Control Strategies for a Combined Batch Reactor/ Batch Distillation Process

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Abstract: A batch reactor may be combined directly with a distillation column by distilling off the light component product in order to increase the reactor temperature or to improve the product yield of an equilibrium reaction. The controllability of such a system is found to depend strongly on the operating conditions, such as reactor temperature and composition of distillate, and on the time during the run. In general, controlling the reactor temperature (one point bottom control), is difficult since the set point has to be specified below a maximum value in order to avoid break-through of heavy component in the distillate. This maximum value may be difficult to know a priori. For the example considered in this study control of both reactor temperature and distillate composition (two-point control) is found to be difficult. As with one point bottom control, the reactor temperature has to be specified below a maximum value. However, energy can be saved since the vapor flow, and thereby the heat input to the reactor, can be decreased with time. Controlling the temperature on a tray in the column (one point column control) is found to give the best performance for the given process with no loss of reactant and a high reactor temperature although no direct control of the reactor temperature is obtained.

Keywords: Reactive batch distillation, controllability, control strategies

1   Introduction

Batch distillation is used in the chemical industry for the production of small amounts of products with high added value and for processes where flexibility is needed, for example, when there are large variations in the feed composition or when production demand is varying. Batch reactors are combined with distillation columns to increase the reaction temperature and to improve the product yield of equilibrium reactions in the reactor by distilling off one or more of the products, thereby driving the equilibrium towards the products.

Most often the control objective when considering batch processes is either i) to minimize the batch time or ii) to maximize the product quality or yield. Most of the papers published on batch distillation focus on finding optimal reflux ratio policies. However, sometimes the control objective is simply to obtain the same conditions in each batch. This was the case for the specific industrial application which was the starting point for our interest in this problem and which is to be presented later.

NEW

Few authors have considered the operation of batch distillation with chemical reaction although these processes are inherently difficult to control. The analysis of such systems in terms of controllability has so far only been considered by Sørensen and Skogestad [11]. Roat *et al.* [8] have developed a methodology for designing control schemes for continuous reactive distillation columns based on interaction measures together with rigorous dynamic simulation. However, no details about their model were given.

Modelling and simulation of reactive batch distillation has been investigated by Cuille and Reklaitis [2], Reuter *et al.* [7] and Albet *et al.* [1]. Cuille and Reklaitis [2] developed a model and solution strategies for the simulation of a staged batch distillation column with chemical reaction in the liquid phase. Reuter *et al.* [7] incorporated the simulation of PI-controllers in their model of a batch column with reaction only in the reboiler. They stated that their model could be used for the investigation of control structure with the aid of Relative Gain Array analysis (RGA) but no details were given. Albet *et al.* [1] presented a method for the development of operational policies based on simulation strategies for multicomponent batch distillation applied to reactive and non-reactive systems.

Egly *et al.* [3], [4] considered optimization and operation of a batch distillation column accompanied by chemical reaction in the reboiler. Egly *et al.* [3] presented a method for the optimization of batch distillation based upon models which included the non-ideal behavior of multicomponent mixtures and the kinetics of chemical reactions. The column operation was optimized by using the reflux ratio as a control variable. Feeding one of the reactants during the reaction was also considered. In a later paper [4], they also considered control of the column based upon temperature measurements from different parts of the column. The optimal reflux ratio policy was achieved by adjusting the distillate flow using a non-linear control system. However, no details where given about neither the column/reactor nor the control system.

The purpose of this paper is to investigate the possible difficulties in controlling a coupled system of a reactor and a distillation column, and also to give some alternative control strategies based on an industrial example. First, a model of the industrial process, consisting of a batch reactor with a rectifying column on top, is developed. Based on a linearized version of this model, we compare different operating points to show how the model differs, that is, whether the same controller settings can be used for different reactor conditions or reactor temperatures. In the various operating points we also consider the stability of the system and the response to step changes in flows. We consider *two-point control*, when both the top and the bottom part are controlled, as well as *one point control*, when only one part of the column/reactor is controlled. A Relative Gain Array (RGA) analysis is used for the investigation of control structures in two point control. Finally, the similarities and differences between our process and a conventional continuous distillation column is considered.

The reaction is reported to be of 0'th order and due to limited data, we also assume the rate to be independent of temperature. However, interesting observations can still be made concerning the coupling between the formation of product in the reboiler and the separation in the column above. Indeed, later work [6], has confirmed that this simplification does not affect the conclusions. The influence of disturbances on the system, e.g. in reaction rate or in temperature measurements, has not been considered in this study.

Column:	6 trays + condenser
Reaction:	$0.5\,R_1 + 0.36\,R_2 + 0.14\,R_3 \rightarrow P(s) + W$
Volatile components:	W $(T_b = 100\ ^{\circ}C)$ and R_2 $(T_b = 188\ ^{\circ}C)$
Non-volatile components:	R_1 $(T_b = 767\ ^{\circ}C)$, R_3 $(T_b = 243\ ^{\circ}C)$ and P (solid)
Vapor pressure:	R_1: $\ln P^s_{R_1} = -4009.3 + 176750.0/T_i + 6300.0 \log T_i$ $\qquad - 0.51168 T_i$ (Pa)
	R_2: $\ln P^s_{R_2} = 25.4254 - 6091.95/(-22.46 + T_i)$ (Pa)
	R_3: $\ln P^s_{R_3} = 231.86 - 18015.0/T_i - 31.753 \log T_i$ $\qquad + 0.025 T_i$ (Pa)
	W: $\ln P^s_W = 23.1966 - 3816.44/(-46.13 + T_i)$ (Pa)
Relative volatility (W and R_2):	8-32
Startup time:	30 min
Total reaction time:	15 hr
Pressure in column/reactor:	1 atm/1.2 atm
Reaction rate, r:	1.25 kmol/hr
Initial vapor flow, V:	16.8 kmol/hr
Hydraulic time constant, τ:	0.0018 hr =6.5 s
Initial holdups:	reactor: 24 kmol
	condenser: 1.6 kmol
	trays: 0.09 kmol
Initial amounts in reactor:	R_1: 10.4 kmol (Acid)
	R_2: 7.5 kmol (Alcohol) (20 % excess)
	R_3: 3.2 kmol (Alcohol)
	P: 0.0 kmol (Ester)
	W: 2.5 kmol (Water)

Table 1: Process data for simulation.

2 Process example

The motivation for this study was an industrial equilibrium esterification reaction of the type

$$\xi_1 R_1 + \xi_2 R_2 + \xi_3 R_3 \rightleftharpoons P(s) + W$$
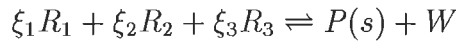
where R_1 is a dibasic aromatic acid, R_2 and R_3 are glycols, P is the solid polymer product and W is the bi-product water. The reaction takes place in a reactor heated by a heating jacket with heat oil. The equilibrium is pushed towards the product side by distilling off the low boiling by-product W from the reactor. Only reactant R_2 and the by-product W are assumed to be volatile, and the binary separation between these two components takes place in the column. The reaction rate was reported to be of zero order; independent of compositions. Due to lack of data we also assume the rate is independent of temperature. A summary of the process data is given in Table 1. In the industrial unit the amount of reactant R_2 in the feed was 20 % higher than necessary to yield complete conversion of the reaction, and this was also assumed in most of our simulations. This was done to account for the possible loss of the reactant in the distillate.

The existing operating practice was to use *one-point top control*; the temperature at the top of the column T_T was kept constant at about 103 $^{\circ}C$ which gave a distillate composition of 0.004 (about 2 weight%) of the heavy component R_2 and thereby a loss of this component. The vapor flow was kept constant by using maximum heating of the
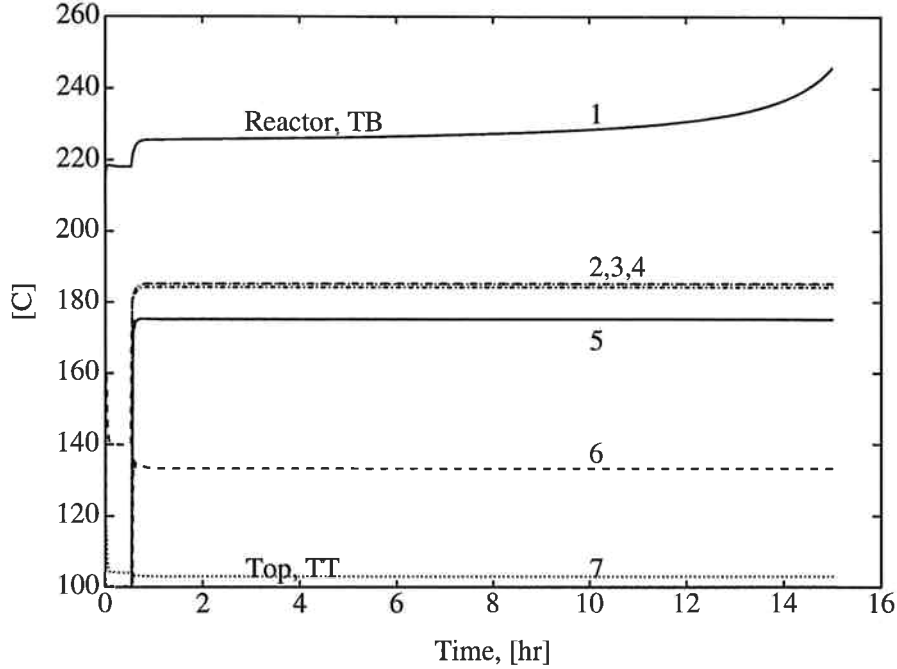
Figure 1: The existing temperature profile in column/reactor.

reactor and the condenser level was controlled by the distillate flow D. The temperature profile at different locations in the column as a function of time is given in Fig. 1. The reactor temperature T_B is almost constant at the beginning but increases as reaction proceeded. The conditions on tray 2, 3 and 4 are practically equal because the column has more stages than needed for the desired separation. With the existing control scheme there is no direct control of the reactor temperature, T_B and more severely, it gives a varying loss of the heavy component, reactant R_2. This leads to a varying quality of the product P between batches.

