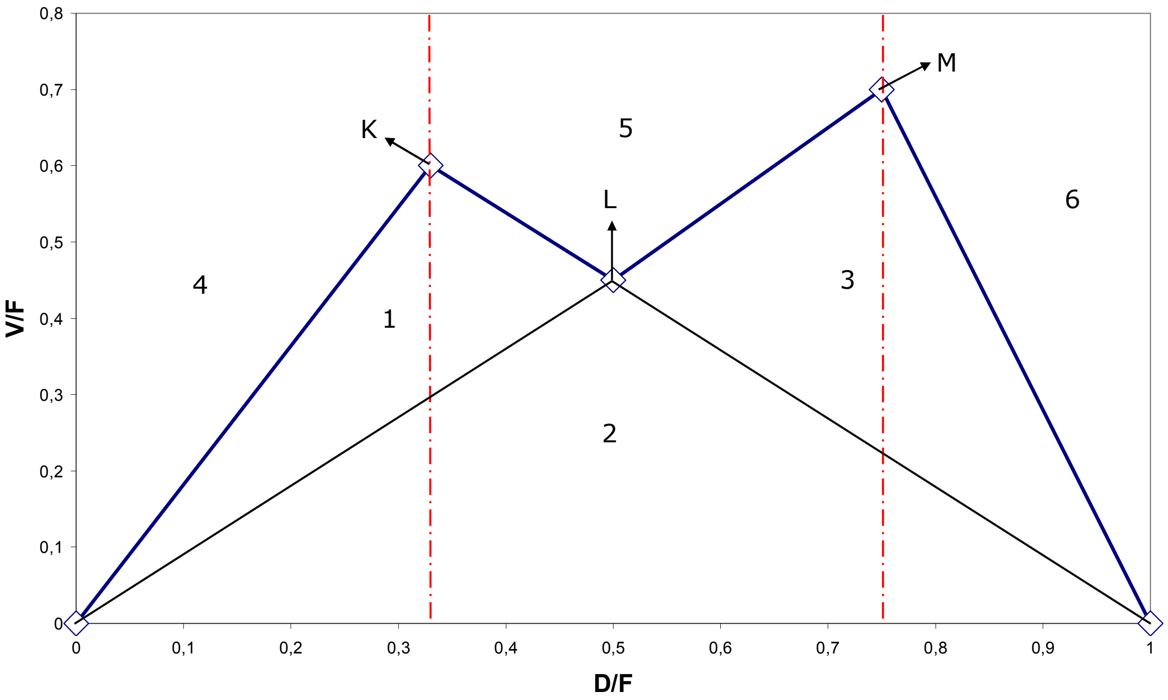


Figure 4: Minimum energy diagram for a 3-component mixture

Outside the mountain there are also three zones of non sharp separation. In zone 4, which is bordered outside the mountain by the red line, the feed is separated in A and ABC. Outside the mountain, between the two red lines, the feed is separated in AB and BC. This is zone 5. In zone six, the feed is separated in ABC and C. In these zones, the separations are done properly, but with more energy than the minimum energy.

All the points and zones are put in Table 1.

Since the height of the peaks represents the needed energy, the highest peak is the energy that is required to perform the most difficult separation.

Table 1: Splits in the 3-component minimum energy diagram

point/zone	destillate	bottom	split type
K	A	BC	sharp
L	AB	BC	preferred
M	AB	C	sharp
1	AB	ABC	non sharp
2	ABC	ABC	non sharp
3	ABC	BC	non sharp
4	A	ABC	non sharp
5	AB	BC	non sharp
6	ABC	C	non sharp

1.5.4 Minimum energy for the separation of a four-component mixture

A similar diagram can be made for the separation of a four component mixture. This is shown in Figure 5.

The feed in this diagram consist of 25% A, 25% B, 25% C and 25% D. The energy values are chosen randomly.

The separations are:

- Point J: separation in A and BCD
- Point K: separation in AB and BCD
- Point L: separation in AB and CD
- Point M: separation in ABC and CD
- Point N: separation in ABC and D
- Point O: preferred A/D split

The non sharp separations are not discussed because there are many areas that can be formed under the energy mountain.

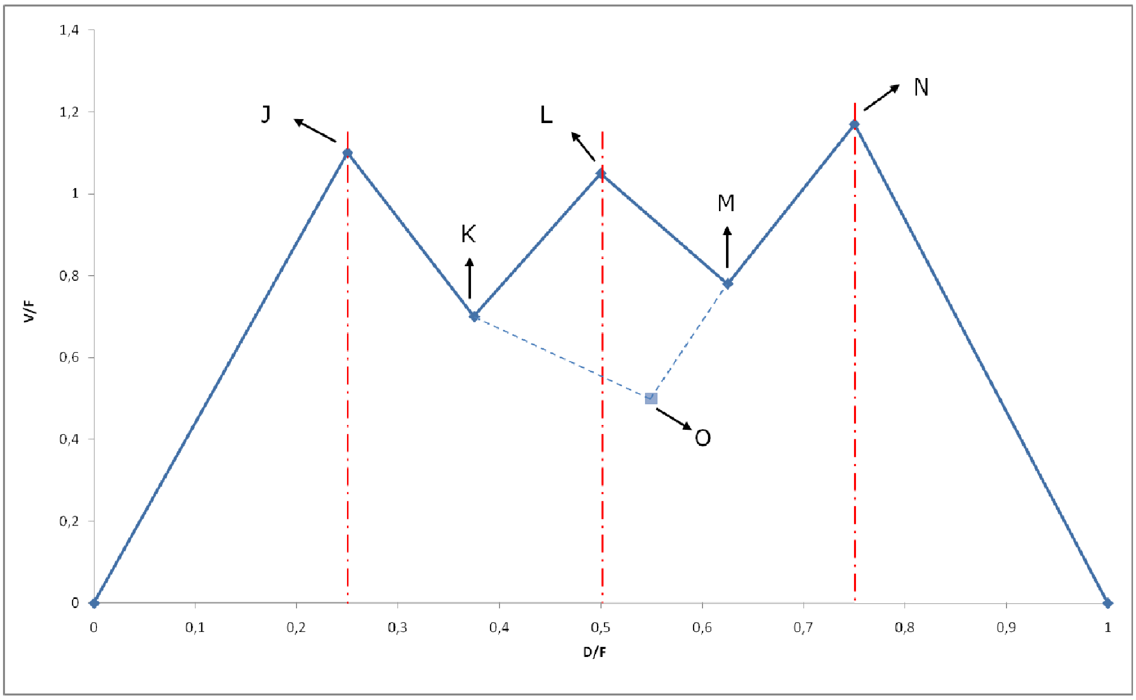


Figure 5: Minimum energy diagram for a 4-component mixture

2 CONTROLLERS

The control of a process is necessary to maintain a certain quality of the product and to run the process within the best possible conditions. This will make the process less energy consuming with high efficiency. The basics of controllers are explained in this chapter.

2.1 Terms in process control

2.1.1 Controller values

There are three important values in process control. The first one is the measured value or the input signal (X). This value is measured in the process and is sent to the controller. In the controller, this value is compared to the desired value, or setpoint (U). The difference between X and U is calculated and the controller will sent a corresponding signal to the process. This signal is presented as ‘Y’ and is the output signal of the controller. The reaction of the controller on a disturbance depends on the type of controller. This is discussed later. A disturbance can be defined as (X-U) or e.

2.1.2 Process values

Each process has different characteristics and will thus react differently. These characteristics can be defined, using some values (Figure 6). A first value is the effective time delay (θ). This is the time between a change in the setpoint and the first change in the measured value. The effective time delay is not always present e.g. when you open a tap at home, water will immediately flow out. It is also possible that the time delay is so small that it barely can be seen.

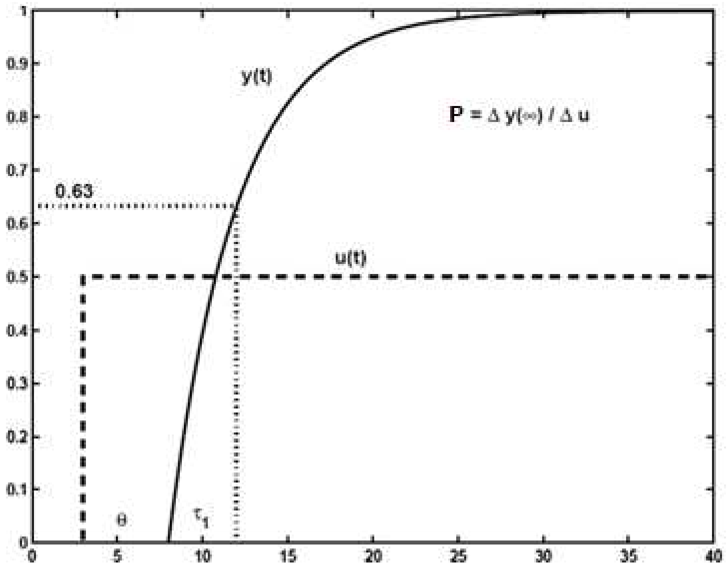


Figure 6: Process values

A second value which defines a process is the dominant time constant (τ_1). This value is the time that the process needs to reach 63% of the new equilibrium value when the setpoint is changed. The process is assumed to reach the new equilibrium at the time that equalizes 5τ.

Another process depending value is the plant gain, P. In the figure this is noted as k. This value represents the process intensification and is calculated by dividing ΔY by ΔU. If this value is smaller than 1, this means that the process weakens a setpoint change.

A large change in the setpoint will cause only a small change in the process. When k is bigger than one, the process intensifies a setpoint change. A small change in the setpoint will have a large effect on the process. If P = 1, the process will react with the same magnitude as the change in the setpoint e.g. if the U increases with one unit, Y will also increase with one unit.

2.2 Different types of controllers

As mentioned before, a controller will react to a disturbance according to its programmed function. There are three basic functions which will be discussed first and then a combination of these functions is discussed.

2.2.1 Proportional controller

A proportional controller will sent an output signal that is proportional to the disturbance. If a step change is made in the setpoint, the output value will also make a step change. The output value can be calculated:

$$Y = K_p \cdot (X - U) + Y_p \qquad 2.1$$

K_P is the controller gain. This gain is similar to the plant gain and is the value that represents the intensification of the controller. (X-U) is the disturbance and Y_p is the steady state output value of the controller. A steady state error also occurs when a P-controller is used. The controller will not be able to restore the old equilibrium but will create a new equilibrium. The difference between those two is the steady state error.

2.2.2 Integral controller

An integral controller will integrate the disturbance. The output value will change with a certain speed, which is a function of the input signal. If a step change is made in the input, the output will change with a constant speed. If the input value increases with a constant speed, the output will change with increasing speed. This is shown in the equation:

$$Y = K_I \int_0^t (X - U) \cdot dt + Y_i \qquad 2.2$$

K_I is the integration constant and is the controller gain. (X-U) is the occurring disturbance and Y_i is the steady state output value. When a proportional and integral controller are combined, the integration time (Ti) or reset time can be determined. This is the time that the integral function needs to make the same change in the output as a proportional controller would do with the same disturbance (see Figure 7).

2.2.3 Derivative controller

The size of the output signal is proportional to the change of speed of the changing input value. If a step change is made in the input, the output will peak and return immediately to its steady state value. If the input changes with a constant speed, the output will make a step change and remain at a certain level as long as the input keeps changing with the same speed. The output value can be calculated with next equation:

$$Y = K_D \cdot \frac{d(X - U)}{dt} + Y_d \qquad 2.3$$

K_D is the differentiation factor, or the differential gain. Just as an integral controller has an integral time, a derivative controller has a derivative time (Td). This is the time which a proportional controller needs to make the same change in output as a derivative function does. This is shown in Figure 8.

2.2.4 PID-controller

A PID-controller is a combination of a proportional, an integral and a derivative controller. They are linked parallel to each other, so they do their thing individually and the results are counted up to one output signal. The output value can be calculated by adding equations 2.1, 2.2 and 2.3. This results in:

$$Y = K_P \cdot (X - U) + K_P \cdot K_I \int_0^t (X - U)dt + K_P \cdot K_D \cdot \frac{d(X - U)}{dt} + Y_p + Y_i + Y_d \qquad 2.4$$

Each part of the controller contributes to achieve an optimal control. The proportional part, which can be tuned by using to proportional gain, stands for a fast response. A larger proportional gain means a faster response, but when the gain is too large oscillation will occur. The integral function is able to eliminate steady state errors. The catch is that it creates an overshoot and some oscillation around the new setpoint will occur before the process stabilizes. The derivative gain is capable of decreasing the overshoot that is caused by the integral gain, but this also means that the total controller will react slower to a disturbance.

Precise tuning of a PID-controller is difficult because the behaviour of the controller depends on K_P, K_I and K_D. These three parameters influence each other and that makes it even more difficult to tune the controller. In most cases a PI-controller is sufficient; the D-function is then turned off.

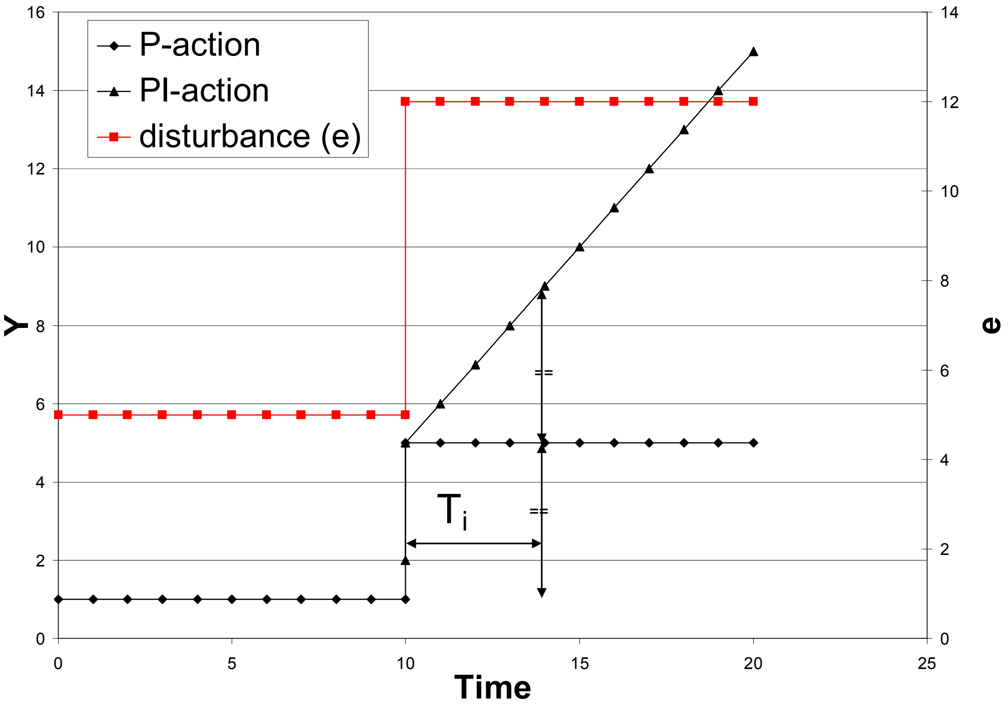


Figure 7: Integration time

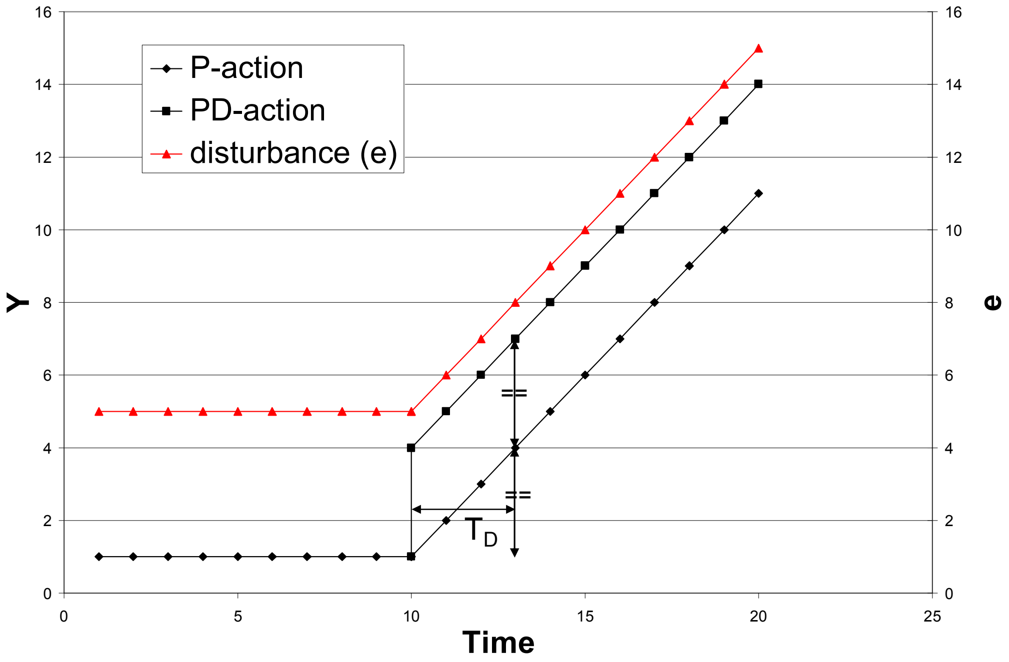


Figure 8: Derivative time

2.3 Loop tuning

The settings of a PID-controller must be chosen correctly. If they are not correct, the process can start to oscillate and this can cause saturation of the product or damage to the plant. The correct settings of a controller can be found by tuning. This can be done in several ways.

2.3.1 Manual tuning

This form of tuning is mostly used if a system needs to stay online and is done by turning the integral and derivative function off. Then, the proportional gain is set on a high value and decreased until it reacts sufficiently. After that, the integral and proportional gains are tuned. This method can be simplified using some tuning rules. This method requires an experienced operator but can not be used when the process is not allowed to oscillate.

2.3.2 Use of tuning rules

Many tuning rules have been developed. The best known are probably the Ziegler-Nichols tuning rules. They are based on the critical gain of a process. This critical gain can be found by adjusting the proportional gain when the integral and derivative functions are turned off. The proportional gain is increased until the process starts to oscillate; this value is the critical gain (K_c). This gain is then used to set the proportional, the integral and the derivative gain.

Another set of rules are the SIMC –PID tuning rules (Skogestad, Simple analytic rules for model reduction and PID controller tuning, 2008). These rules are also based on the critical gain, but the gain is calculated from the effective time delay, the oscillation period, the plant gain and the dominant time constant. These rules are very effective to apply when a process is online and is oscillating. With some calculations the right settings for the controller can be found immediately.

2.3.3 Software

A last possibility to tune a controller is the use of software. The process is put in a model on a computer and the best settings for the controller can be found by simulating the process. This method is mostly used when a new process is developed and started.

3 DIVIDED WALL COLUMN

This chapter describes the development of the divided wall columns.

3.1 Separation of multi-component mixtures

When a mixture of *n* components is separated, this requires *n-1* distillation columns (Kaibel, 1987). If four components need to be separated, three distillation columns are needed to perform the separation. The installation- and operation costs for three columns are high. To reduce the investment costs, an arrangement can be made which consists of a main column with side columns attached to it. In such an arrangement, the bottom product of the first column is sent to the second column, the bottom product of the second is sent to the third etc. Each column saves either on a reboiler or a condenser, but the number of columns is not reduced ((Kaibel, 1987). A disadvantage of these first arrangements was that only the bottom was connected to the next column. This gave impure side streams.

3.2 Development of combined columns

3.2.1 Arrangement of Brugma

A solution for this problem was given in 1937 by Antoine J. Brugma (Brugma, 1937). He presented a thermally coupled column to separate an eight-component mixture. This arrangement is shown in Figure 9. This arrangement consists of a main column with a side column and a second side column, which is attached to the first side column. Thermally coupled, or heat integrated means that the heat from inside the system is used to perfom vapourization and condensation inside the column. Vapour from the first column will be used to vapourize a part of the liquid in the main column. Liquid coming from the main column will condensate some of the vapour in the prefractionator. The bottom of the prefractionator is heated with the vapour from the main column.

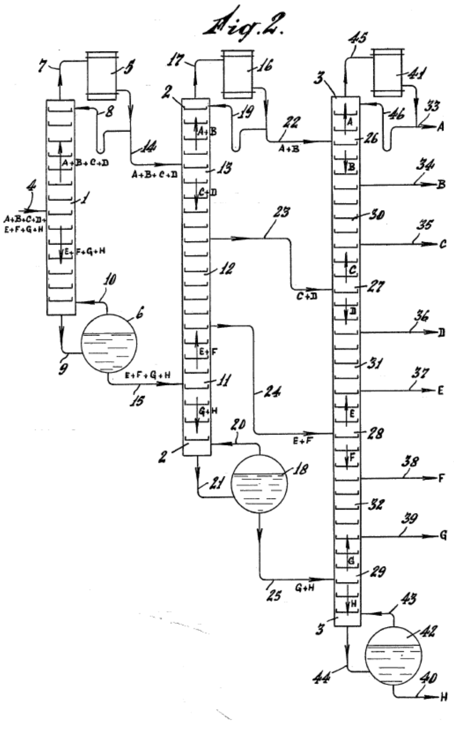


Figure 9: arrangement of Brugma

3.2.2 Petlyuk arrangement

The Brugma arrangement was complex and there is no indication that it was used on industrial scale (Kaibel, 1987). In 1965, Petlyuk made an arrangement to separate a 3-component mixture (Pletyuk, Platonv, & Slavinskii, 1965). This arrangement consists of 2 columns, which are placed above each other and a side column. The feed is introduced in the side column. The heavy components leave through the bottom in the lowest column, which has a reboiler. The light components of the feed leave through the top to the upper column, which has a condenser in the top. The two main columns are connected with each other. Vapour from the lowest column enters the upper column and liquid from the upper column enters the lower column. This setup has potential energy savings from 20 up to 50% compared to a conventional distillation setup to separate a 3-component mixture. Figure 10 is a sketch of this arrangement.

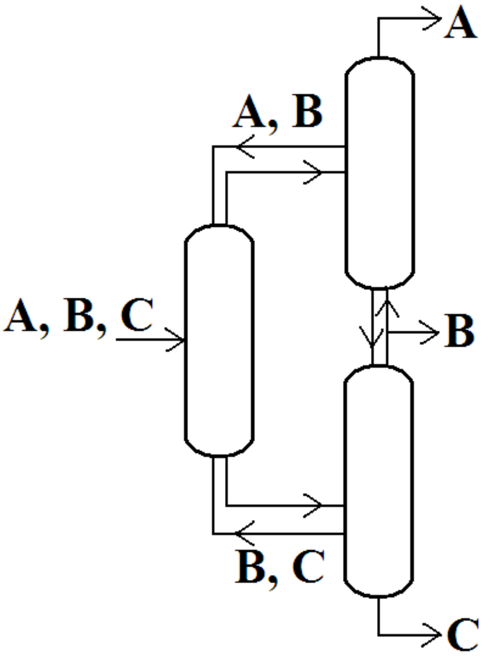


Figure 10: Petlyuk arrangement

3.2.3 Columns with vertical partition

The Petlyuk model can be simplified by using a vertical partition inside a column. This vertical partition had already been introduced by Wright in 1946 (Wright, 1949), but has been further developed by Gerd Kaibel (Kaibel, 1987) for BASF. He was also the first one to apply the column for industrial use at BASF. Such a column is also known as a divided wall column (DWC) "The simplest example of application of a distillation column with a vertical partition is the separation of a 3-compontent mixture. The vertical partition, which in the simplest case consists of a metal sheet, forms two separate parts in the centre section of the column, above and below the inlet and the outlet." (Kaibel, 1987) In terms of thermodynamics a Petlyuk arrangement and a DWC are equal.

The separation in the column creates two sections: the feed section and the discharge section. The feed enters the feed section of the column where it separates in two streams. The most volatile product leaves this section, going upwards. The heavy components leave the feed section, going downwards. The medium boiling component leaves the feed section in both directions. The only important matter is that no heavy components leave at the top and no light components leave at the bottom (Kaibel, 1987). With the use of one wall a mixture of 4 products can be separated, using only one column and one reboiler. It is also possible to use more vertical partitions, as shown in Figure 11, so mixtures with more than four components can be separated. Practically, the number of fractions is limited because more fractions need more theoretical trays to separate. When more theoretical trays are used, the column will be higher, this creates a larger pressure drop and this causes undesirably high temperatures in the reboiler (Kaibel, 1987).

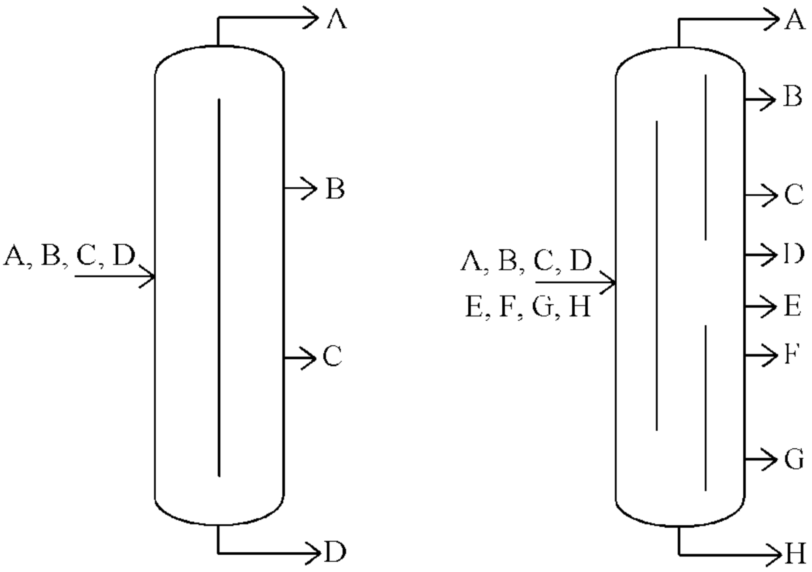


Figure 11: Divided wall columns

3.3 Benefits and disadvantages of DWCs

As mentioned before, DWCs are able to separate a multi-component mixture using only one column. This reduces the capital costs and the physical space that is required to perform the separation. This is possible because the separation can take place in one single shell instead of in multiple shells. Most important is probably the energy saving that can be done. A DWC uses only one reboiler and condenser and is thus able to save up to 35% of energy (Kaibel, 1987). This reduction of energy decreases the exhaust of CO_2. Because the industry stands for a large amount of the exhaust, a little energy saving in industry could make a big difference in the total emission (Kvernland, 2009).

A disadvantage is that it is more difficult to operate the column. There are more parameters than a conventional column has and the energy savings are only possible if the column is operated well.

Another disadvantage is that the columns are higher than conventional columns because, technically, they are columns stacked on top of each other. Because the separations take place in one shell it is also not possible to use different pressures, the whole column operates at the same pressure. This makes them less flexible in use.

4 COLUMN DYNAMICS

This chapter describes the optimal operation of the pilot plant at the NTNU (Norges teknisk-naturvitenskapelige universitet), but it can be applied for any divided wall column which separates a four component mixture.

4.1 Prefractionator

The heated feed (ABCD) enters the column in the prefractionator, creating a rectification (1) and a stripping section (2) (see Figure 12). The prefractionator works optimal if it is able to separate the feed (ABCD) sharp in AB and CD. The minimum energy that is required for this performance can be found in the V_{min}-diagram. The amount of energy that is needed is lower than the minimum energy to perform the most difficult separation. The vapour is divided between the prefractionator and the main column by the vapour split. Hence it is possible to sent the right amount of vapour in the prefractionator to get the optimal separation.

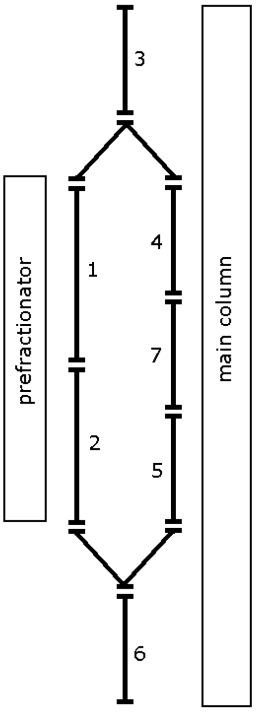


Figure 12: Sections in the Kaibel column

4.2 Main column

The main column can be divided into five parts. The junction parts are not counted since they don't contain packing. The vapours that leave the prefractionator enter the main column between sections 3 and 4. In these two sections, A and B are separated from each other. The minimum energy that is needed to do this separation can be derived from the V_{min}-diagram.

The liquid that leaves the prefractionator enters the main column between sections 5 and 6. In these two sections, C and D are separated. The minimum energy can also be derived from the V_{min}-diagram.

The seventh section is a special section. As it is placed in the main column, one would expect that it separates B and C. But this separation should not be performed here, since it was already done in the prefractionator. In the most ideal case, no mass transportation takes place here. The only thing that should be transported through this section is energy to the upper part of the column. In theory, this part could be replaced by a heat exchanger that heats up liquid from the upper section with the vapour from the lower section. This is practically not applied because some impurities will always occur in the different sections. These impurities need to be able to reach the right section through section number seven.

Each component has an optimal way to travel through the column. For components A and B, this is leaving through the top of the prefractionator (Figure 13). For components C and D, the most ideal way is through the bottom of the prefractionator. Unfortunately it happens that a part of the components doesn't take the ideal way (Figure 14). It is possible that some B leaves through the bottom of the prefractionator and climbs back up in the main column.

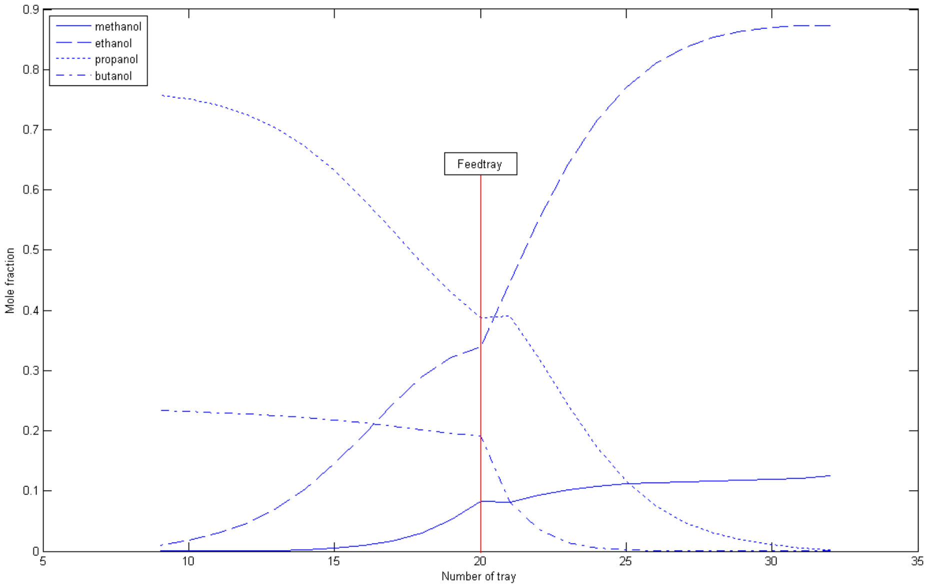


Figure 13: Optimal separation in the prefractionator

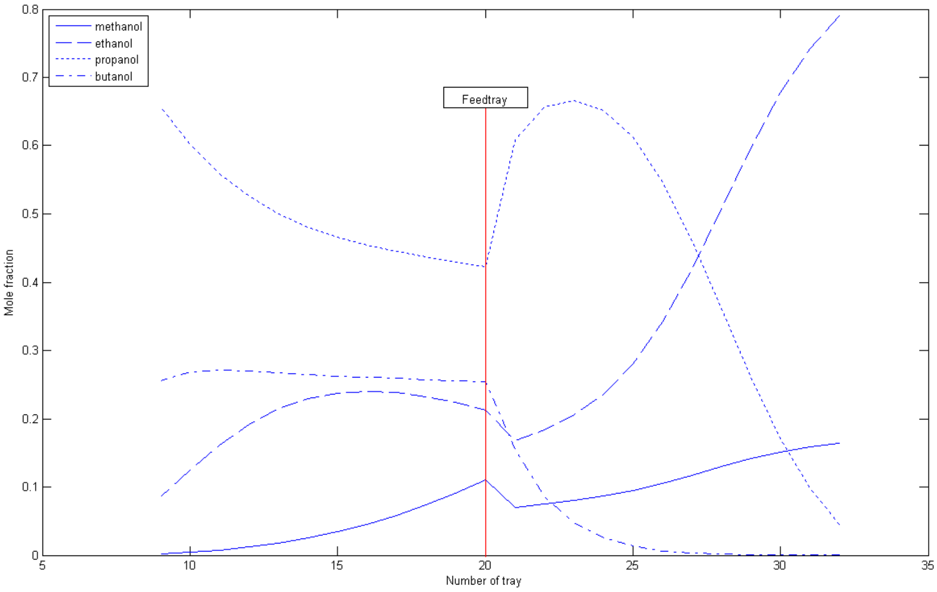
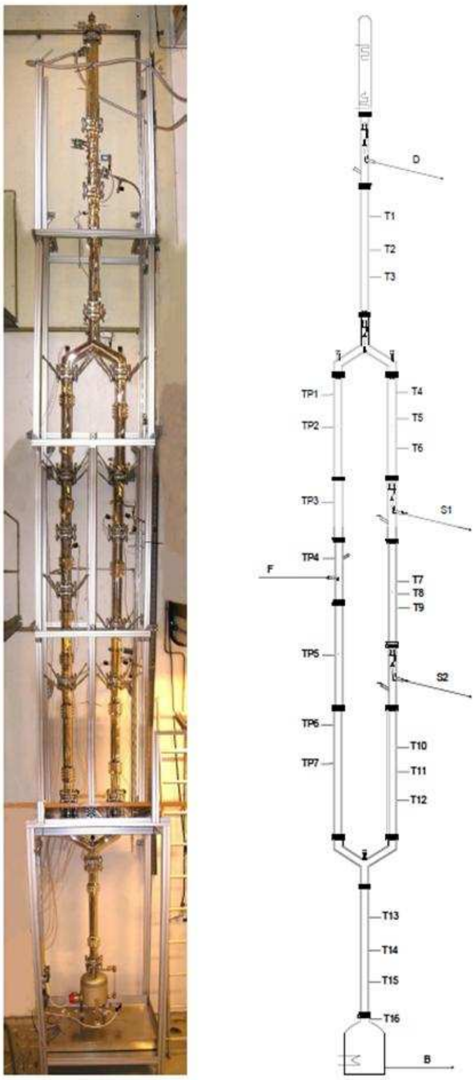


Figure 14: Non optimal separation in the prefractionator

5 PILOT PLANT AT THE NTNU

In 2006, PhD student Jens Strandberg built a pilot plant of a Kaibel distillation column in the Experimental Hall of the department of Chemical Engineering, which is part of the NTNU



a) Picture of the pilot plant *b) Scaled sketch*

Figure 15: Pilot plant at the NTNU

5.1 Construction

The construction physically differs from the construction as described in chapter three (Figure 11). This column starts as a single column, but splits in two columns, which join each other again in the top. The left hand column is the prefractionator, where the heated feed enters the column. This feed is a mixture of methanol, ethanol, propan-1-ol and butan-1-ol. In the prefractionator, methanol and ethanol are separated from propanol and butanol. These last two are the heaviest components and leave the prefractionator through its stripping section. Methanol and ethanol leave through the rectification section of the prefractionator. A table with the boilingpoint of the alcohols can be found on page 31.

The right hand column is the main distillation column. In the upper part, methanol and ethanol are separated. Methanol is drained off as distillate (D) and ethanol is drained off as side stream 1 (S_1). In the lower part of the main column, propanol is drained off as side stream 2 (S_2) and butanol.

The column can be divided into seven parts, with a total height of eight metres and an internal diameter of 5 centimetres. The column is filled with glass Raschig rings and has thus no physical stages. The number of theoretical stages in the model is 64, which makes its equivalent size larger than the pilot plant. But this means that the pilot plant has faster dynamic responses. In order to fit the model, the column has an open vent; the pressure inside the column will always tend to the atmospheric pressure.

5.2 Control and operation

5.2.1 Inputs and disturbances

The Kaibel distillation column has eight input values and five disturbances. The input values are the liquid and vapour splits, the vapour boil up, the reflux, side stream 1, side stream 2, distillate flow and bottom flow.

The liquid split can be found in the top of the column, where the prefractionator and the main column join each other. The split divides the liquid that comes from the top of the column between the prefractionator and the main column.

The vapour split is found in the bottom of the column, where it splits in two. This split divides the vapour between the prefractionator and the main column and is very important. If the vapour is not divided properly between the two columns, they are not able to work efficiently. The control of this split is difficult in practise and thus it is regarded as a disturbance.

Since the feed consists of only four components, it is enough to know the fraction of three products. The sum of the fraction is always one. The value z_F is the composition of the feed, which can be split in z_D, z_{S1}, z_{S2} and z_b.

The inputs and disturbances, with their nominal values from the model, are shown in Table 2 and in Figure 16.

