# Robust Control of Homogeneous Azeotropic Distillation Columns

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Presented at AIChE Annual Meeting, Chicago, nov. 1990

#### Abstract

The entrainer feed used in homogeneous azeotropic distillation provides an extra degree of freedom in the steady state design compared to simple distillation. In this paper we discuss the control of azeotropic distillation columns in the region close to minimum entrainer feed. Both industrial experience and previous research indicate that this is a difficult task. However, by considering the high frequency behavior (initial response) of the column, we show that tight and robust control can be obtained with simple single-loop PI-controllers. The results depends on the presence of high-frequency phenomena such as flow dynamics.

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#### 1 Introduction

Binary mixtures forming minimum boiling azeotropes are commonly encountered in the chemical industry. The separation of such mixtures into the pure components is not possible by simple distillation and must be accomplished by other means. In homogeneous azeotropic distillation the separation is made feasible by adding a third component, called the entrainer. The entrainer alters the thermodynamic properties of the mixture, thereby enabling separation of the binary azeotrope into the pure components. This type of distillation is widespread in the process industry and the economic potential of improved operation is usually high.

Distillation is the unit operation that has received most attention in the process control community. However, almost all the work so far has been concentrated on ideal binary distillation. The main reason for this is probably that even simple distillation columns are hard to understand and control. In homogeneous azeotropic distillation several complexities are added; non-ideal thermodynamics, multicomponent mixtures and multiple feeds. Most papers in this area have concentrated on modelling the thermodynamics, selection of entrainers (eg. Doherty and Caldarola, 1985, Laroche et.al., 1990), steady state design (eg. Levy and Doherty, 1985a-b) and optimizing the separation (eg. Knight and Doherty 1989). Only a few papers have been devoted to the control problem; Abu-Eishah and Luyben (1985), Andersen et.al (1989), Anderson (1989), Bozenhardt (1988) and Gilles et.al (1980). Most of these papers have studied specific cases and few general conclusions are drawn. Andersen et.al. (1989) presented a more general analysis, but mainly based on steady state arguments. The most significant conclusion to draw from the papers is that homogeneous azeotropic distillation columns seems to be much more difficult to control than simple distillation columns. This seems to be supported also by industrial experience.

The entrainer used in homogeneous azeotropic distillation columns provides an extra degree of freedom compared to simple distillation. As several authors have shown (eg. Andersen et.al.,1989), this degree of freedom may be used to optimize the operation in terms of entrainer and utility (heating and cooling) consumption. However, steady state arguments indicate a much more difficult control problem in the steady state optimal operating point than in operating points with higher utility consumption (Andersen et.al., 1989). This has has led some authors to concentrate also on non-optimal operating points (eg. Knapp and Doherty, 1990). Industrial experience also shows that it is usual to operate far from the steady state economic optimum, and this may be due to a belief of easier operation with increased entrainer consumption.

In this paper we will concentrate on the high frequency (initial response) dynamics of the model. It is the initial response of the open-loop model which is most important for feedback control properties. The frequency region around the expected closed-loop bandwidth is most important. In our analysis we will make use of the frequency dependent Relative Gain Array (RGA) (Bristol, 1966, 1978) and the Closed-Loop Disturbance Gain (Hovd and Skogestad, 1990). The frequency dependent Relative Gain Array has proven to be an efficient tool for evaluating controllability of simple distillation columns, and the Closed-Loop Disturbance Gain seems to have promising properties when evaluating effect of disturbances under feedback control.

We start the paper by giving a short presentation of the separation sequence in homogeneous azeotropic distillation. Thereafter we present the model used in the analysis. This model includes important high-frequency characteristics usually excluded in control studies of distillation columns. An analysis of the model is presented next. In this analysis we study both different operating points as well as different control configurations. The selection of an appropriate control configuration has proven to be essential in control of simple distillation columns. The ultimate test of controllability is obviously design of controllers with an optimized performance. In this paper we make use of the structured singular value (Doyle, 1982) as a criteria. This way we may include the effect of uncertainty which is of outmost importance when designing controllers for ill- conditioned plants (Skogestad and Morari, 1988a). At the end of the paper we discuss how optimal operation may be obtained in practice.

We will throughout the paper make use of an example column which separates a mixture of Acetone, Heptane and Toluene (AHT). Acetone and Heptane forms a minimum boiling azeotrope and Toluene is used as entrainer. Toluene is the least volatile component in the mixture. Data for the column is given in Table 1. Control design results will also be presented for a column separating a system of Ethanol, Water and Ethylene glycol. Data for this column is given in Table 2.