3 Mathematical model

In this section we consider the mathematical description of the batch distillation column and reactor shown in Fig. 2 and described in the previous section. The equations for the individual stages consist of the total mass balance, the mass balance for each component, tray hydraulics and phase equilibrium and are valid under the following assumptions:

A1 A staged model is used for the distillation column.

A2 A multicomponent mixture in the reactor, but a binary mixture in the distillation column is considered.

A3 Perfect mixing and equilibrium between vapor and liquid on all stages is assumed.

A4 The vapor phase holdup is negligible compared to the liquid phase holdup.

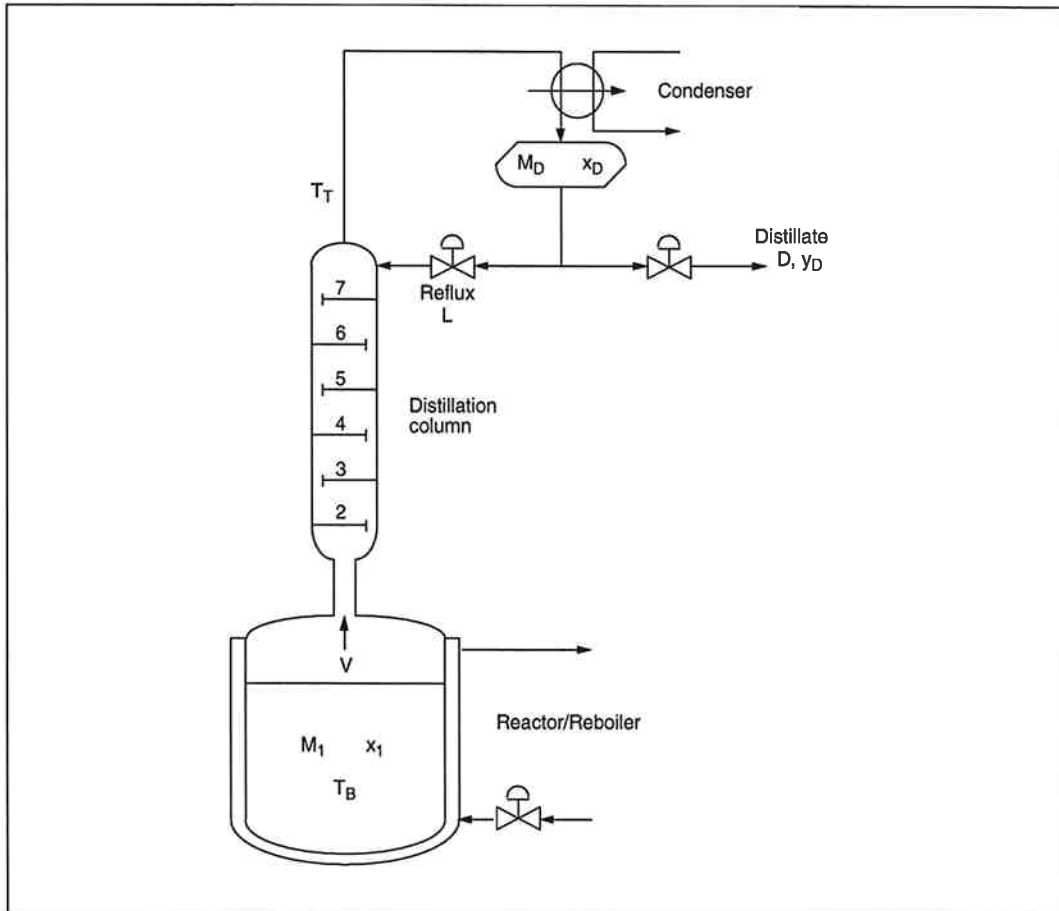**A5** The stage pressures and the plate efficiencies are constant.

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Figure 2: Batch distillation column/reactor.

A6 Constant molar flows are assumed (no energy balance).

A7 Linear tray hydraulics is considered.

A8 Total condensation with no subcooling in the condenser is assumed.

A9 The chemical reaction is limited to the reactor.

A10 Raoult's law for the vapor-liquid equilibrium holds.

Below i denotes the stage number and j the component number ($j = 1, 2$ are the volatile components W and R_2). The following differential and algebraic equations result.

reactor/reboiler, i=1 :

$$dM_1/dt = L_2 - V + \sum_{j=1}^{4} \xi_j r \tag{1}$$

$$d(M_1 x_{1,j})/dt = L_2 x_{2,j} - V y_{1,j} + \xi_j r \quad , j = 1 \tag{2}$$

reaction components not distilled :

$$d(M_1 x_{1,j})/dt = \xi_j r \quad , j = 3, 4 \tag{3}$$

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column tray, i=2,N :

$$dM_i/dt = L_{i+1} - L_i \tag{4}$$

$$d(M_i x_{i,j})/dt = L_{i+1} x_{i+1,j} + V y_{i-1,j} - L_i x_{i,j} - V y_{i,j} \quad , j = 1 \tag{5}$$

condenser, i=N+1 :

$$dM_{N+1}/dt = V - L_{N+1} - D \tag{6}$$

$$d(M_{N+1} x_{N+1,j})/dt = V y_{N,j} - L_{N+1} y_{D,j} - D y_{D,j} \quad , j = 1 \tag{7}$$

linearized tray hydraulics :

$$L_i = L_{oi} + \frac{M_i - M_{oi}}{\tau} \tag{8}$$

liquid-vapor equilibrium :

$$y_{i,j} = \frac{\alpha_i x_{i,j}}{1 + (\alpha_i - 1)\, x_{i,j}} \tag{9}$$

relative volatility :

$$\alpha_i = f\left(T_i\right) = \frac{P_1^s\left(T_i\right)}{P_2^s\left(T_i\right)} \tag{10}$$

temperatures :

$$P_i = \sum_{j=1}^{2(4)} x_j P_j^s\left(T_i\right) \tag{11}$$

On each stage the composition of component $j = 2$ (R_2) is obtained from $\sum x_j = 1$. Note that all four components were used to calculate the reactor temperature using eq. 11, but only the two lightest components were considered in the column. The model is highly non-linear in vapor composition y and in temperature T. On vector form the differential equation system to be solved can be written

$$d\mathbf{x}/dt = \mathbf{f}[\mathbf{x}(t), \mathbf{u}(t)] \tag{12}$$

In addition there is a set of algebraic equations, equations (8)-(11)

$$0 = \mathbf{g}[\mathbf{x}(t), \mathbf{u}(t)] \tag{13}$$

Eq. (12-13) constitute a set of differential-algebraic equations (DAE). The equations are solved using the equation solver LSODE [5]. The startup conditions are total reflux and no reaction.

3.1   Linear model

In order to investigate the controllability of a process using available tools, a linear model is needed. Based on the non-linear model described by eq. (12) and (13) a linear model can be developed by linearizing the equation system at a given operating point. For continuous processes normally only one operating point considered; that of the steady state conditions. The linear model is then found by linearizing around this operating point and will be valid for small deviations from the steady state. When considering batch processes there is no such steady state; the conditions in the reactor or column are changing with time and the model is linearized along a trajectory. A linearized model of

Controlled variables (y):	Manipulated variables (u):
condenser holdup M_D	distillate flow D
distillate composition y_D	reflux flow L
reactor temperature T_B	vapor flow V

Table 2: Controlled and manipulated variables.

the process, representing deviations from the "natural drift" along the trajectory with D, L and V constant, can be described by the following equations:

$$
\begin{aligned}
dx/dt &= Ax + Bu \\
y &= Cx
\end{aligned}
\tag{14}
$$

Where

$$
\begin{aligned}
\mathbf{x} &= [\Delta x_1, \Delta M_1.., \Delta n_3,..]^T \\
\mathbf{y} &= [\Delta M_D, \Delta y_D, \Delta T_B]^T \\
\mathbf{u} &= [\Delta D, \Delta L, \Delta V]^T
\end{aligned}
$$

Laplace transformation yields:

$$
\mathbf{y}(s) = G(s)\mathbf{u}(s)
\tag{15}
$$

The control problem will thus have the controlled and manipulated variables as given in Table 2. It is then assumed that the vapor flow V can be controlled directly, even though the real manipulated variable is the heat input to the reactor.