Table 2: Inputs and disturbances

	Variable	Explanation	Nominal value	Unit
INPUTS	R_L	liquid split	0,2572	ratio
	R	reflux	2,8492	mol/min
	V	vapour boil up	3,0000	mol/min
	S_1	side stream 1	0,2494	mol/min
	S_2	side stream 2	0,2497	mol/min
	D	distillate	0,2508	mol/min
	B	bottom product	0,2503	mol/min
DISTURBANCES	R_V	vapour split	0,3770	ratio
	F	feed stream	1,0000	mol/min
	z_D	mole fraction of D in feed	0,2500	ratio
	z_{S1}	mole fraction of S1 in feed	0,2500	ratio
	z_{S2}	mole fraction of S2 in feed	0,2500	ratio
	q	liquid fraction of feed	0,9000	ratio

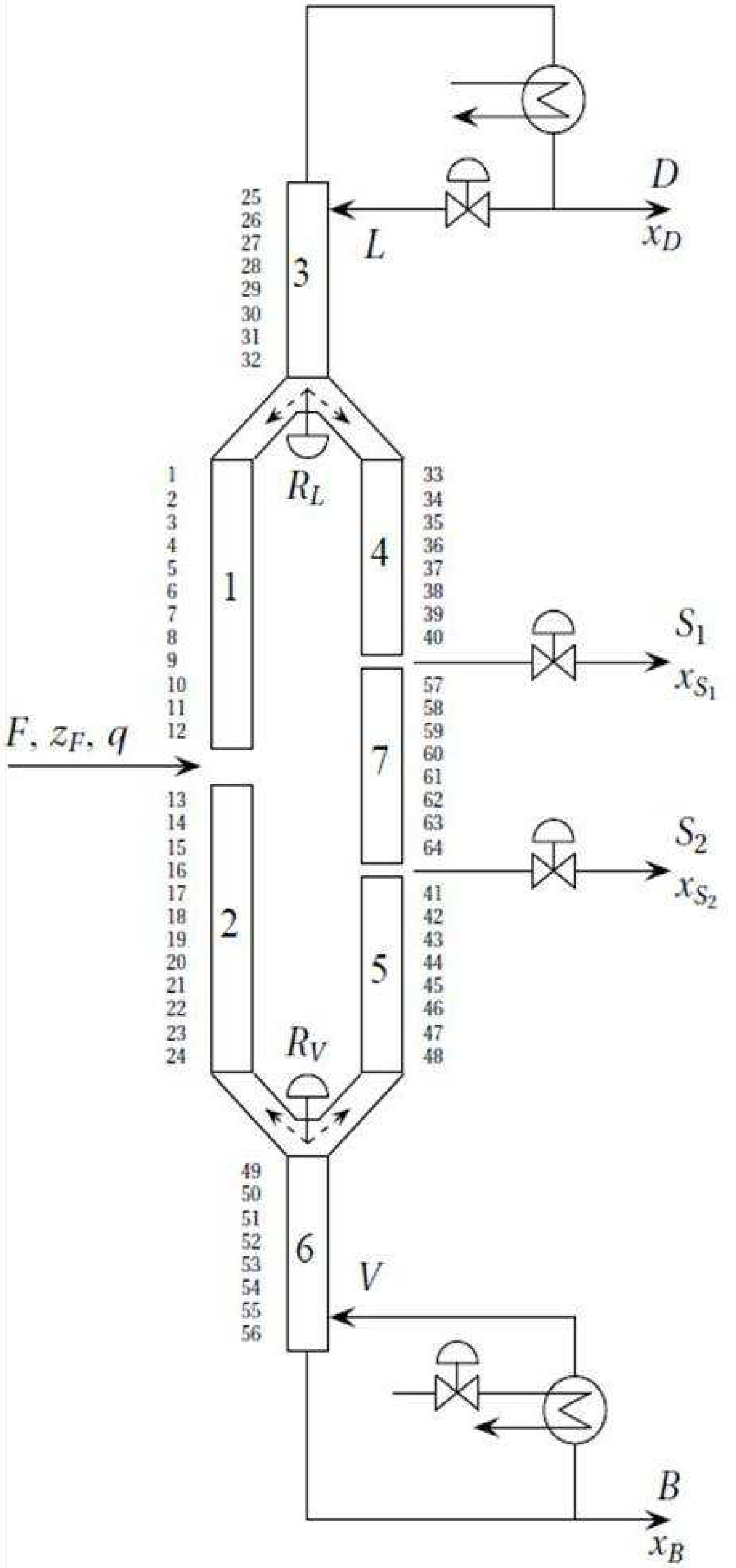


Figure 16: Inputs and disturbances

5.2.2 Operation

The column can be operated in two different ways. The most energy is saved when the column is operated at the minimum energy of the preferred split. This involves the risk that when a change is made in e.g. a setpoint or the feed, the required minimum energy shifts up or down. When it shifts up, the desired separation won’t be done properly anymore.

This is the reason why the column is operated above the minimum energy. This allows small changes to happen, without the risk of operating the column below the minimum energy.

5.2.3 Valves

The valves that are used in the liquid split and to drain of the products are electro-magnetic funnels (Figure 17). These are glass funnels with a piece of metal in it. Outside the column, next to the valve is an electromagnet. When current flows through the coil, it will attract the funnel and the product is drained off. The magnet is operated with a pulse width signal that comes from the computer. The input value is between zero and one. The timer uses timeintervals of 10 seconds. For the product valves, (distillate, S_1 and S_2) a value of 1 means that the valve is closed. A value of 0 means that the valve is completely open. For the liquid split, a value of 1 means that all the liquis goes in the prefractionator and a value of 0 means that all the liquid goes in the main column. The R/L valve has an upper and lower limit to prevent that all the liquid goes in one side of the column. These settings are local settings.

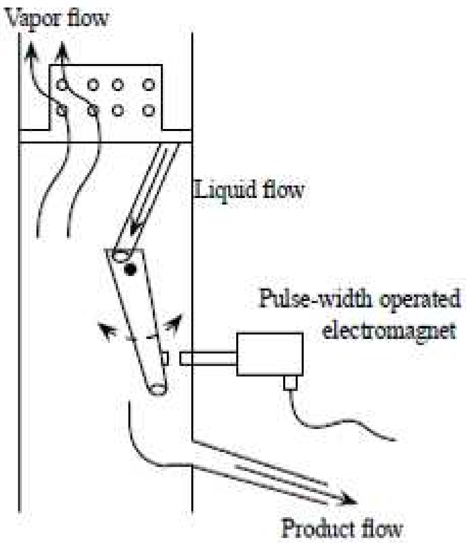


Figure 17: Electromagnetic funnel

5.2.4 Control loops

The column is controlled by temperature measurements. This technique is preferred, because the temperature is a good indication for the composition of the product. In 24 places in the column is the temperature measured, see Figure 15, but only four of these measurements are used to control and operate the valves. These four loops are shown in Figure 18 and are set to correspond with the boiling temperatures of the alcohols.

Table 3: Boiling points and setpoints

Alcohol	Boiling point (°C)	Temperature (nr. of tray in fig 16)	setpoint (°C)
methanol	64,7	30	72,0
ethanol	78,3	17	83,0
propanol	97,3	59	86,0
butanol	117,7	49	105,0

The measured temperatures are sent to the computer, where they are compared with the setpoint. The program that is used to run the column is written in LabVIEW™ and uses PI controllers. As can be seen in Table 3, the setpoints for all temperatures do not match with the corresponding boiling temperatures. This is done because the temperatures are measured a bit lower than the location of the valves.

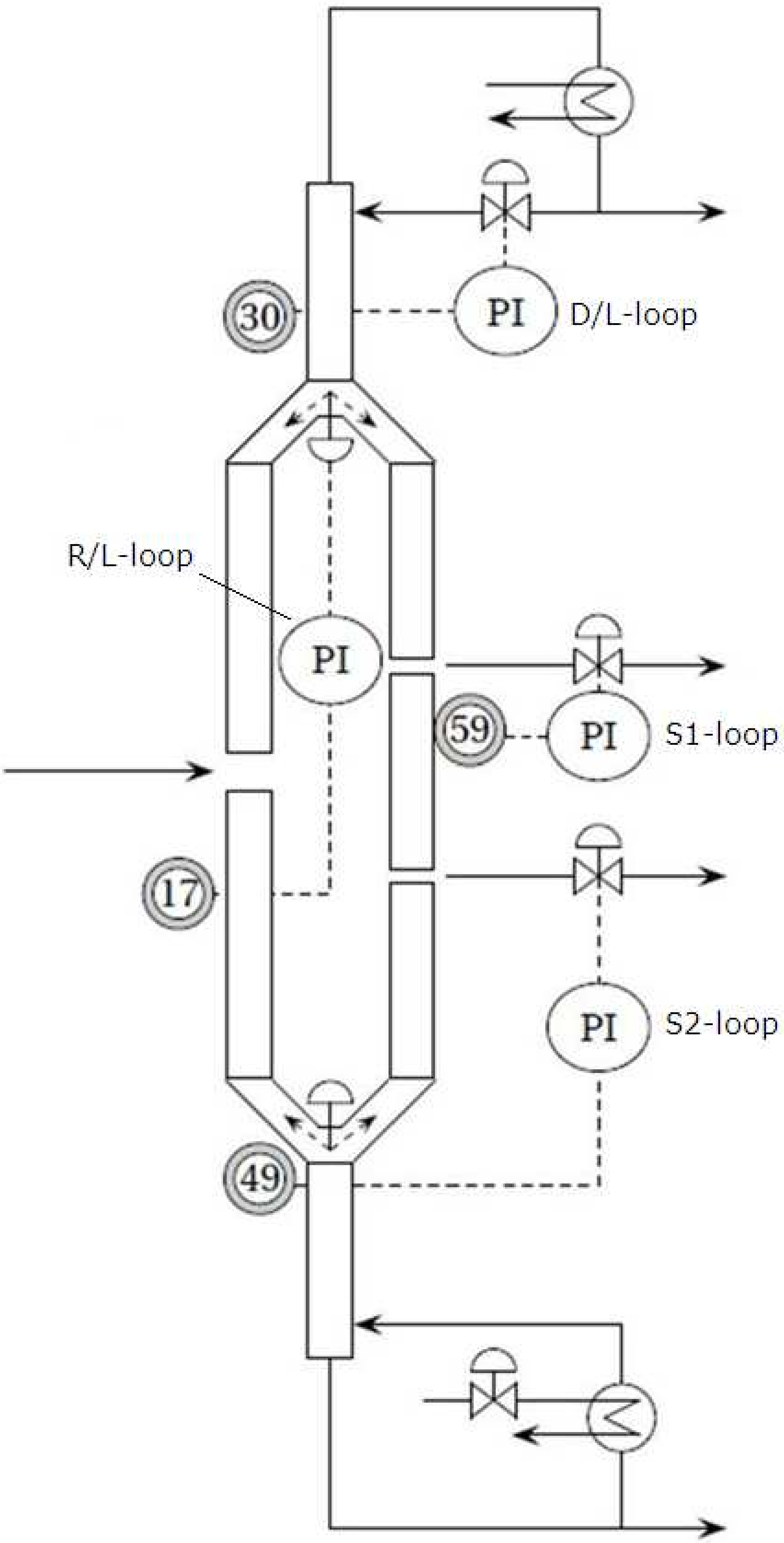


Figure 18: Temperature control loops

6 COMPUTER MODEL

6.1 Model description

The model is built up as in Figure 16 and is implemented in MATLAB/Simulink. The model evolved step by step since many persons have contributed in the development of the column. It has 13 inputs; these are all the values that are shown in Table 2. Since not all inputs are controllable, they are divided in inputs and disturbances. The disturbances are the inputs that can not be controlled with exception of the vapour split. This one can be controlled but this is very difficult in practise and is thus regarded as a disturbance.

The model has also a level controller for the distillate and the bottom. The pilot plant has no reflux hold-up. This is simulated in the model by using a very large stock. Since both the bottoms and distillate level are held constant, they have no further influence in the model.

As mentioned before, the column exists of 7 parts. Part one and part two form the prefractionating zone. In the model, both sections have 12 trays but this can be easily changed in the model. The third part is the top section of the column and consists of eight trays. The main column consists of three sections, which are numbers 4, 7 and 5, counted from the top to the bottom. The seventh section is section number 6 and is the part above the reboiler. Sections 3, 4, 5, 6 and seven all contain eight trays in the model. This brings the total number of trays in the column at 64.

The model is based on three differential equations. Two are used to calculate the mass fraction and the mole fraction. The third is used to update the temperature inside the column.

6.2 Summary of the model

The calculations in the model can be summarized in 5 steps (Kvernland, 2009).

1. The liquid and vapour flow for each stage (tray) in the column is calculated;
2. The mass change on each stage is calculated;
3. The vapour mole fraction for each component at each stage is calculated;
4. The liquid mole fraction for each component at each stage is calculated;
5. The temperature on each stage is updated.

7 TUNING OF THE COLUMN CONTROLLERS

7.1 Purpose of the experiment

The aim of this first run is to see if the column can work in a stable way. In the past, there have been a lot of issues with getting the column stable. Some modifications have been done to run the column in a more stable way. Once the column is stabilized, it is tested to see how it responds to setpoint changes. This is done by changing the setpoints and let the column stabilize. The controller settings are taken from the model and are adapted using the SIMC-PID tuning rules

7.2 Description of the tests

The loops are shown on page 33, in Figure 18.

7.2.1 R/L-loop

The setpoint of the loop is increased from 84°C to 83°C. The new setpoint is reached in about half an hour, after some oscillation around the setpoint. The output for the R/l-valve increases from 35% to 45%. This means that 45% of the reflux is sent in the prefractionating section. The S_1 temperature loop is able to reach it se point, but the valve almost saturates. The value of the distillate-loop increases, but it is not able to return to its setpoint: The valve saturates at its upper limit (1). It is thus not able to compensate the disturbance. The S_2 valve saturates at the lower limit (0,2). This means that 20% of the product is drained off. The graphs are displayed in Chart 1.

7.2.2 Feed rate

In the second test, the feed rate was increased from 3L/h to 4L/h. All temperature loops are able to return to their setpoint, except the S_2-loop. It saturates at the lower limit of 0,2 and is not able to reach its setpoint. See Chart 2.

7.2.3 S_2 control loop

In the next test, the S_2 setpoint is decreased from 107,7°C to 107°C. The new setpoint is reached in 10 minutes, and all the other control loops react OK. This is shown in Chart 3.

7.2.4 S_1 control loop

Then, the S_1-controltemperature is set from 86,4°C to 87,4°C. In the first attempt, the temperature oscillates a lot. The loop was retuned and a more aggressive tuning parameter (τ_C) is applied. With this new parameter, the setpoint can be reached in 8 minutes and all loops perform well (Chart 4).

7.2.5 D/L-loop

For the fifth test, the D/l-control temperature is changed from 72°C to 73°C. The new setpoint is reached in 10 minutes. When the temperature is lowered back to 72°C, the valve-output saturates at the upper limit. Probably, some of the heavy products escaped to the top section, which causes a major disturbance. The D/l-loop is put in manual mode and the column is stabilized. See Chart 5.

7.2.6 Steady state operation

The last figure, Chart 6, shows the stabilized column, with all loops in automatic mode. This is the column in a steady state.

7.2.7 Explanation of the charts

The charts always show two columns. The left hand column shows the temperature measurement (oscillating lines) and the step change that is made. The right hand column shows the output value that is sent to the valve.

7.3 Discussion

The initial tuning parameters were taken from the computer models. This didn't mean that they are the best in practice, as can be seen in the results. This experiment gave an idea about how good they are and which ones had to be adapted. The results of this experiment are used to modify the model.

The setpoints that are used to control the valves are also taken from the models. These experiments can not say if these are the right setpoints or not. To know if these setpoints are good enough, samples must be taken and analysed. This performance test is described in the next chapter.

7.4 Conclusion

The column operates in a stable manner. The measured values are close to the setpoints. Although they seem to oscillate around the setpoints, the column works more stable than in the past. The results are satisfactory when they are compared to previous results.

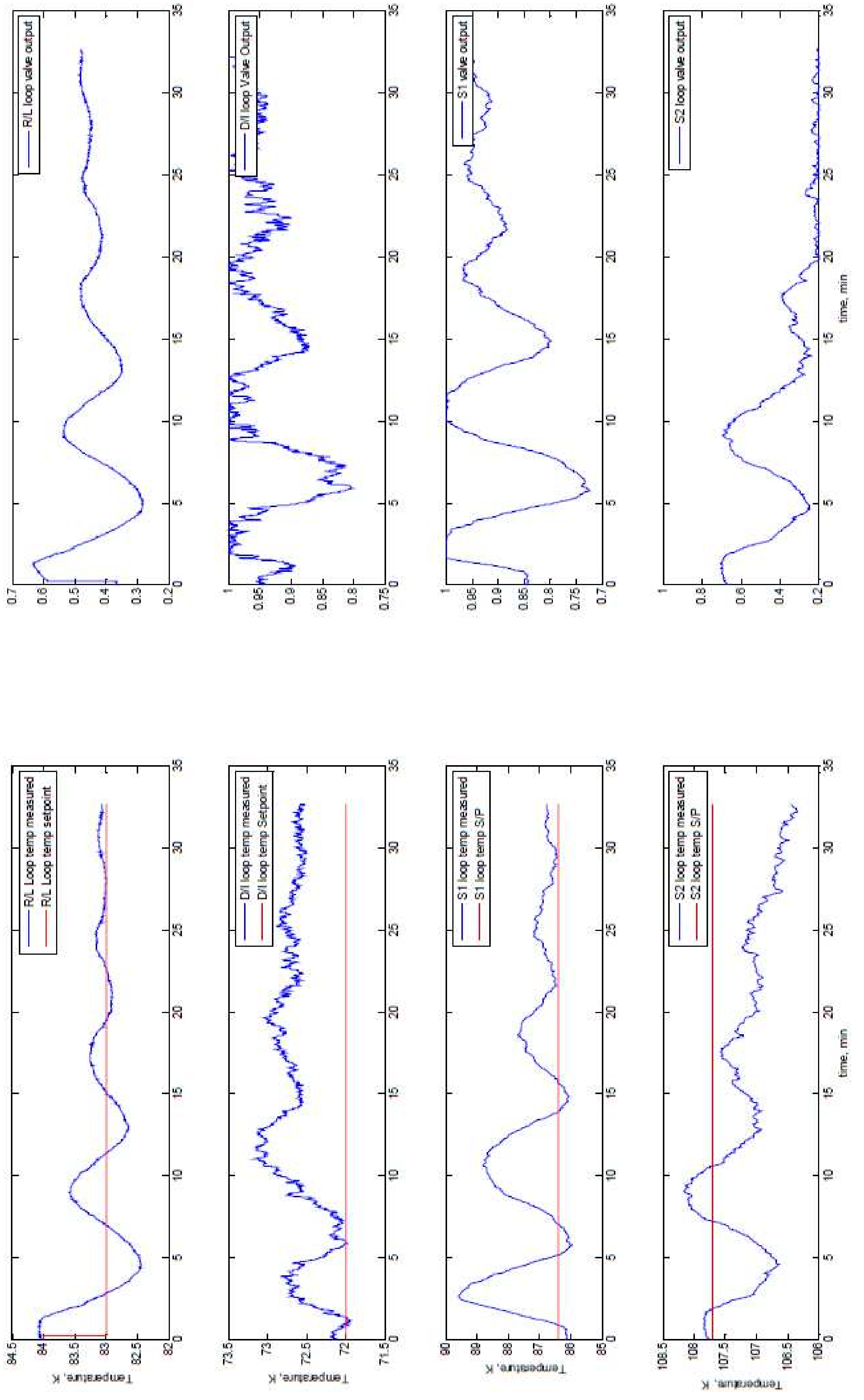


Chart 1: R/L loop setpoint change

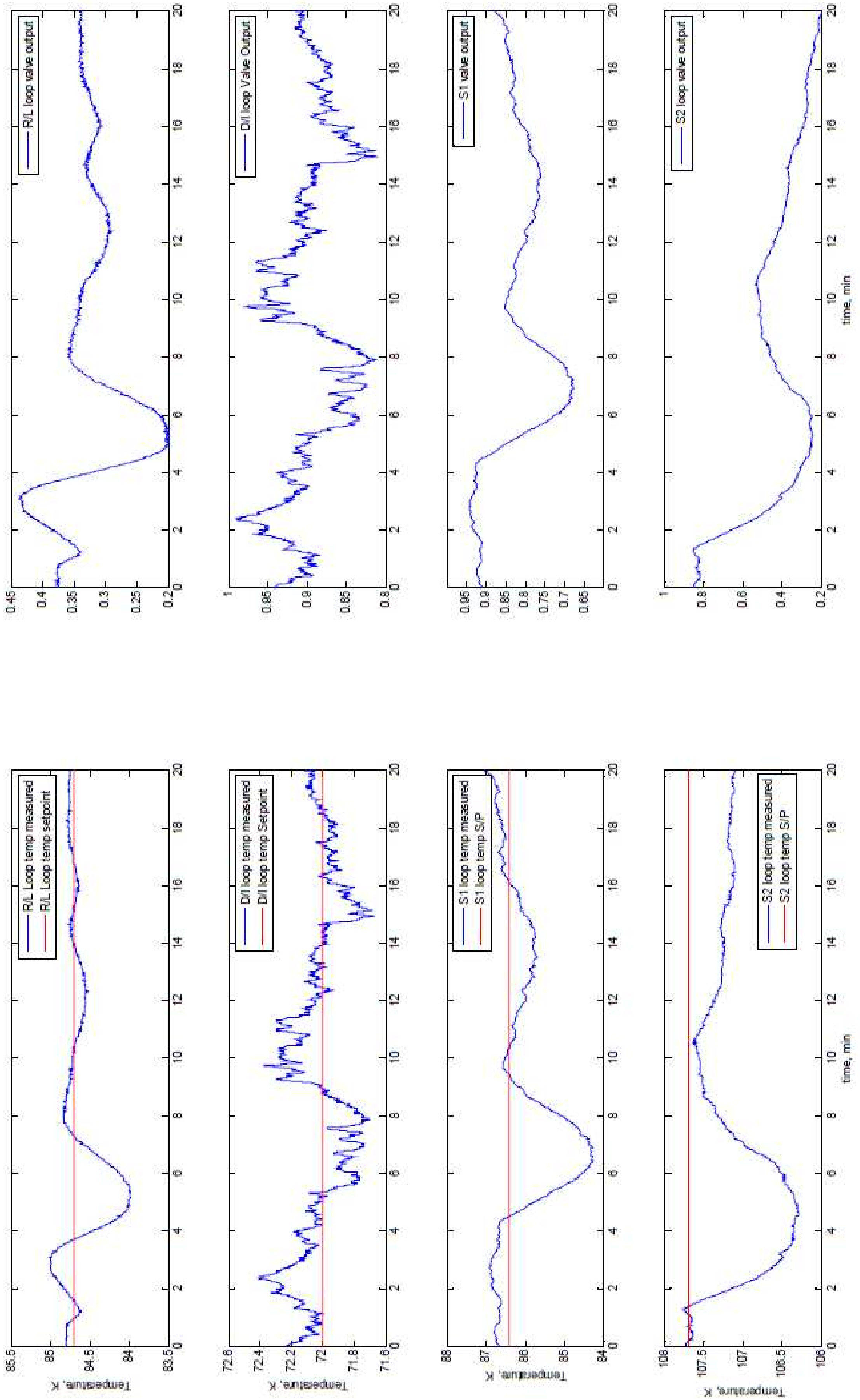


Chart 2: Feedrate change

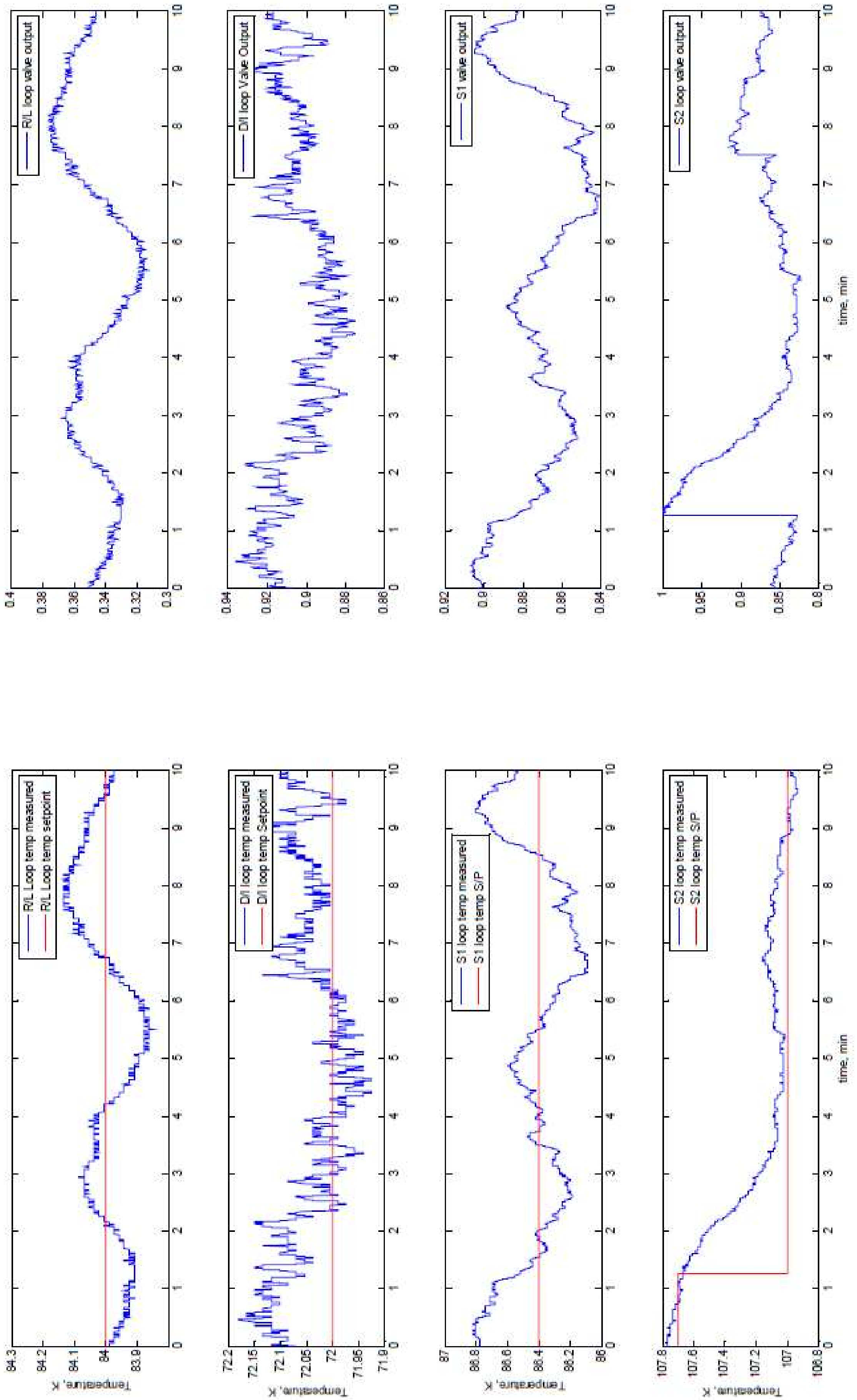


Chart 3: S2 setpoint change

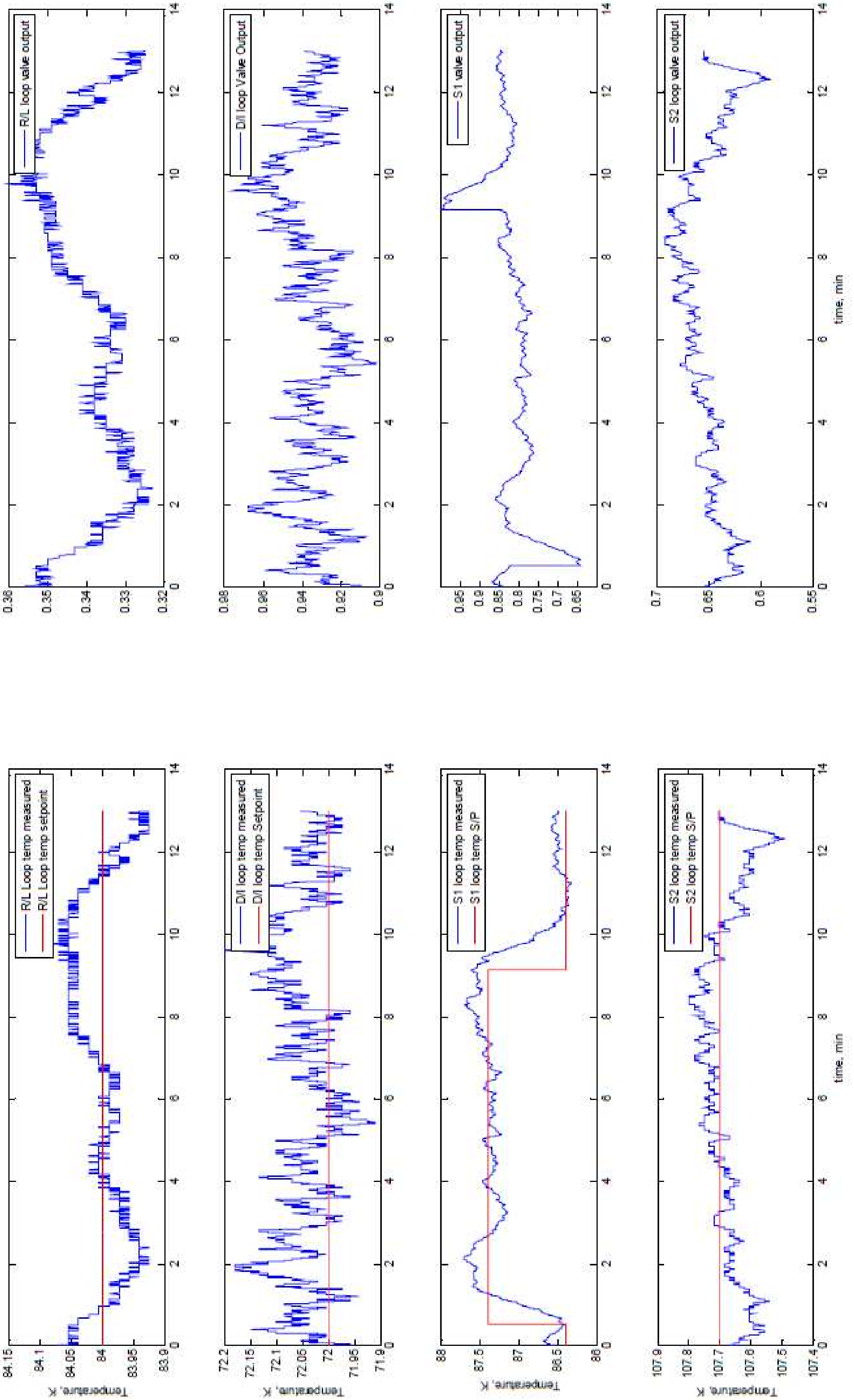


Chart 4: S1 setpoint change

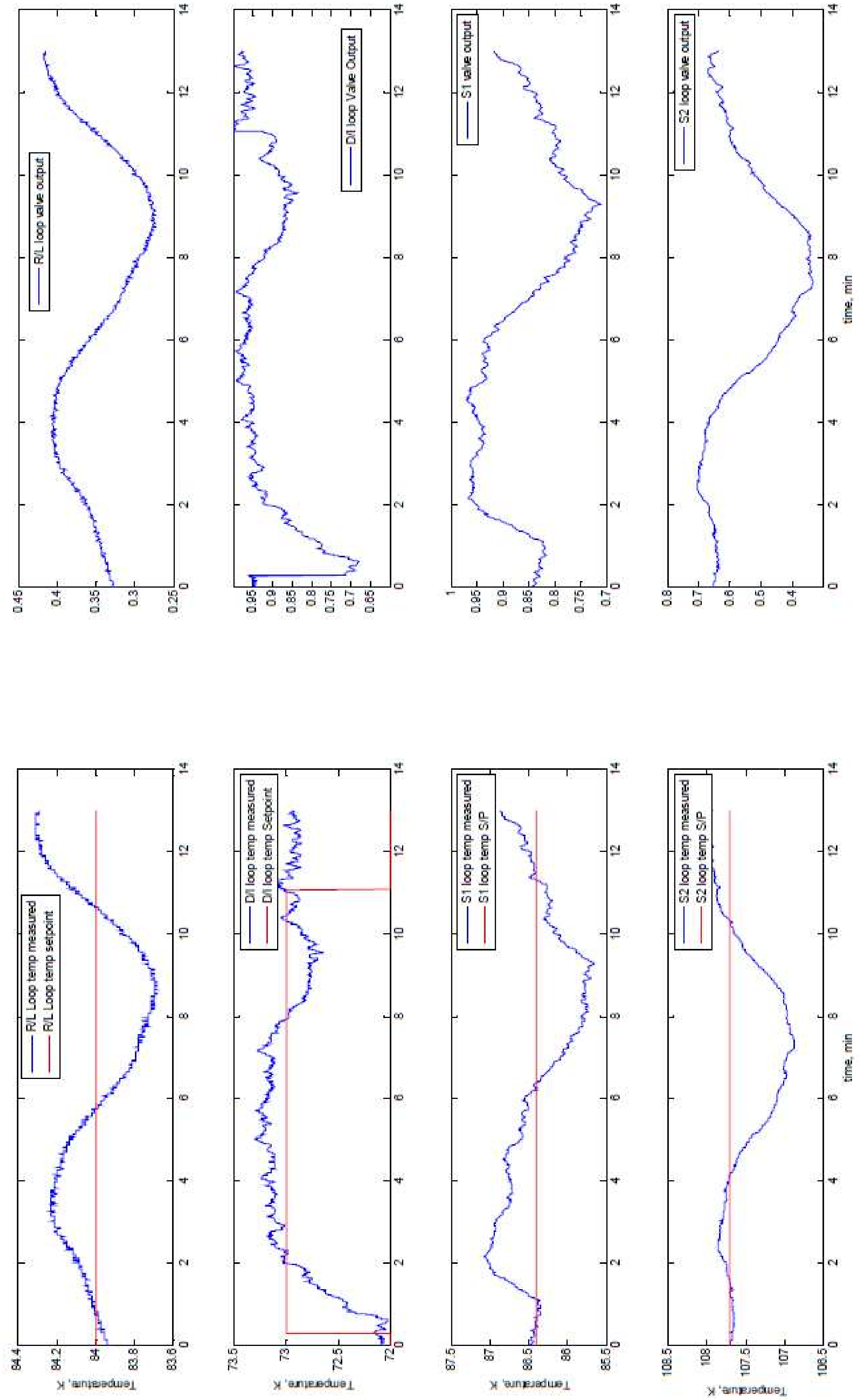


Chart 5: D/L setpoint change

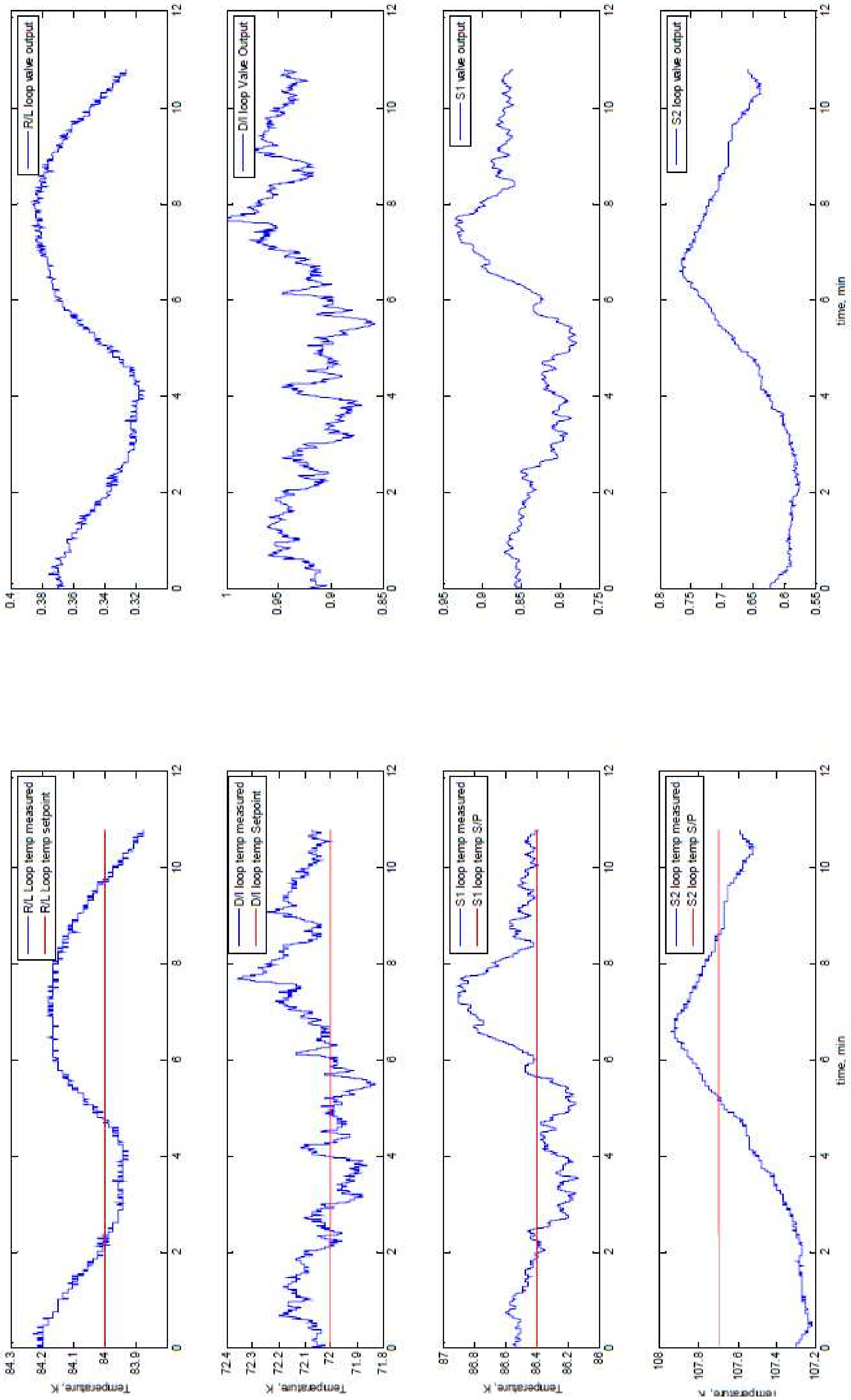


Chart 6: Steady state operation

8 COLUMN PERFORMANCE

In order to say something about the setpoints that are used to run the column, the performance of the column is studied. This study is done with two experiments. The first is a flow measurement to see how much of the products is drained off. The second test is analysis of samples that are taken directly after the flow measurements. The composition of these samples will give an idea about the separation in the column.

8.1 Flow measurement

Before the test is started, the column was brought to a steady state. This insures that all controllers are working and that the flow is constant.

8.1.1 Setup

During the flow measurement, the valves of the storage tanks of the distilled products are closed; half an hour after, the tanks are emptied and the volume is measured. This test is repeated three times and samples are taken. The experiment was done in two days. The first measurement was done on the first day and the second and third measurements were done on the second day.

8.1.2 Results

Table 4: Flowtest results

Measurement	V_{dist} (mL)	V_{S1} (mL)	V_{S2} (mL)	Total (mL)
26/02/2010	160	470	560	1190
01/03/2010 (1)	150	(200)	790	1140
01/03/2010 (2)	150	260	830	1240

Notes:

- The V_{S1}-value of the first measurement of 01/03/2010 is not correct. The valve was opened towards the pump in stead of draining off the product. Some of the product was thus not measured and the value is too low.

8.1.3 Discussion

The first measurement of 01/03/2010 is rejected because of the measurement fault in V_{S1}.

The amount of top product is constant in all the three measurements. For the V_{S1} and V_{S2}, a distinction can be made between the two days. On the first day, much more V_{S1} but less V_{S2} was drained off. To say something about the quality of the products, the results of the analysis will also need to be involved.

The total amount of drained off product is also in the same range. This was expected because the feed rate of the column was the same during the three measurements. The amount of product in the reboiler can not be measured, unless it is completely drained off. This is not possible when the column is operating.

8.2 Analysis of the samples

During two flow tests, samples were taken to analyse the composition. This will give an idea about the performance of the column. The samples are diluted 20 times with water and analysed with HPLC. The resulting chromatograms are compared with the standard values in the computer.

8.2.1 Results of the analysis

The first set of samples was taken after the flow test of 26/02/2010. After the volume of collected product was measured, a sample of each fraction was taken.