## 2 The Separation Sequence

In homogeneous azeotropic distillation an entrainer is added to make the separation feasible. The system will therefor consist of three components, and two columns are needed to separate the mixture into its pure components. The sequence in which this separation takes place will be determined by the type of entrainer that is used. The entrainer most commonly used in the industry is a heavy boiler, that is the entrainer is the least volatile component of the mixture. We only consider this type of system in this work. In the case of heavy entrainer we recover in the first column, called the extractive column, the lightest component in the top and a mixture of the intermediate and heavy component in the bottom. The bottom product is fed to the second column, the entrainer recovery column, for separation of the binary mixture. The entrainer product from the recovery column is fed back to the extractive column. In the case of heavy entrainer the entrainer will be fed into to the upper part of the column. The separation sequence is illustrated in Fig.1.

As there are two feeds to the extractive column we will have three sections in the column compared to two in the ideal binary case (see Fig.1.). The main purpose of the extractive section is to separate the components of the binary azeotrope. In the stripping section the entrainer is removed from the top product while in the rectifying section light component is removed from the bottom product. For a more detailed discussion on the significance of the different sections we refer to the work by Andersen et.al. (1989).

As pointed out by Andersen et.al. one must be careful when making specifications in the bottom product of the extractive column as this will have a strong influence on achieveable separation in the recovery column. More specifically, all the light component in the bottom product of the extractive column will enter the top product of the recovery column as impurities. In order to make sure that the fraction of impurities does not exceed a desired value we must limit the fraction of light to intermediate component in the bottom product of the first column. As specifications for the extractive column we will therefor use

 $y_D$  - fraction of light component in the distillate

 $r_{12}$  - ratio of light to intermediate component in the bottom product.

The specifications for the AHT-column are  $y_D = 0.998$  and  $r_{12} = 0.005$ .

## 2.1 Steady State Optimal Operation

As stated above the entrainer feed may be used to optimize the column for a given separation. Andersen et.al. showed that by varying the entrainer feed flow and adjusting the other flows accordingly for a given separation, one obtains a continous set of possible operating points. They described the set as entrainer feed versus boilup. For the AHTcolumn studied in this paper we obtain the set of operating points shown in Fig.2. The set of operating points may be divided into three distinct regions. In region I (see Fig.2.) a decrease in entrainer feed leads to a decrease in boilup and thereby to a more optimal operation with decreased entrainer consumption. At a certain point the boilup reaches a minimum  $(V_{min})$  and a further decrease in entrainer feed results in increased boilup (region II). This will continue untill we reach the minimum entrainer needed for the separation  $(E_{min})$ . The optimal operating point will be between  $E_{min}$  and  $V_{min}$ , ie. in region II. The exact location of the optimal operating point will be determined by the cost of entrainer and heating/cooling. The third region (region III) is caused by an input multiplicity in the system and was discovered by Andersen et.al. In this region an increased entrainer feed gives a sharp increase in boilup. Andersen et.al. have discussed in detail the origin of the different regions.

The rest of the paper is devoted to analysing the controllability in the different regions. The operating points we will consider are named  $I_A$ ,  $I_B$ ,  $V_{min}$ ,  $II_A$ ,  $II_B$ ,  $III_A$  and  $III_B$  and are shown in Fig.2. We will assume operating point  $II_A$  to be optimal and will hence concentrate on this operating point. Operating point  $II_B$  is located almost at  $E_{min}$ .

We will not discuss the control of the recovery column as this basically may be considered as an ideal binary column. The coupling of the two columns is discussed briefly towards the end of the paper.

#### 3 Modelling

The full non-linear dynamic model used in this paper have three states per tray. The states are expressed as fraction of two components plus liquid holdup on each tray. For the AHT- column this implies a total of 102 states. The flow-dynamics are described by a linear relation between liquid flow and liquid holdup:

$$L_i = L_i^o + (M_i - M_i^o)/\tau_L \tag{1}$$

where superscript o denotes nominal steady state values.  $\tau_L$  is computed from a linearized Francis weir formula

 $\tau_L = \frac{2}{3} \frac{M_{oi}}{Li} \tag{2}$ 

where  $M_{oi}$  denotes liquid over weir and  $L_i$  is the liquid flow. We use a holdup on each tray equal to  $M_i/F = 0.5min$  and assume half of the liquid over weir. The flow-dynamics are usually not included in models for control studies of distillation columns, but has an important high-frequency effect. As shown by Skogestad et.al.(1990a) the flow dynamics introduces a lag in reflux from the top to the bottom of the column which leads to a decoupling at high frequencies.