4 Analysis of linear model

4.1 Operating procedures

The linear model depends on the operating point. To study these variations we initially considered four different operating procedures:

I The existing operating practice, $T_T = 103^0$ C (one point top control, V constant)

II $T_B = 200^0$ C (one-point bottom control, V constant)

III $T_B = 222^0$ C (one-point bottom control, V constant)

IV $T_B = 228^0$ C (one-point bottom control, V constant)

Temperature profiles for the four operating procedures are given in Fig. 3. For operating procedure I, II and III the conditions are more or less constant with time, whereas procedure IV has a changing temperature profile with large variations at the beginning of the batch but more or less stabilizing midway through the batch. For operating point I ($T_T = 103\ ^0C$), the front between light and heavy component is kept high in the column giving a loss of the heavy component R_2. For procedure II ($T_B = 200\ ^0C$), the front is low and the composition of heavy component R_2 is almost negligible from tray 3 and up, giving a very pure distillate. However, the reactor temperature is low and it is unlikely
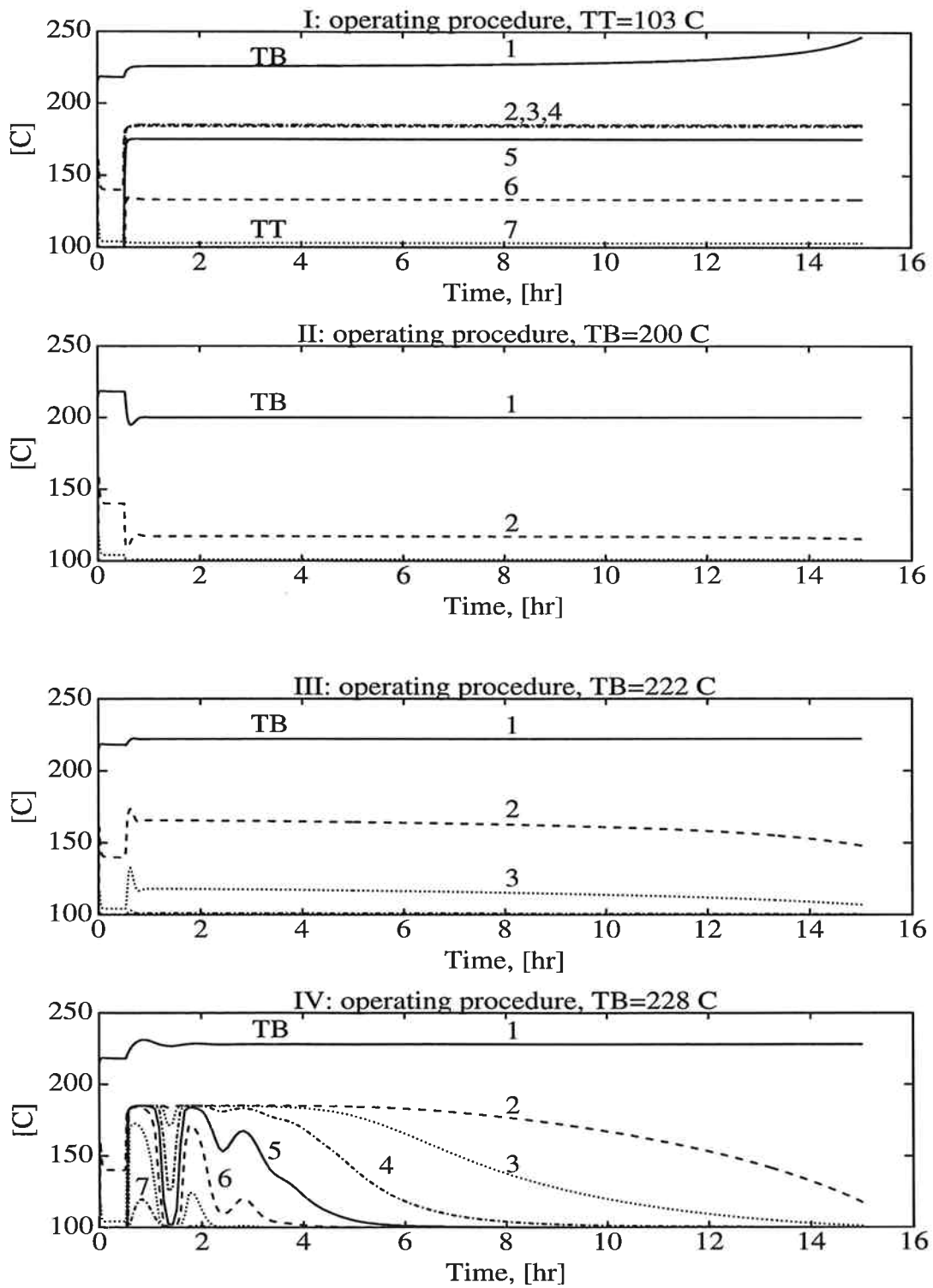
Figure 3: Temperature profiles in column/reactor for different operating procedures.

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that the assumed reaction rate will be achieved. When the reactor temperature is increased to $T_B = 222 \, ^0C$ for procedure III the composition of R_2 in the column section increases pushing the light/heavy component front upwards in the column. At the end of the batch the front detracts slightly giving more light component in the bottom part. For procedure IV, at $T_B = 228 \, ^0C$, the front between light and heavy component is lifted so high up in the column that it leads to a "break-through" of heavy component R_2 in the distillate and thereby causing the large variations in the profile. After the loss of R_2 the light/heavy component front detracts continuously during the batch.

Of the four operating procedures, procedure III ($T_B = 222 \, ^0C$) is the only one with both a high reactor temperature and at the same time no loss of reactant R_2. Procedure IV ($T_B = 228 \, ^0C$) gives a substantial loss of reactant R_2 and is therefore not considered further.

4.2   Linear open-loop model

To illustrate how the process behavior changes during the batch the equation system (eq. 12 and 13) is linearized at different operating points; that is at different reactor conditions or times during a batch. (Notation: An operating point is specified as *procedure-time*, eg. I-8 is the conditions with operating procedure I after 8 *hr* reaction time.) These linear models were found by first running a non-linear simulation of the process with control loops implemented (level control in the condenser and temperature control of tray 1 or of the reboiler) in order to obtain a given profile in the column/reactor. The simulations were then stopped at the specified time, all the controller loops opened and the model linearized numerically. (We would get the same responses, in Δy_{DH} and ΔT_B from steps in ΔL and ΔV if the condenser level loop was to remain closed with the distillate flow D during the linearization. This is because L and V have a direct effect on compositions and the effect of the level loop is a second order effect which vanishes in the linear model.) The resulting linear model is thus an *open loop description* of the process at the given time and conditions; it describes how the system responds to changes when no controllers (or only the level controller) are implemented.