Table 5: Analysis of the first set of samples

	Volume%				
sample	MeOH	EtOH	PrOH	BuOH	Total
D	83,38 %	13,62 %	0,00 %	0,00 %	97,00 %
S1	8,56 %	64,84 %	24,07 %	0,00 %	97,47 %
S2	0,00 %	17,99 %	77,19 %	4,35 %	99,53 %
bottoms	0,00 %	0,14 %	7,50 %	92,89 %	100,53 %
feed	10,07 %	18,83 %	31,61 %	37,83 %	98,34 %
	mole %				
sample	MeOH	EtOH	PrOH	BuOH	Total
D	89,82 %	10,18 %	0,00 %	0,00 %	100,00 %
S1	12,86 %	67,55 %	19,59 %	0,00 %	100,00 %
S2	0,00 %	22,19 %	74,38 %	3,42 %	100,00 %
bottoms	0,00 %	0,21 %	8,98 %	90,80 %	100,00 %
feed	17,67 %	22,92 %	30,05 %	29,36 %	100,00 %

The second set of samples was taken on 01/03/2010. The liquid was taken directly from the outlet of the column, instead of the stock tanks.

Table 6: Analysis of the second set of samples

	Volume%				
sample	MeOH	EtOH	PrOH	BuOH	Total
D	83,07 %	11,83 %	0,00 %	0,00 %	94,90 %
S1	9,63 %	64,81 %	20,29 %	0,00 %	94,73 %
S2	0,31 %	14,62 %	64,48 %	20,76 %	100,17 %
bottoms	0,00 %	0,00 %	7,06 %	93,41 %	100,47 %
feed	6,77 %	25,72 %	48,80 %	17,93 %	99,22 %
	mole %				
sample	MeOH	EtOH	PrOH	BuOH	Total
D	91,01 %	8,99 %	0,00 %	0,00 %	100,00 %
S1	14,69 %	68,55 %	16,76 %	0,00 %	100,00 %
S2	0,57 %	18,58 %	64,02 %	16,83 %	100,00 %
bottoms	0,00 %	0,00 %	8,47 %	91,53 %	100,00 %
feed	11,48 %	30,25 %	44,83 %	13,45 %	100,00 %

8.2.2 Discussion

Because of a fault in the calibration, the volumefractions don't count up to 100%. In the calculations, these values were used as if they count up to 100%. That is why there is a difference between the sum of the volume and molefraction. The analysis were not self performed, but were done by the analysis lab of the NTNU. HPLC is used to perform the analysis.

The results show clearly that the separation isn’t done properly. The component that should be drained off has always the highest fraction, but the concentration is not high enough. The setpoints that were chosen are not good enough to perform a proper separation.

The sum of the fractions is also not 100%. This is due to errors associated with the measurement method.

An explanation can be that there are pure products in the column, but that they are not drained off enough. And hence the products are pushed to other parts of the column, causing impurities.

8.3 Conclusion

The setpoints that are applied are not good. The separation is insufficient. The results will be used to adjust the model and then new setpoint values can be retrieved and tested.

9 ADJUSTING TEMPERATURE SETPOINTS

The temperature setpoints applied on the column are not ideal. If the concentrations from chapter eight are regarded, mainly the sidestreams S_1 and S_2 are not pure enough. During experimental runs that are described below, new sets of setpoints are applied. These should increase the purity of the distilled products, mainly sidestream 1 and sidestream 2. All the three sets of setpoints should give a more or less equal temperature profile in the column.

9.1 First case

9.1.1 Setspoints

Table 7: First set of new setpoints

Loops	Old setpoint	New setpoint
D/L loop	72,0°C	manual
S1 loop	86,4°C	91,8°C
S2 loop	107,7°C	111,5°C
R/L loop	84,0°C	90,0°C

As can be seen in Table 7, the distillate temperature loop is in manual mode. This was done because the temperature measurement gave an error, which can be seen in Chart 7.

9.1.2 Analysis of the samples

Table 8: Analysis of the first case

	methanol	ethanol	propanol	butanol	Total
distillate	77,82282%	22,17718%	0,00000%	0,00000%	100%
S1	3,95448%	63,32168%	32,72384%	0,00000%	100%
S2	0,00000%	6,23111%	81,55102%	12,21787%	100%
bottoms	0,00000%	1,32720%	15,86550%	82,80730%	100%
feed	9,80609%	26,38208%	44,21140%	19,60043%	100%

All the results are molefractions.

9.1.3 Discussion

When compared to the cases from chapter eight, the purities of the distillate and bottoms are almost 10% lower. The impurities in the top can be explained by the manual mode of the D/L loop. The purity of the S1 stream becomes also lower (less ethanol), but the purity of S2 becomes higher (more propanol).

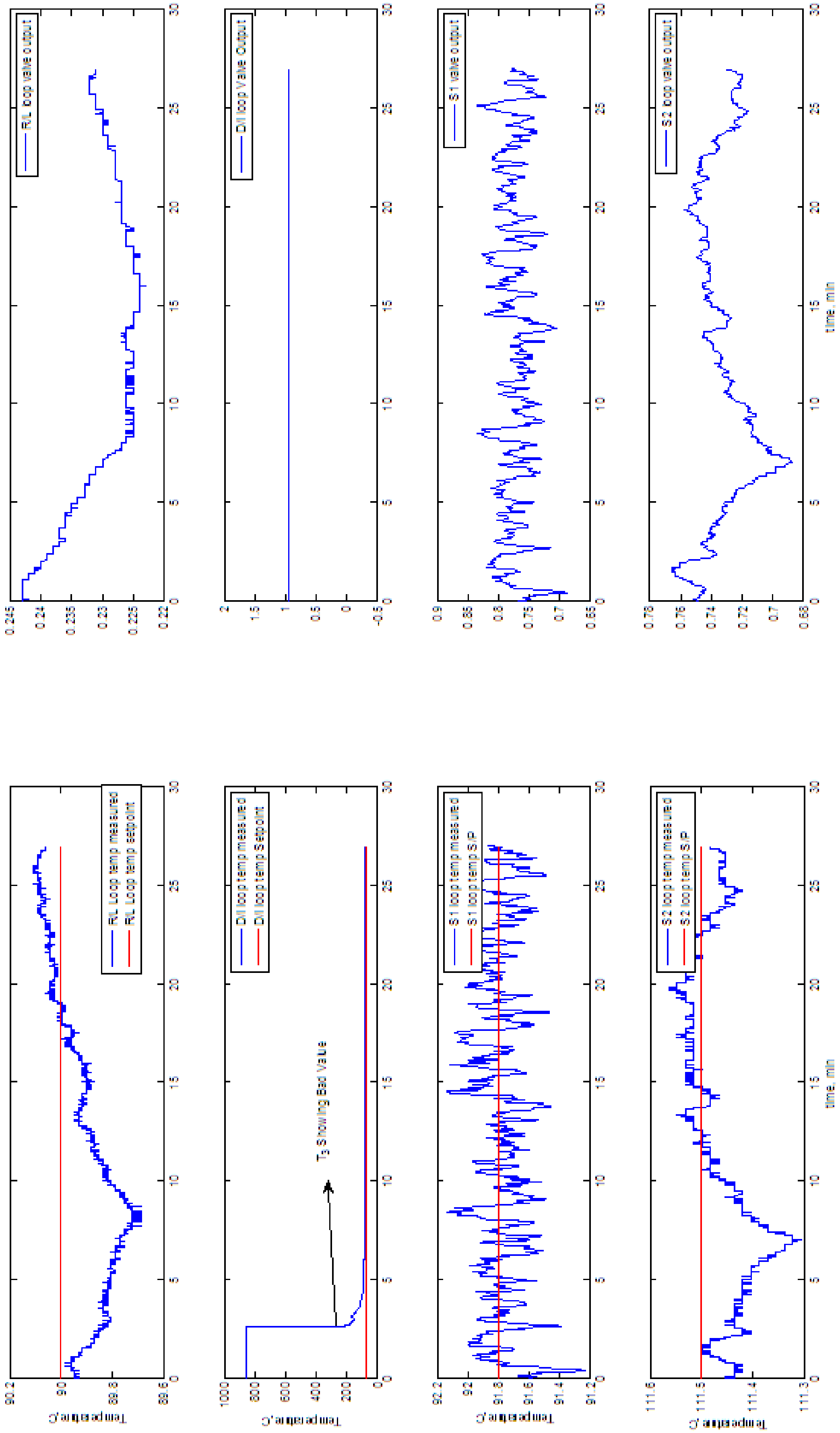


Chart 7: Behaviour during case 1

9.2 Second case

9.2.1 Setpoints

Table 9: Setpoints case 2

Loops	Old setpoint	New setpoint
D/L loop	manual	72°C
S1 loop	91,8°C	91,8°C
S2 loop	111,5°C	120,0°C
R/L loop	90,0°C	91,5°C

The location of the temperature measurement was relocated to the temperature measurement which is the closest to the top of the column. This made it possible to put the controller back in automatic mode.

9.2.2 Analysis of the second set of samples

Table 10: Results of the analysis

	methanol	ethanol	propanal	butanol	Total
distillate	92,69409%	7,305909%	0,00000%	0,00000%	100%
S1	17,23875%	51,54409%	31,21717%	0,00000%	100%
S2	0,00000%	5,38886%	89,63234%	4,978797%	100%
bottoms	0,00000%	0,00000%	6,68279%	93,31721%	100%
feed	20,38695%	17,64041%	26,67070%	35,30194%	100%

Like in the first case, all the concentrations are mole fractions.

9.2.3 Discussion

All the purities increased, except the concentration of ethanol in the first side stream. It is mainly the concentration of methanol that has increased. This can be due to the fact that the D/L control loop is turned automatic, at the same temperature. This temperature is 7°C above the boiling point of methanol.

The control loops behave quite well, as can be seen on.

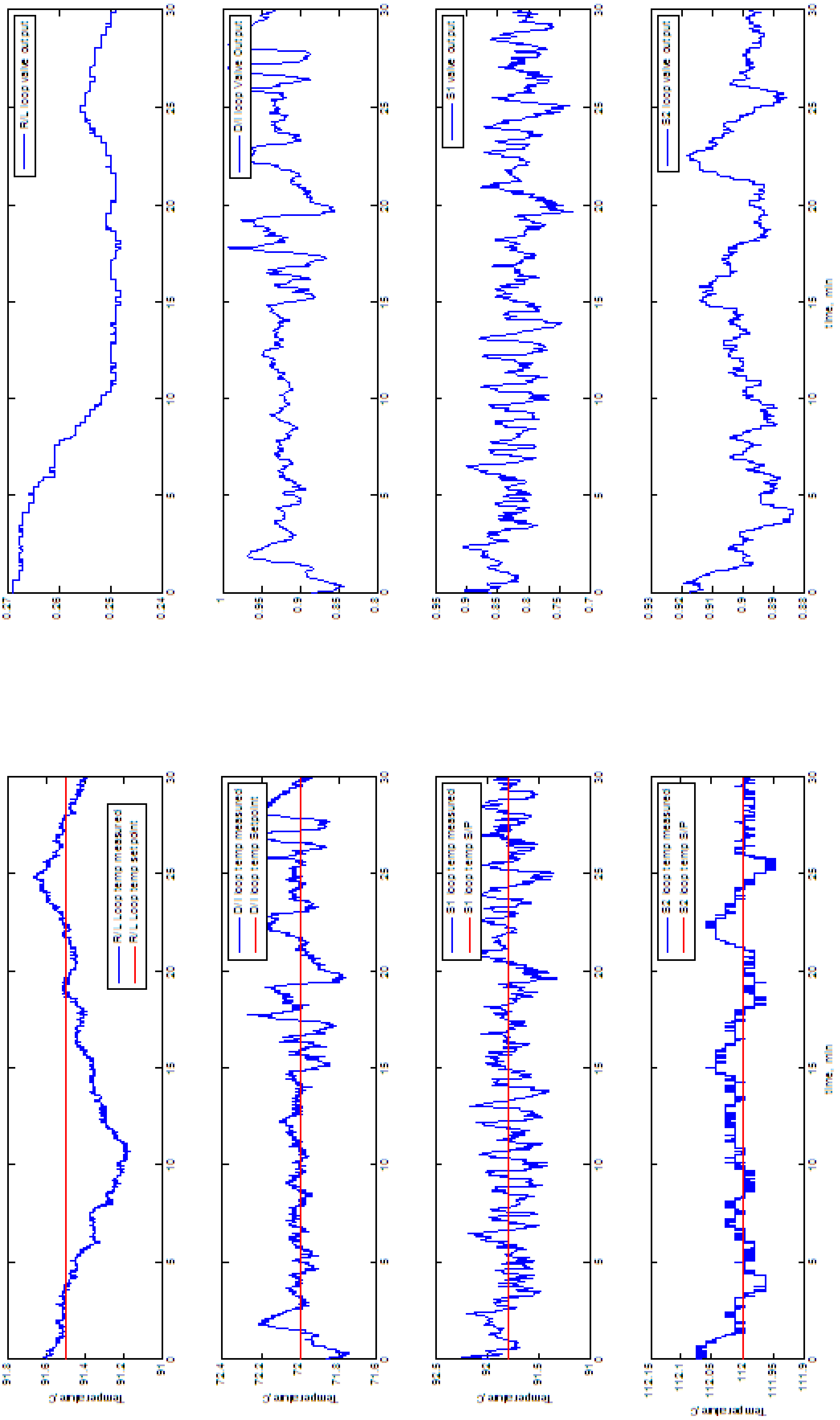


Chart 8: Control loop behaviour during case 2

9.3 Third case

9.3.1 Setpoints

Table 11: Setpoint for case 3

Loops	Old setpoint	New setpoint
D/L loop	72°C	71°C
S1 loop	91,8°C	86°C
S2 loop	120,0°C	112°C
R/L loop	91,5°C	96°C

9.3.2 Analysis of the samples

Table 12: Analysis of the samples from case 3

	methanol	ethanol	propanal	butanol	Total
distillate	94,39448%	5,605523%	0,00000%	0,00000%	100%
S1	26,30131%	56,34609%	17,35260%	0,00000%	100%
S2	0,00000%	10,14515%	86,33347%	3,521385%	100%
bottoms	0,00000%	0,00000%	6,68279%	93,54654%	100%
feed	16,30592%	19,01968%	28,30867%	36,36573%	100%

All concentrations are moleconcentrations

9.3.3 Discussion

The composition of the first sidestream remains bad. Even more methanol comes down, despite the setpoint temperature of D/L was decreased. But since all temperatures, exept R/L, were decreased, the effect could be worked away. Chart 9 shows that the controllers were working quite steady.

9.4 Conclusion

The achieved purities are some of the best that have been achieved so far; they are not good enough though. The second and third sets give equal results in purity. To improve the purities, better setpoints need to be found that allow larger temperature drops in the sections between the valves, in this way that the temperature at the valve is the boiling point of the desired component.

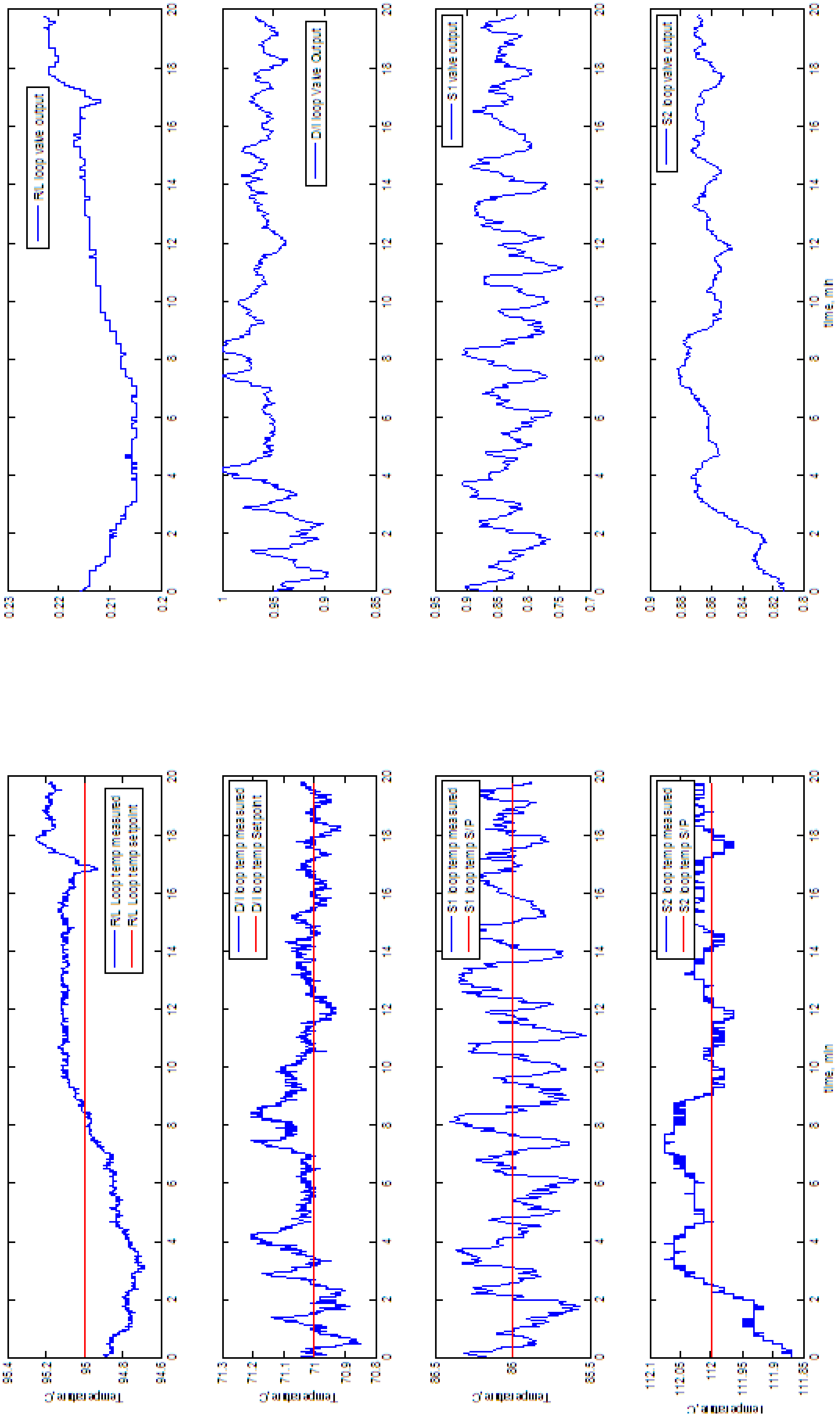


Chart 9: Controller behaviour in case 3

10 DETERMINE THE NUMBER OF TRAYS

The determination of the number of trays in the column is a rather important experiment. It allows to adjust the model more precisely to the real column. Up to now, the number of trays that was applied in the model, was an estimated guess. With the results from this experiment, a more accurate number can be applied.

10.1 Preparation of the column

10.1.1 Changes in setup

In order to perfom the experiment, some changes had to be made to the experimental setup. First, the feedline and distillate tank were disconnected. Then the feedline was connected to the distillateline so the feed is pumped through this line, in the top of the column. (see Figure 19)

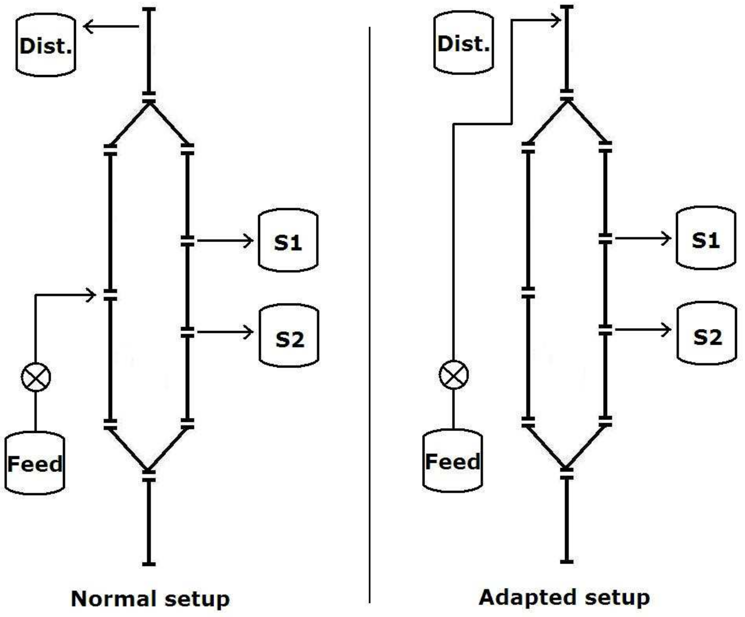


Figure 19: Adapted setup for the determination of the number of trays

After the change was made, the column was rinsed with ethanol. Ethanol is used because the mixture that is used to determine the number of trays is methanol-ethanol. This however is not a good mixture because the difference in boilingpoint between methanol and ethanol is 13°C. It was used because it doesn’t insert contamination in the column.

10.1.2 Changes in the controlstructure

In order to determine the number of trays, the column needs to work as a normal distillation column, which means that the temperatureprofiles in the prefractionator and the main column needs to be equal. To do this, a change was made in LabVIEW™. An extra function was added that calculates the difference two temperature measurements and uses this difference to control the liquid split (R_L). This allows to measure a temperature in the prefractionator and the main column and use these two temperatures to adjust R_L in that way that the temperatures are equal.

This change in the controlstructure makes it possible to determine the ratio of the vapour split (R_V). Since the temperatures in both sections needs to be equal, the value of R_L will be adapted so that liquid is sent in each section in the same ratio as vapour is sended in each section. E.g. if 40% of vapour goes in the prefractionator, R_L should also allow 40% of liquid to go into the prefractionator. This should be the case in the most ideal case, when each section contains an equal number of trays.

10.2 Experimental runs

10.2.1 Start up

An equimolar mixture of methanol and ethanol was brought in the feedtank and pumped into the column. After that, the pump was shut down and the valve on the distillate line was closed. Also the valves leading to the S_1 and S_2 stocktanks were closed. Some extra methanol was brought in the feedtank. This was used to adjust the temperature profiles in the column.

Next, heating was started and the column was brought to an equilibrium. When the profiles were stable, the R_L controlloop was turned on, so it could be tuned. Once it was tuned, the column came to a new equilibrium with equal temperatures in both sides of the column. In order to get the average temperature of the two boilingpoints in the middle of the column, some extra methanol had to be pumped into the column. A snapshot of the temperature profile in the prefractionator and the main column can be found in Chart 10 and Chart 11.

Chart 12 shows the steady state profiles that were achieved during the run.

Some problems occur during the run:

- An error in the programming code was discovered. A wrong connection had been made which caused a double reading of a temperature instead of reading two different temperatures. This problem was solved easily.
- During the run, the temperatures in the column kept raising, up to the boiling point of ethanol. A leak in the disconnected feedline was found. Product leaked from the prefractionator in the disconnected part of the feedline. This caused a waste of some product and a major temperature upset in the profiles. The leak was closed.

During both runs, R_L stabilized around the value of 0,42. Which means that 42% of the liquid is sent into the prefractionator.

10.2.2 Sample taking

As mentioned before, the mixture of methanol and ethanol is not an ideal mixture to perfom this experiment. This could clearly be seen in the temperature profile: the temperature in the top is 66°C, which is near to the boilingpoint of methanol; the temperature in the bottoms is 80°C, which is higher than the boilingpoint of ethanol. The bias of the temperature measurements is not included in these values, which explains why they deviate from the normal values.

Samples were taken by collecting liquid in the draining valves. Next to each valve is a samplepoint, were the liquids where drained off. The samples were taken at the distillate valve, the S_1 and S_2 valve and from the bottoms.

Since the top and bottom products are expected to be pure methanol and ethanol, the samples from the S_1 and S_2 valve can be important. The difference in concentration between those two is determined by only one section. Since the main column consists of 5 equal sections, the total number of trays can be determined, using the samples from S_1 and S_2.

Chart 10: Temperature profile in the main column

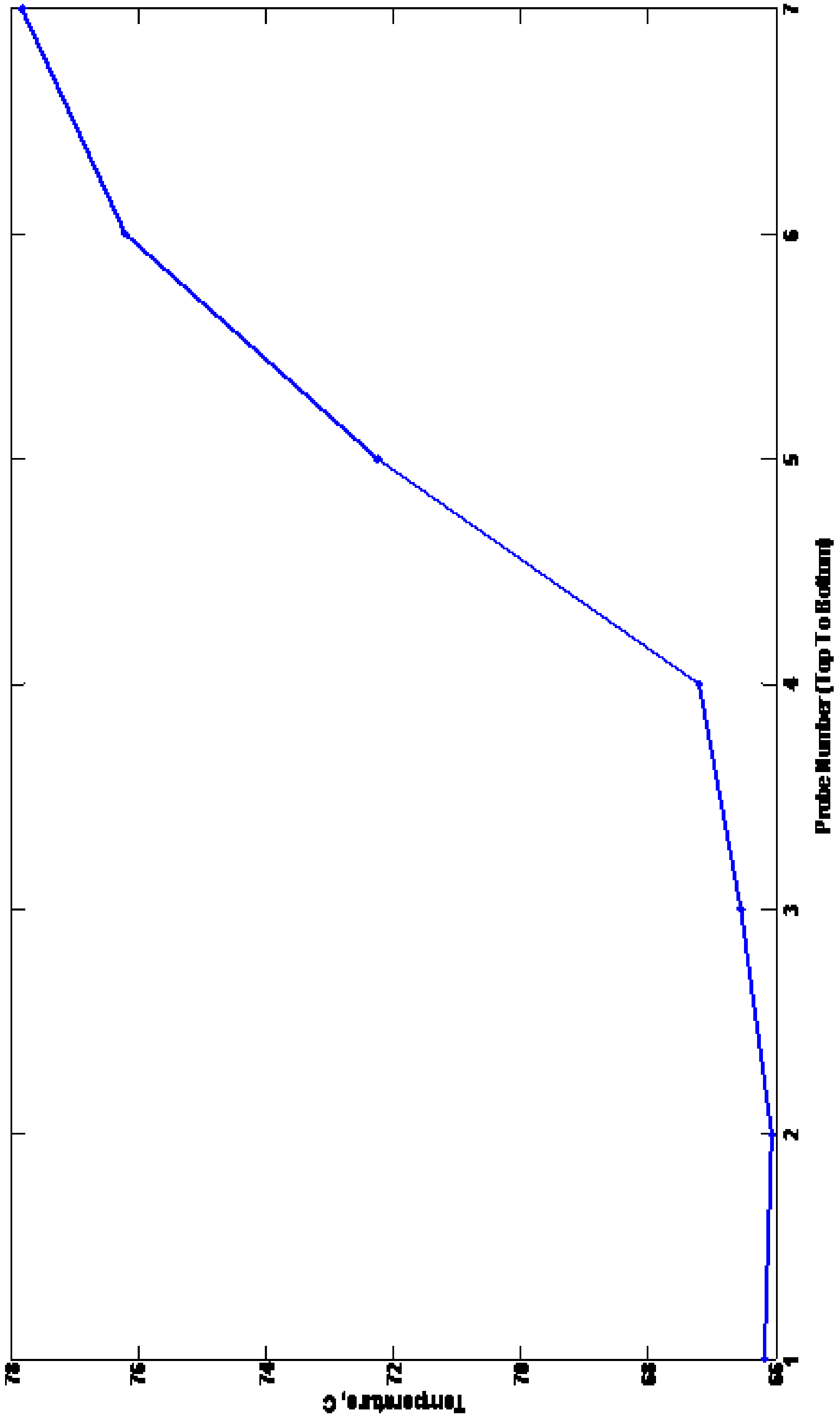


Chart 11: Temperature profile in the prefractionator

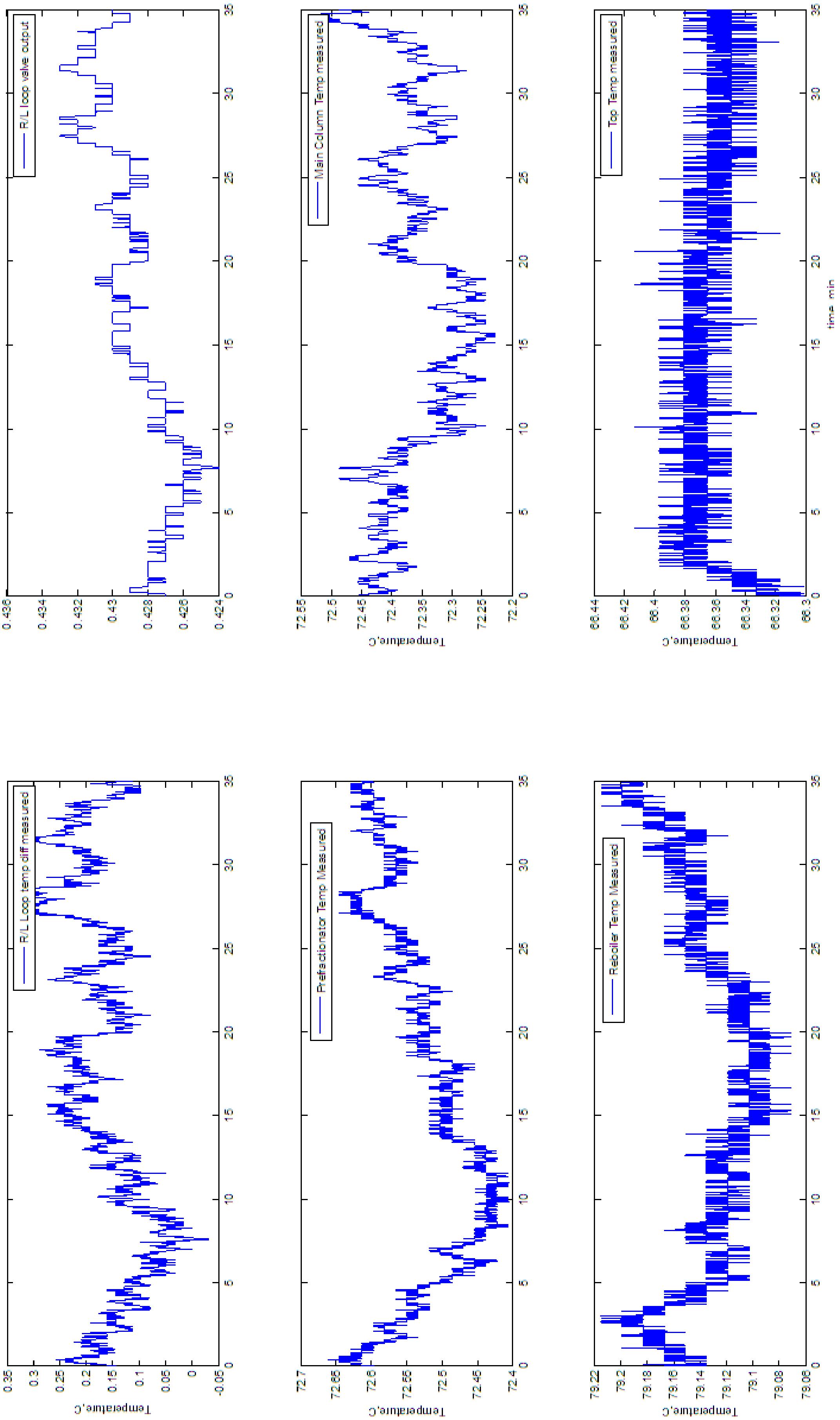


Chart 12: Steady state profile

10.3 Analysis of the samples

The experiment was performed two times. This gives a better indication in how well the experiment was performed. If the calculated numbers are close to each other, the experiment was performed well.

Table 13: Samples from the first run

	MeoH	EtOH
Dist.	96,80819	3,19181
S1	80,33284	19,66716
S2	22,32877	77,67123
Bottoms	46,45513	53,54487

Table 14: Samples from the second run

	MeoH	EtOH
Dist.	86,75458	13,24542
S1	75,76138	24,23862
S2	20,75281	79,24719
Bottoms	3,53254	96,46746

The results are given in molarfractions.

As can be seen in Table 13 and Table 14, the difference between the two runs is large. Especially for the distillate and bottoms there is a significant difference. The compositions of the distillate and bottoms also doesn't seem correct, since the temperature in the top was 66°C, which is the boiling point of pure methanol. The temperature in the bottom was 80°C, which is the boilingpoint of ethanol. Remember there is some bias in the measurements, which explains the slight difference from the real boilingpoints. The values of the distillate and the bottoms are thus not used.

The compositions of S1 and S2 are quite equal to each other. These values will be used to determine the number of trays in one section. When this number is divided by the height of this section, an estimation for the other sections can be done, using the Height Equivalent Theoretical Plate (H.E.T.P.)

10.4 Determination of the number of trays and H.E.T.P.

The number of trays can be determined using the Fenske equation:

$$n = \frac{\ln\left[\left(\frac{x_D}{1 - x_D}\right) \cdot \left(\frac{1 - x_B}{x_B}\right)\right]}{\ln \beta}$$

In this formula, n is the number of trays and includes the reboiler. The number of tray (without reboiler) is thus n-1

The relative volitality of the methanol-ethanol mixture is 1,717. The number of trays between S1 and S2 can be determined:

10.4.1 First run

$$n = \frac{\ln\left[\left(\frac{0{,}8033284}{1 - 0{,}8033284}\right) \cdot \left(\frac{1 - 0{,}2232877}{0{,}2232877}\right)\right]}{\ln 1{,}717}$$

n = 4,904

n-1= 3,904

10.4.2 Second run

$$n = \frac{\ln\left[\left(\frac{0{,}7576138}{1-0{,}7576138}\right)\cdot\left(\frac{1-0{,}2075281}{0{,}2075281}\right)\right]}{\ln 1{,}717}$$

n = 4,587

n-1= 3,587

The average of the first and second run is 3,745 plates.

10.4.3 H.E.T.P. and number of trays in the other sections

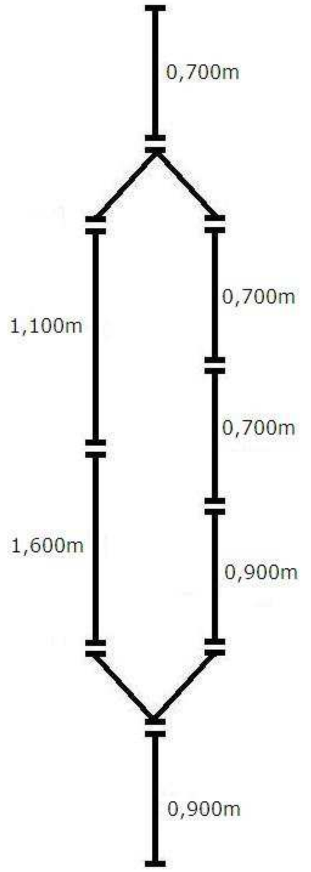


Figure 20: Height of the sections

The height of the section between S_1 and S_2 is 0,7 metres. The H.E.T.P. of this section is thus:

$$\text{H.E.T.P.} = \frac{0{,}700\text{m}}{3{,}745\ \text{plates}}$$

H.E.T.P. = 0,187m/plate

With this value, the number of tray in the other sections can be calculated easy with nex formula:

$$n = \frac{\text{height (m)}}{0{,}187\text{m/plate}}$$

For the sections with a height of 0,900 metres, the number of trays becomes:

n = 4,813 plates

For the section with a height of 1,100m, the number of trays becomes:

n = 5,882 plates

For the section with a height of 1,600m, the number of trays is:

n = 8,556

The main column contains: ((3*3,745)+(2*4,813)) = 20,051 plates, from bottom to top. The same calculation can be done for the prefractionator: (5,882+8,556) = 14,438 plates.

10.5 Discussion

The number of plates, as determined above, is not an integer number. When this experiment is repeated, there will be some difference in the result, but the number should be in the same range.

These numbers also explain why the liquid split is not 0,5, but 0,42. The number of trays in the prefractionator is about 14, but the number of trays in the same part from the main column is (3,745*2)+4,813 = 12,303. This section of the main column has thus fewer plates, which means fewer resistance. Vapour will try to follow the path of the least resistance and more vapour will go through the main column in stead of through the prefractionator.

10.6 Conclusion

The number of trays in the main column is about 20 plates. The prefractionator has 14 or 15 plates. The R_v ratio is about 0,42. 42% of the vapour goes into the prefractionator.

11 INFLUENCE OF THE VAPOUR SPLIT ON PRODUCT PURITIES

The vapour split divides the vapour between the prefractionator and the main column. If the vapour split is decreased, less vapour enters the prefractionator. This should reduce the amount of propanol in the upper section of the prefractionator. On the other hand, more heat will be sent in the main column, causing heavy fractions to go up in the column. The valves will need to be closed more to maintain the temperature profile. This could lead to flooding in the column when not enough products can be drained off.

11.1 About the simulations

The simulations help to get a better insight in what happens in the column when a change is made. For every simulation, three charts are shown. The first two charts show the composition profiles in the prefractionator and the main column; the thirth chart shows the temperature profile in the prefractionator and the main column. In the two tables, which can be found with each simulation, the inputs and results are shown.

The simulations are done with a model that has not been adapted to the results of determination of the number of trays, since this experiment was done at the end of the internship.

11.2 Basecase

11.2.1 Tables and discussion

Table 15: Inputs and disturbances

Degrees of freedom		stage efficiency	
product D (Td) T33	75,9699°C	section	alpha
product S_1 (Ts1) T60	88,7960°C	1	0
product S_2 (Ts2) T49	98,6284°C	2	0
bottoms (Vapour boil-up)	2,3000mol/min	3	0
prefractionator (R_L) T15	85,4560°C	4	0
R_V	0,3770	5	0
R_L	0,2885	6	0
		7	0

Table 16: Results

	%A	%B	%C	%D	flow (mol/min)
F	18	23	30	29	1,0000
D	79,93	20,07	0,00	0,00	0,2044
S_1	0,43	80,64	18,93	0,00	0,1981
S_2	0,10	8,39	91,06	0,45	0,2753
B	0,00	0,01	17,93	82,06	0,3222
				TOTAL	1,0000

A	methanol
B	ethanol
C	propanol
D	butanol

This is the base case for this set of simulations. The final input values and composition results are shown in Table 15 and Table 16.

Chart 13 and Chart 14 show the composition profiles in the prefractionator and main column. Chart 15 shows the temperature profiles in the main column and the prefractionator.