4.3   Step responses

To illustrate how the process behavior changes with conditions in the reactor we consider step changes to the linearized models. The effect of a step in the vapor flow V on y_{DH} and T_B (deviation from nominal value) for three different operating procedures after 8 hr is given in Figure 4. The variation of the linear model within batch III is illustrated by Fig. 5. The responses in y_{DH} for different reactor conditions (top part of Fig. 4) are similar but differ in magnitude. This is because in operating point II-8, where we have a very low reactor temperature, we have a very pure distillate. The increase in reflux will only increase the purity marginally. Whereas in operating point I-8, we have a distillate which is less pure so the increase will be larger. We note from Fig. 5 that the variations within the batch are large for the response in reactor temperature. The main reason for the changes in the responses for this temperature (lower part of Fig. 4 and 5) is the concentration of water in the reactor. That is, a higher water concentration gives a larger effect.
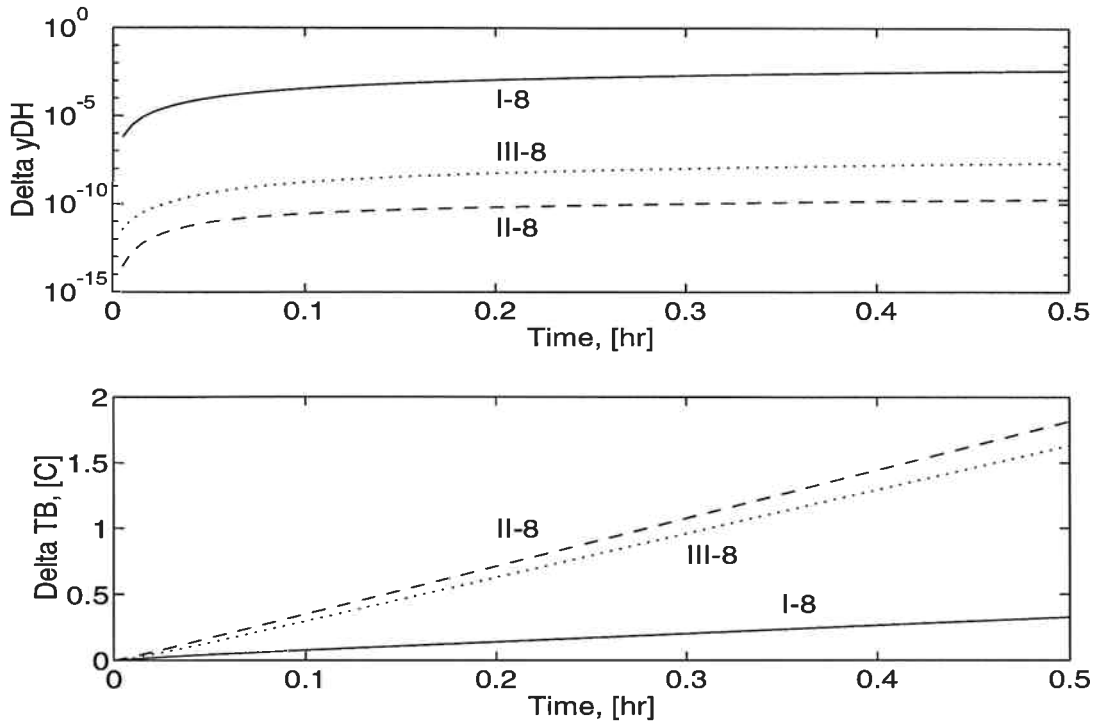
Figure 4: Step in vapor flow ($\Delta V = 0.1$) for linear model: effect on Δy_{DH} and ΔT_B for procedure I-8 ($T_T = 103\ ^0C$), II-8 ($T_B = 200\ ^0C$) and III-8 ($T_B = 222\ ^0C$).
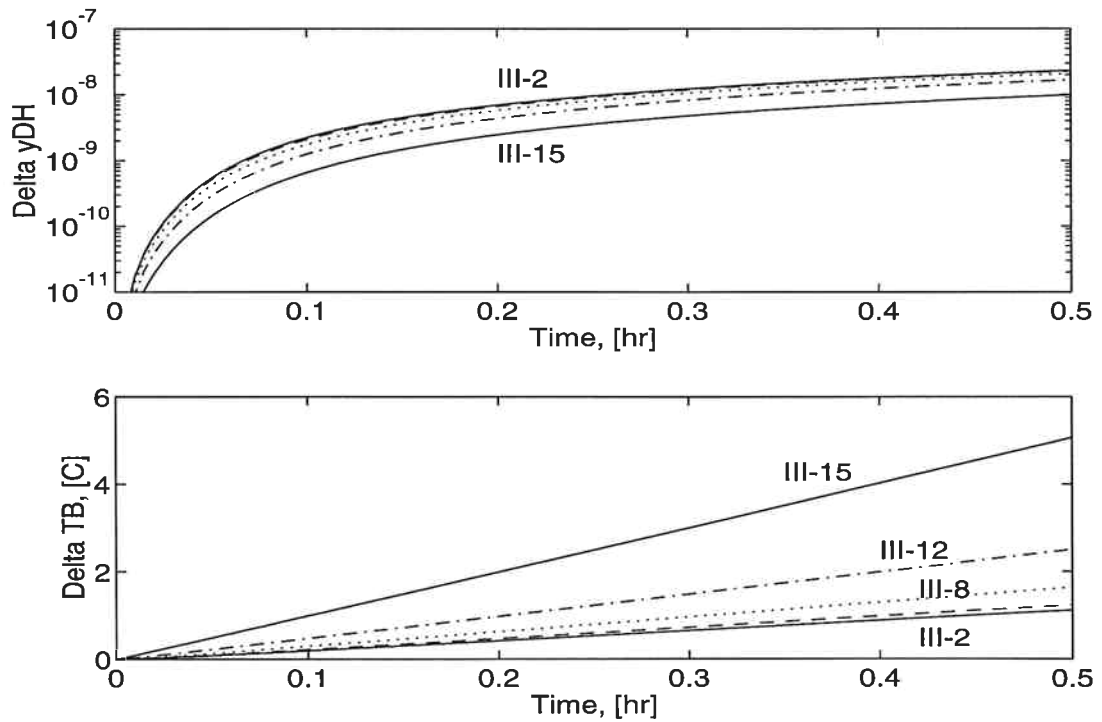

Figure 5: Step in vapor flow ($\Delta V = 0.1$) for linear model: effect on Δy_{DH} and ΔT_B for procedure III-2 to III-15 ($T_B = 222\ ^0C$).
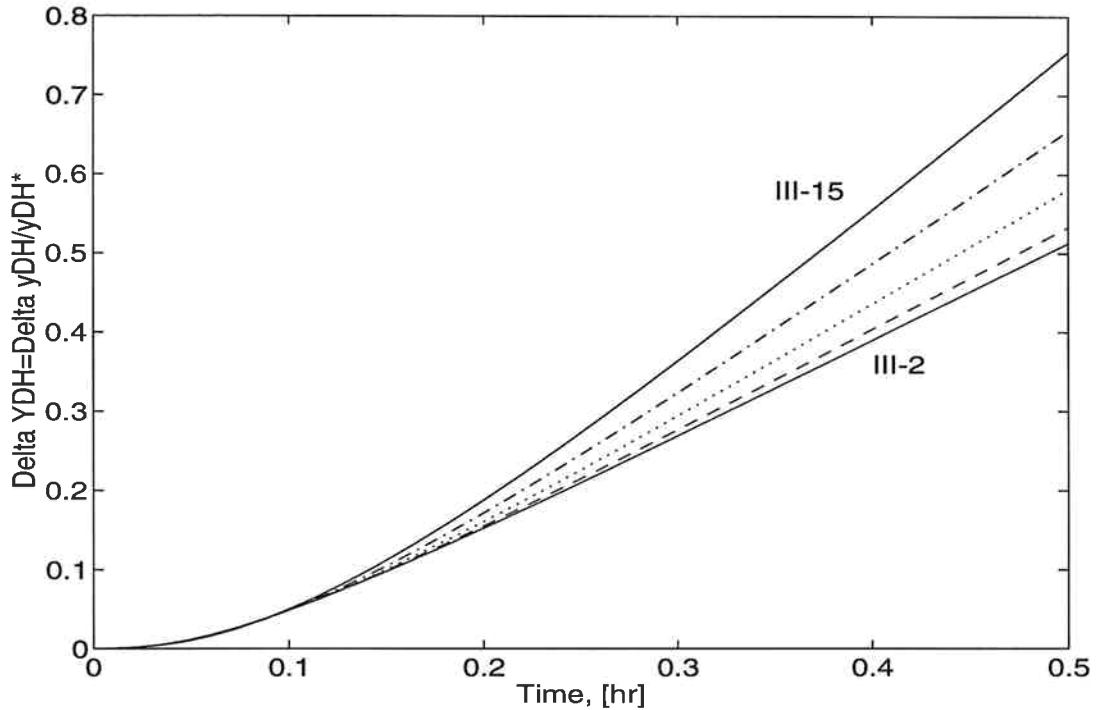
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Figure 6: Logarithmic transformation for linear model: Different times during batch for procedure III ($T_B = 222\ ^0C$).

4.4   Reducing the non-linearity for top composition

An interesting feature in Fig. 4 and 5 is that the responses in y_{DH} to step changes have a similar initial shape on a log-scale. This is actually a general property for distillation [9]. The inherent nonlinearities in this variable can therefore be reduced by using a log transformation on the distillate composition y_D:

$$Y_D = -\ln\left(1 - y_D\right) \tag{16}$$

which in deviation variables becomes

$$\Delta Y_D = \frac{\Delta y_D}{1 - y_D^*} \quad , \quad \Delta Y_{DH} = \frac{\Delta y_{DH}}{y_{DH}^*} \tag{17}$$

The responses in mole fraction of heavy component R_2 in the distillate after the transformation, Y_{DH}, is given in Fig. 6 for operating points III-2 to III-15. These responses are of the same order of magnitude and the non-linearity is thereby reduced. From Fig. 4 and 5 there is no obvious transformation that can be suggested to deal with the non-linear effect for the reactor temperature.

5   Control strategies

The varying loss of reactant R_2 in the distillate and the lack of direct control of the reactor temperature were the major problems with the existing operating practice. In the control part of this study the following control strategies are compared:

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- one-point bottom control (controlling the reactor temperature directly)

- two-point control (controlling both the distillate composition and the reactor temperature)

- one-point column control (controlling the temperature on a tray in the column)

The control parameters for the PI-controllers used in the simulations are given in Table 3. Note that an integral time of $\tau_i = 0.1hr = 6min$ was used in all the simulations and that the transformed variable Y_D was used instead of y_D for two-point control.

level control:	$K_p = -500$ and $\tau_i = 0.1$ ($M_D \rightarrow D, L$)
bottom control:	$K_p = 1.0$ and $\tau_i = 0.1$ ($T_B \rightarrow L$)
two-point control:	$K_p = 0.456$ and $\tau_i = 0.1$ ($Y_D \rightarrow L$)
	$K_p = -4.0$ and $\tau_i = 0.1$ ($T_B \rightarrow V$)
column control:	$K_p = 1.0$ and $\tau_i = 0.1$ ($T_5 \rightarrow L$)

Table 3: Control parameters used in the simulations.

5.1 One-point bottom control

The objective with one-point bottom control is to keep the reactor temperature constant at the highest possible temperature as this will maximize the rate of reaction for a temperature dependent reaction. The reflux flow is used as manipulated variable and the vapor flow is kept at its maximum value ($V = V_{max} = 16.8$ kmol/hr). However, it is very difficult to achieve a high value of T_B and at the same time avoid "break-through" of the heavy component R_2, in the distillate. This is illustrated in Fig. 7 which shows how the mole fraction of R_2, y_{DH}, changes when the set point for the temperature controller in the reactor increases from $T_{B,set} = 224.5^0C$ to $T_{B,set} = 225^0C$. An increase of 0.5^0C causes the mole fraction of reactant R_2 to increase by a factor of 25. The loss of reactant is only temporary, and y_{DH} is reduced to ≈ 0 after about 1 hr. The break-through is caused by the fact that when the specified temperature is above a certain maximum value where most of the light component W is removed, then a further increase is only possible by removing the heavy component, reactant R_2. If the set point temperature is specified below the maximum value, in this case $\approx 224.0^0C$, good control of the system ($T_B \approx T_{B,set}$ and $y_{DH} \approx 0$) is achieved. The system can, however, become unstable at the end of the batch depending on the choice of control parameters in the PI-controller. This due to the non-linearity in the model causing the system to respond differently to changes at different times during the batch as illustrated in Fig. 5.