11.2.2 Charts

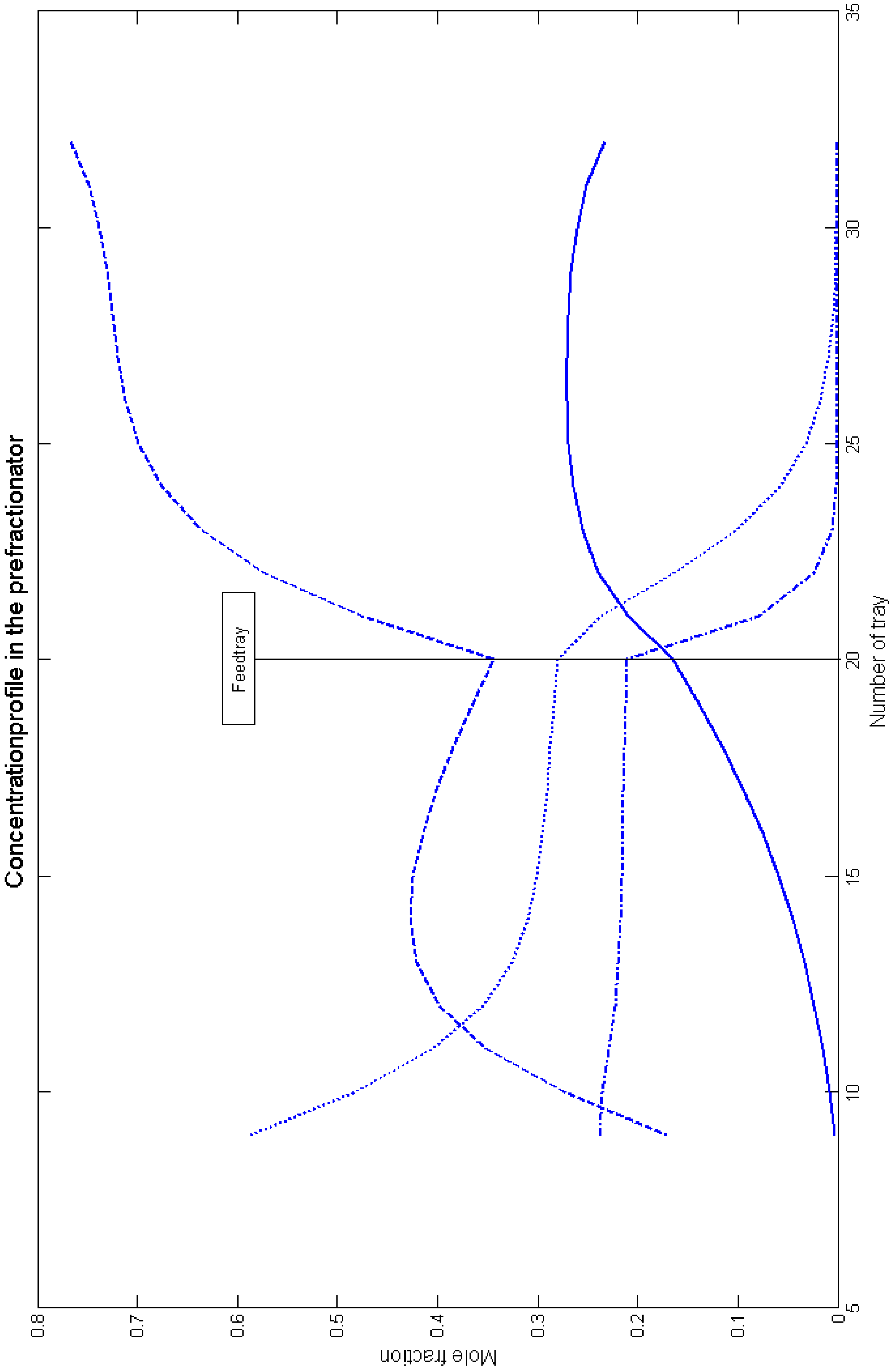


Chart 13: Basecse - composition in prefractionator

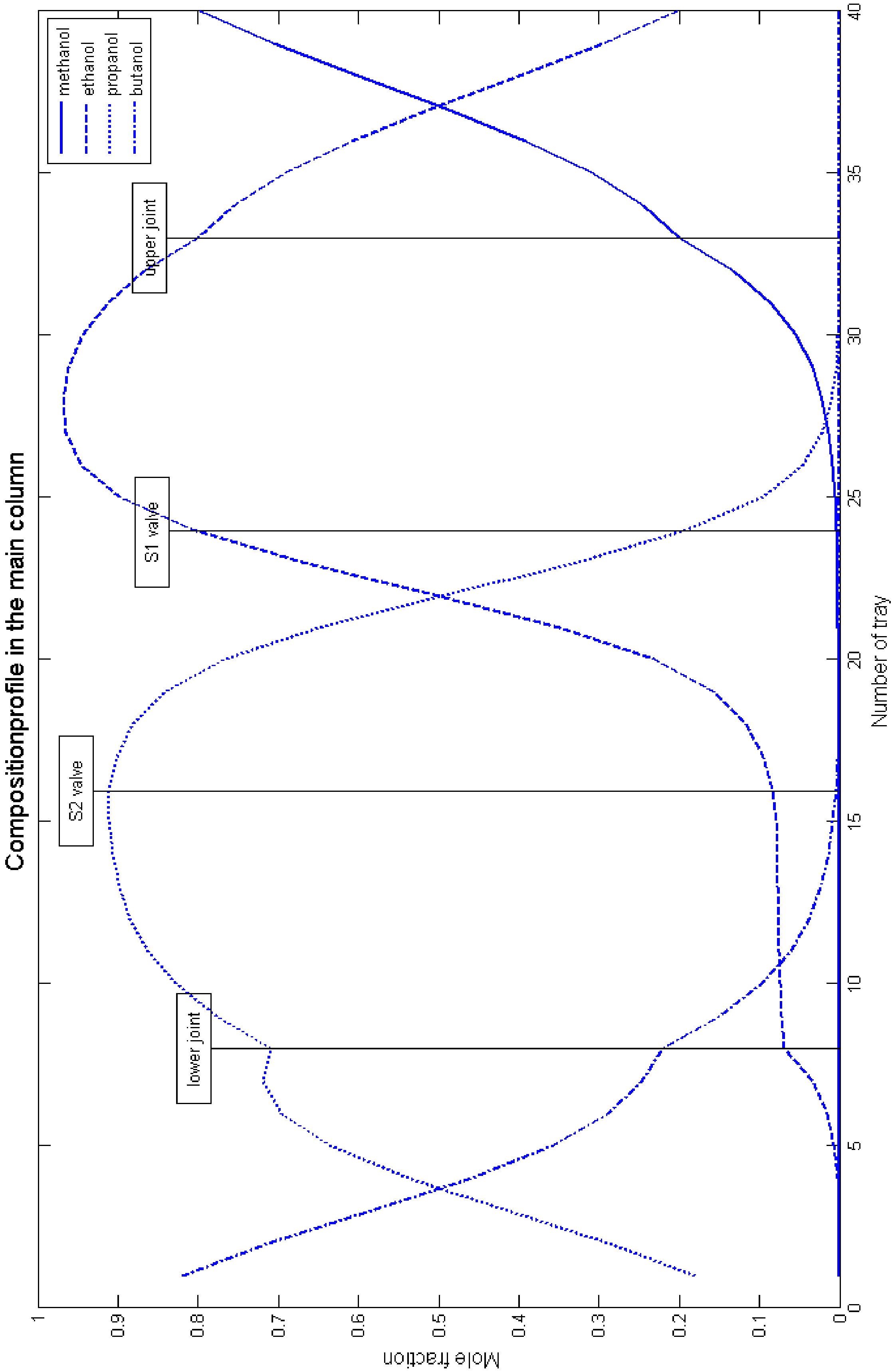


Chart 14: basecase - compostion in main column

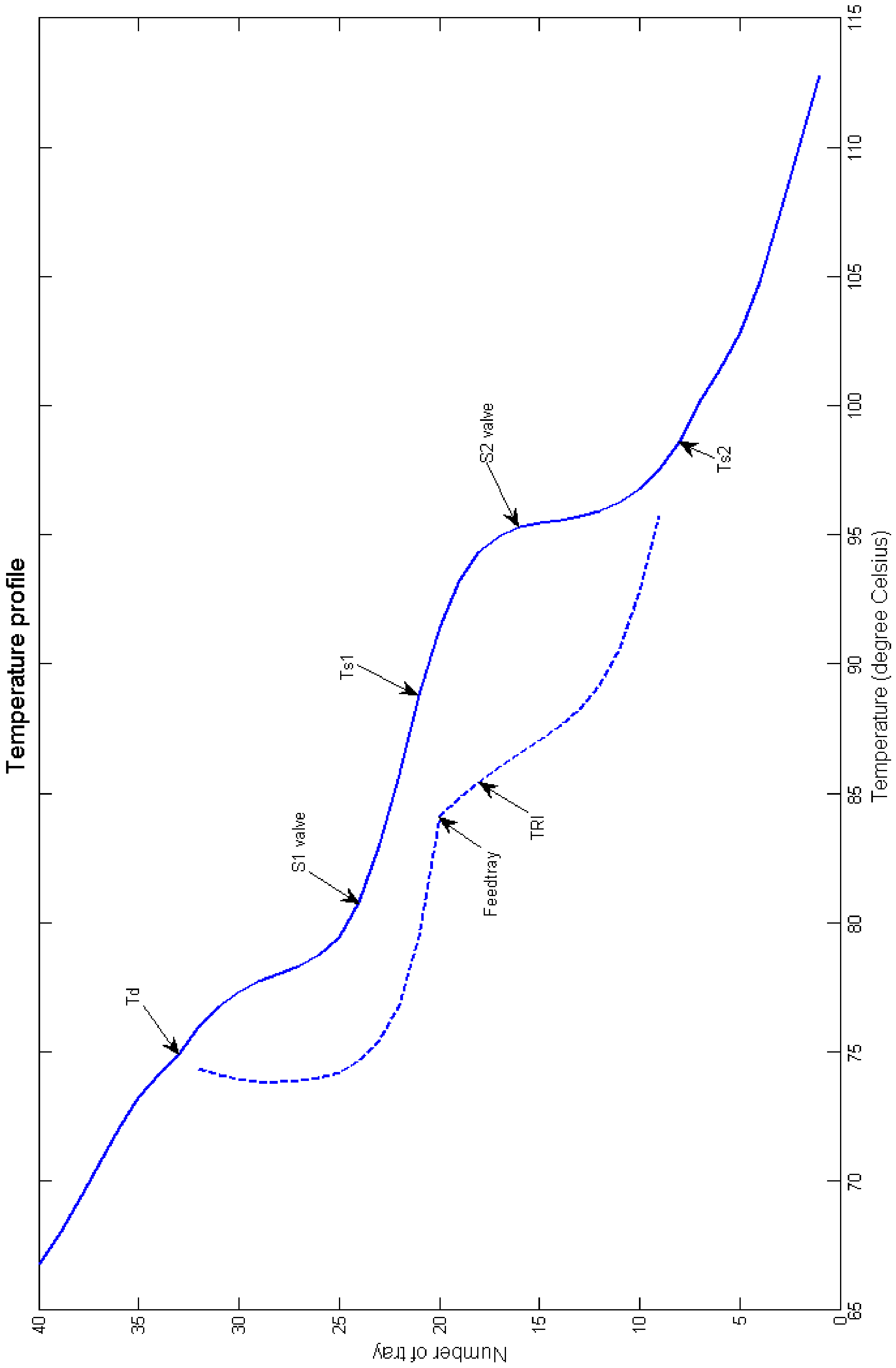


Chart 15: Basecase - temperature profile

11.3 R_v ratio -1%

11.3.1 Charts

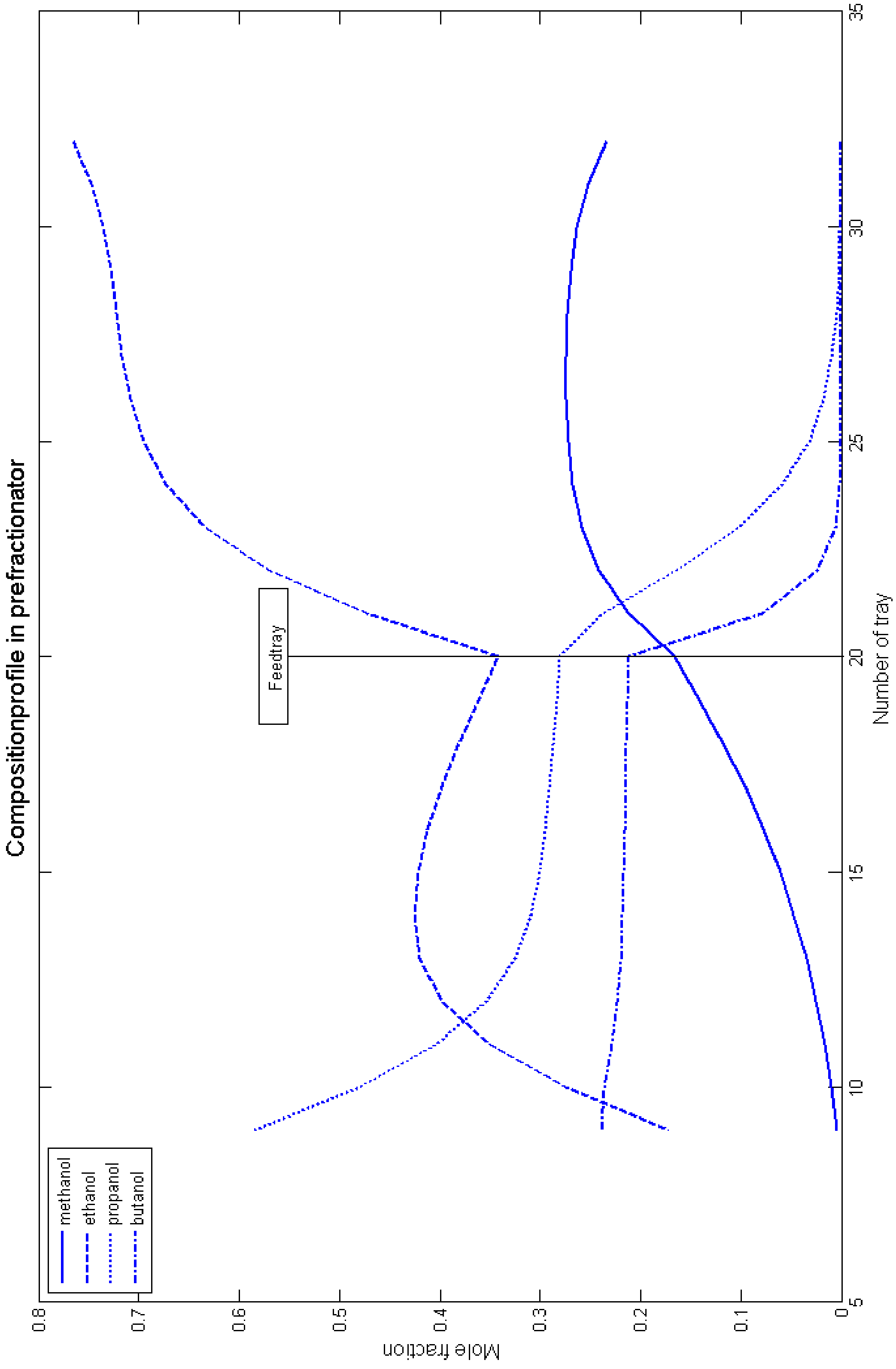


Chart 16: Rv-1% - Composition in prefractionator

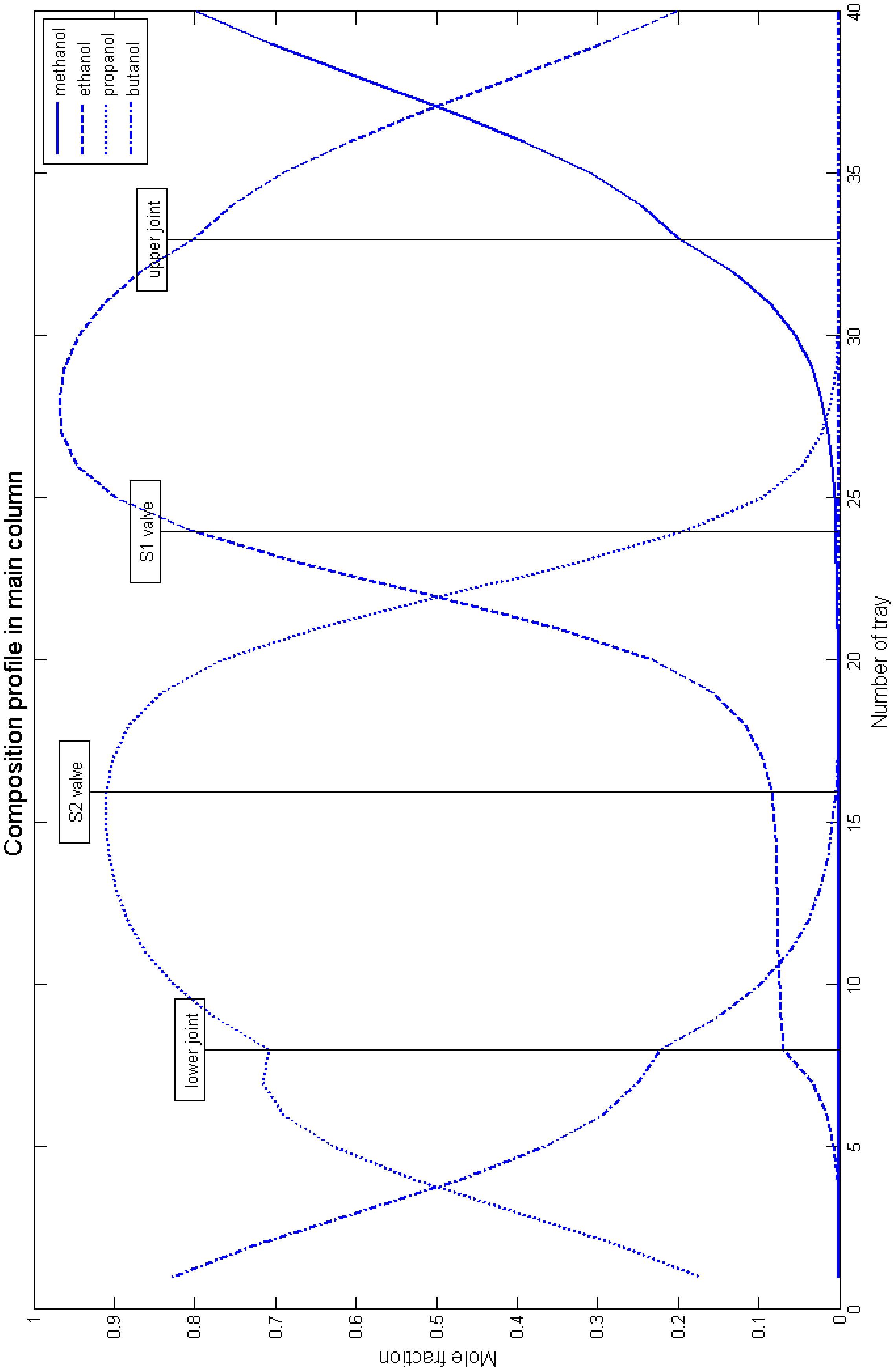


Chart 17: Rv-1% - composition in main column

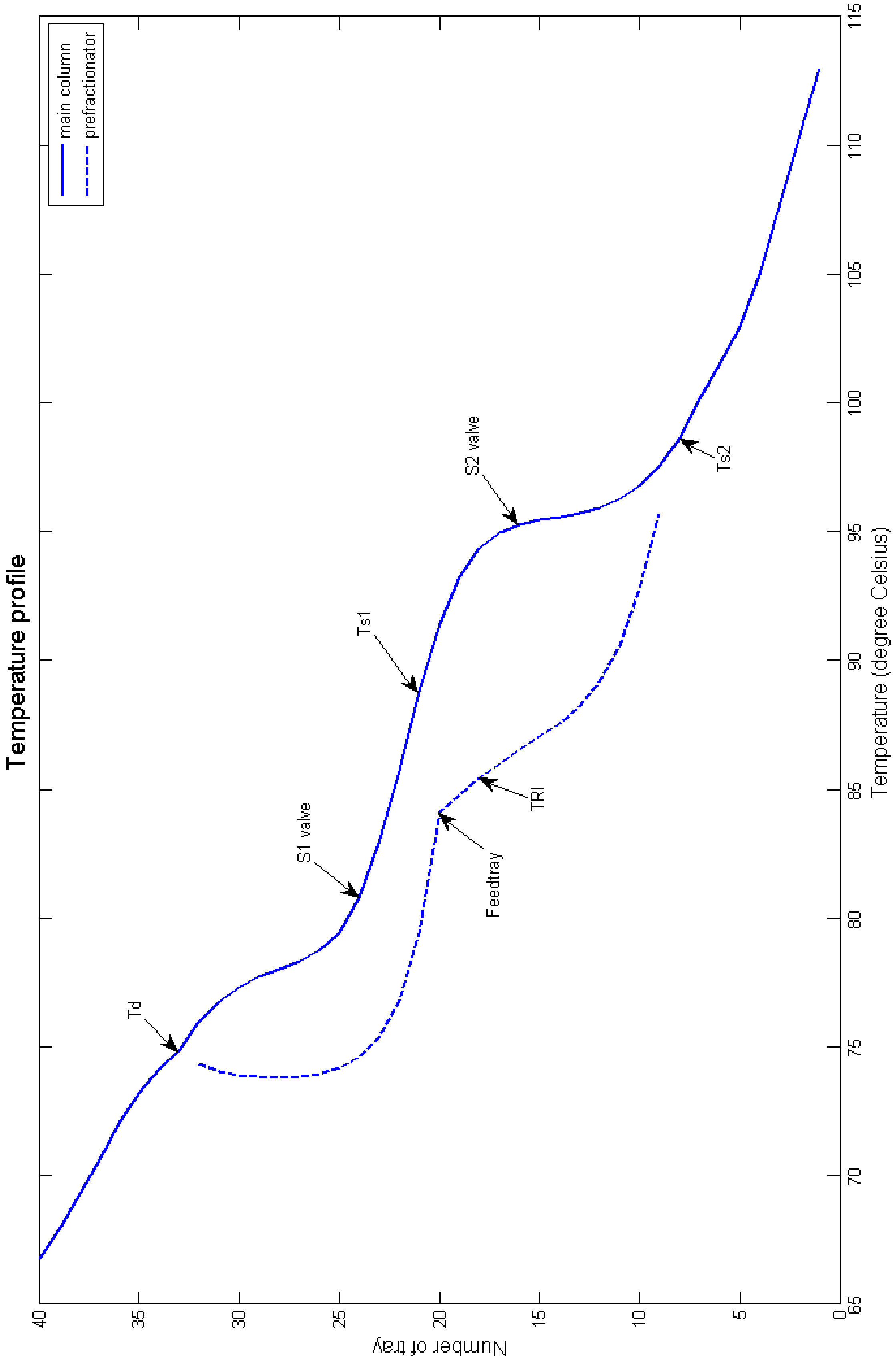


Chart 18: Rv-1% - temperature profile

11.3.3 Tables and discussion

Table 17: Inputs and disturbances

Degrees of freedom	setpoint	stage efficiency	
product D (Td) T33	75,9697°C	section	alpha
product S_1 (Ts1) T60	88,7951°C	1	0
product S_2 (Ts2) T49	98,6457°C	2	0
bottoms (Vapour boilup)	2,3000mol/min	3	0
prefractionator (R_L) T15	85,4322°C	4	0
R_V	0,3732	5	0
R_L	0,2851	6	0
		7	0

Table 18: Results

	%A	%B	%C	%D	flow (mol/min)
F	18	23	30	29	1,0000
D	79,96	20,04	0,00	0,00	0,2043
S_1	0,44	80,59	18,97	0,00	0,1980
S_2	0,11	8,41	91,02	0,46	0,2772
B	0,00	0,01	17,15	82,84	0,3205
				TOTAL	1,0000

A	methanol
B	ethanol
C	propanol
D	butanol

As can be seen in Table 17, the vapour split ratio was reduced by 1%. When the product compositions (Chart 16 and Chart 17) are compared to those in the base case, no remarkable difference can be noticed. Also Chart 18 looks similar to the one from the basecase.

11.4 RV ratio -2%

11.4.1 Charts

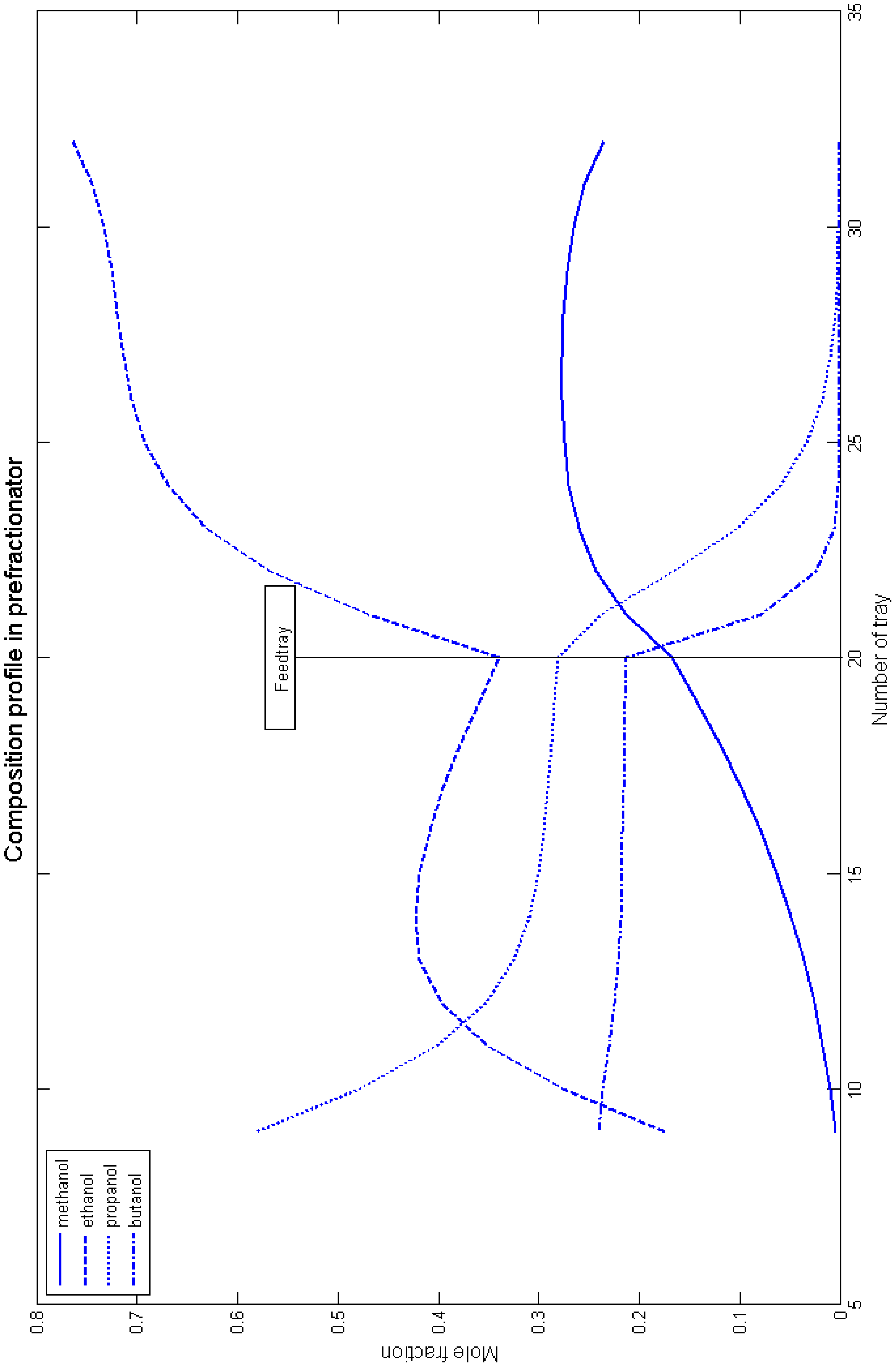


Chart 19: Rv-2% - composition in prefractionator

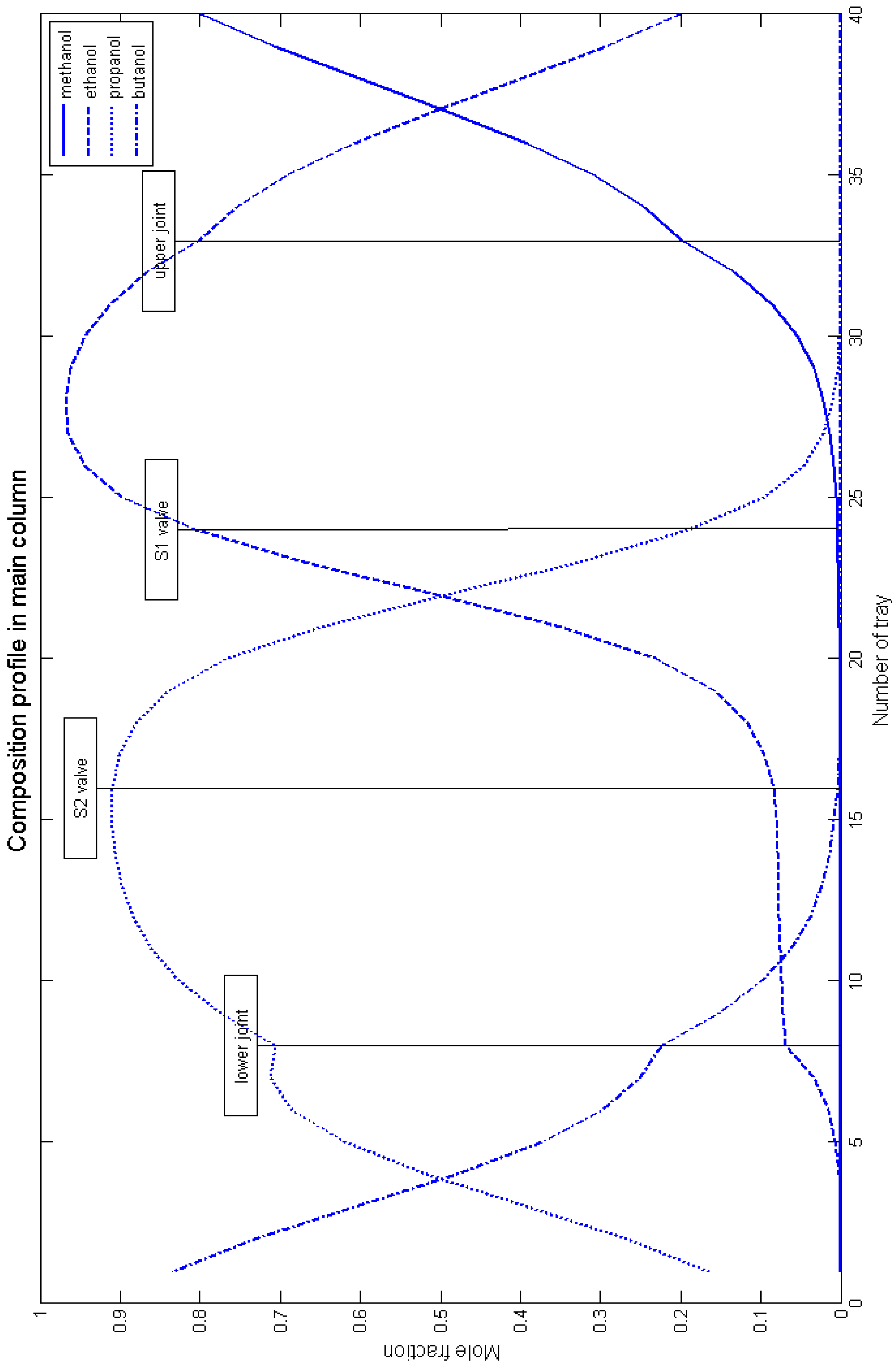


Chart 20: Rv-2% - composition in main column

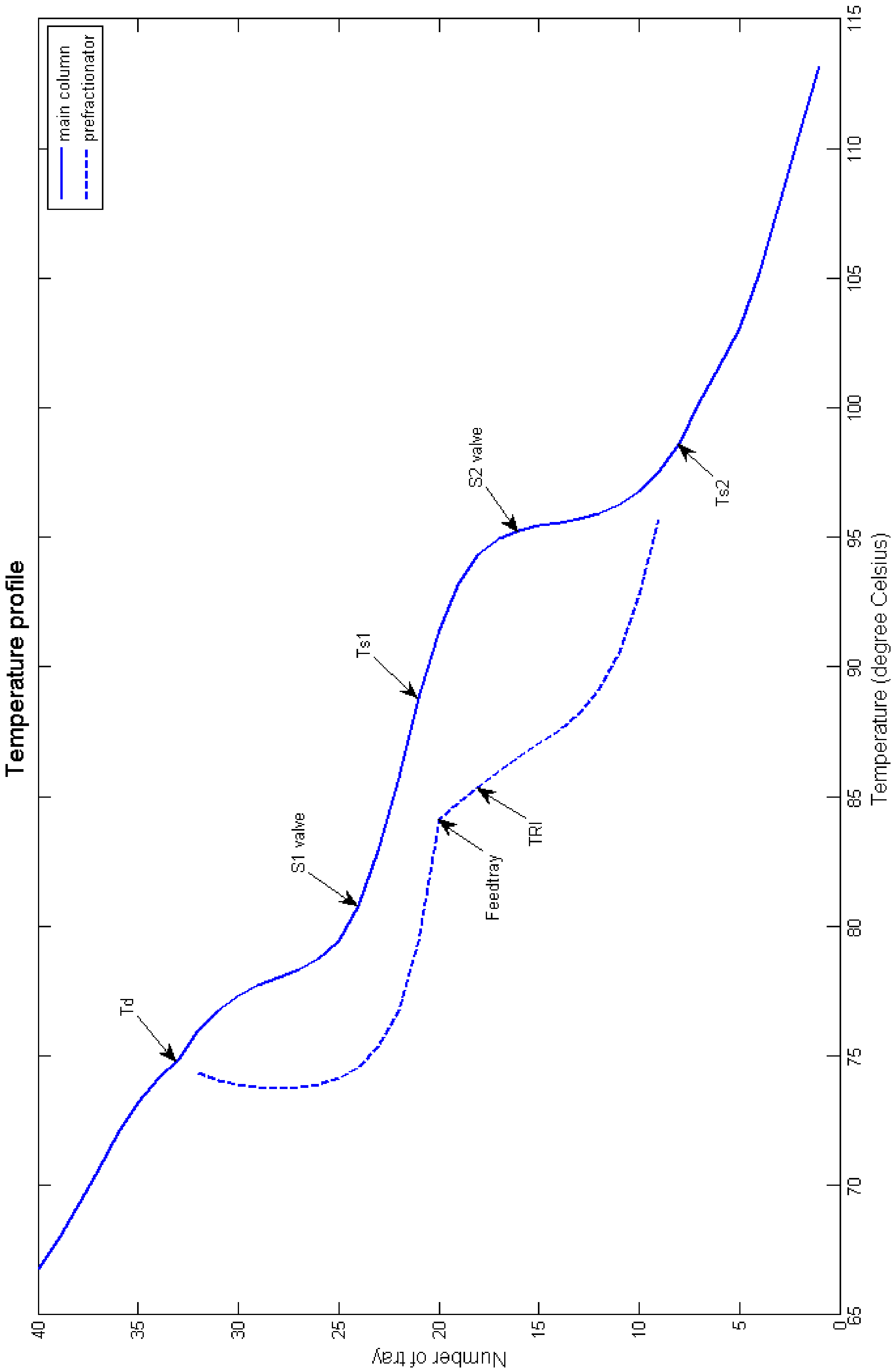


Chart 21: Rv-2% - temperature profile

11.4.2 Tables and discussion

Table 19: Inputs and disturbances

Degrees of freedom	setpoint	stage efficiency	
		section	alpha
product D (Td) T33	75,9695°C	1	0
product S_1 (Ts1) T60	88,7943°C	2	0
product S_2 (Ts2) T49	98,6621°C	3	0
bottoms (Vapour boilup)	2,3000mol/min	4	0
prefractionator (R_L) T15	85,4087°C	5	0
R_V	0,3695	6	0
R_L	0,2817	7	0

Table 20: Inputs and disturbances

	%A	%B	%C	%D	flow (mol/min)
F	18	23	30	29	1,0000
D	79,99	20,01	0,00	0,00	0,2042
S_1	0,46	80,54	19,00	0,00	0,1979
S_2	0,11	8,43	90,99	0,47	0,2788
B	0,00	0,01	16,48	83,51	0,3191
				TOTAL	1,0000

A	methanol
B	ethanol
C	propanol
D	butanol

When table Table 19 and Table 20 are compared with the same tables from the basecase, there is as good as non difference between the two cases. Chart 19, Chart 20 and Chart 21 are almost identical to the charts from the basecase. A small decrease of 1 or 2% doesn’t have an influence on the purity of the products.

11.5 R_V ratio -5%

11.5.1 Charts

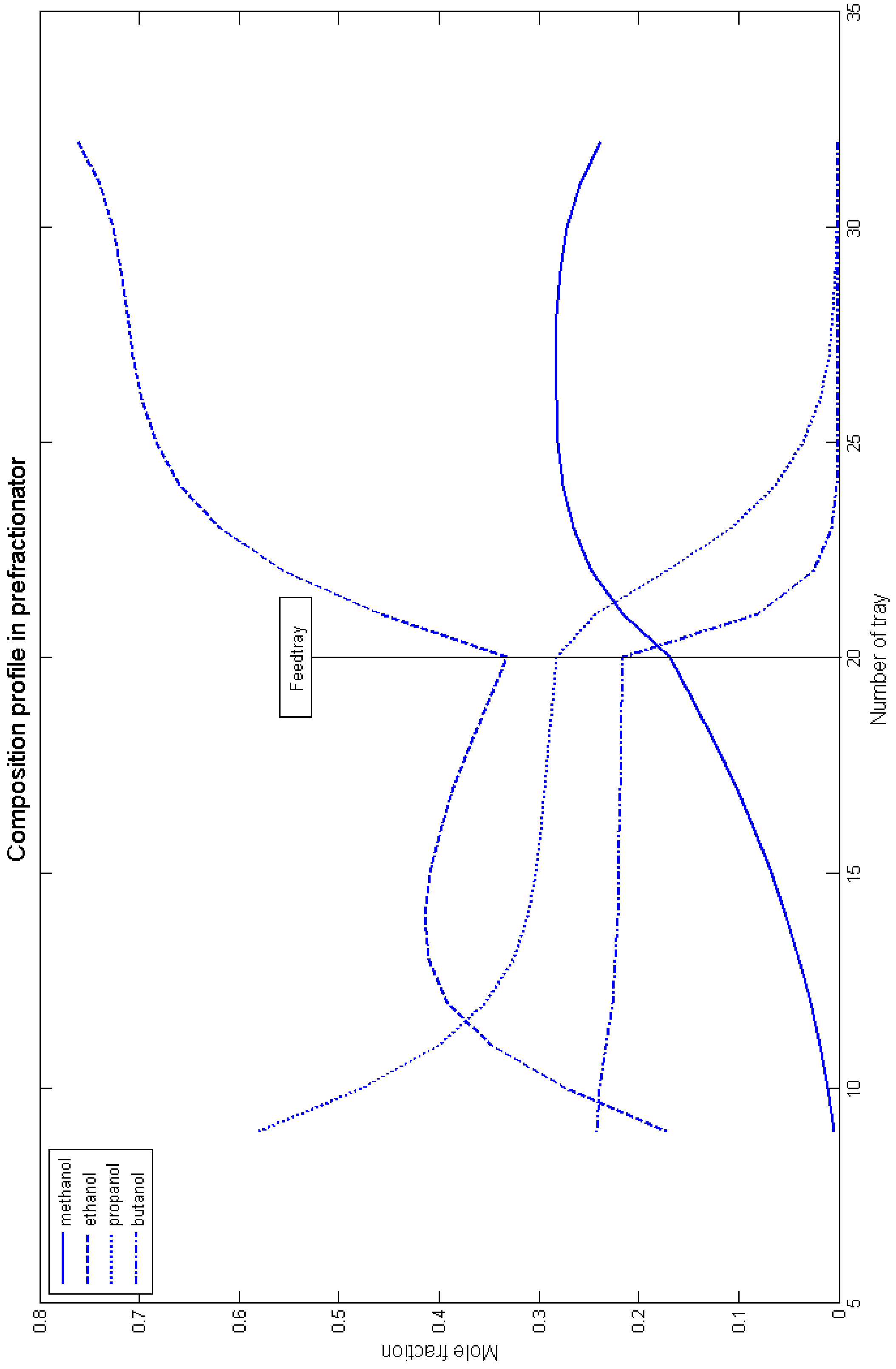


Chart 22: Rv-5% - composition in prefractionator

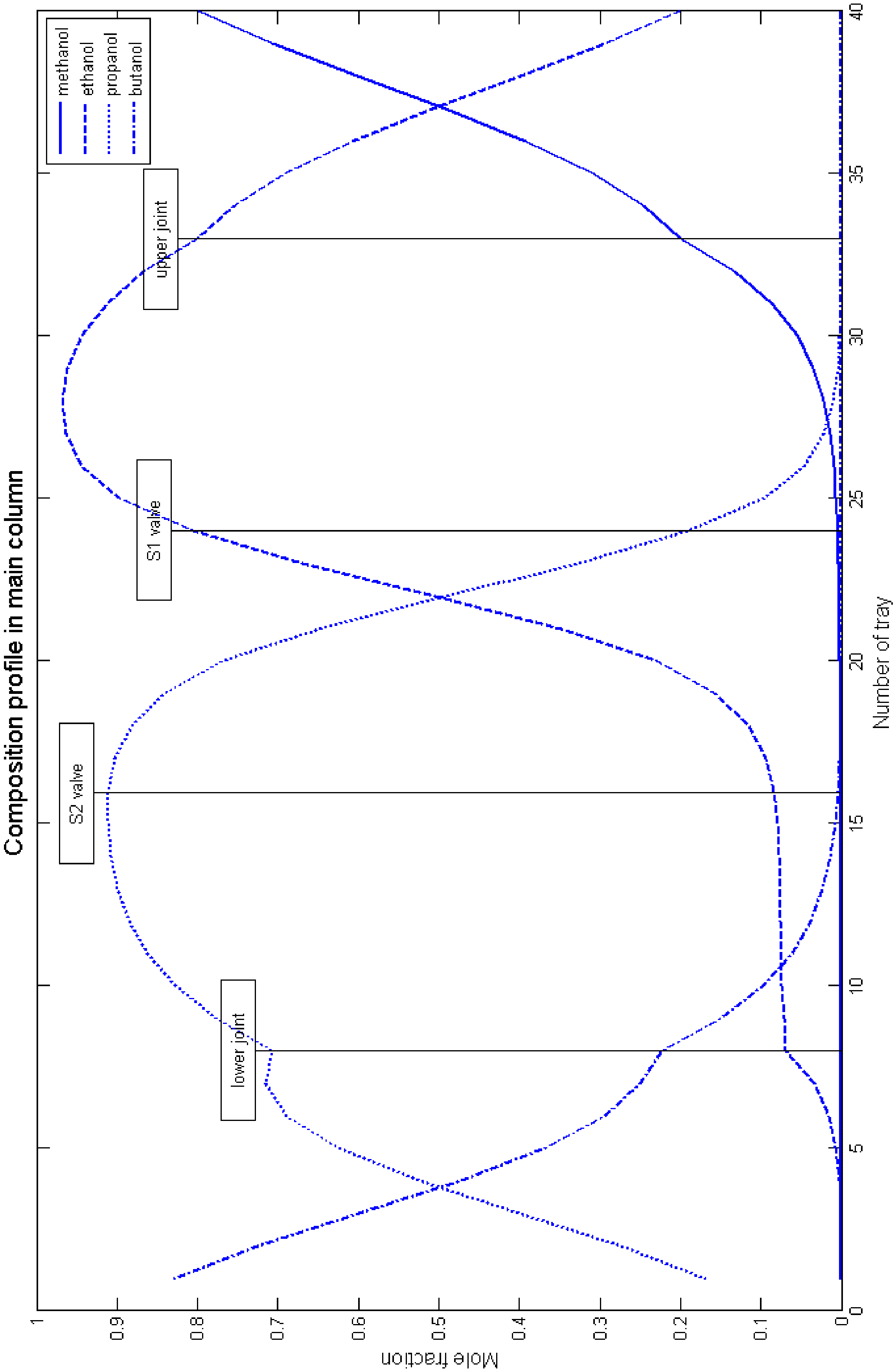


Chart 23: Rv-5% - composition in main column

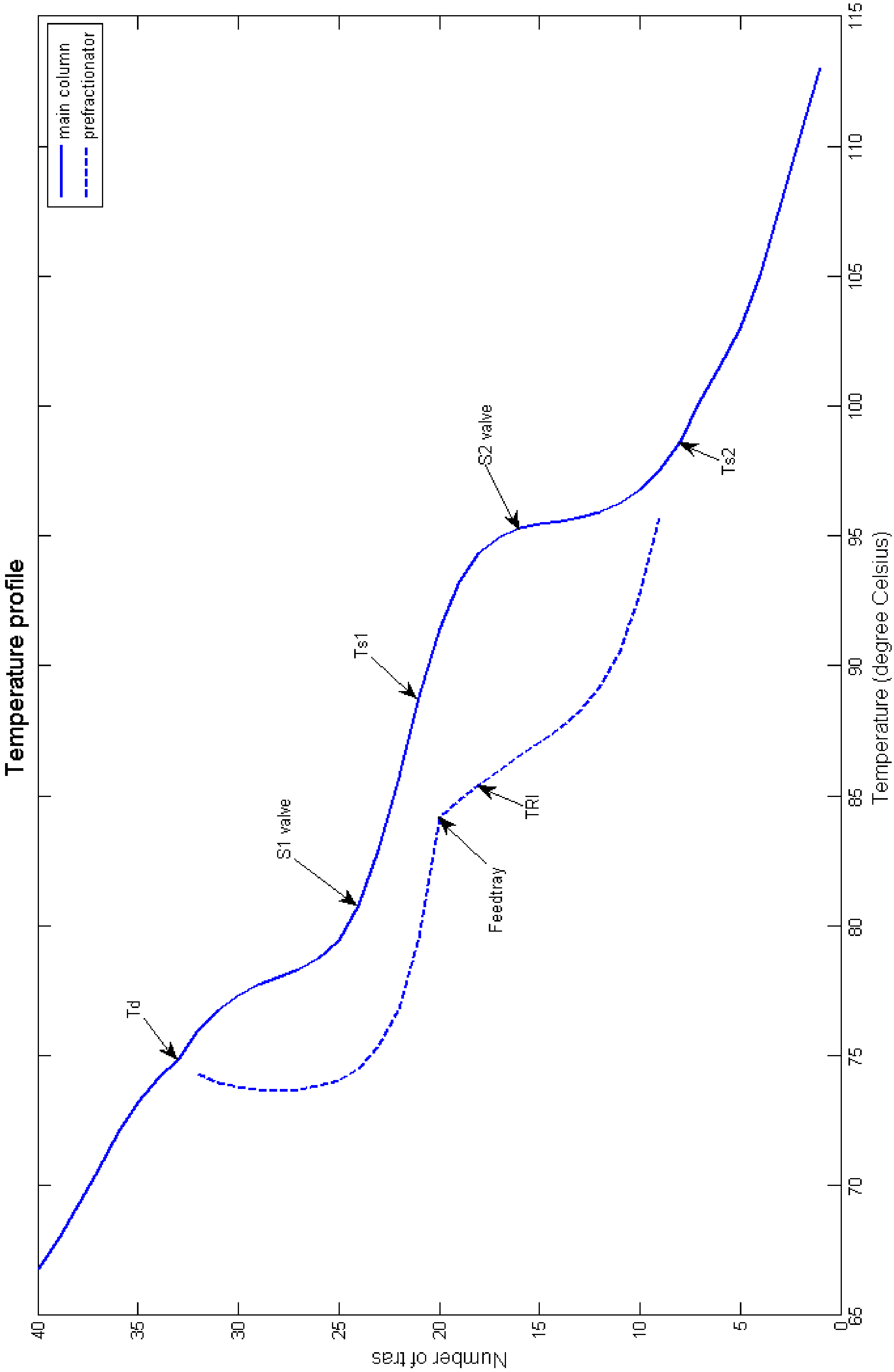


Chart 24: Rv-5% - Temperature profile

11.5.2 Tables and discussion

Table 21: Inputs and disturbances

Degrees of freedom	setpoint	stage efficiency	
product D (Td) T33	75,9697°C	section	alpha
product S_1 (Ts1) T60	88,7945°C	1	0
product S_2 (Ts2) T49	98,6645°C	2	0
bottoms (Vapour boilup)	2,3000mol/min	3	0
prefractionator (R_L) T15	85,4336°C	4	0
R_V	0,3582	5	0
R_L	0,2696	6	0
		7	0

Table 22: Results

	%A	%B	%C	%D	flow (mol/min)
F	18	23	30	29	1,0000
D	80,00	20,00	0,00	0,00	0,2041
S_1	0,49	80,53	18,98	0,00	0,1984
S_2	0,13	8,34	91,09	0,44	0,2773
B	0,00	0,01	16,96	83,03	0,3202
				TOTAL	1,0000

A	methanol
B	ethanol
C	propanol
D	butanol

The compositions remain in the same range (Table 21 and 22). The biggest change with the base case is that the purity of the bottoms increases with 1%. Also Chart 22, Chart 23 and Chart 24 remain equal to the base case.

As can be seen in Chart 23, the temperature profile in the column is not optimal. Between the temperature measurement for the S1 control loop and the S1 valve is a large temperature drop. On Chart 23, this results in a concentration peak for ethanol above the valve. Which means that the temperature setpoint is not correct.

11.6 R_V ratio -10%

11.6.1 Charts

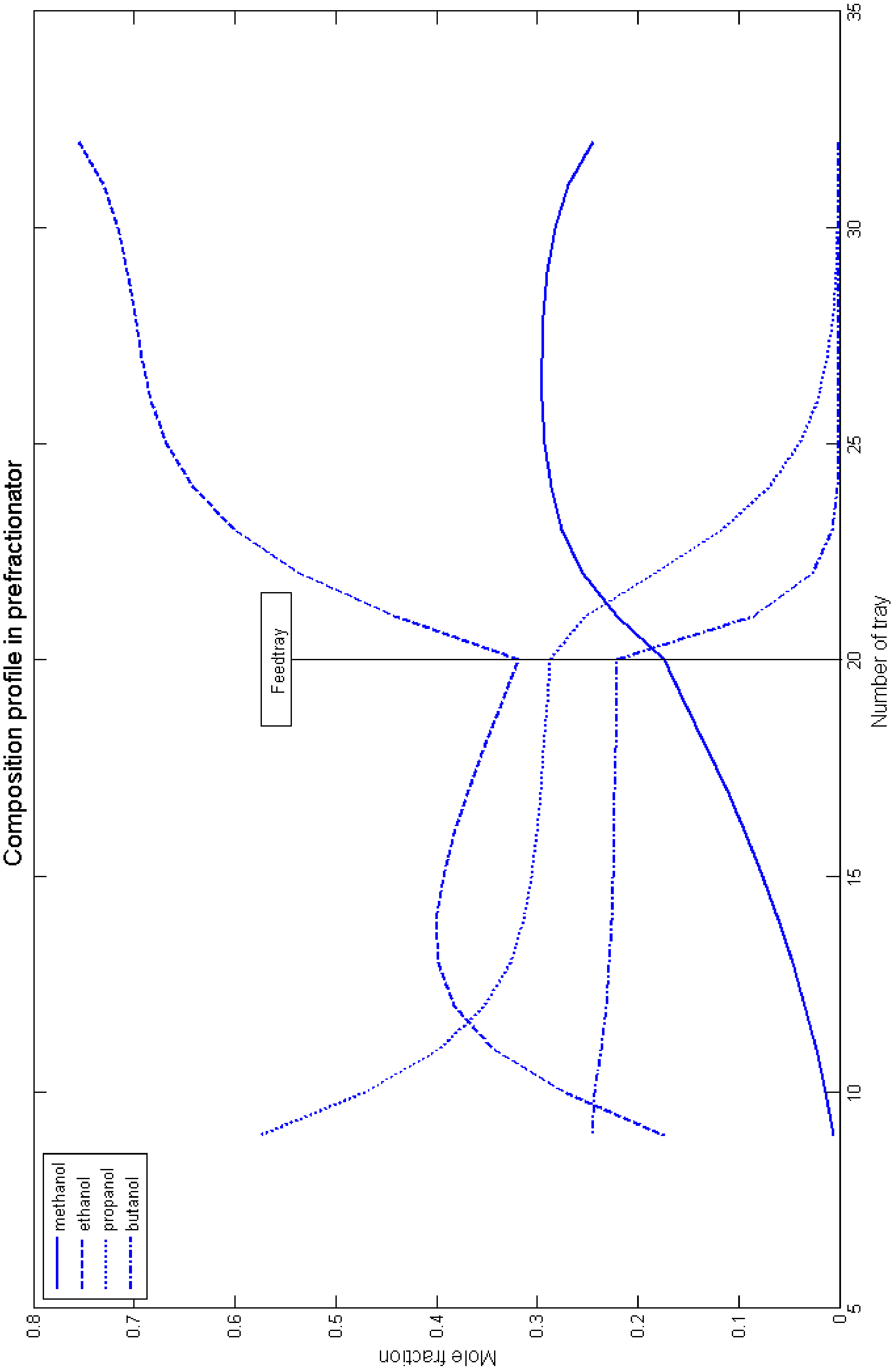


Chart 25: Rv-10% - composition in prefractionator

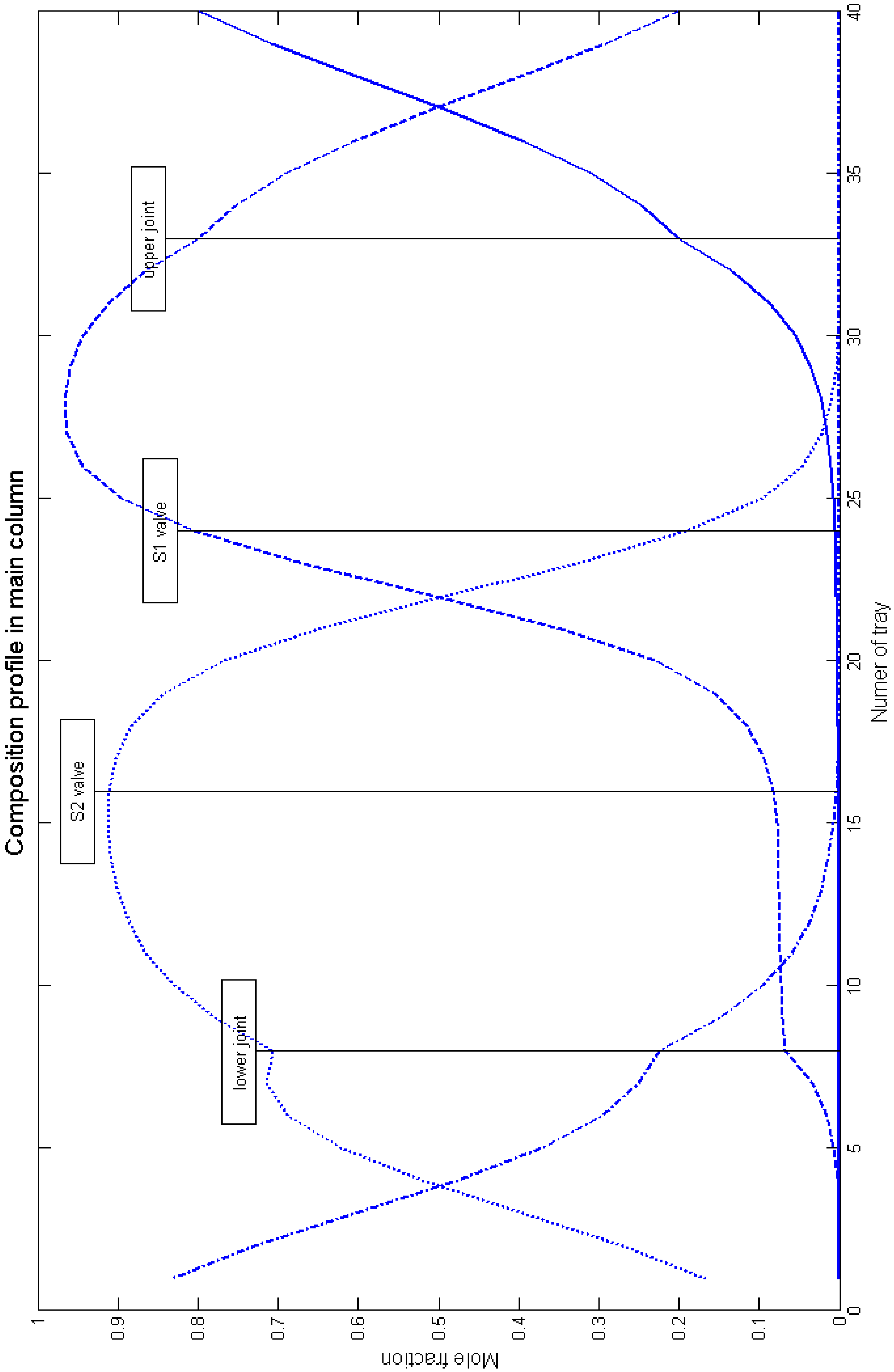


Chart 26: Rv-10% - composition in main column

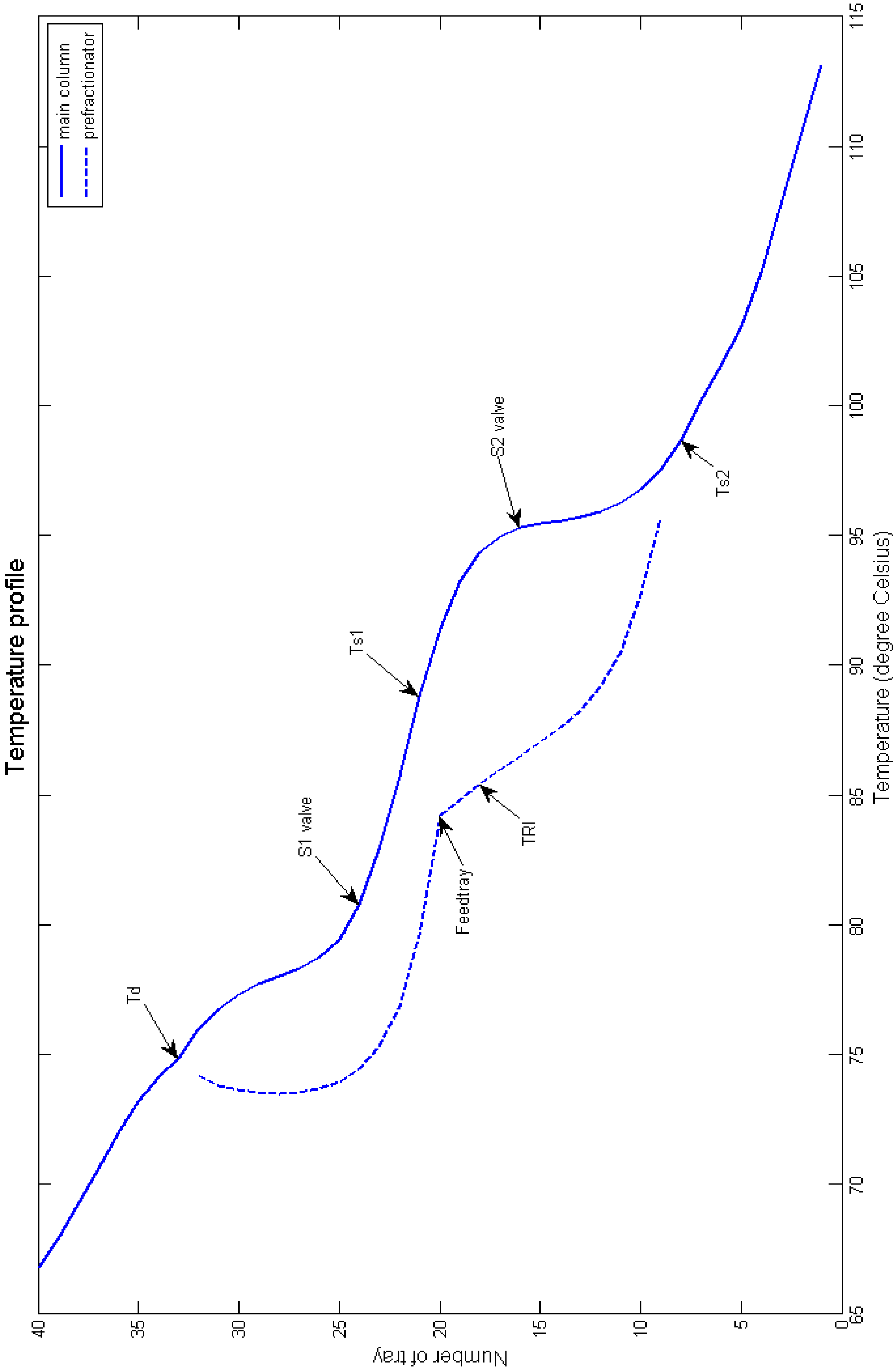


Chart 27: Rv-10% - temperature profile

11.6.2 Tables and discussion

Table 23: Inputs and disturbances

Degrees of freedom	setpoint	stage efficiency	
product D (Td) T33	75,9696°C	section	alpha
product S_1 (Ts1) T60	88,7937°C	1	0
product S_2 (Ts2) T49	98,6857°C	2	0
bottoms (Vapour boilup)	2,3000mol/min	3	0
prefractionator (R_L) T15	85,4149°C	4	0
R_V	0,3393	5	0
R_L	0,2503	6	0
		7	0

Table 24: Results

	%A	%B	%C	%D	flow (mol/min)
F	18	23	30	29	1,0000
D	80,03	19,97	0,00	0,00	0,2038
S_1	0,56	80,43	19,01	0,00	0,1990
S_2	0,15	8,26	91,17	0,42	0,2776
B	0,00	0,01	16,68	83,31	0,3196
				TOTAL	1,0000

A	methanol
B	ethanol
C	propanol
D	butanol

As can be seen in Table 23, the temperature setpoints remain equal to the base case, which is normal. Also in Table 24 is not much difference. The only major change is that the concentration of butanol in the bottoms has increased a bit, compared to the previous case. The charts (25, 26 and 27) remain equal.

11.7 R_V ratio -20%

11.7.1 Charts

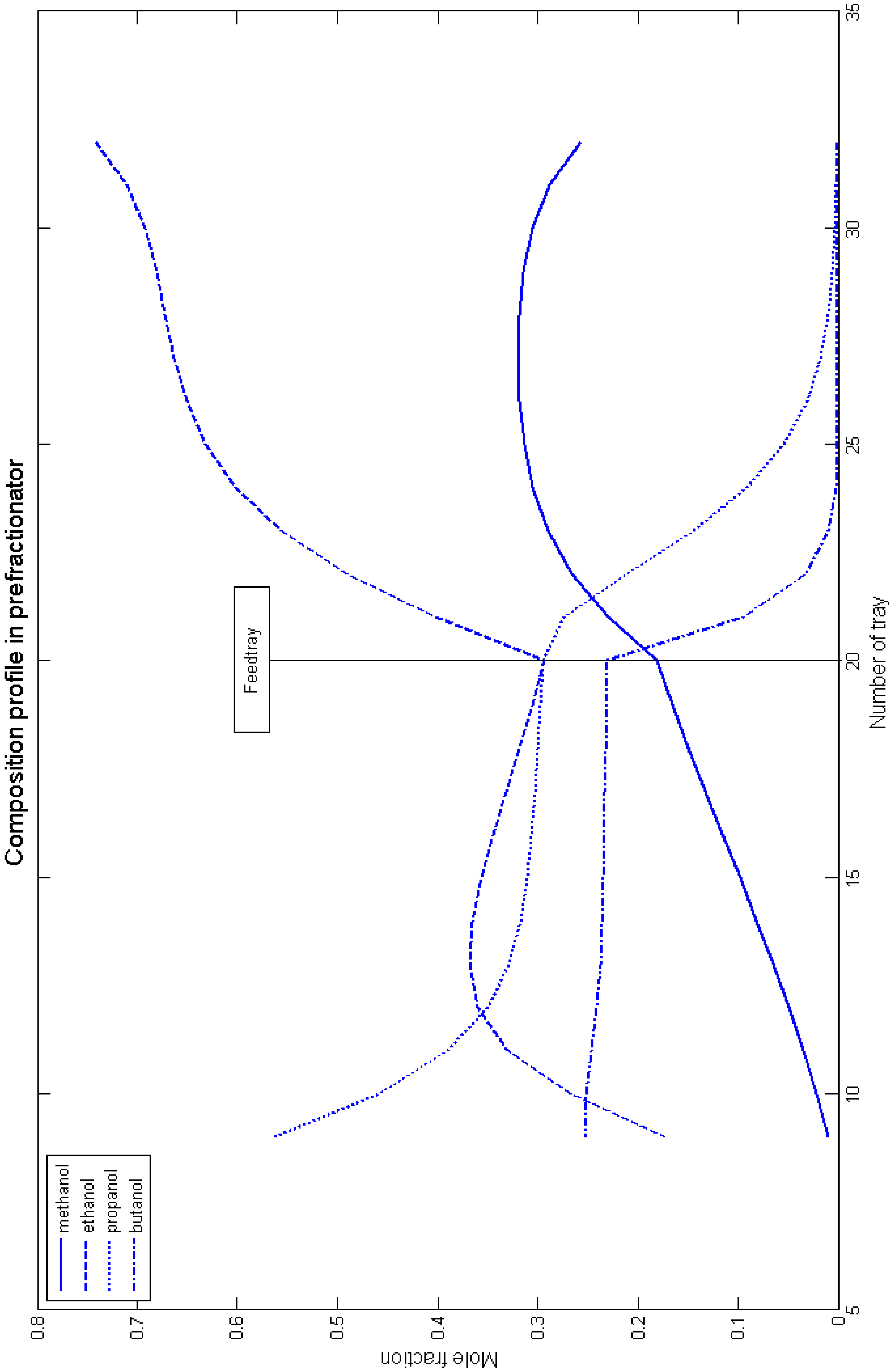


Chart 28: Rv-20% - composition in prefractionator

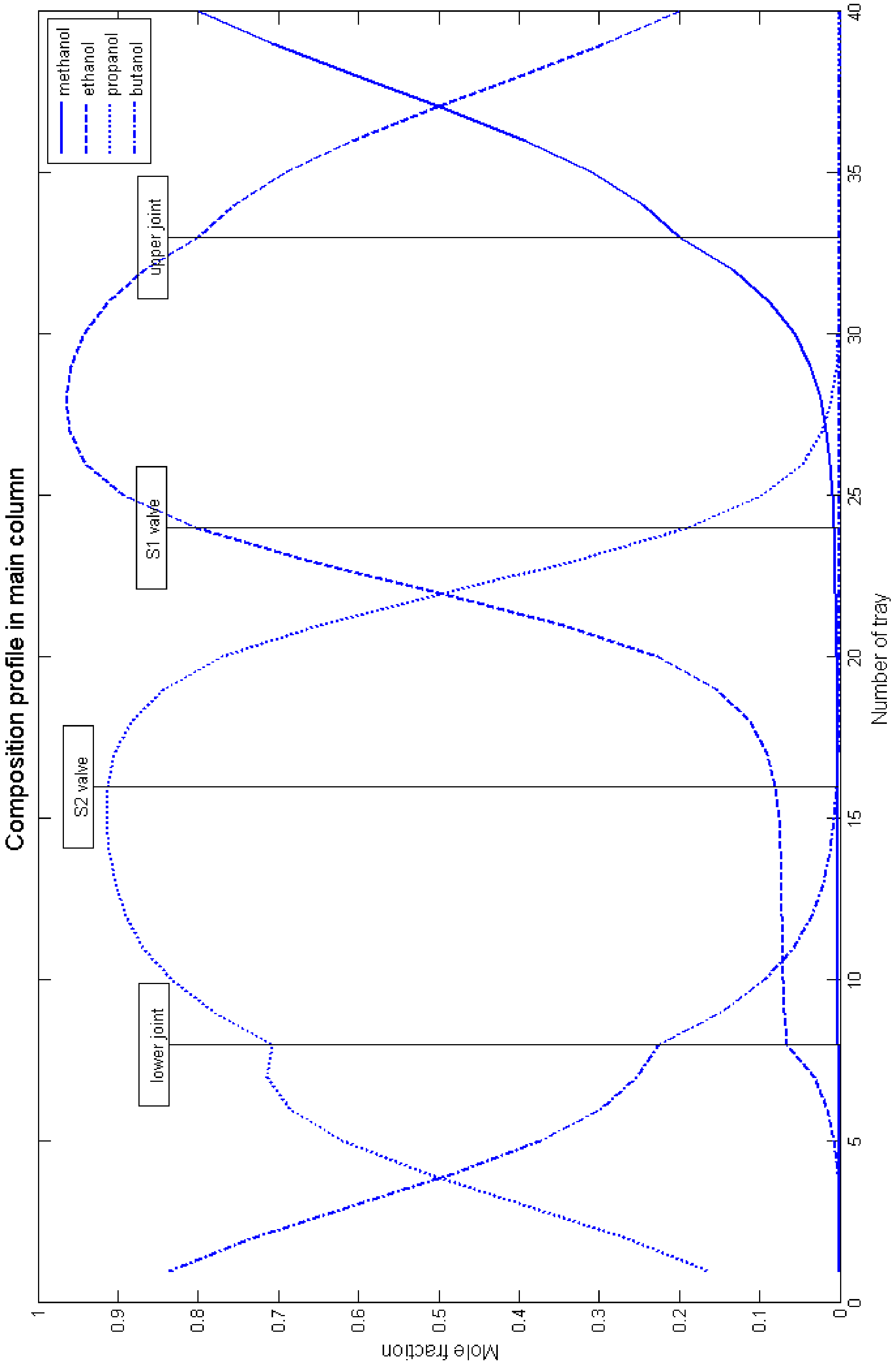


Chart 29: Rv-20% - composition in main column

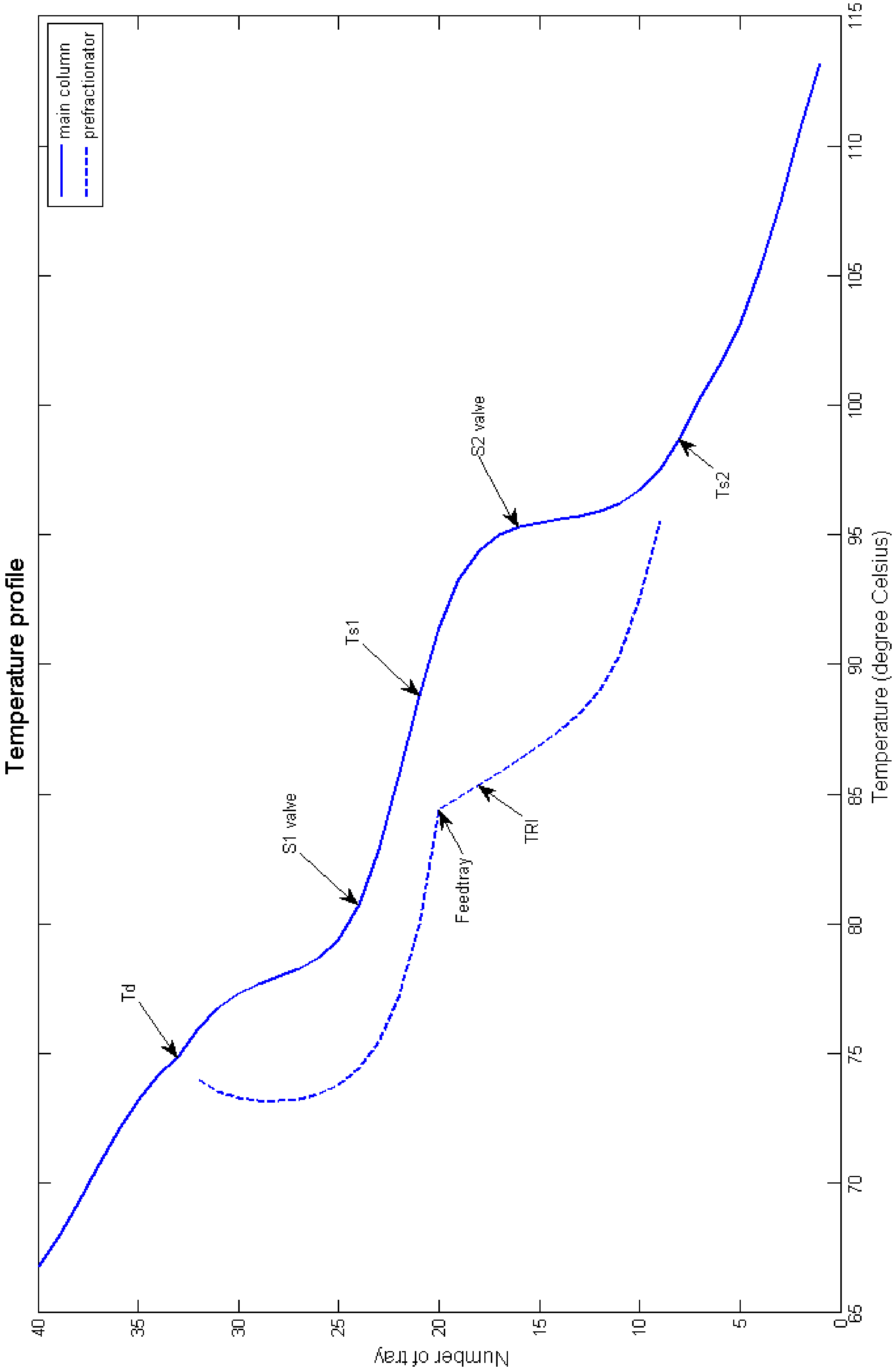


Chart 30: Rv-20% - Temperature profile

11.7.2 Tables and discussion

Table 25: Inputs and disturbances

Degrees of freedom	setpoint	stage efficiency	
product D (Td) T33	75,9690°C	section	alpha
product S_1 (Ts1) T60	88,7924°C	1	0
product S_2 (Ts2) T49	98,7235°C	2	0
bottoms (Vapour boilup)	2,3000mol/min	3	0
prefractionator (R_L) T15	85,3645°C	4	0
R_V	0,3016	5	0
R_L	0,2116	6	0
		7	0

Table 26: Results

	%A	%B	%C	%D	flow (mol/min)
F	18	23	30	29	1,0000
D	80,02	19,98	0,00	0,00	0,2030
S_1	0,77	80,18	19,05	0,00	0,2005
S_2	0,24	8,01	91,36	0,39	0,2740
B	0,00	0,01	16,36	83,63	0,3191
				TOTAL	0,9966

A	methanol
B	ethanol
C	propanol
D	butanol

Tables 25 and 26 remain equal, only the concentration of butanol in the bottoms has increased a bit more. The charts remain almost equal. In Chart 28, the concentration of methanol in the lower section of the prefractionator decreases linear, where in the base case, the increase is more parabolic. This can be an indication that not enough heat is sent in the prefractionator. The concentration of ethanol in the lower section becomes lower. This is probably also an indication that the amount of heat in the prefractionator becomes to little. Chart 29 and Chart 30 remain equal to the basecase.