Another alternative for raising the reaction temperature, and thereby the reaction rate for a temperature dependent reaction, is to let the set point follow a given trajectory, e.g. a linear increase with time. Again, the maximum reactor temperature to avoid break-through will limit the possible increase and break-through is inevitable if it is specified too high. Fig. 8 illustrates a run when the set point follows a linear trajectory from 220^0C at $t = 0.5$ hr to 245^0C at $t = 15$ hr. The loss of reactant R_2 is substantial, almost 10 % of the feed of this component. By lowering the endpoint temperature to 230 ^0C, loss of reactant is avoided (not shown).
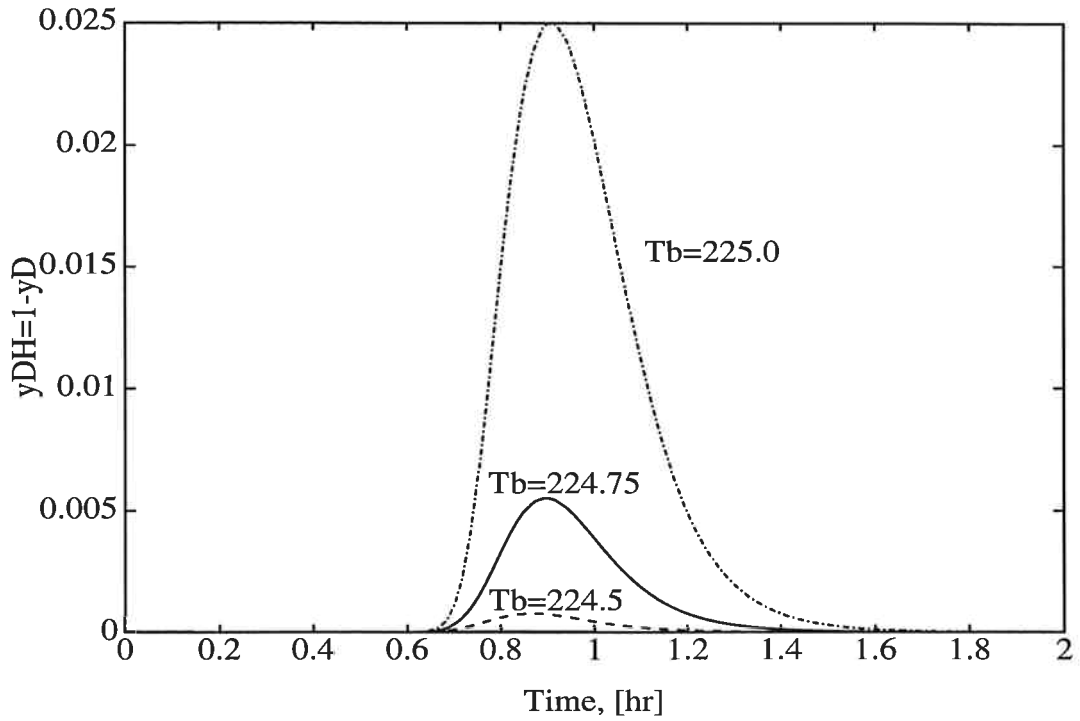
Figure 7: One-point bottom control. Heavy component in distillate at different $T_{B,set}$ for the first two hours of the batch.
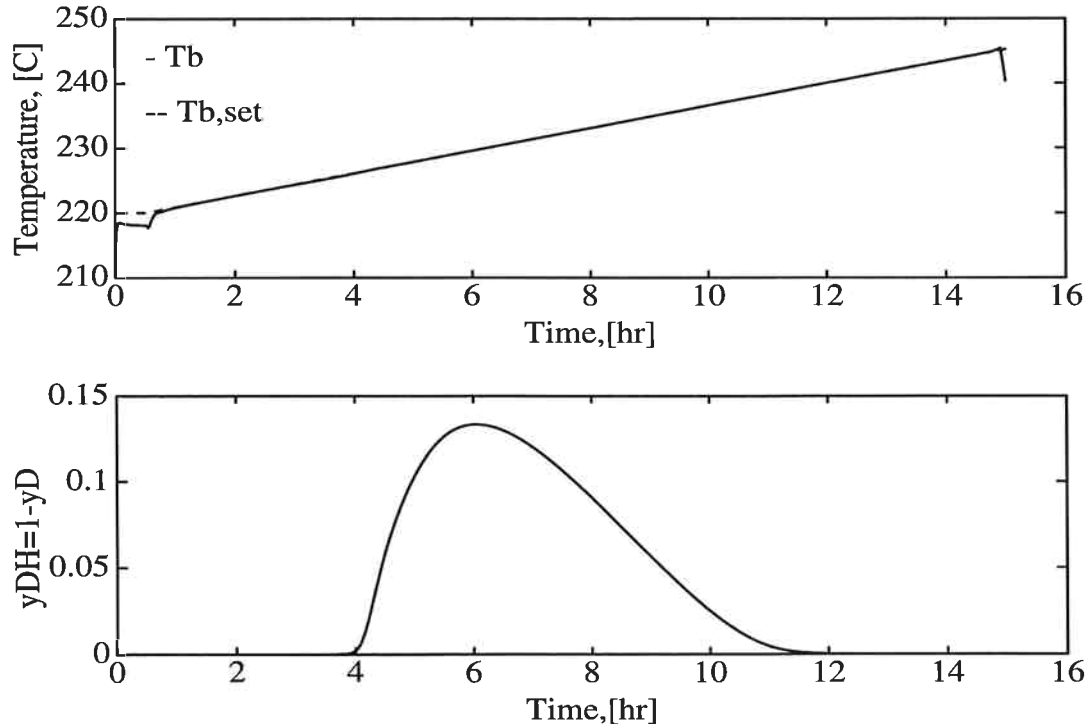

Figure 8: One-point bottom control. Reactor temperature for T_B increasing from 220^0C (t=0.5) to 245^0C (t=15).

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5.2   Two-point control

By using *two-point control* it may be possible to control both the top and the bottom part of the distillation column by implementing two single control loops in the system. In this way energy consumption can be reduced since it will no longer be necessary to keep the vapor flow V, and thereby the temperature or amount of heating oil, at its maximum value. In the case of the esterification process, it is desirable to control not only the reactor temperature T_B but also the composition of the distillate y_D, i.e. the loss of reactant R_2.

Two different control configurations are considered for the batch column:

LV-**configuration**   Controlling the condenser level using the distillate flow D leaving the reflux flow L and the vapor flow V to control the distillate composition Y_D and the reactor temperature T_B:

$$M_D \longleftrightarrow D$$
$$Y_D, T_B \longleftrightarrow L, V$$

DV-**configuration**   Controlling the condenser level using the reflux flow L leaving the distillate flow D and the vapor flow V to control the distillate composition Y_D and the reactor temperature T_B:

$$M_D \longleftrightarrow L$$
$$Y_D, T_B \longleftrightarrow D, V$$

5.2.1   Controllability analysis of two point model

Open-loop step responses for both configurations are given in Fig. 9 and 10 for operating point III-8 ($T_B = 222\ ^0C$ at $t = 8hr$). The term "open-loop" should here be put in quotes because we are not talking about an uncontrolled column, but assume that the condenser level is perfectly controlled ($M_D \longleftrightarrow D$ or $M_D \longleftrightarrow L$) and we consider the effect of the remaining independent variables on the composition and reactor temperature.

From Fig. 9 it can be seen that for the LV-configuration the responses to steps in L and V are similar but in opposite direction. For the DV-configuration the responses by a step in D are similar as for the step in V for the LV-configuration. However, the responses to a step in V is very small. This is a general property for distillation.

In a distillation column there are large interactions between the top and the bottom part of the column, a change in the conditions in one end will lead to a change in the other end as well. Because of these interactions a distillation column can be difficult or almost impossible to control. The interactions in a system can be analyzed by various tools (see e.g. Wolff [12]), amongst them the RGA, or Relative Gain Array. Systems with no interactions will have an RGA-value of 1. The larger the deviation from 1, the larger the interaction and the more difficult the process is to control. Pairing control loops on steady-state RGA-values less than 0 should be avoided.

The magnitude of the 1,1-element of the RGA for both the LV- and DV-configuration is given as a function of frequency in Fig. 11 for operating procedure III-8 ($T_B = 222^0C$). From the figure it can be seen that for the LV-configuration the RGA is very high at low frequencies (when the system is approaching a steady state). This shows that the interaction reduce the effect of the control input (L, V) and make control more difficult.
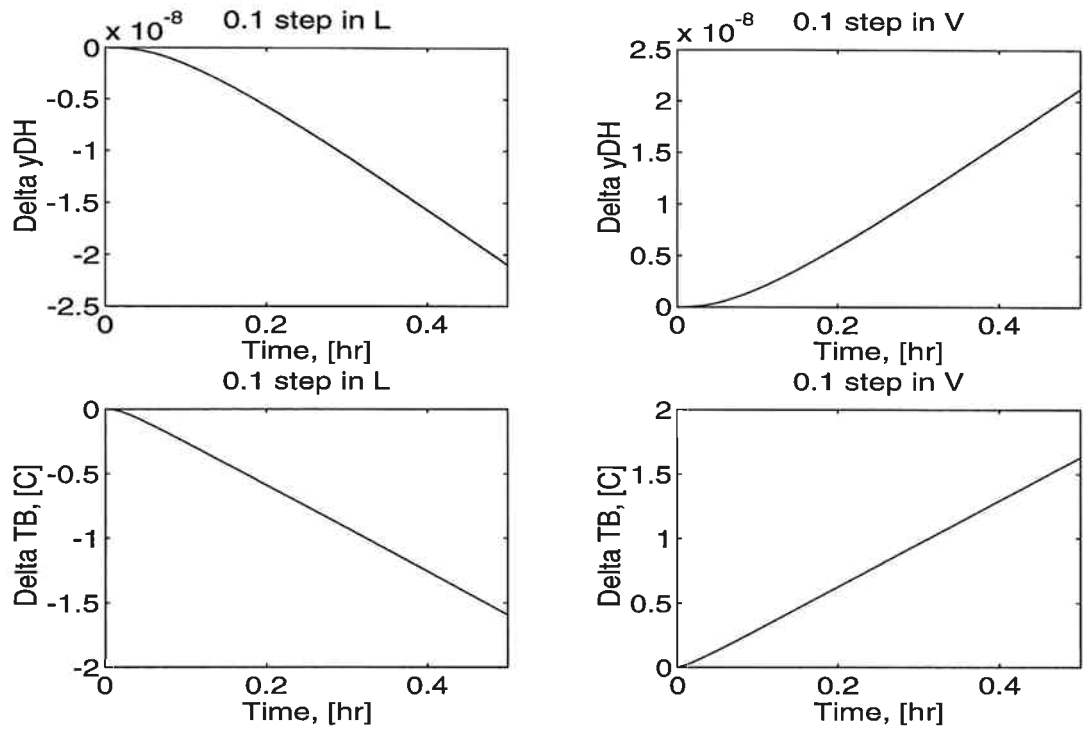
Figure 9: Linear open-loop step responses for LV-configuration for operating point III-8.
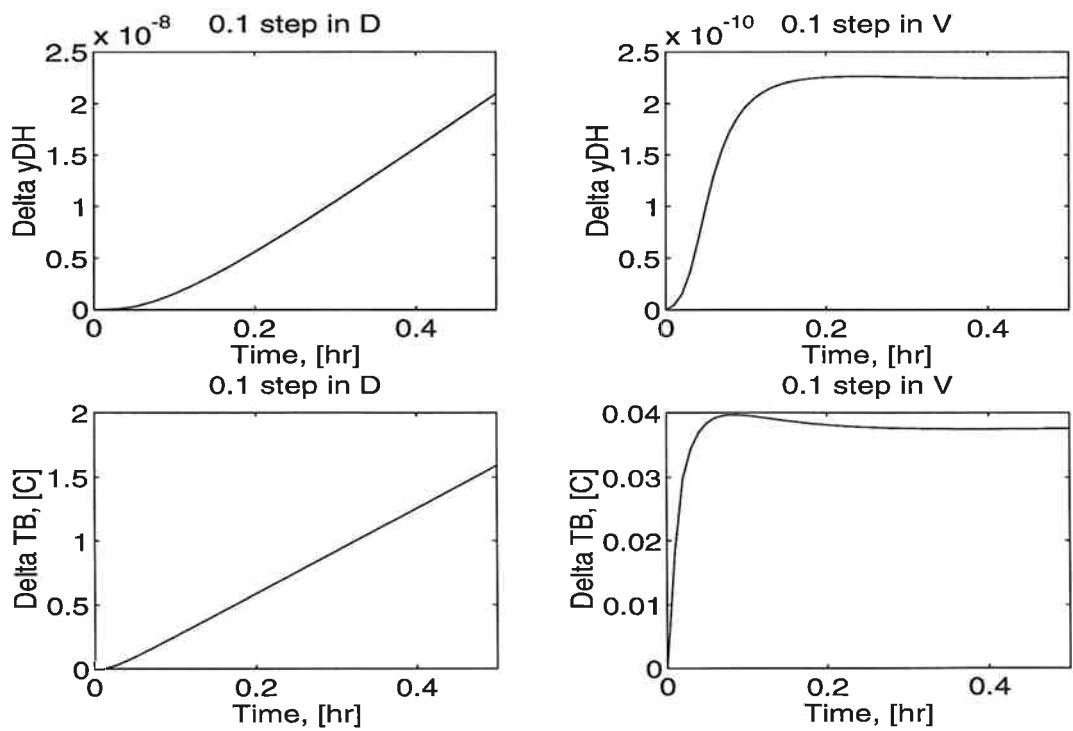

Figure 10: Linear open-loop step responses for DV-configuration for operating point III-8. (Note that the y-axis scaling is 100 times smaller for changes in V).
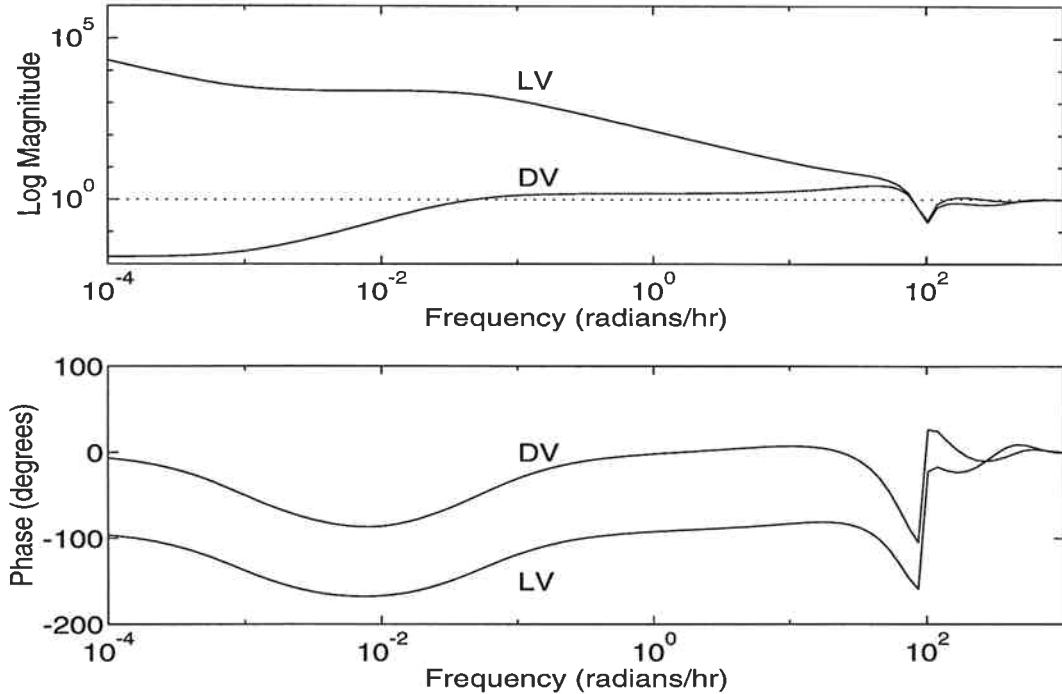
Figure 11: RGA for LV- and DV-configuration for linear model in operating point III-8.

RGA for *DV* is generally lower at all frequencies. This difference between configurations is the same as one would observe in a continuous distillation column.

However, the control characteristics from the RGA-plot for the *LV*-configuration are not quite as bad as it may seem. For control the steady-state values are generally of little interest (particularly in a batch process since the process will never reach such a state), and the region of interest is around the system's closed-loop bandwidth (response to changes), which is in the frequency range around 10 *rad/hr* (response time about 6 min). We note that the RGA is closer to 1 here and that the difference between the two configurations is much less. From the high-frequency RGA, which is close to 1, we find that for decentralized control, the loop pairing should always be to use the vapor flow V to control the reactor temperature T_B and either the reflux flow L or the distillate flow D to control the distillate composition or the loss of reactant R_2, Y_D. This is in agreement with physical intuition.

$$T_B \longleftrightarrow V$$
$$Y_D \longleftrightarrow L, D$$
$$M_D \longleftrightarrow L, D$$

5.2.2 Non-linear simulation of two-point model

Closed-loop simulations confirm that two-point control may be used if fast feedback control is possible. However, as in the case for one-point bottom control, we still have the problem of specifying a reasonable set-point for the bottom temperature to avoid breakthrough of reactant R_2 in the distillate. An example of two-point control of the process

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using the LV-configuration is given in Figure 12 with the following set point for the controllers: $T_{B,set} = 225\ ^0C$ and $y_{DH,set} = 0.0038$. (Note that we control the transformed distillate composition Y_D instead of y_D in order to reduce the non-linearity in the model.) It can be seen that only a minor break-through of reactant occurs during the run. The reactor temperature T_B is kept at its set point while the distillate composition y_{DH} is slightly lower than its set point showing that it is difficult to achieve tight control of both ends of the column at the same time. It should also be noticed how the vapor flow decreases with time which shows that energy can be saved using two-point control. Control using DV-configuration give similar results (not shown).