11.8 R_V ratio -30%

11.8.1 Charts

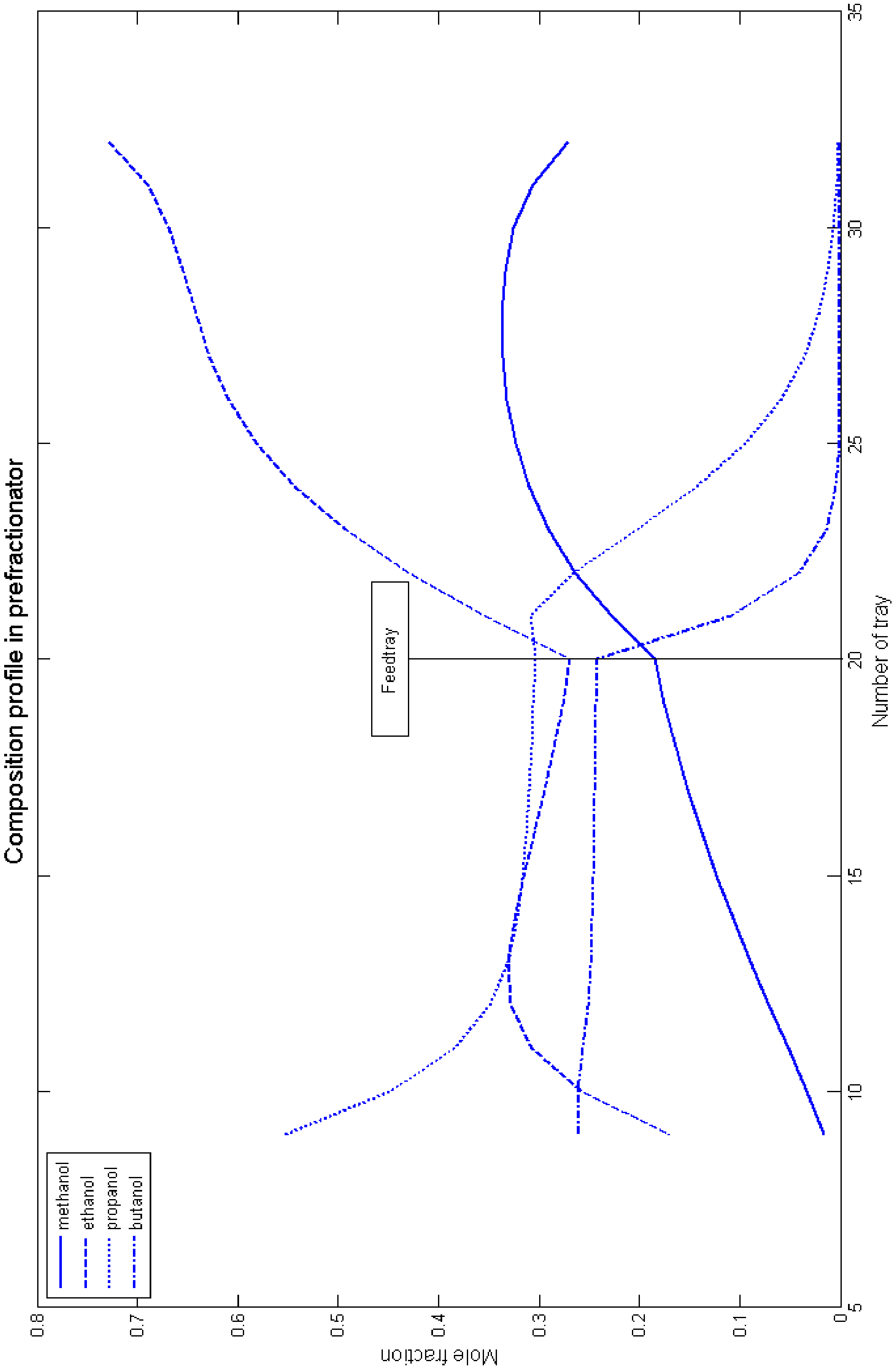


Chart 31: Rv-30% - composition in prefractionator

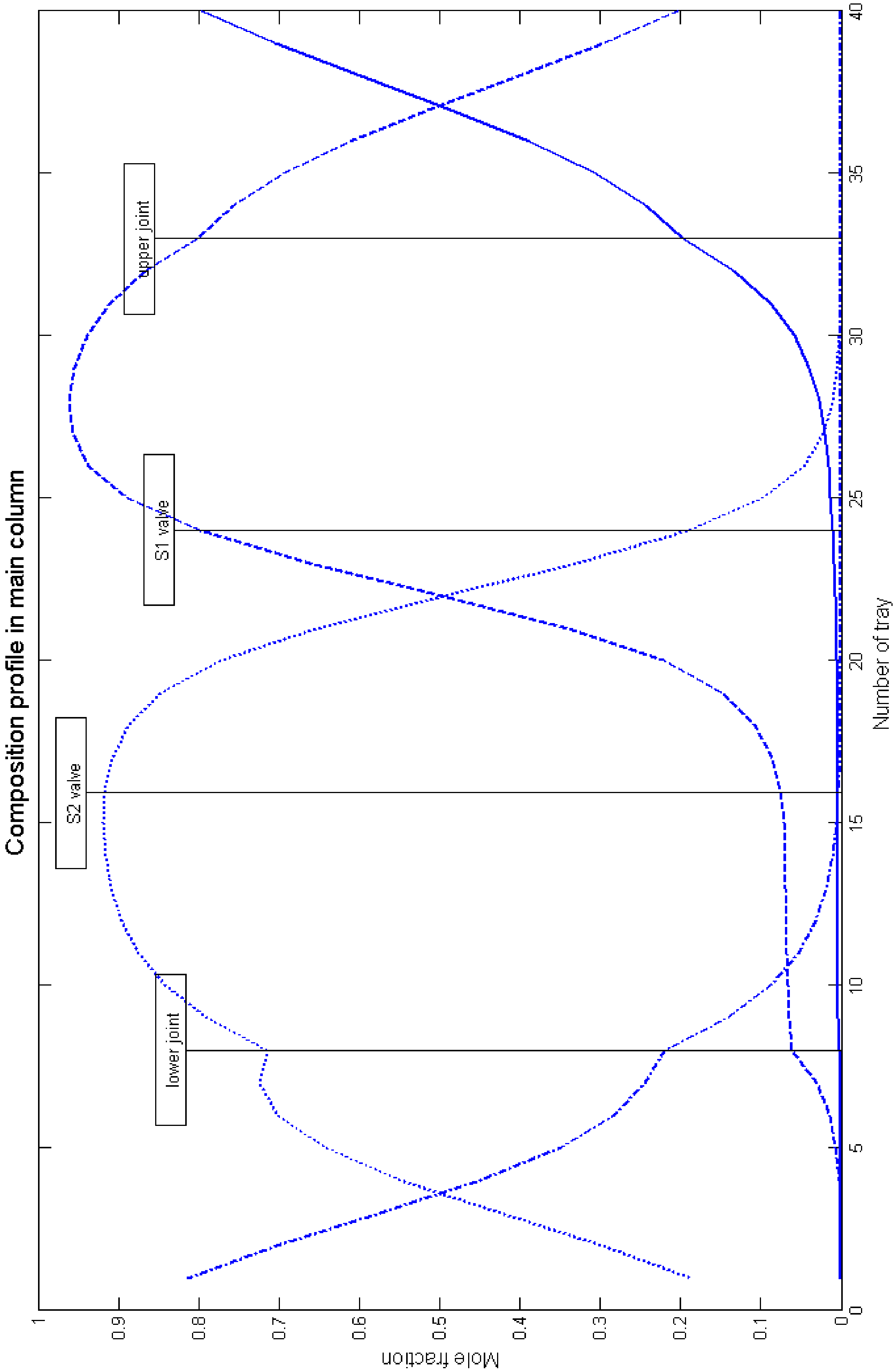


Chart 32: Rv-30% - composition in main column

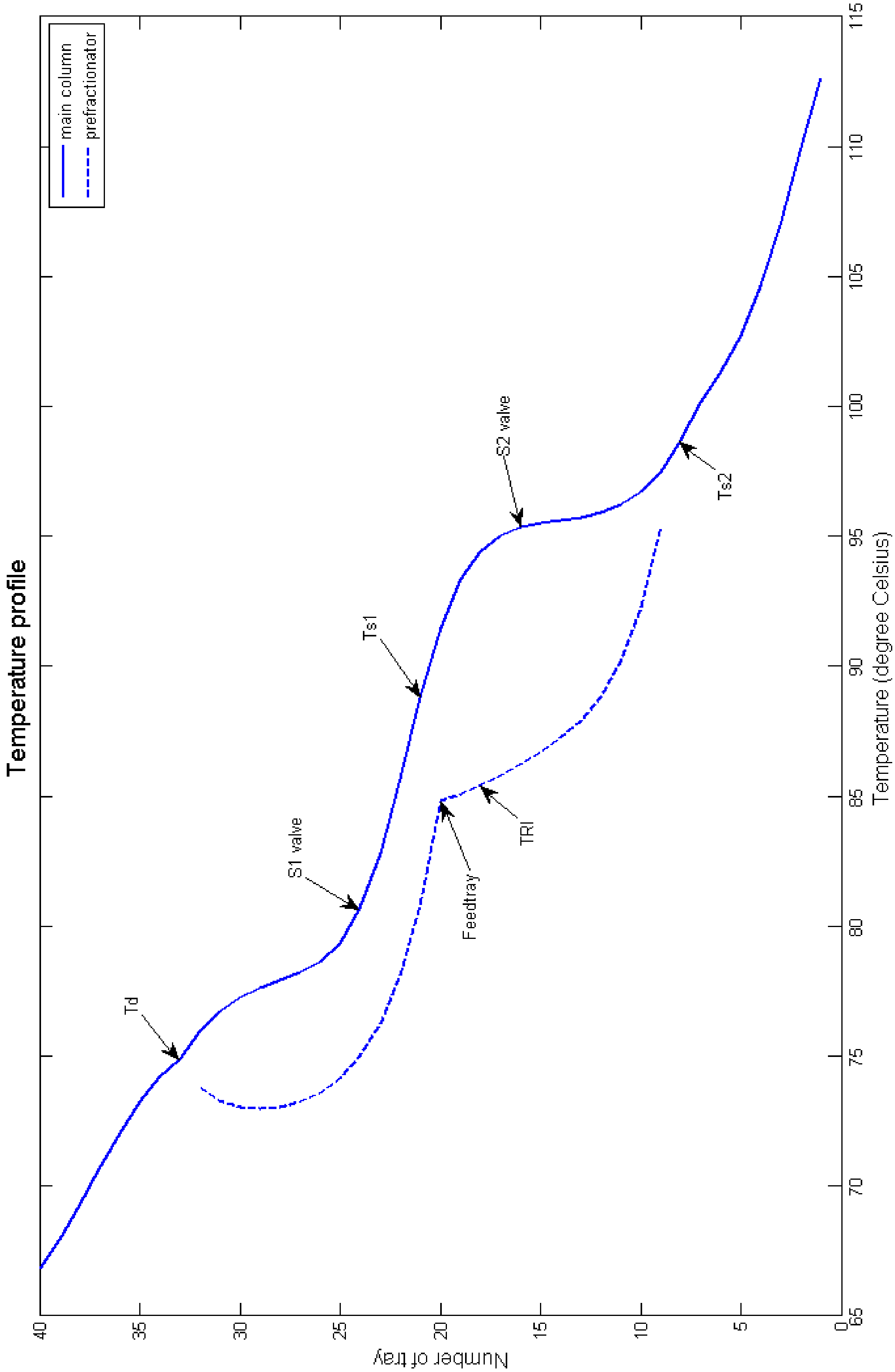


Chart 33: Rv-30% - temperature profile

11.8.2 Tables and discussion

Table 27: Inputs and results

Degrees of freedom	setpoint	stage efficiency	
product D (Td) T33	75,9696°C	section	alpha
product S_1 (Ts1) T60	88,7943°C	1	0
product S_2 (Ts2) T49	98,6820°C	2	0
bottoms (Vapour boilup)	2,3000mol/min	3	0
prefractionator (R_L) T15	85,4290°C	4	0
R_V	0,2639	5	0
R_L	0,1718	6	0
		7	0

Table 28: Results

	%A	%B	%C	%D	flow (mol/min)
F	18	23	30	29	1,0000
D	79,84	20,16	0,00	0,00	0,2020
S_1	1,11	79,84	19,05	0,00	0,2031
S_2	0,37	7,61	91,68	0,34	0,2708
B	0,00	0,01	18,63	81,36	0,3241
				TOTAL	1,0000

A	methanol
B	ethanol
C	propanol
D	butanol

The concentrations of the products (Table 28) don’t change a lot. But they all seem to drop a bit, when compared to the previous case. But compared to the base case, they all remain equal. The effect of the decrease of vapour split ratio can be handled well by the column. The temperatures (Table 27) remain equal.

In Chart 31, the concentration of propanol shows a bump above the feedtray. This can be explained by the fact that not enough heat is sent in the prefractionator, but the needed heat is derived from the feedheat. This heat enables the propanol to vapourise. The other charts (32 and 33) remain equal.

11.9 R_V ratio -40%

11.9.1 Charts

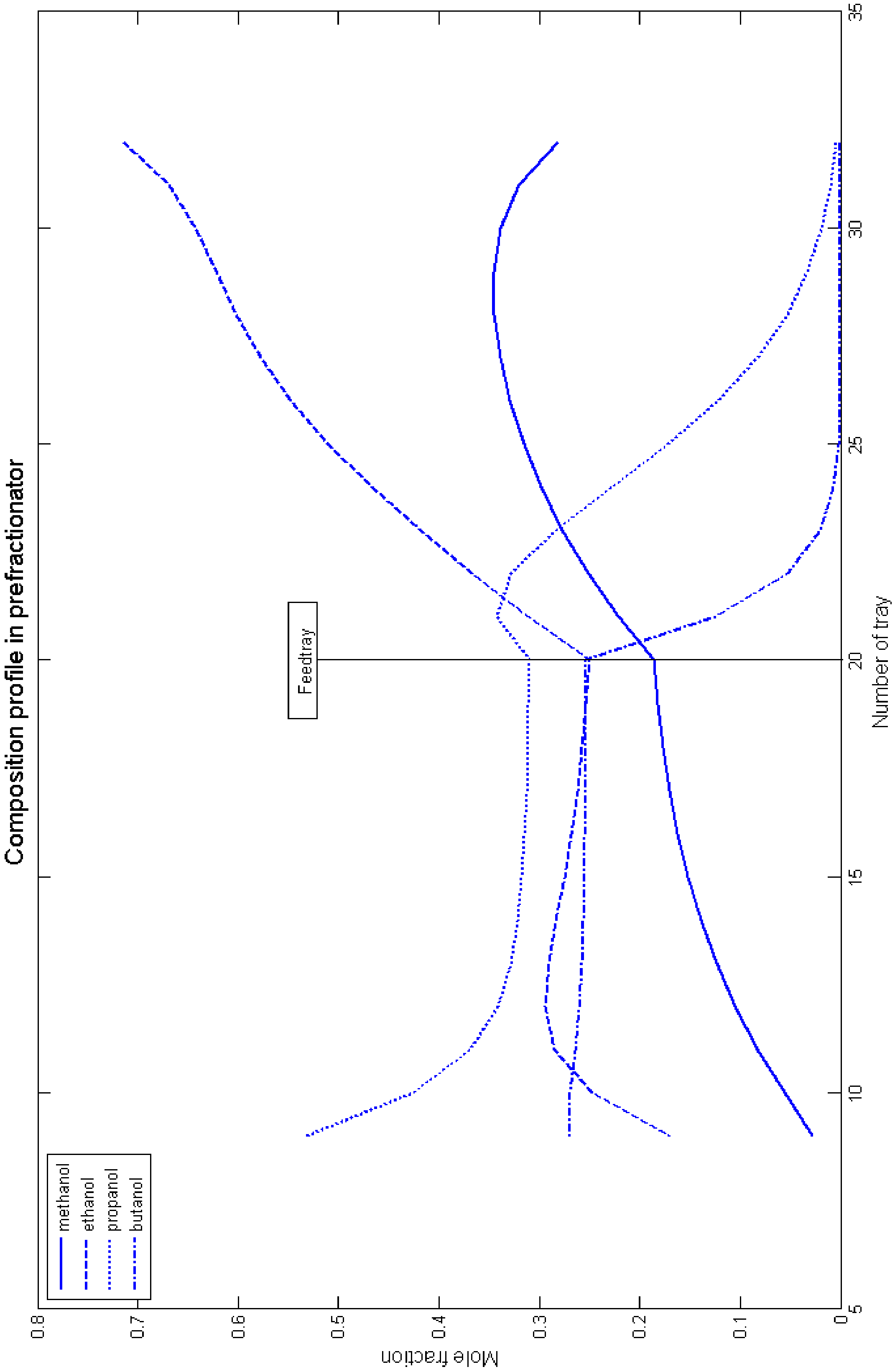


Chart 34: Rv-40% - composition in prefractionator

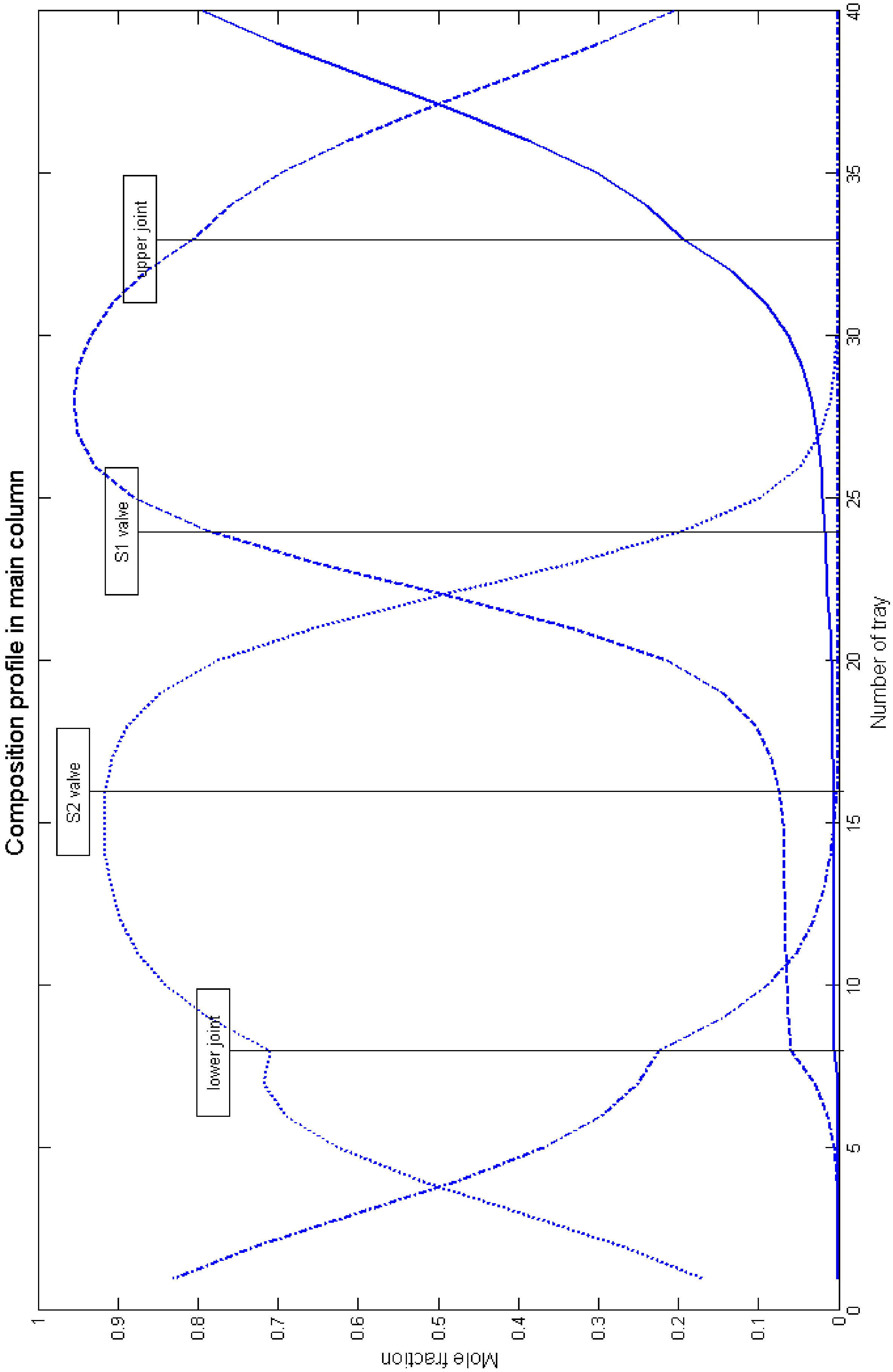


Chart 35: Rv-40% - composition in main column

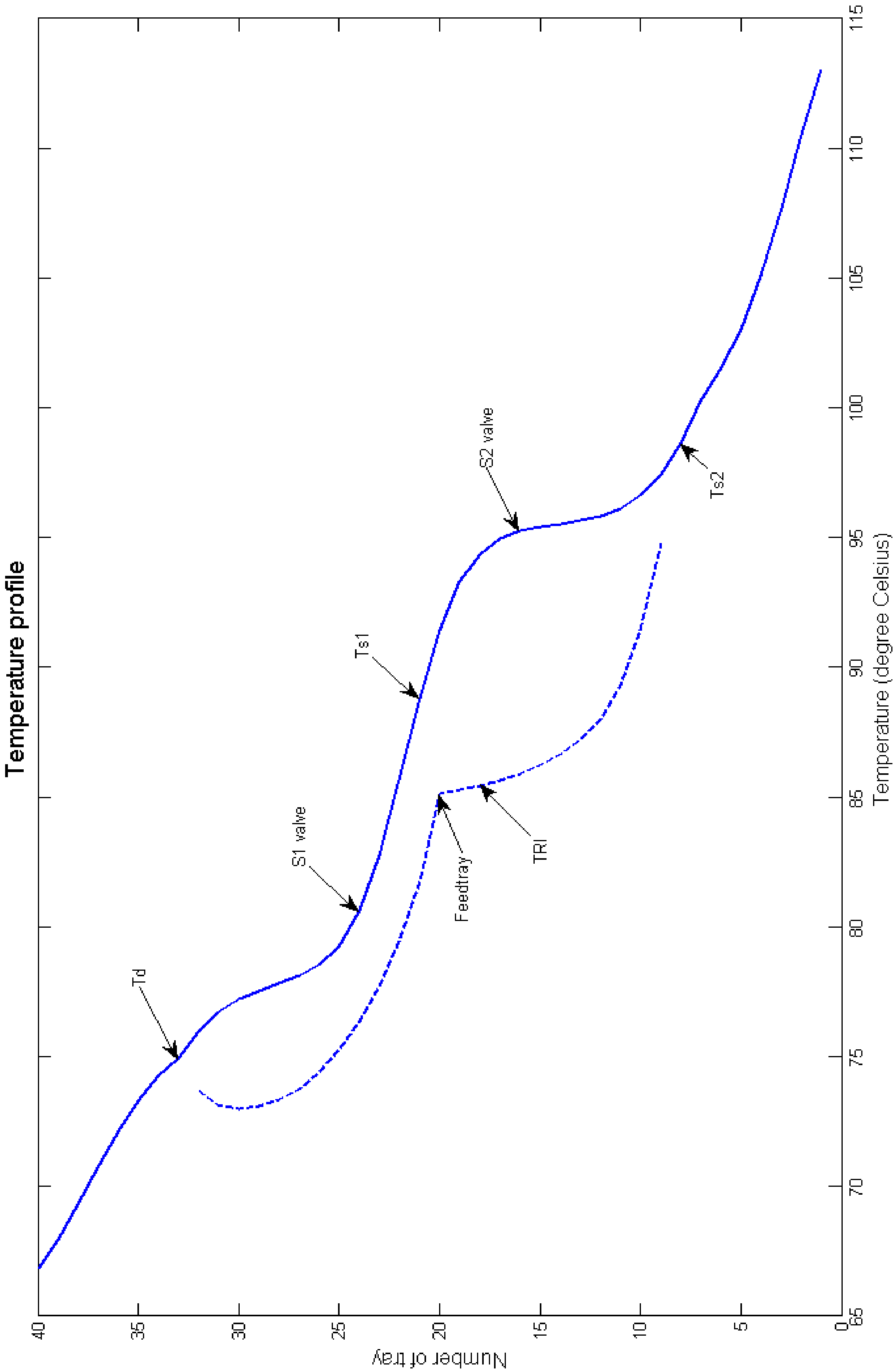


Chart 36: Rv-40% - temperature profile

11.9.2 Tables and discussion

Table 29: Inputs and disturbances

Degrees of freedom	setpoint	stage efficiency	
product D (Td) T33	75,9697°C	section	alpha
product S_1 (Ts1) T60	88,7953°C	1	0
product S_2 (Ts2) T49	98,6510°C	2	0
bottoms (Vapour boilup)	2,3000mol/min	3	0
prefractionator (R_L) T15	85,4391°C	4	0
R_V	0,2262	5	0
R_L	0,1366	6	0
		7	0

Table 30: Results

	%A	%B	%C	%D	flow (mol/min)
F	18	23	30	29	1,0000
D	79,57	20,43	0,00	0,00	0,1999
S_1	1,79	78,78	19,43	0,00	0,2056
S_2	0,64	7,39	91,64	0,33	0,2743
B	0,00	0,01	16,89	83,10	0,3201
				TOTAL	0,9999

A	methanol
B	ethanol
C	propanol
D	butanol

The temperature setpoints (Table 29) remains equal, just as the compositions (Table 30). But in the charts, some things happen. In Chart 34 it becomes clear, that not sufficient energie enters through the bottom of the column. The concentration of methanol in the lower part remains higher. The propanol bump above the feedtray becomes bigger, which indicates that a major part of the heat is extracted from the feedheat. The other two charts(Chart 35 and Chart 36) remain more or less equal.

11.10 R_V +1%

11.10.1 Charts

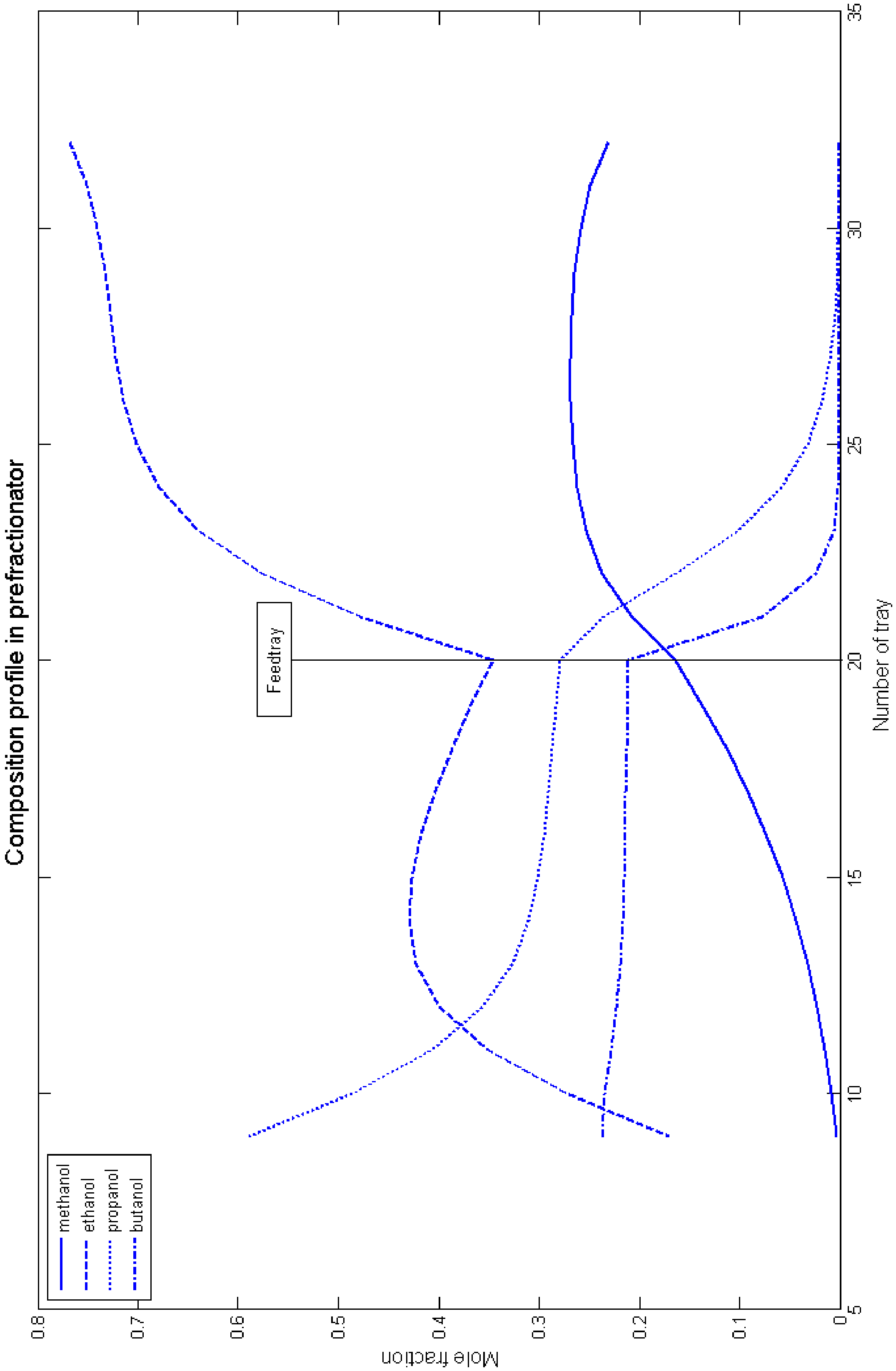


Chart 37: Rv+1% - composition in prefractionator

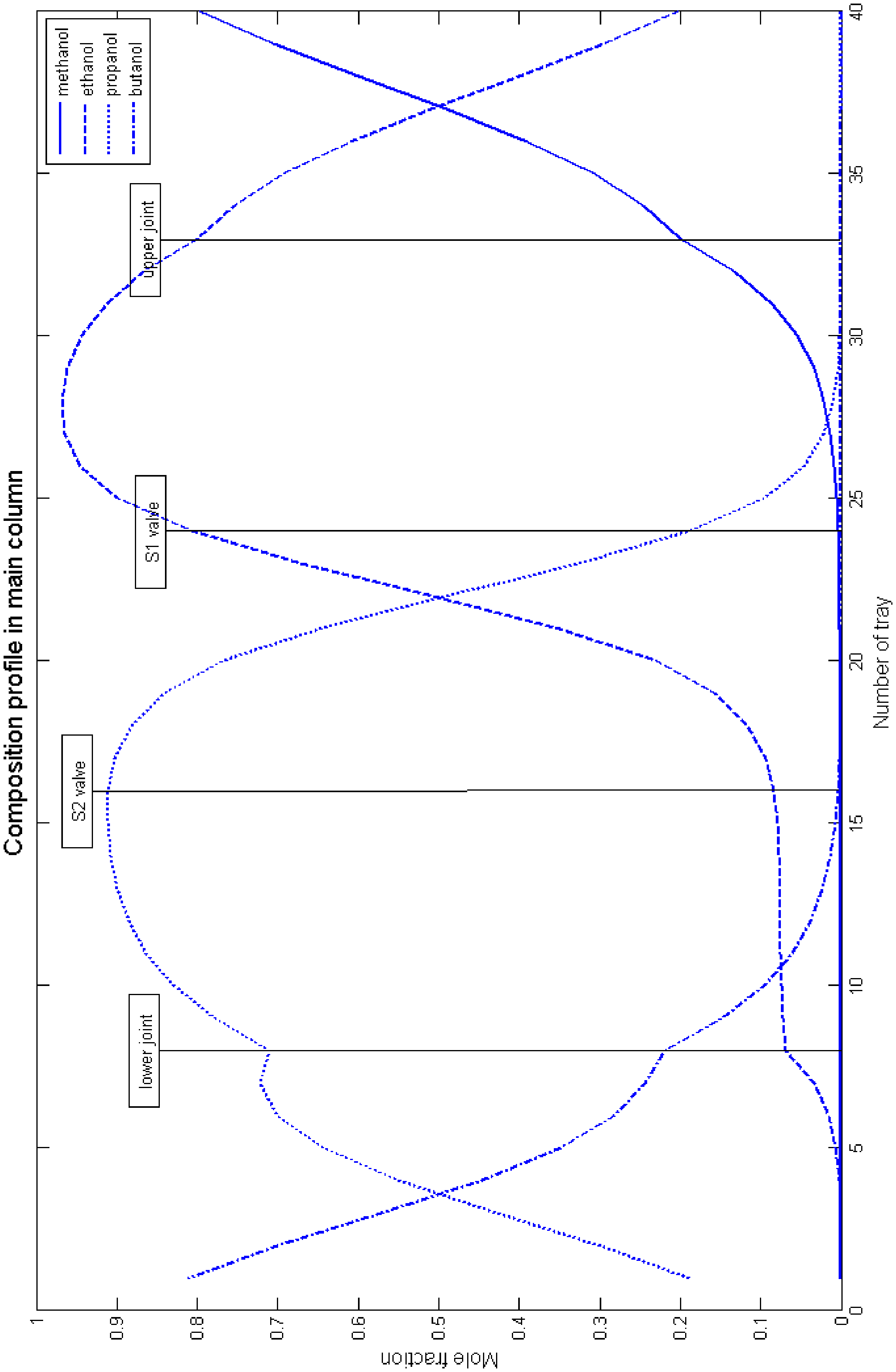


Chart 38: Rv+1% - composition in main column

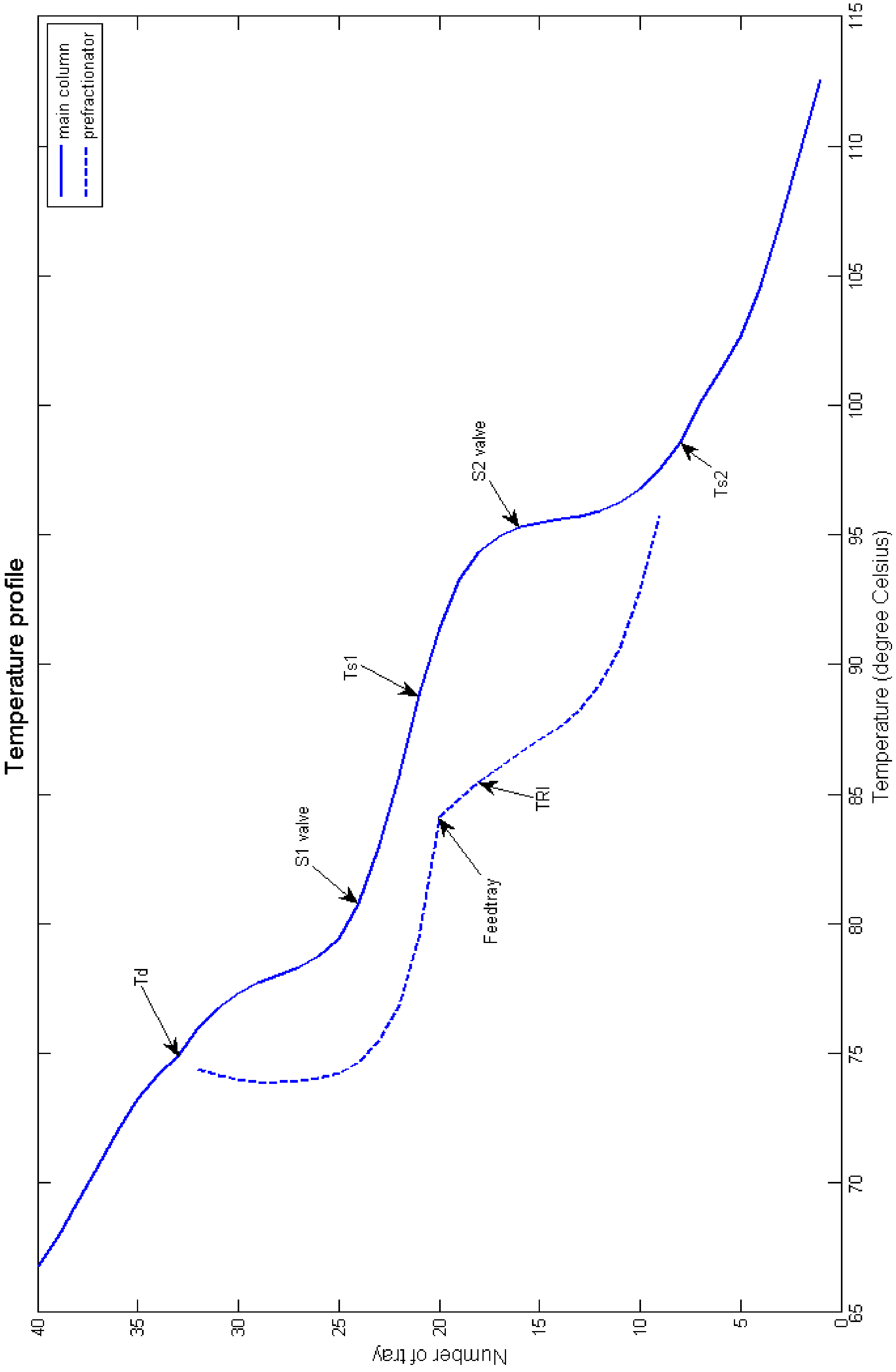


Chart 39: Rv+1% - temperature profile

11.10.2 Tables and discussion

Table 31: Inputs and disturbances

Degrees of freedom	setpoint	stage efficiency	
product D (Td) T33	75,9701°C	section	alpha
product S_1 (Ts1) T60	88,7969°C	1	0
product S_2 (Ts2) T49	98,6108°C	2	0
bottoms (Vapour boilup)	2,3000mol/min	3	0
prefractionator (R_L) T15	85,4803°C	4	0
R_V	0,3808	5	0
R_L	0,2919	6	0
		7	0

Table 32: Results

	%A	%B	%C	%D	flow (mol/min)
F	18	23	30	29	1,0000
D	79,89	20,11	0,00	0,00	0,2045
S_1	0,42	80,69	18,89	0,00	0,1983
S_2	0,10	8,36	91,10	0,44	0,2731
B	0,00	0,01	18,79	81,20	0,3232
				TOTAL	0,9991

A	methanol
B	ethanol
C	propanol
D	butanol

The concentration of butanol in the bottoms (Table 32) decreases a bit, but stays in the same order as the base case. Thus the concentration of propanol in the bottoms increases. Beside that, not much changes in Chart 37, 38 and 39. The temperature setpoint (Table 31) remain equal.