5.3   One-point column control

In the existing operating practice the temperature at the top of the column was controlled. The set point was 103 ^0C which gave a composition of 0.4 % of reactant R_2 in the distillate. By lowering the set point to e.g 100.1 ^0C the distillate would be purer, but the column would become very sensitive to measurement noise, and this system would not work in practice.

One alternative is to measure the composition y_D and use this for feedback. However, implementing an analyzer (or possibly an estimator based on the temperature profile) is costly and often unreliable. A simpler alternative is to place the temperature measurement further down in the column, e.g. a few of trays below the top tray, since this measurement will be less sensitive to noise. In this investigation the temperature on tray 5 is chosen as the new measurement to be used instead of the one on the top tray. The vapor flow is kept fixed at its maximum value ($V = V_{max} = 16.8$ kmol/hr). With this control configuration ($T_5 \leftrightarrow L$) there is no direct control of the reactor temperature. However, with an appropriate choice of set point, $T_{5,set}$, loss of reactant R_2 could easily be avoided and one of the main causes of the operability problems thereby eliminated.

The temperature profile for one-point column control with set point $T_{5,set} = 130\ ^0C$ is shown in Fig. 13. The conditions are "stable" (i.e. no break-through of reactant R_2) throughout the batch. The reactor temperature increases towards the end and the mole fraction of heavy component in the distillate y_{DH} is less than or equal to 0.0001 at all times. Also note that this control procedure with V fixed at its maximum will yield the highest possible reactor temperature. This may be important in some cases when the reaction is slow.

6   Reducing amount of reactant

The proposed operating procedure with one-point column control gives a lower reactor temperature than the existing one-point top control procedure with $T_T = 103\ ^0C$. In the existing procedure the amount of reactant R_2 in the feed is about 20 % higher than needed for the reaction and all the above simulations were based on this. This is done to account for the loss of the reactant during the run. By using one-point column control with $T_5 = 130\ ^0C$, loss of reactant can be avoided and the surplus of R_2 is therefore not needed. By removing the excess 20 % of the reactant from the feed (such that the initial charge of R_2 is 6.25 kmol) the obtainable reactor temperature increases by about 2^0C at the beginning of the batch to about 40^0C towards the end as illustrated in Fig.
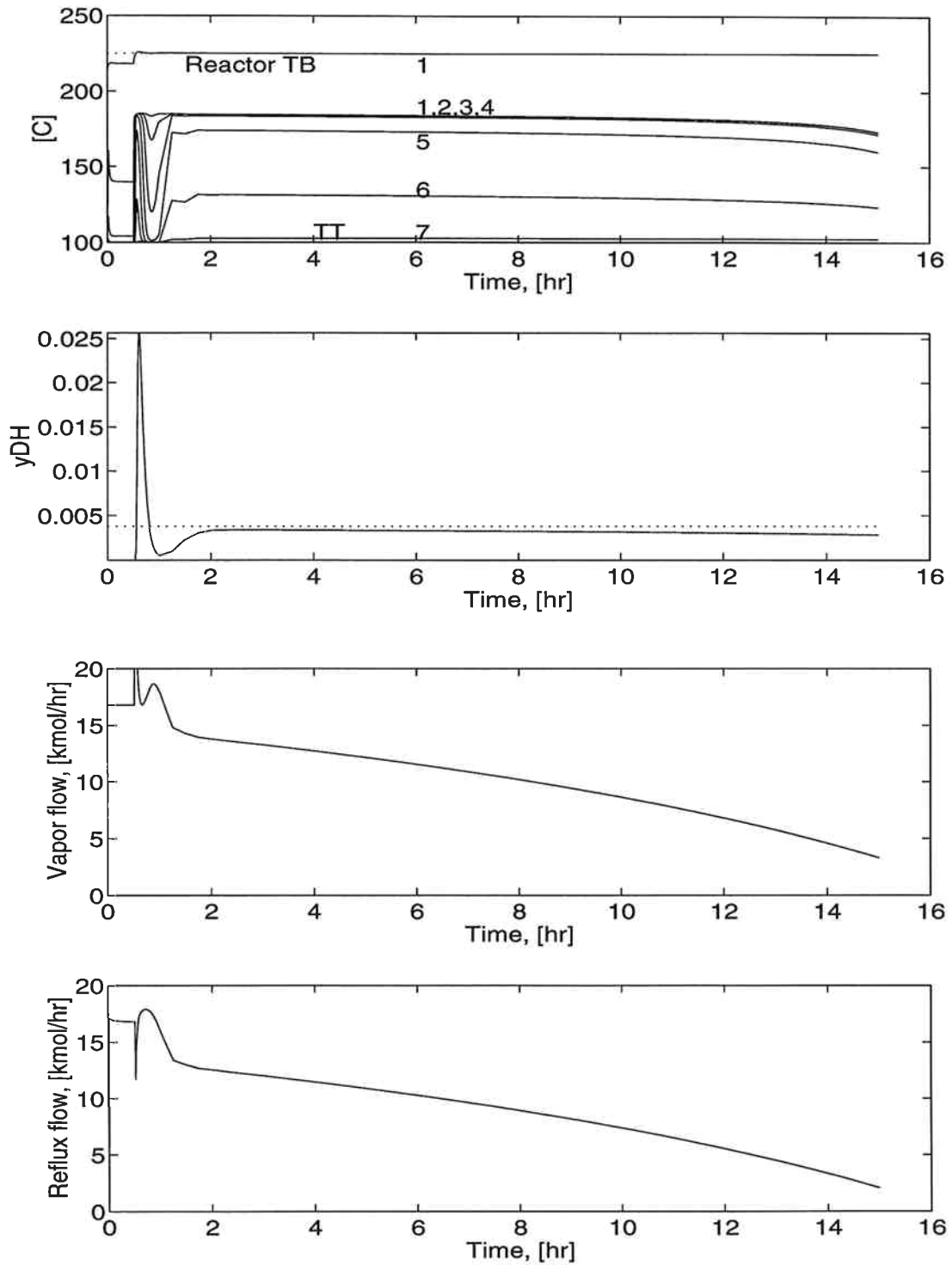
Figure 12: Two-point control. Temperature profile, distillate composition, vapor flow and reflux flow for LV-configuration with set points $T_{B,set} = 225\ ^0C$ and $y_{DH,set} = 0.0038$.
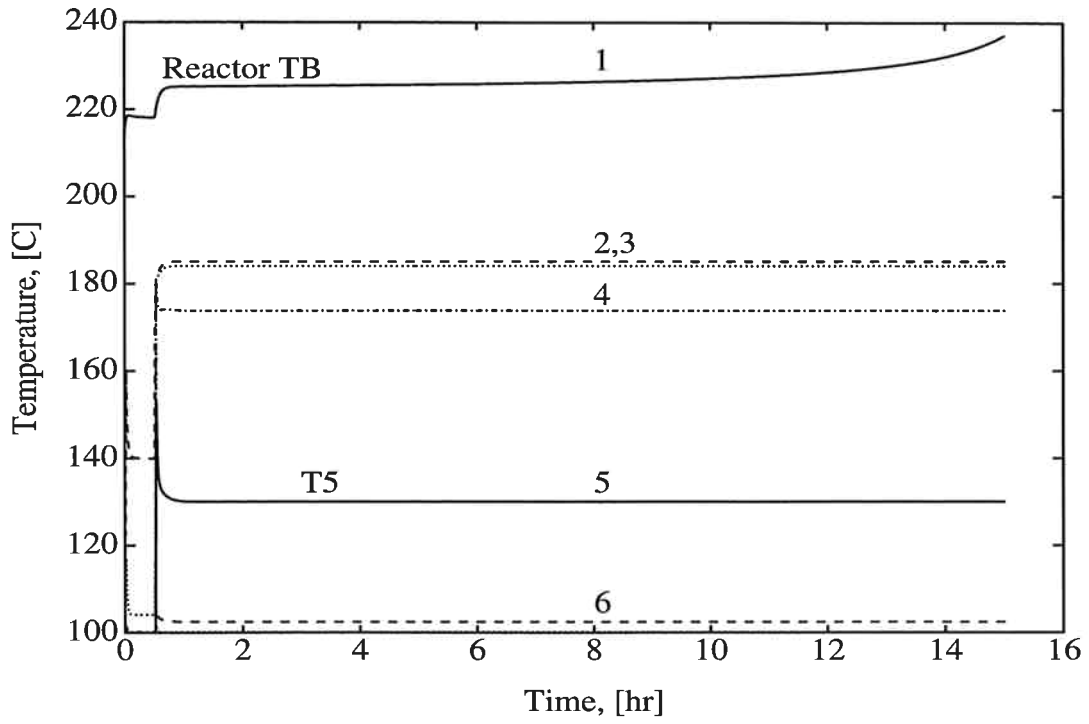
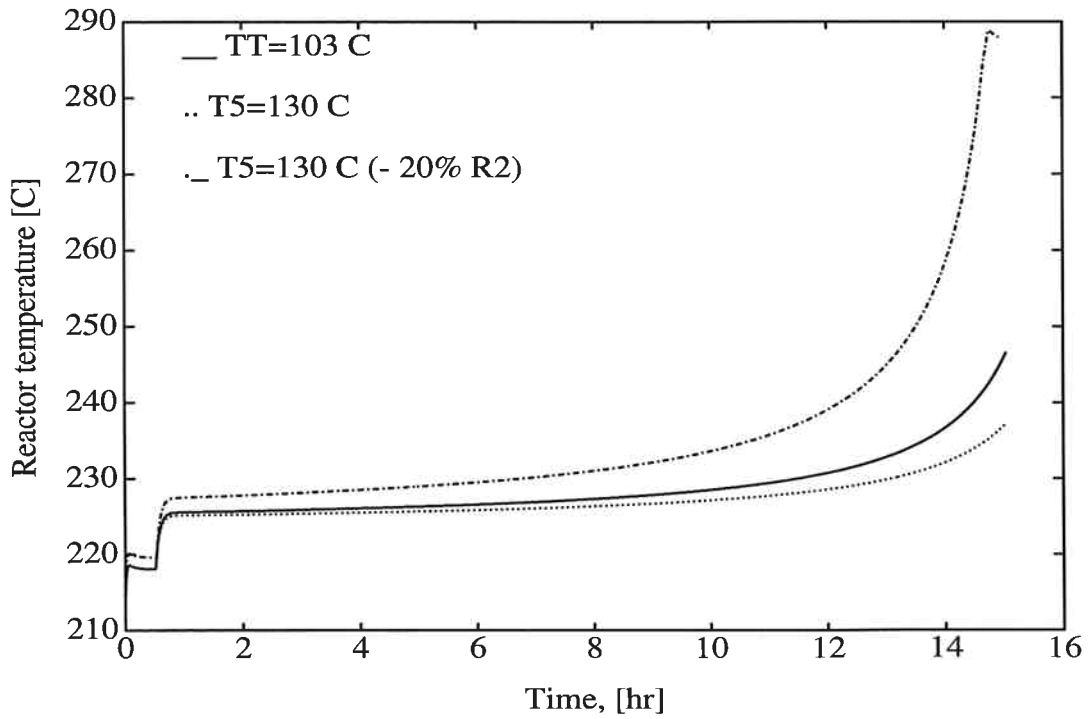Figure 13: One-point column control. Temperature profile with $T_{B,set} = 130\ ^{\circ}C$.