11.11 R_V +2%

11.11.1 Charts

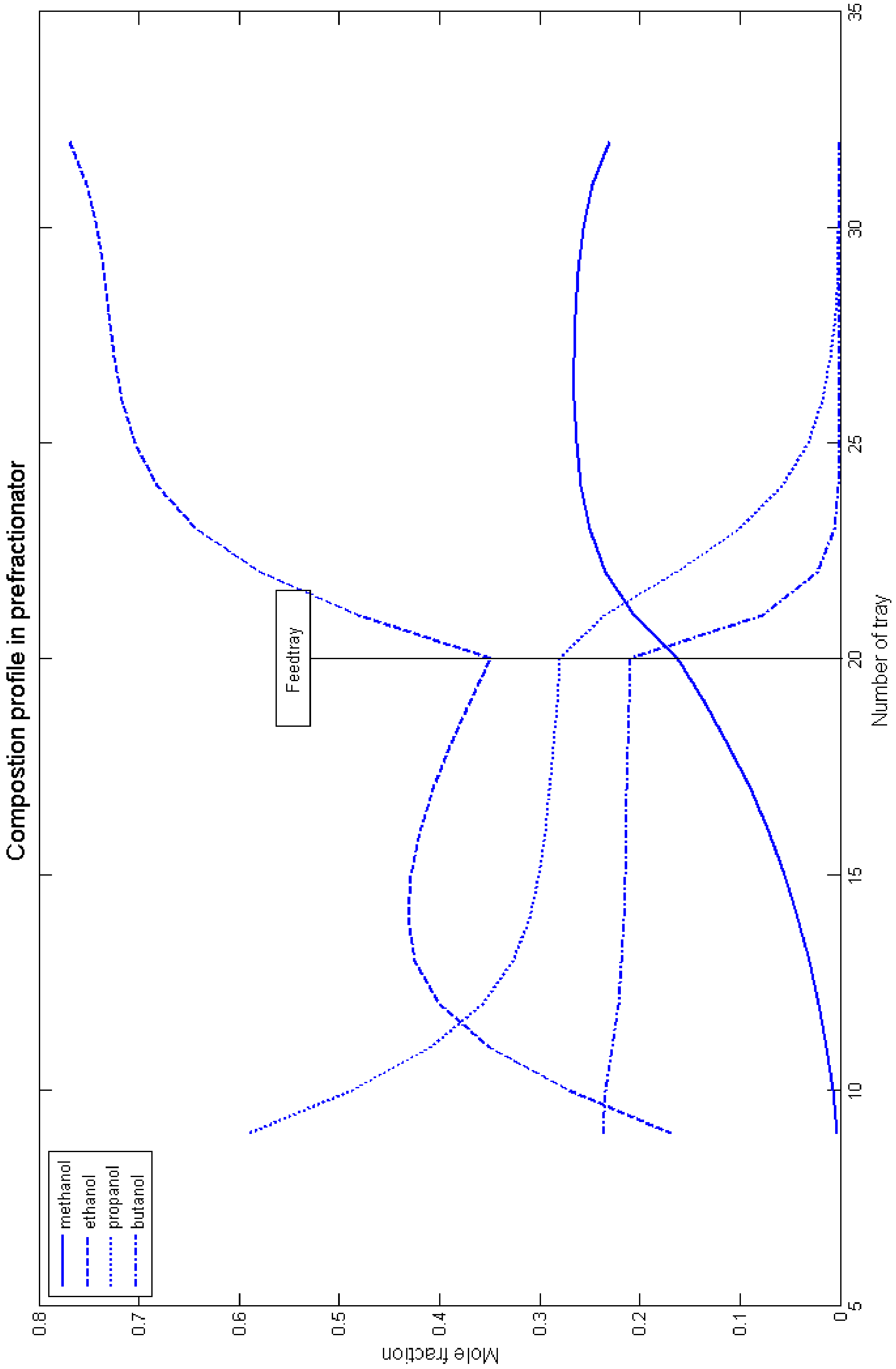


Chart 40: Rv+2% - composition in prefractionator

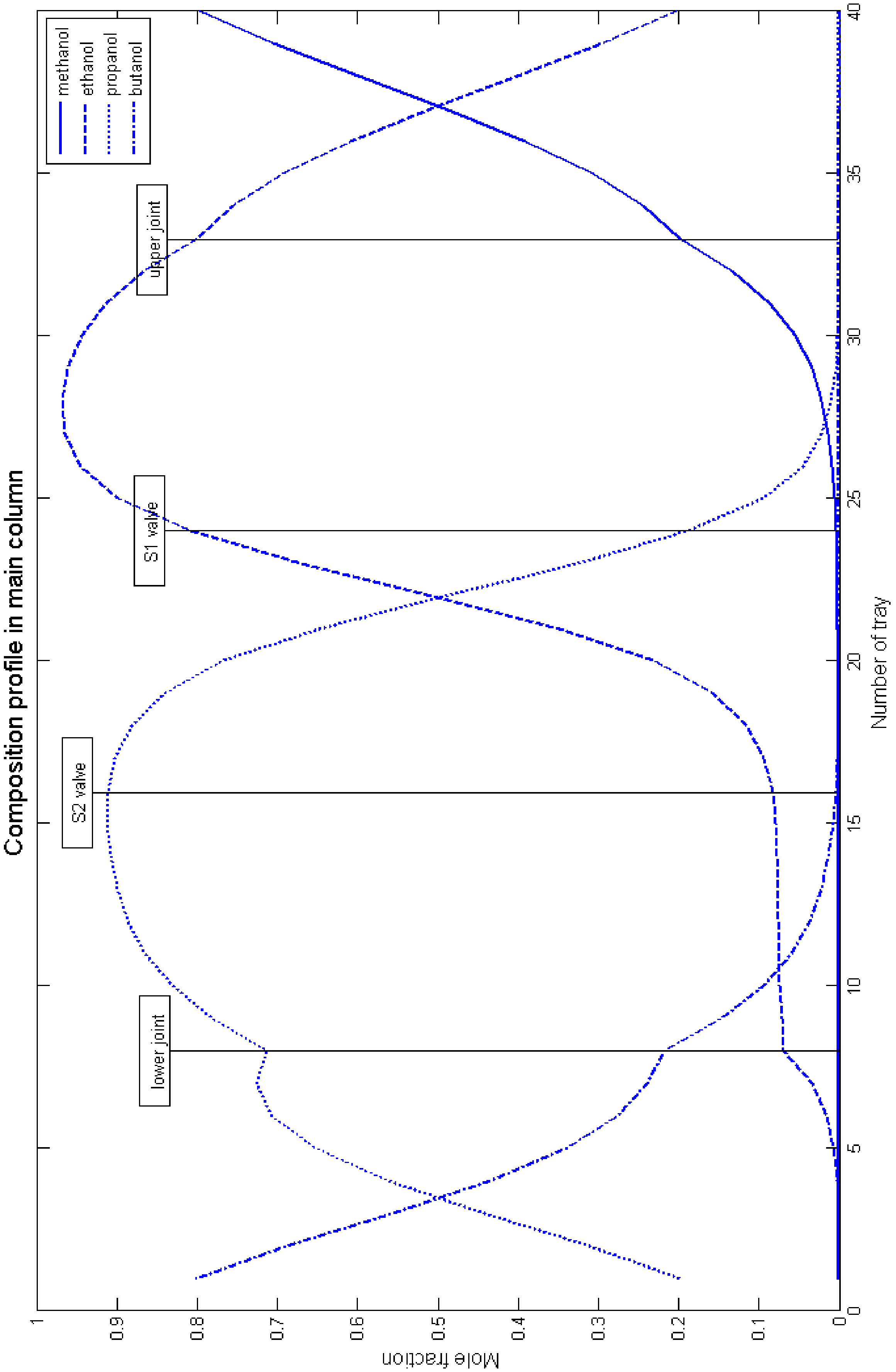


Chart 41: Rv+2% - composition in main column

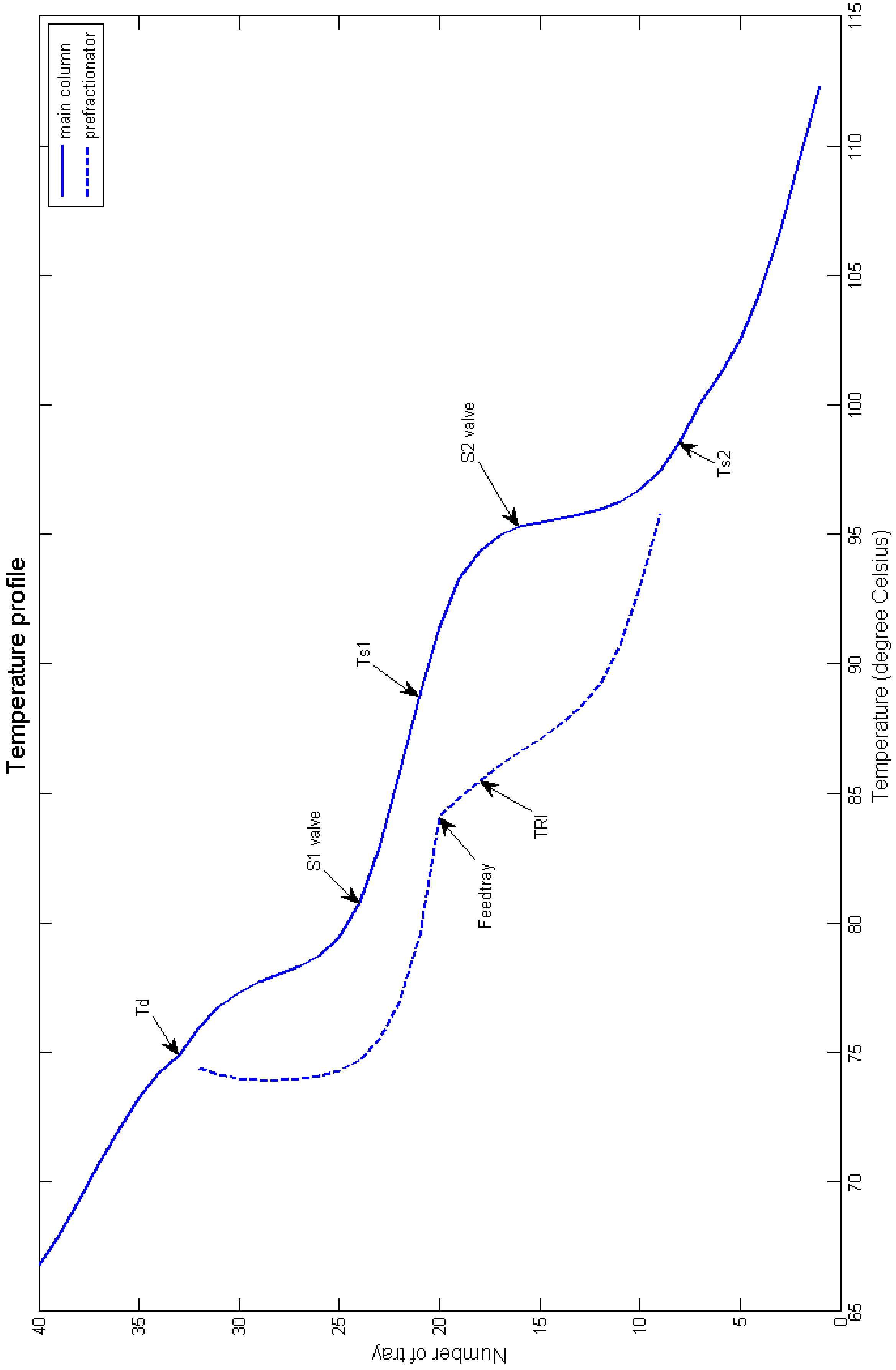


Chart 42: Rv+2% - temperature profile

11.11.2 Tables and discussion

Table 33: Inputs and disturbances

Degrees of freedom	setpoint	stage efficiency	
product D (Td) T33	75,9703°C	section	alpha
product S_1 (Ts1) T60	88,7978°C	1	0
product S_2 (Ts2) T49	98,5933°C	2	0
bottoms (Vapour boilup)	2,3000mol/min	3	0
prefractionator (R_L) T15	85,5049°C	4	0
R_V	0,3846	5	0
R_L	0,2953	6	0
		7	0

Table 34: Results

	%A	%B	%C	%D	flow (mol/min)
F	18	23	30	29	1,0000
D	79,86	20,14	0,00	0,00	0,2046
S_1	0,41	80,74	18,85	0,00	0,1985
S_2	0,09	8,33	91,14	0,44	0,2706
B	0,00	0,02	19,76	80,22	0,3264
				TOTAL	1,0001

A	methanol
B	ethanol
C	propanol
D	butanol

More propanol ends up in the bottoms, which means that the concentration of butanol decreases (Table 34). In charts 40, 41 and 42 however, not much changes. The temperature setpoint, shown in Table 33, remain equal.

11.12 R_V +5%

11.12.1 Charts

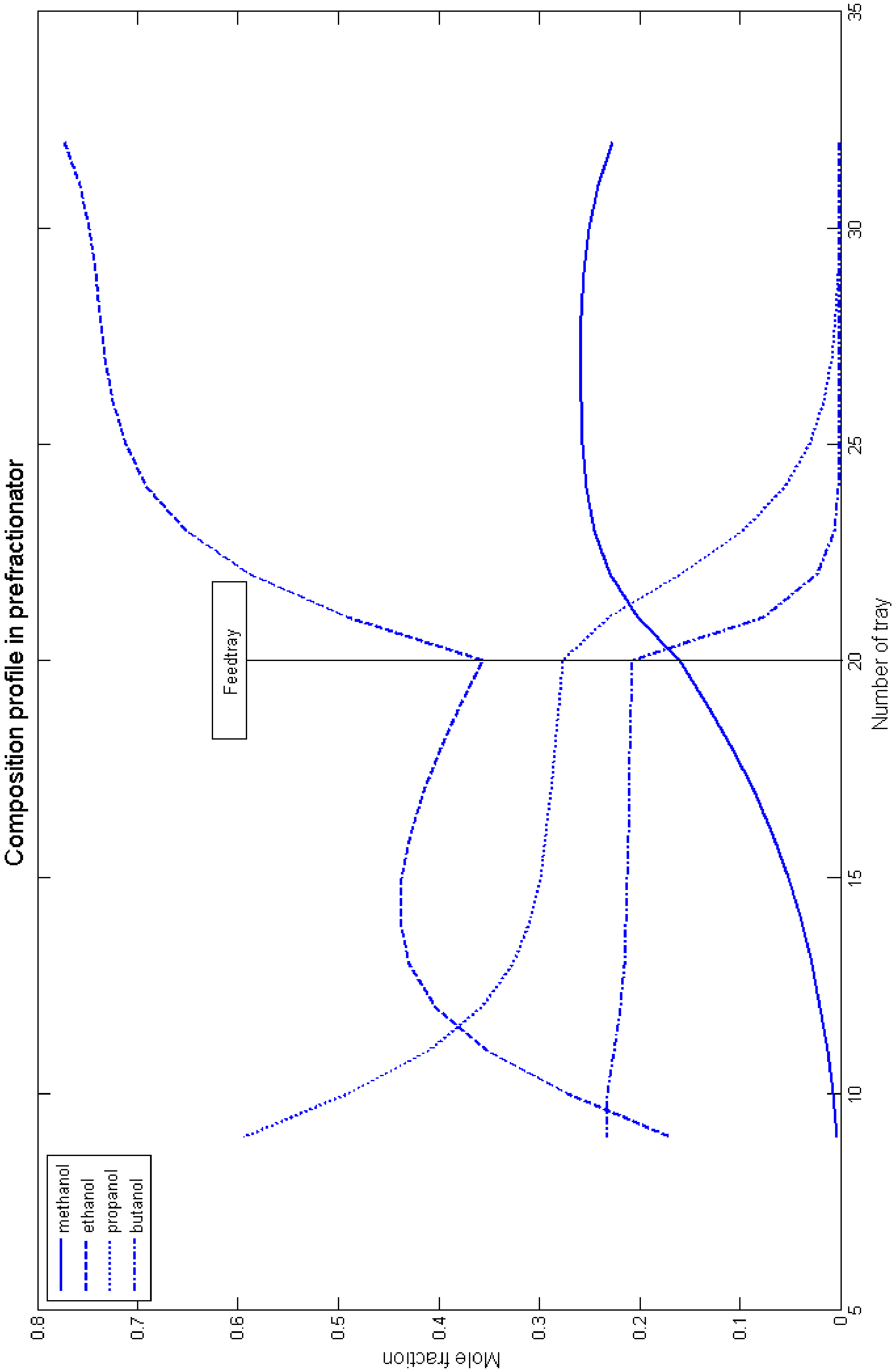


Chart 43: Rv+5% - composition in prefractionator

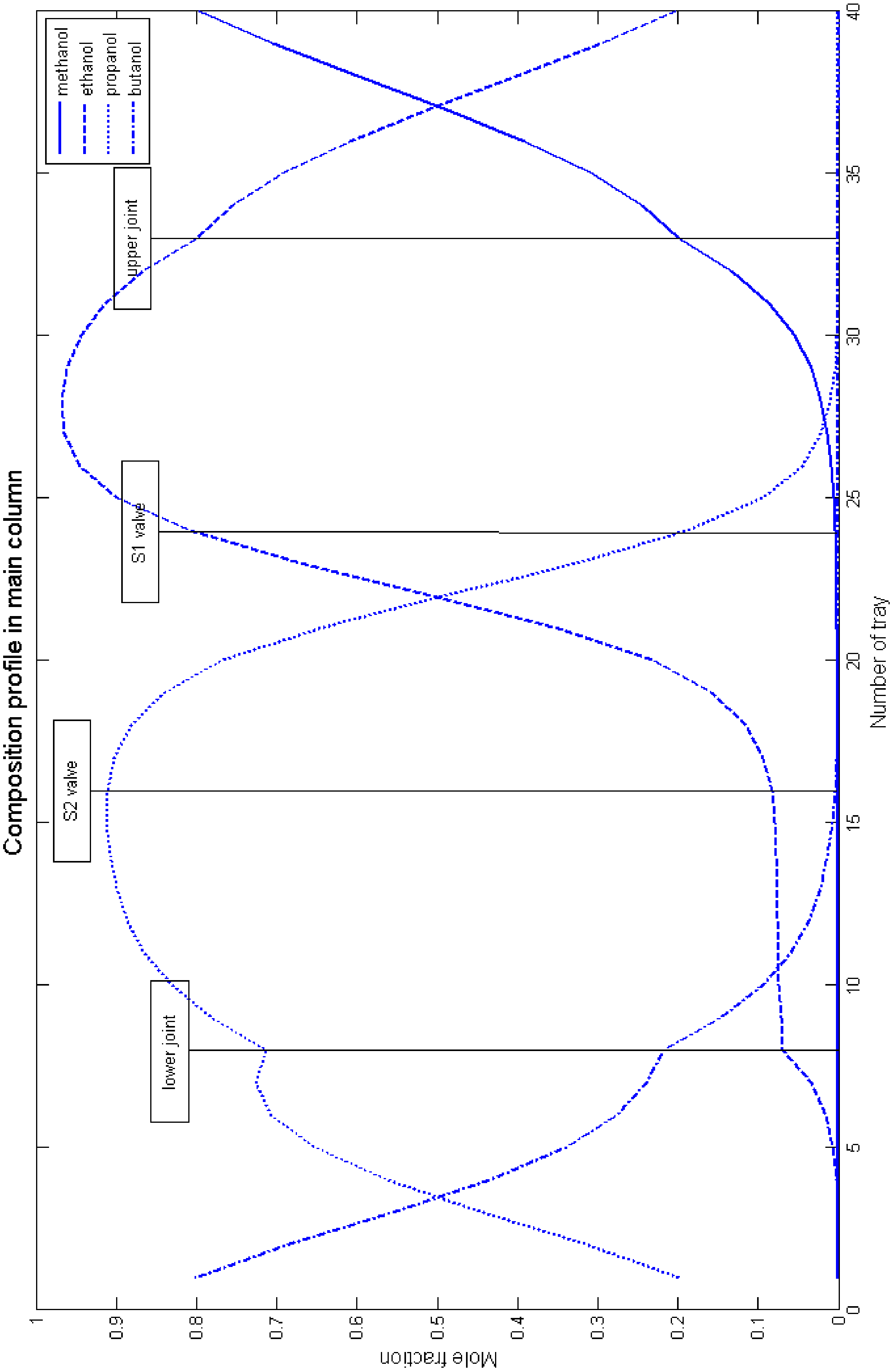


Chart 44: Rv+5% - composition in main column

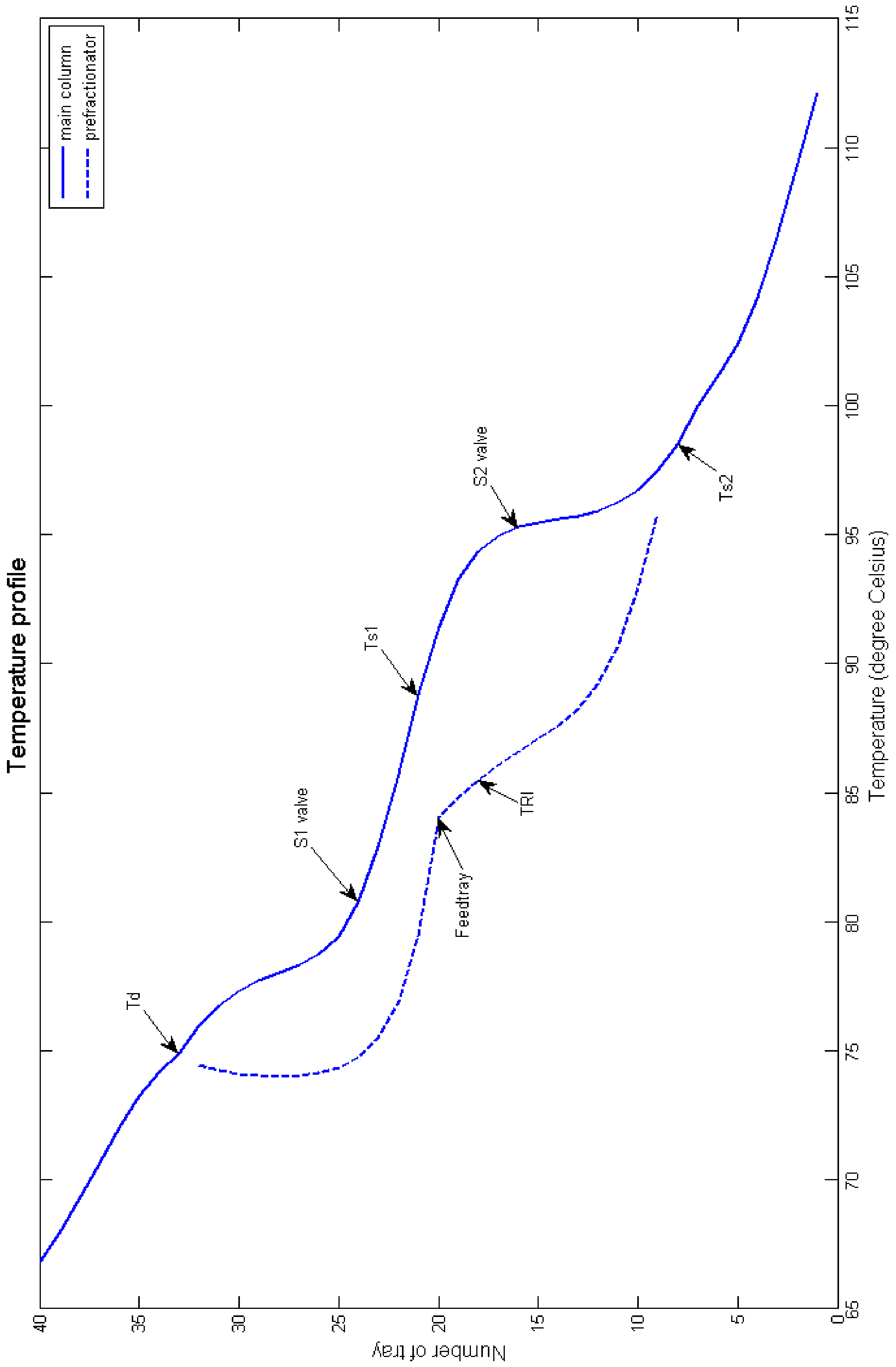


Chart 45: Rv+5% - temperature profile

11.12.2 Tables and discussion

Table 35: Inputs and disturbances

Degrees of freedom	setpoint	stage efficiency	
product D (Td) T33	75,9702°C	section	alpha
product S_1 (Ts1) T60	88,7987°C	1	0
product S_2 (Ts2) T49	98,5594°C	2	0
bottoms (Vapour boilup)	2,3000mol/min	3	0
prefractionator (R_L) T15	85,4848°C	4	0
R_V	0,3959	5	0
R_L	0,3070	6	0
		7	0

Table 36: Results

	%A	%B	%C	%D	flow (mol/min)
F	18	23	30	29	1,0000
D	79,82	20,18	0,00	0,00	0,2047
S_1	0,39	80,76	18,85	0,00	0,1983
S_2	0,08	8,39	91,08	0,45	0,2689
B	0,00	0,02	20,49	79,49	0,3280
				TOTAL	0,9999

A	methanol
B	ethanol
C	propanol
D	butanol

The concentration of propanol keeps increasing in the bottoms (Table 36). This can be explained by the fact that less vapour enters the main column, which enables propanol to go down in the column. Since more heat enters the prefractionator, more liquid will enter from the top to cool it down (Table 35). Thus less liquid goes in the main column. This means that it would heat up, but since the setpoint temperatures remain equal the concentration profile in the main column will shift a little (Chart 44). This however doesn’t effect the side stream purities. Charts 43 and 45 remain equal.

11.13 R_V +10%

11.13.1 Charts

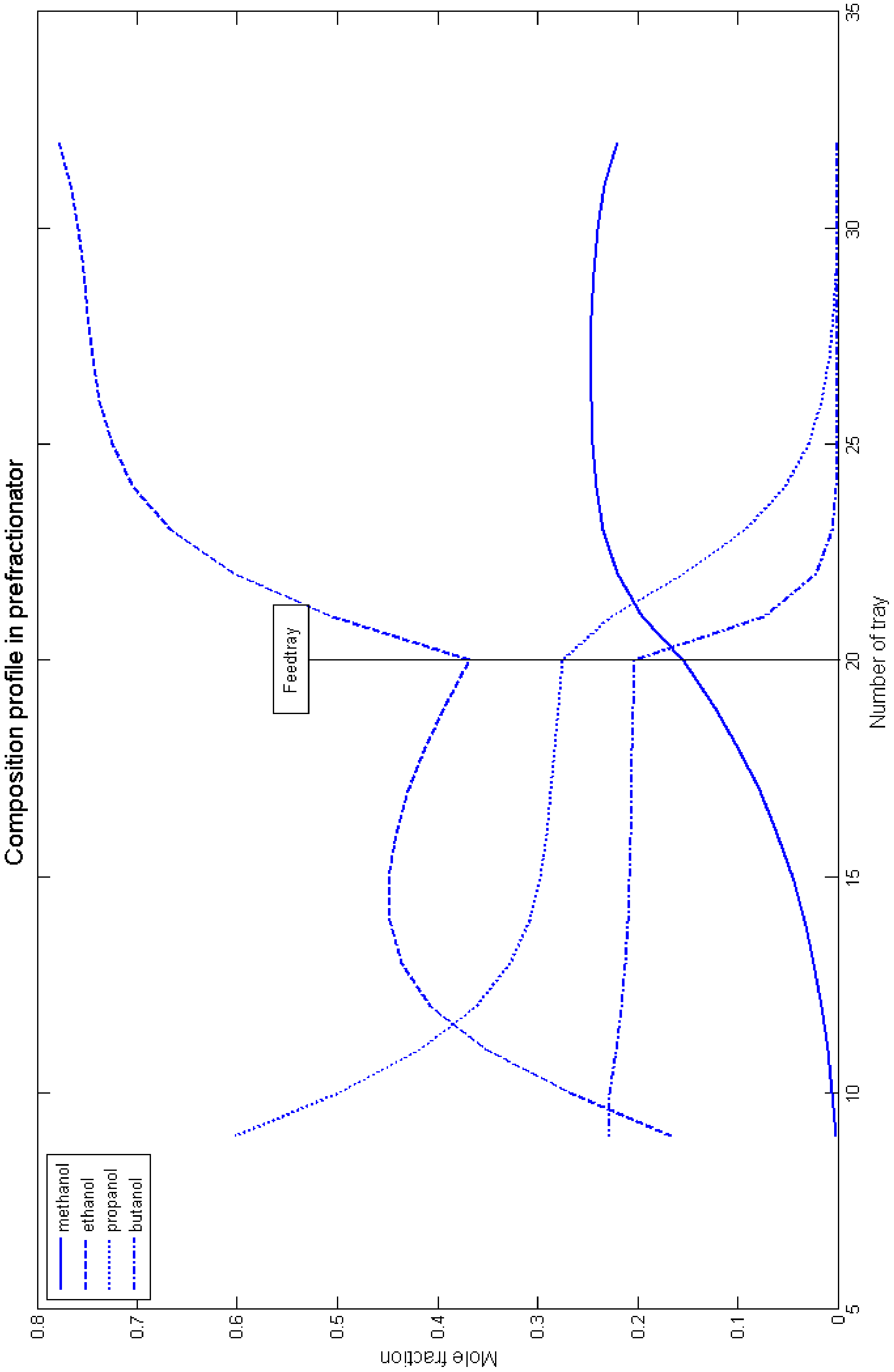


Chart 46: Rv+10% - composition in prefractionator

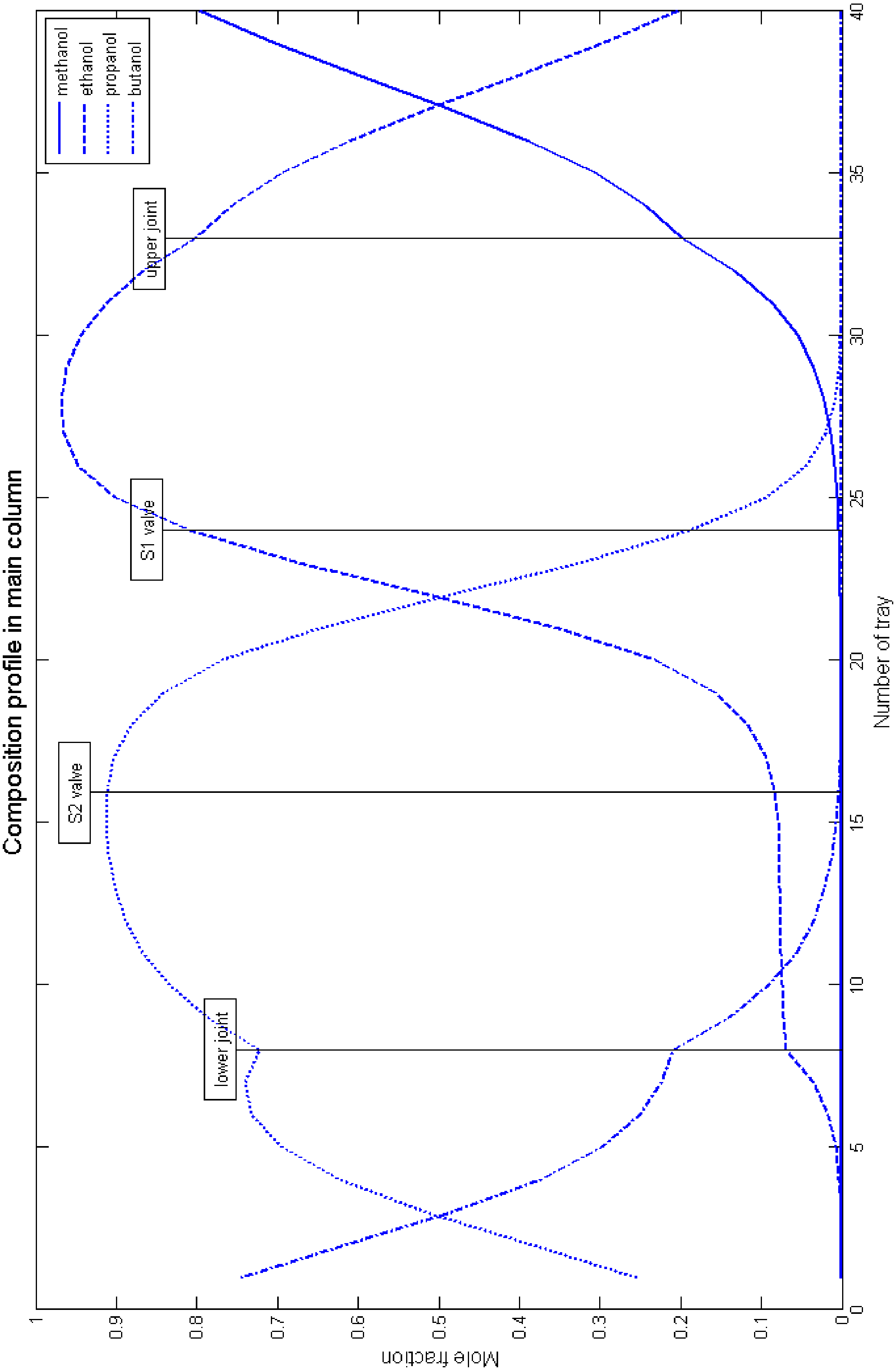


Chart 47: Rv+10% - composition in main column

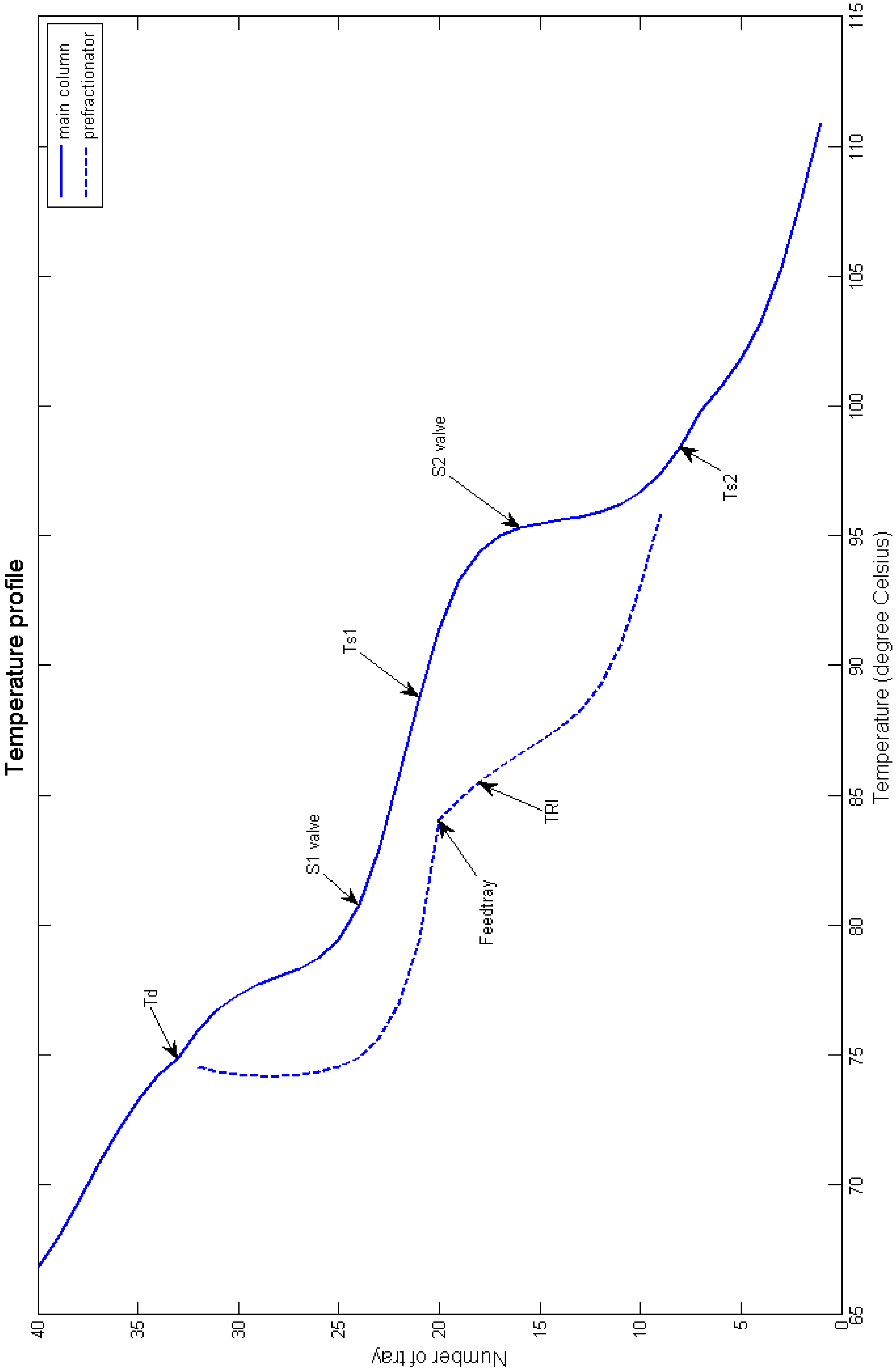


Chart 48: Rv+10% - temperature profile

11.13.2 Tables and discussion

Table 37: Inputs and disturbances

Degrees of freedom		stage efficiency	
product D (Td) T33	75,9704°C	section	alpha
product S_1 (Ts1) T60	88,8025°C	1	0
product S_2 (Ts2) T49	98,4628°C	2	0
bottoms (Vapour boil-up)	2,3000mol/min	3	0
prefractionator (R_L) T15	85,5199°C	4	0
R_V	0,4147	5	0
R_L	0,3249	6	0
		7	0

Table 38: Results

	%A	%B	%C	%D	flow (mol/min)
F	18	23	30	29	1,0000
D	79,66	20,34	0,00	0,00	0,2051
S_1	0,35	80,90	18,75	0,00	0,1993
S_2	0,07	8,35	91,17	0,41	0,2548
B	0,00	0,02	25,49	74,49	0,3408
				TOTAL	1,0000

A	methanol
B	ethanol
C	propanol
D	butanol

The concentration of propanol in the bottoms has increased with almost 10% when compared to the base case (Table 38). The strange thing is that the rest of the compositions remain equal. The flow of S_2 has decreased a bit, to maintain the temperature setpoints (Table 37). The charts (46, 47 and 48) remain equal as well.

11.14 R_V +20%

11.14.1 Charts

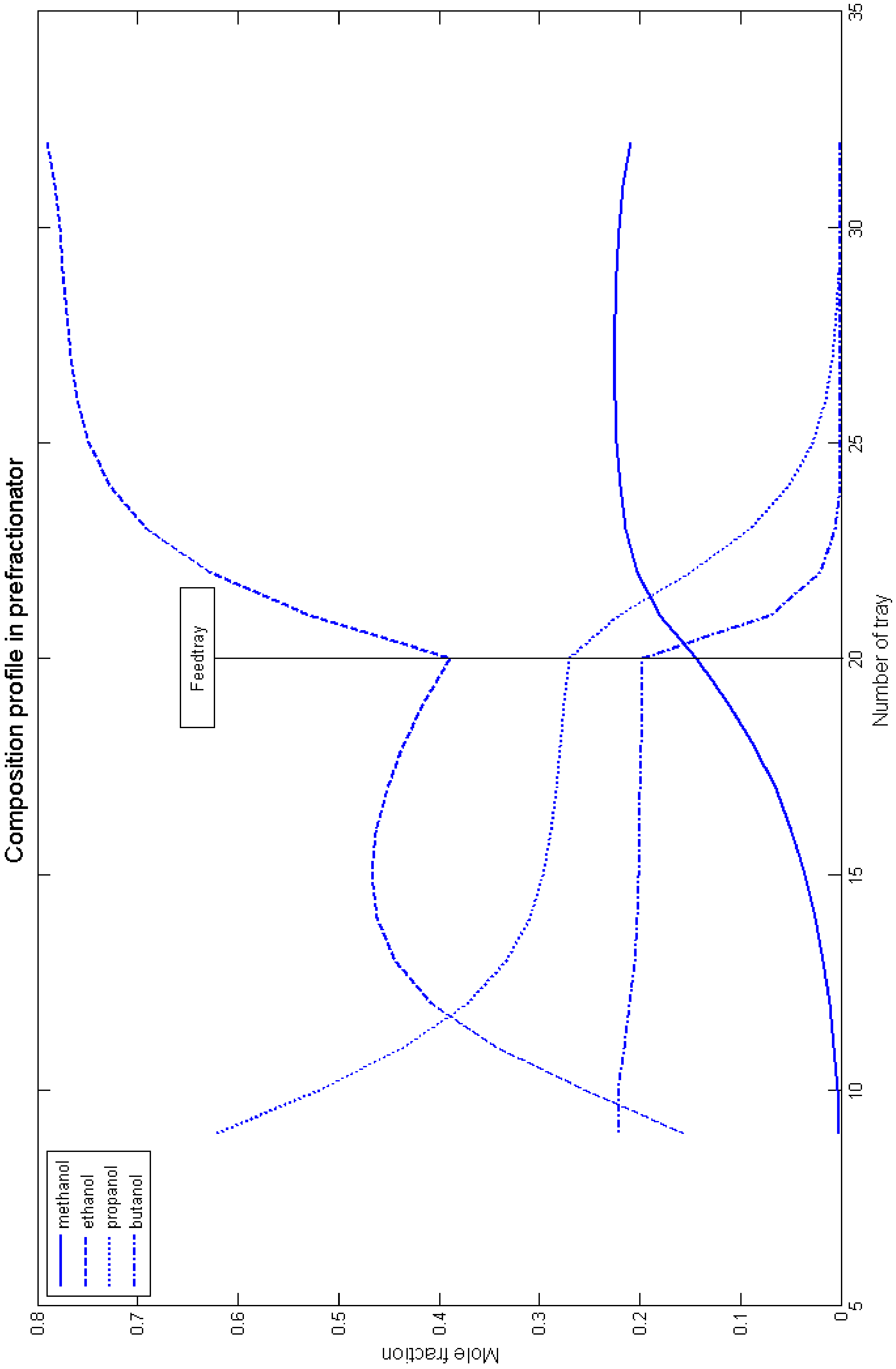


Chart 49: Rv+20% - composition in prefractionator

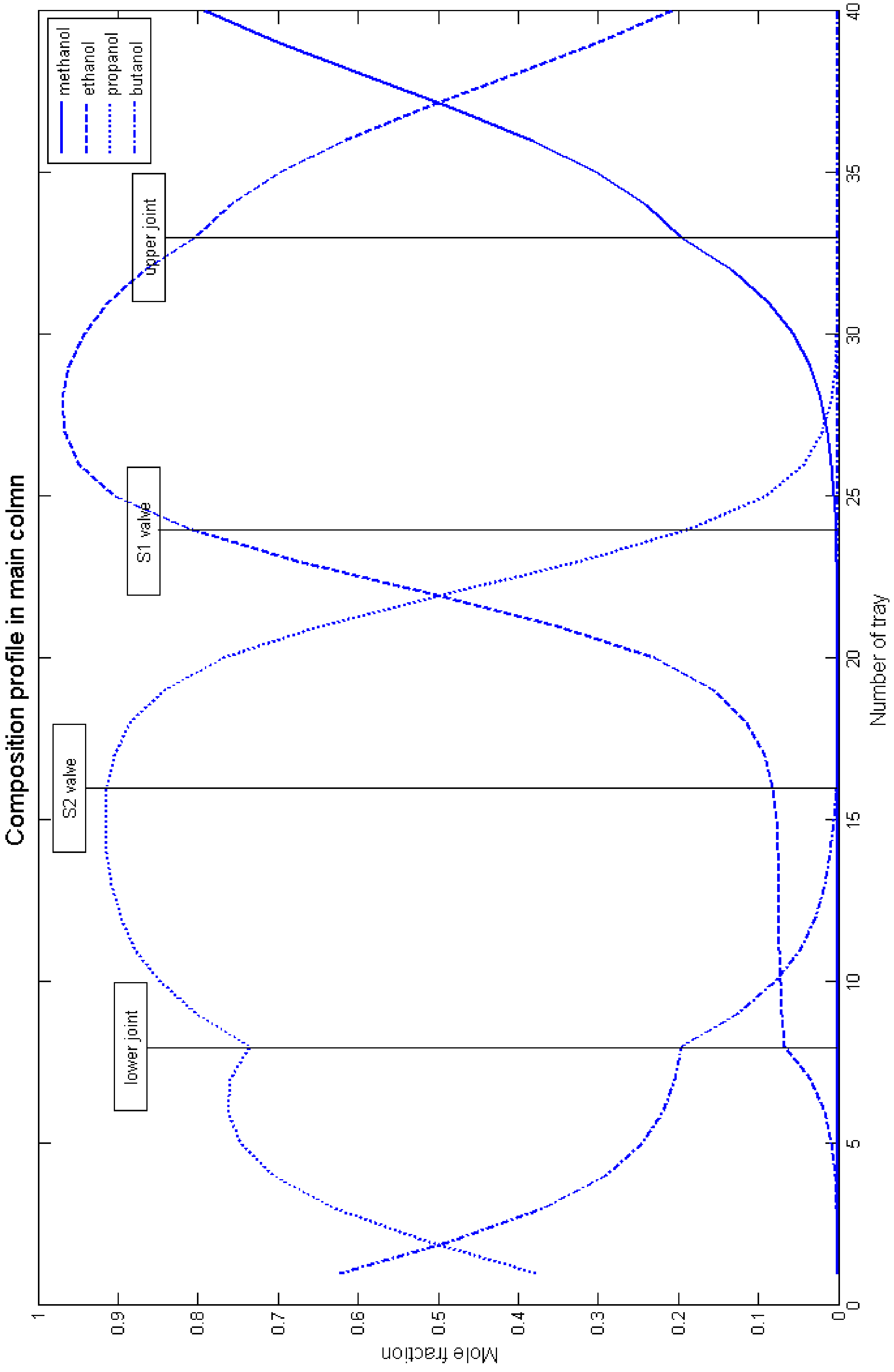


Chart 50: Rv+20% - composition in main column

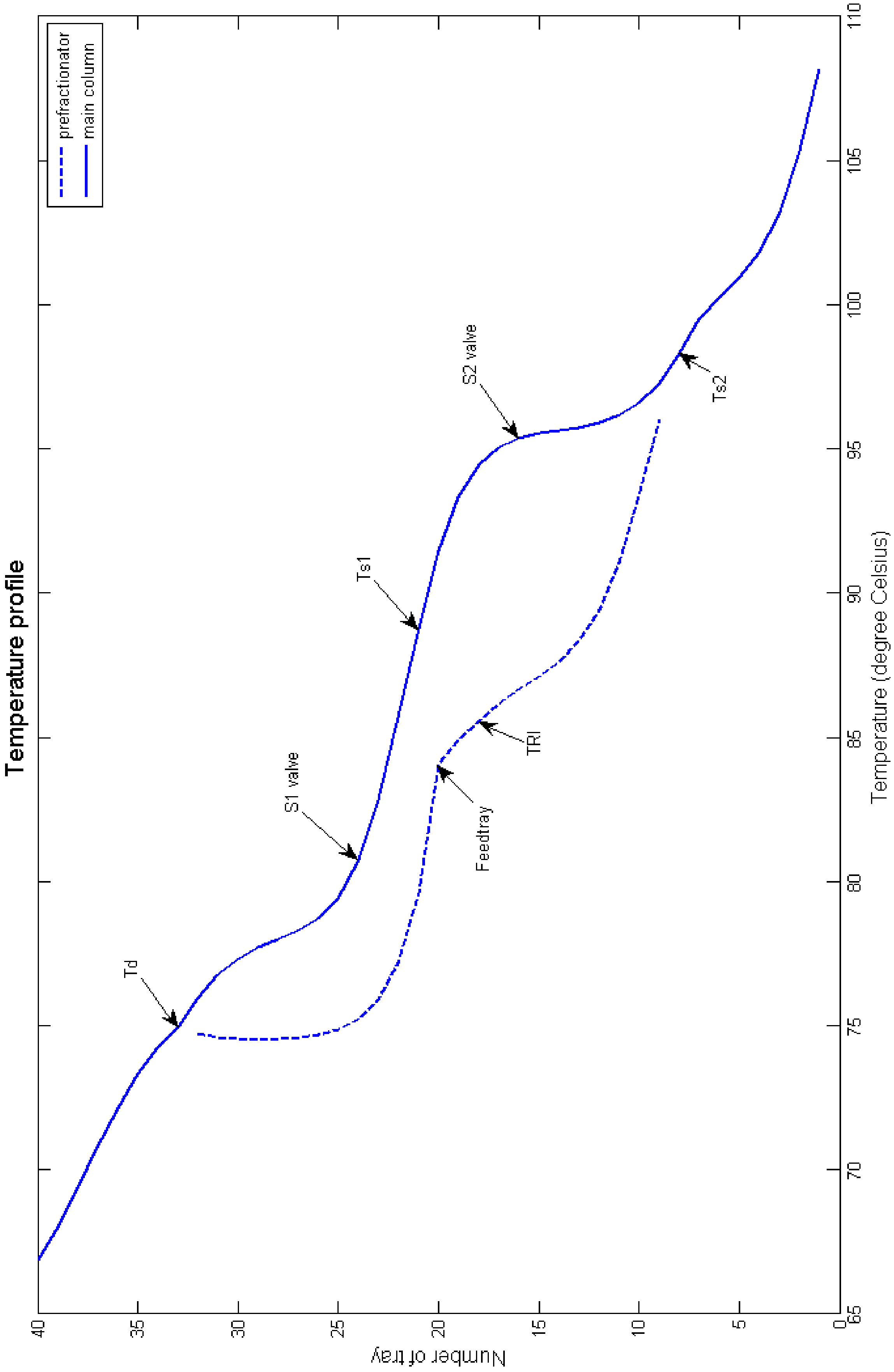


Chart 51: Rv+20% - temperature profile

11.14.2 Tables and discussion

Table 39: Inputs and disturbances

Degrees of freedom	setpoint	stage efficiency	
product D (Td) T33	75,9709°C	section	alpha
product S_1 (Ts1) T60	88,8070°C	1	0
product S_2 (Ts2) T49	98,3375°C	2	0
bottoms (Vapour boilup)	2,3000mol/min	3	0
prefractionator (R_L) T15	85,5848°C	4	0
R_V	0,4524	5	0
R_L	0,3594	6	0
		7	0

Table 40: Results

	%A	%B	%C	%D	flow (mol/min)
F	18	23	30	29	1,0000
D	79,22	20,78	0,00	0,00	0,2059
S_1	0,31	81,24	18,44	0,01	0,2028
S_2	0,04	8,18	91,46	0,32	0,2103
B	0,00	0,03	37,62	62,35	0,3809
				TOTAL	0,9999

A	methanol
B	ethanol
C	propanol
D	butanol

The trend of increasing propanol concentrations in the bottoms keeps on continuing. See Table 40. The temperature setpoint although remain perfect in the same range of the base case. (Table 39). The increasing concentration can also be seen on the composition plot in the main column (Chart 50). The concentration profiles in the prefractionator (Chart 49) remain equal, just as the temperature profiles (Chart 51).

11.15 R_V +30%

11.15.1 Charts

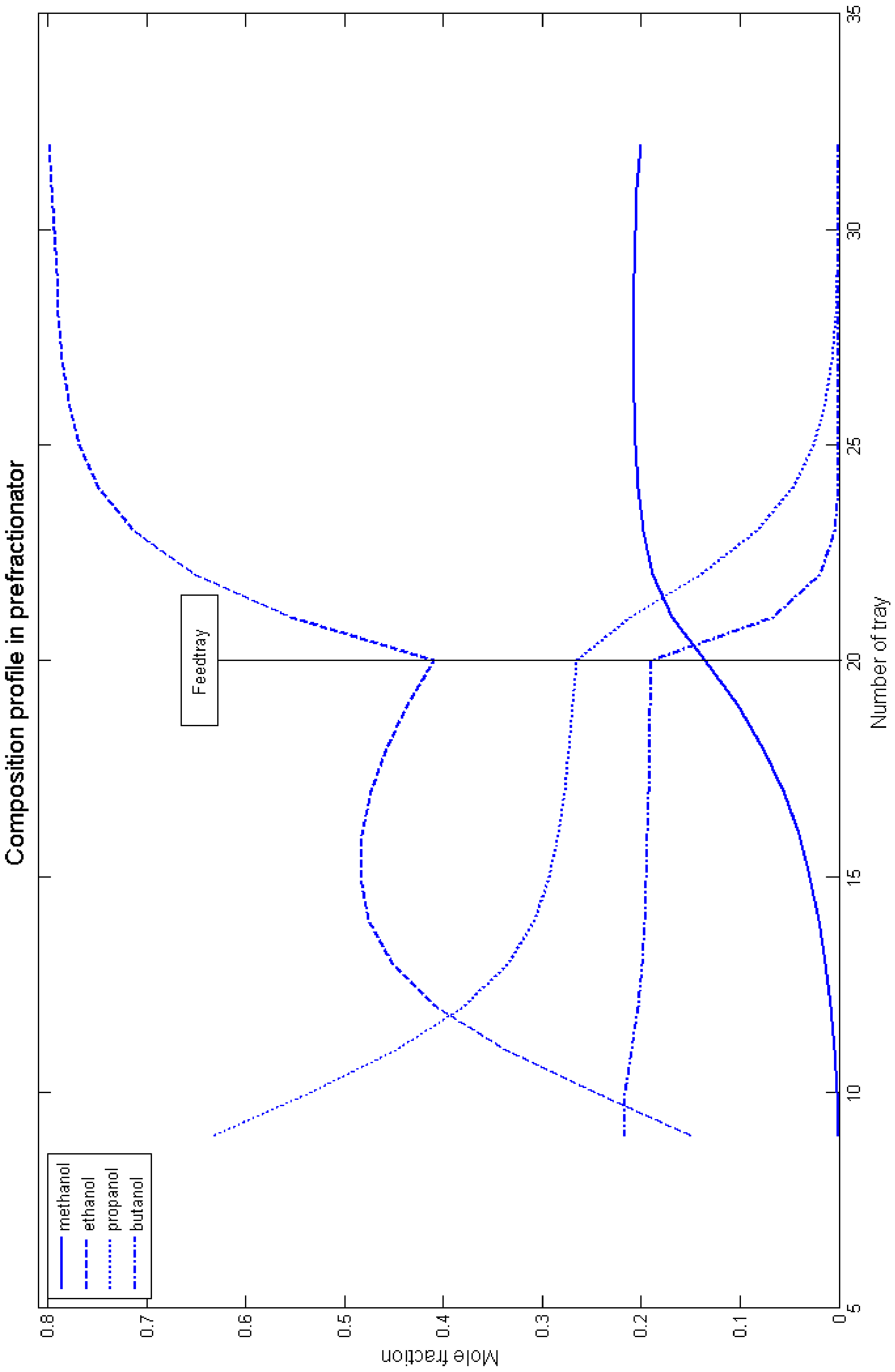


Chart 52: Rv+30% - composition in prefractionator

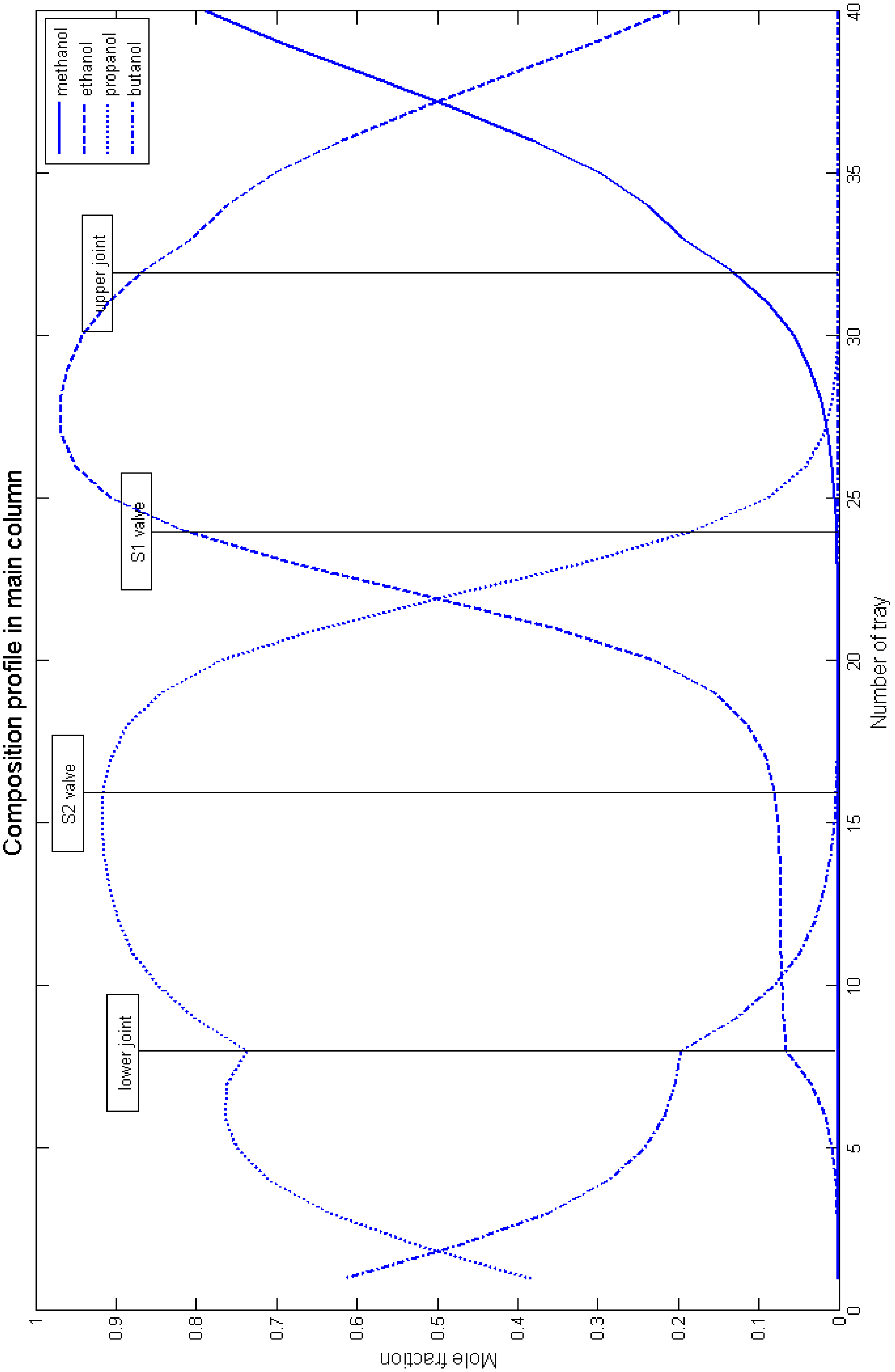


Chart 53: Rv+30% - composition in main column

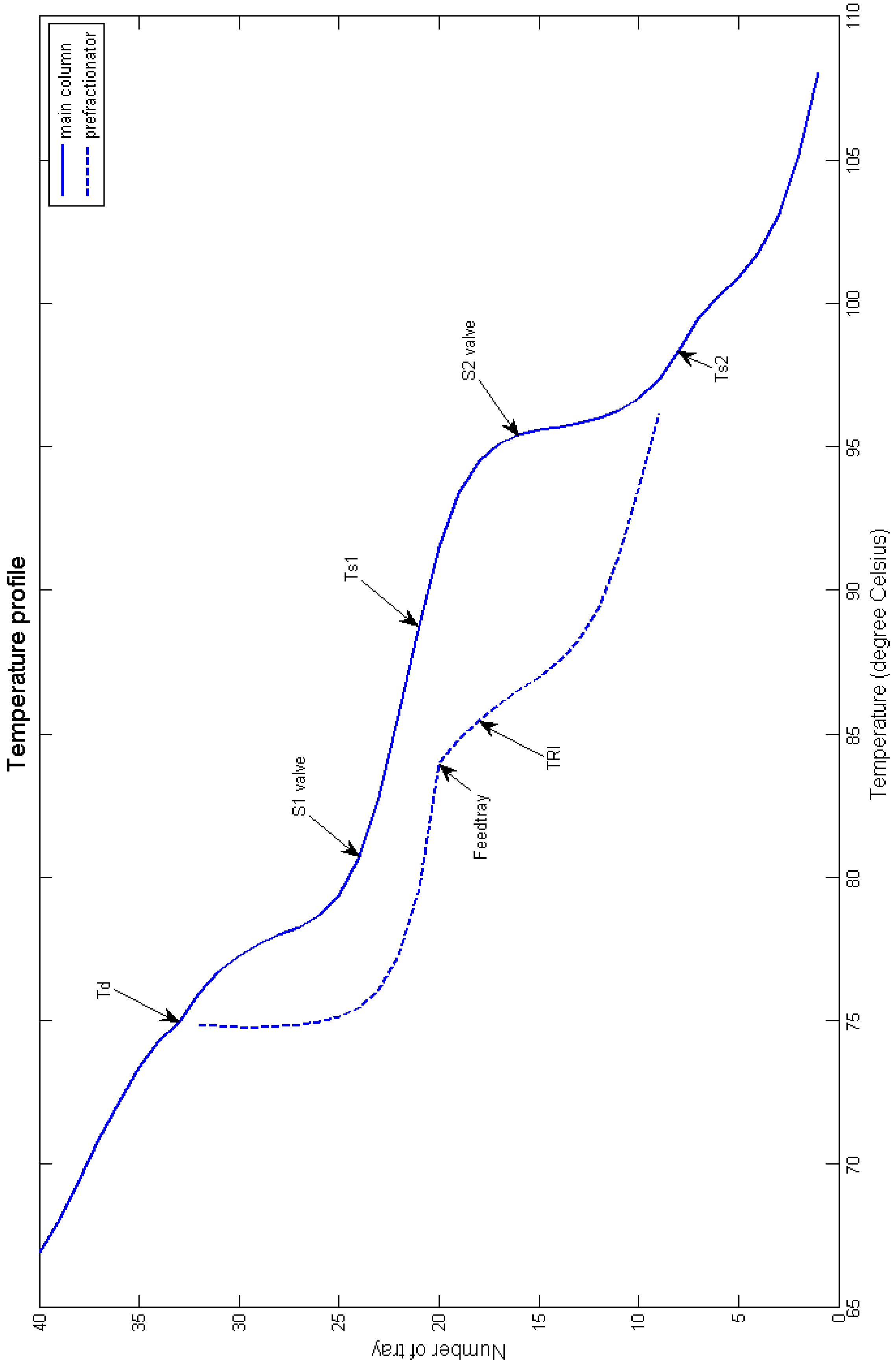


Chart 54: Rv+30% - temperature profile

11.15.2 Tables and discussion

Table 41: Inputs and disturbances

Degrees of freedom	setpoint	stage efficiency	
product D (Td) T33	75,9702°C	section	alpha
product S_1 (Ts1) T60	88,8047°C	1	0
product S_2 (Ts2) T49	98,3782°C	2	0
bottoms (Vapour boilup)	2,3000mol/min	3	0
prefractionator (R_L) T15	85,4931°C	4	0
R_V	0,4901	5	0
R_L	0,3962	6	0
		7	0

Table 42: Results

	%A	%B	%C	%D	flow (mol/min)
F	18	23	30	29	1,0000
D	78,83	21,17	0,00	0,00	0,2066
S_1	0,30	81,54	18,16	0,00	0,2022
S_2	0,03	8,01	91,62	0,34	0,2074
B	0,00	0,03	38,31	61,66	0,3837
				TOTAL	0,9999

A	methanol
B	ethanol
C	propanol
D	butanol

The concentration of propanol in the bottoms is more than double the concenetration of the base case (Table 42). Apperently, this causes to decrease the concentration of methanol in the S_2 stream. The setpoints remain constant (Table 41).

The temperature in the top of the prefractionator becomes a constant. This can be seen clearly in Chart 54. The composition in the prefractionator remains equal (Chart 52). The compostion in the main column is in the same order of the profile in the previous case. (Chart 53)

11.16 R_V +40%

11.16.1 Charts

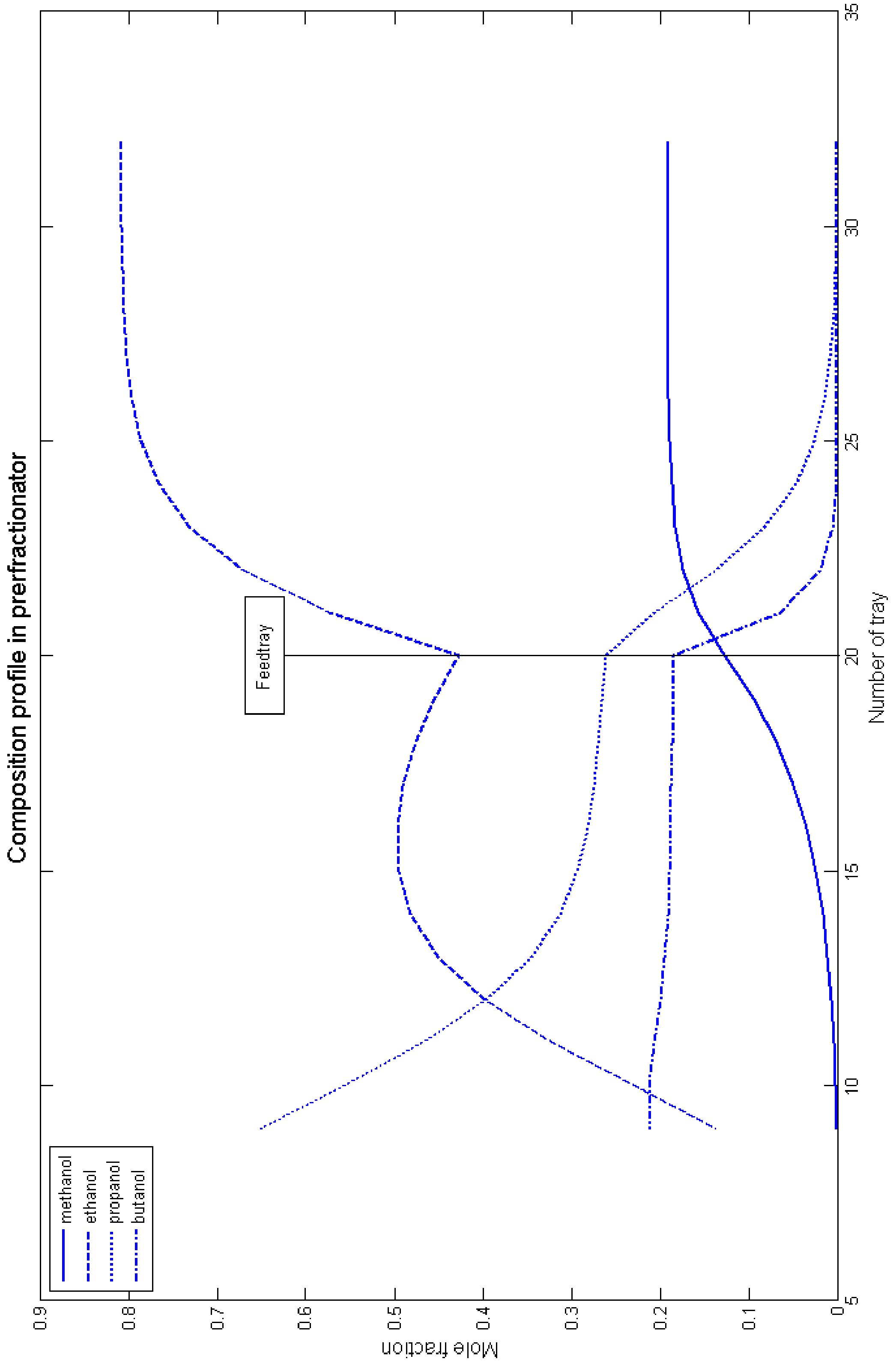


Chart 55: Rv+40% - composition in prefractionator

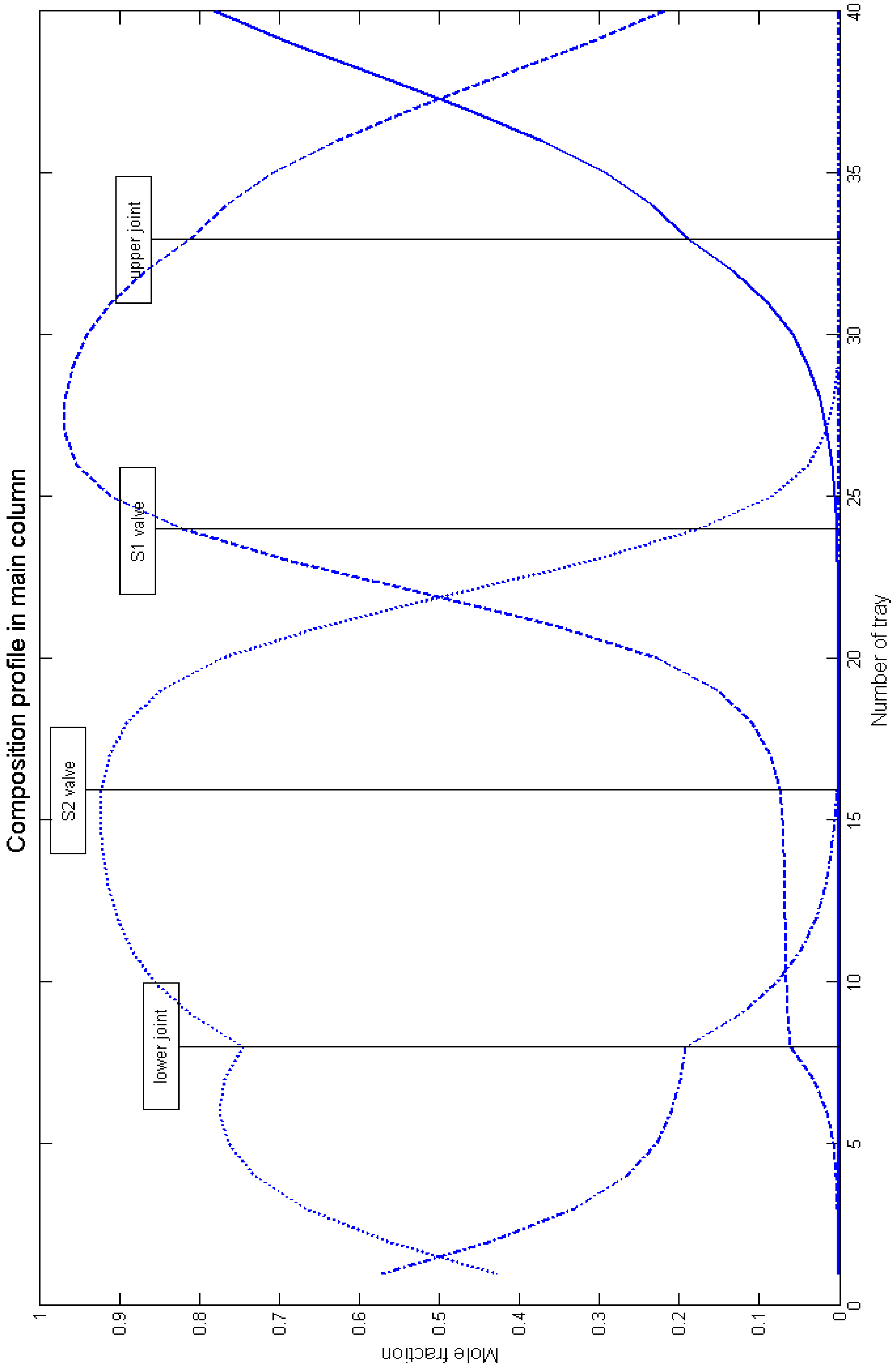


Chart 56: Rv+40% - composition in main column

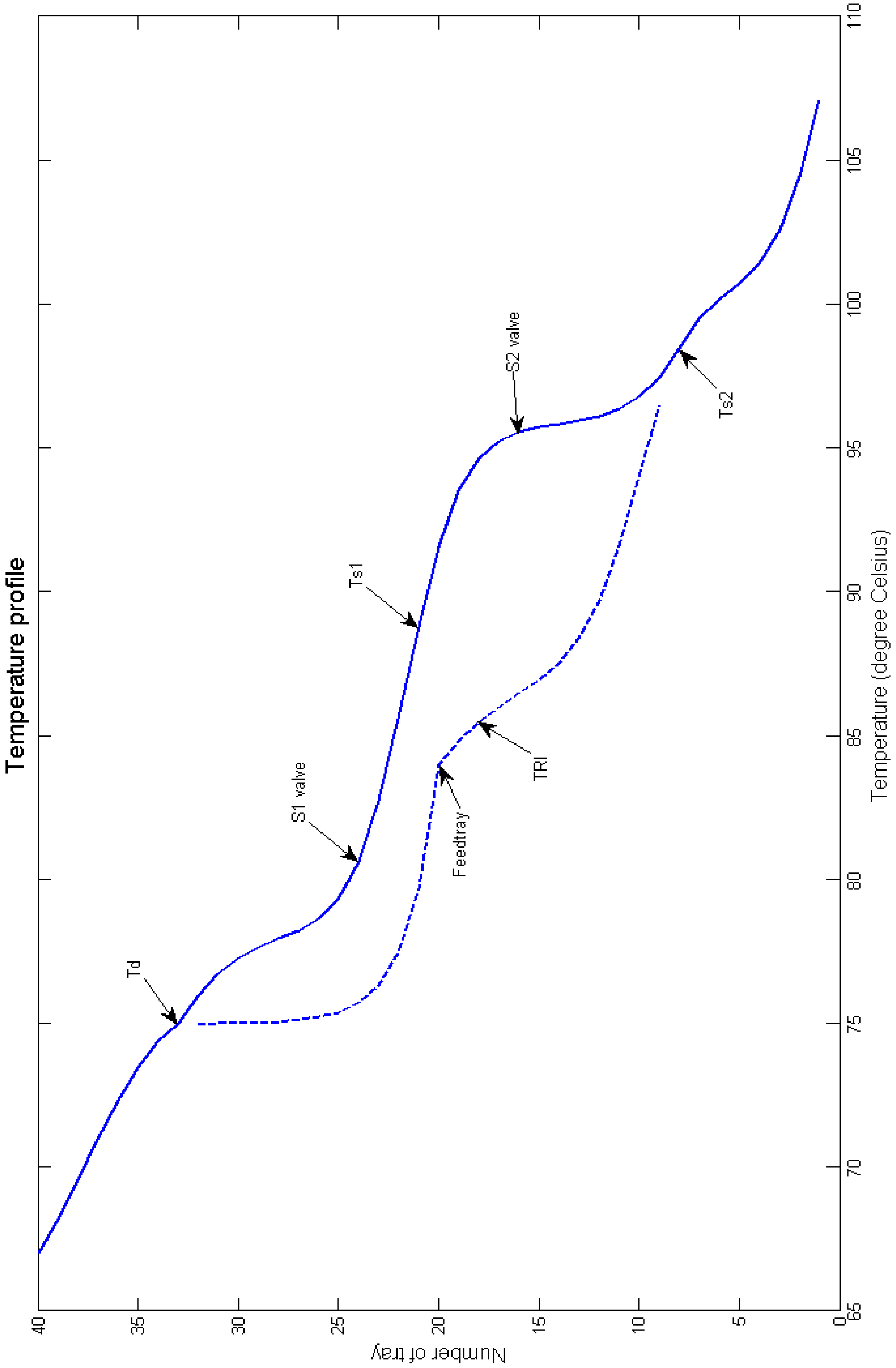


Chart 57: Rv+40% - temperature profile

11.16.2 Tables and discusison

Table 43: Inputs and disturbances

Degrees of freedom	setpoint	stage efficiency	
product D (Td) T33	75,9700°C	section	alpha
product S_1 (Ts1) T60	88,8013°C	1	0
product S_2 (Ts2) T49	98,4639°C	2	0
bottoms (Vapour boilup)	2,3000mol/min	3	0
prefractionator (R_L) T15	85,4673°C	4	0
R_V	0,5278	5	0
R_L	0,4304	6	0
		7	0

Table 44: Results

	%A	%B	%C	%D	flow (mol/min)
F	18	23	30	29	1,0000
D	78,20	21,80	0,00	0,00	0,2077
S_1	0,30	82,07	17,62	0,01	0,2033
S_2	0,02	7,51	92,15	0,32	0,1851
B	0,00	0,03	42,82	57,15	0,4038
				TOTAL	0,9999

A	methanol
B	ethanol
C	propanol
D	butanol

The concentration of propanol in the bottoms heads towards 50% (Table 44), while the temperature setpoints remain the same (Table 43).

In the pervious case, the temperature in the top of the prefractionator became stable. This trend is continued in this way that the composition of the product leaving through the top of the prefractionator becomes constant. This can be seen in Chart 55 and Chart 57. On the composition profile in the main column (Chart 56), the dominace of propanol can clearly be seen. This can be explained by the fact that not enough vapour is sent in the main column, which enables propanol to come down.

11.17 R_V – R_L ratio

In all the above simulations, the temperature setpoints remain as good as constant and the product flows differ only slightly. A look at the setpoint table can give an explanation. The R_L value changes together with the R_V value. This can be explained as followed.

If R_V decreases, less heat will enter the prefractionator. If R_L remains constant, the temperature, measured for the setpoint will drop. In order to reduce the drop, R_L is also reduced. The same happens when R_V increases.

All facts are placed in Table 45.

Table 45: Overview of Rv and Rl

change	Rv	Rl	Rl/Rv
-40	0,2262	0,1366	0,6039
-30	0,2639	0,1718	0,6510
-20	0,3016	0,2116	0,7016
-10	0,3393	0,2503	0,7377
-5	0,3582	0,2696	0,7527
-2	0,3695	0,2817	0,7624
-1	0,3732	0,2851	0,7639
0	0,3770	0,2885	0,7653
1	0,3808	0,2919	0,7665
2	0,3846	0,2953	0,7678
5	0,3959	0,3070	0,7754
10	0,4147	0,3249	0,7835
20	0,4524	0,3594	0,7944
30	0,4901	0,3962	0,8084
40	0,5278	0,4304	0,8155

The table shows that the ratio is not constant. This is against first thoughts. The ratio decreases as the decrease of R_V becomes larger. On the other hand, the ration tends to a constant as the increase of R_V becomes larger. This is shown in Chart 58.

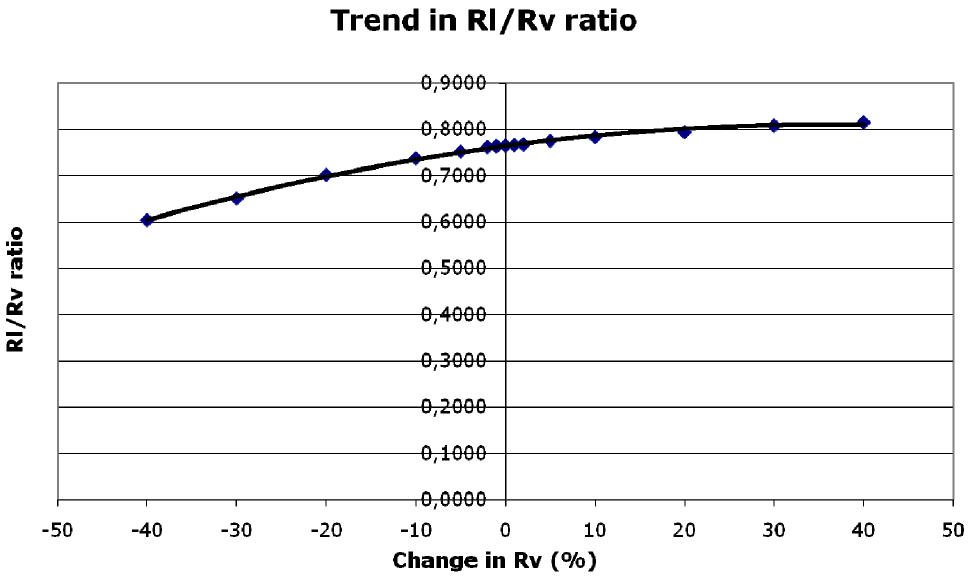


Chart 58: Trend in Rl/Rv ratio

11.18 Discussion

The effect of decreasing R_V doesn't affect the purity of the products as much as thought. An increase of R_V however makes a change in the composition of the bottom product. Propanol, the second heaviest product will come down in the main column and reach the reboiler.

The impurity of the distillate and the S_1 sidestream can be explained by non ideal setpoints. The peak in ethanol concentration lays higher than the valve where it is drained off. The setpoints in the upper part of the column needs to be lowered to get a better separation.

11.19 Conclusion

The influence of R_V on the product purities is not as big as assumed.

CONCLUSION

The column can be operated in a stable way, after some retuning of the control loops had been done. This made it also possible to get higher product purities.

The simulations have been used to study the influence of R_V on the product purities. Since R_V cannot be controlled in the lab, it was useful to do some simulations to get a better understanding in the influence of R_V. The simulations have shown that R_V doesn’t influence the purities as much as thought, although is plays an essential part in the column’s operation.

In this way, the simulations help to understand how the column works. This is useful when experiments are done. The experiments on the other hand will reveal problems that a simulator is not able to calculate, because it works with ideal conditions. That is why it is important to do experiments. With the results of the experiments, the model can be further optimized.

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