Figure 14: Effect of reducing the amount of reactant R_2 in the feed.

14. The reason for this is the high vapor pressure of the component R_2 which lowers the temperature as given by Eq. 11. Since the temperature is considerably higher towards the end of the batch when the excess R_2 is removed, the total batch time can therefore be reduced for a temperature dependent reaction.

In conclusion, by moving the location of the temperature lower down in the column, we

1. Increase the reactor temperature and thus reduce the batch time

2. Avoid loss of reactant R_2

3. Maintain more constant reactor conditions.

7   Comparison with conventional distillation columns

A comparison of our column with a conventional batch distillation column, shows significant differences in terms of control. For example, the common "open-loop" policy of keeping a fixed product rate (D) or reflux ratio (L/D) does not work for our column because of the chemical reaction (see also [6]). If the distillate flow D is larger than the amount of light component W formed by the reaction, the difference must be provided for by loss of the intermediate boiling reactant R_2. For optimal performance we want to remove exactly the amount of bi-product W formed. Therefore *feedback from the top is needed*. In fact, our column is very similar to a conventional <u>continuous</u> distillation column, but with the feed replaced by a reaction and with no stripping section.

By comparing our reaction batch column with a conventional continuous column we find that most conclusions from conventional columns carry over. As for a continuous column RGA(1,1) $\approx$ 0 at steady state (low frequency) for the DV-configuration for a pure top product column (see Fig. 11) implying that the reflux flow should be used to control the reactor temperature [10]. However, for control the pairing must be selected based on the RGA(1,1)-values around the bandwidth (10 rad/hr) implying that the vapor flow should always be used to control the reactor temperature for two-point control as was done in the simulations.

8   Conclusion

In this paper a dynamic model of a combined batch reactor/distillation process has been developed. Based on a linearized version of the model the controllability of the process depending on different reactor conditions and different times during a batch has been analyzed. The responses of the industrial example has been found to change considerably with operating point.

Controlling the reactor temperature directly using *one-point bottom control*, will give a more consistent product quality. However, since the response changes with time (gain between T_B and V), a non-linear controller might be needed to avoid instability. Moreover, because of the moving light/heavy component front in the column it is difficult to find the right set point temperature that does not give a break-through of heavy component

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in the distillate. This set point temperature will therefore in practice have to be specified low enough to ensure an acceptable performance.

Two-point control allows both the reactor temperature and the distillate composition to be controlled. By using two-point control energy will be saved compared with one-point control as the vapor flow can be reduced. However, one encounters the same problems of specifying the set point for the reactor temperature as for one-point bottom control.

The existing operating practice, controlling the temperature at the top of the column, is poor, sensitive to noise and leads to a varying loss of reactant R_2 and thereby varying product quality. The measuring point should therefore be moved from the top tray and further down in the column. The proposed new procedure of *one-point column control*, where the temperature on tray 5 is controlled, has several advantages:

- No loss of reactant R_2 (compared to controlling the top temperature)

- Need not worry about maximum attainable reactor temperature (compared to controlling the reactor temperature directly by one-point bottom control)

- No interactions with other control loops (compared to two point control)

With this new operating policy addition of excess reactant R_2 to the initial batch can be avoided. Thus, the batch temperature can be increased and the batch time thereby reduced.

NOTATION

A	system matrix	T_i	temperature, K
B	system matrix	T_b	boiling point, C
C	system matrix	T_B	reactor temperature, K
D	distillate flow, $kmol/hr$	T_T	temperature at top of column, K
$G(s)$	transfer function	$\mathbf{u}$	control vector
L	reflux flow, $kmol/hr$	V	vapor flow, $kmol/hr$
L_i	internal liquid flow, $kmol/hr$	$\mathbf{x}$	state vector
L_{0i}	initial liquid flow, $kmol/hr$	$x_{i,j}$	molfraction of light comp. (W) in liquid
M_i	liquid holdup, $kmol$	y_D	molfraction of light comp. (W) in distillate
M_B	liquid holdup in reactor, $kmol$	y_{DH}	molfraction of heavy comp.(R_2) in distillate
M_D	liquid holdup in cond., $kmol$		$= 1 - y_D$
M_{0i}	initial liquid holdup, $kmol$	Y_D	logarithmic molfraction of light comp. (W)
P_i	pressure on tray i, Pa		in distillate $= -\ln(1 - y_D)$
P_j^s	vapor pressure, Pa	$y_{i,j}$	molfraction of light comp. (W) in vapor
r	reaction rate, $kmol/hr$	$\mathbf{y}$	measurement vector

Greek	*letters*		*scripts*	
α_i	relative volatility		i	tray number
Δ	deviation from operating point		j	component number
τ	hydraulic time constant, h^{-1}		*set*	set point
ξ_j	stoichiometric coefficient		*	nominal value

References

[1] Albet, J., J.M. Le Lann, X. Joulia and B. Koehret: "Rigorous Simulation of Multicomponent Multisequence Batch Reactive Distillation", Proc. COPE'91, Barcelona, Spain, 75-80 (1991).

[2] Cuille, P.E. and G.V. Reklaitis: "Dynamic Simulation of Multicomponent Batch Rectification with Chemical Reactions", Comp. Chem. Engng., 10(4), 389-398 (1986).

[3] Egly, H., V. Ruby and B. Seid: "Optimum design and operation of batch rectification accompanied by chemical reaction", Comp. Chem. Engng., 3, 169-174 (1979).

[4] Egly, H., V. Ruby and B. Seid: "Optimization and Control of Batch Rectification Accompanied by Chemical Reaction", Ger. Chem. Eng., 6, 220-227 (1983).

[5] Hindmarsh, A.C.: "LSODE and LSODI, two new initial value ordinary differential equation solvers", SIGNUM Newsletter, 15(4), 10-11 (1980).

[6] Leversund, E.S. S. Macchietto, G. Stuart and S. Skogestad: "Optimal control and on-line operation of reactive batch distillation", Comp. Chem. Eng., Vol. 18, Suppl., S391-395 (1994) (supplement from ESCAPE'3, Graz, July 1993).

[7] Reuter, E., G. Wozny and L. Jeromin: "Modeling of Multicomponent Batch Distillation Processes with Chemical Reaction and their Control Systems", Proc. CHEMDATA'88, Gothenburg, 322-329 (1988).

[8] Roat, S.D., J.J. Downs, E.F. Vogel and J.E. Doss: " The integration of rigorous dynamic modeling and control system synthesis for distillation columns: An industrial Approach", Presented at CPC'3 (1986).

[9] Skogestad, S. and M. Morari: "Understanding the dynamic behavior of distillation columns", Ind. & Eng. Chem. Research, 27(10), 1848-1862 (1988).

[10] Shinskey, F.G: "Distillation Control", 2. ed., McGraw-Hill Inc., 1984.

[11] Sørensen, E. and S. Skogestad: "Controllability analysis of a combined batch reactor/distillation process", AIChE 1991 Annual Meeting, Los Angeles, paper 140e (1991).

[12] Wolff, E.A., S. Skogestad, M. Hovd and K.W. Mathisen: "A Procedure for controllability analysis", presented at the IFAC Workshop on interactions between process design and control, Imperial College, London, Sept. 6-8, 1992.