The equilibrium is modelled by the Van Laar activity coefficient model. We assume

constant molar flows, that is we exclude the energy balance.

In the analysis and controller design we make use of linearized models. Due to the high order of the full models we use reduced models with 20 states. The models were reduced by means of a balanced minimal realization.

In simple distillation one has been successful using simplified models with only one or two time constants (eg. Skogestad and Morari, 1988b). We found it difficult to obtain good and general models in this way for the extractive column, and will hence not use such models here.

Skogestad and Morari (1988b) suggested using logarithmic measurements in controllers for simple distillation columns. They found that this gave a good scaling of the problem and that it also tended to linearize the dynamics. We will adopt this for the extractive column and will hence use the following measurements in the rest of the paper

$$log(1 - y_D) \tag{3}$$

$$log(r_{12}) \tag{4}$$

This leads to the following scaling of the outputs

$$\Delta y_D^s = \frac{\Delta y_D}{1 - y_D} \tag{5}$$

$$\Delta r_{12}^s = \frac{\Delta r_{12}}{r_{12}} \tag{6}$$

where superscript s denotes scaled variables.

## 4 Control Configurations

In control terms a distillation column may be viewed as a 5x5 system. That is, we have 5 manipulated variables  $(L, V, D, B, V_T)$  and 5 primary outputs  $(y_D, r_{12}, M_D, M_B, P)$ . The feed streams are here assumed to be given (disturbances). One could design a full multivariable controller where all manipulated variables are coupled to all the measurements. This would of course be optimal from a theoretical point of view. However, for more practical reasons it usual to design a decentralized controller with five single loops. Such a controller is easier to understand and retune and will also be more failure tolerant. We will assume such a decentralized controller stucture also in this work.

When using a decentralized control structure the first decision usually made is selection of level loops. The selection of which inputs to be used for level control is often considered as a part of the column design. However, the decision made here is of vital importance for the remaining control problem. Different control configurations will have different dynamic characteristics. This has been pointed out by several authors for the case of simple distillation (eg. Shinskey, 1984; Skogestad et.al., 1990a). In this paper we will consider four different configurations; the LV-, DV-, DB- and (L/D)(V/B)-configuration. The names indicate which inputs that are left for composition control, eg. the LV-configuration refers to using reflux L and boilup V to control compositions. The DB-configuration would be rejected from steady state arguments (D+B=F) but as shown by Skogestad et.al (1990b) it may be a good choice for simple distillation columns due to its high-frequency characteristics. This also shows how misleading steady state arguments may be when evaluating processes for controlability. Skogestad et.al. (1990a) found the (L/D)(V/B)-configuration to be the overall best selection for a series of simple distillation column they studied.

### 5 Open-Loop Dynamics

Region III. Andersen et.al. (1989) found that the operating points in the undesirable region III had severe right half plane zeros. They found that these right half plane zeros were due to a negative effect of internal flows (dL = dV) on separation in this region. The rhp-zeros also explains the input multiplicity found in the set of operating points (see Fig.2). For the AHT-column we find the worst rhp-zeros at 0.234 in operating point  $III_A$  and at 0.128 in operating point  $III_B$ . Due to these rhp-zeros which are serious bandwidth limitations and the fact that the operating points in region III are clearly non-optimal it is unlikely that the column would be operated in this region. We will therefor exclude these operating points from any further analysis.

Region I and II. In order to get an idea of how the dynamics of the extractive column varies with operating conditions we will consider the open-loop gains of the LVconfiguration in the different operating points. That is we consider the gains in the transfer-matrix

 ${\binom{dlog(1-y_D)}{dlog(r_{12})}} = G^{LV}(\frac{dL}{dV})$ (7)

Figure 3. shows the open-loop gains as a function of frequency for the LV-configuration in the different operating points. Note that the outputs are scaled according to Eq.5 and Eq.6. We see that for the bottom composition  $r_{12}$  there are only small variations in the gains for different operating points. The responses in the bottom are essentially first order, as in simple distillation, with a time constant of approximately 500 min. For responses in the top composition  $y_D$  we do however observe somewhat higher order dynamics. There is a relatively fast initial response with a time constant of approximately 4 min., and then a slower response with a time constant around 500 min. The initial effect is due to a change in operating lines and this effect seems to dominate in region I. This is also confirmed by the fact that in region I the steady state effect of changes in internal flows (dL = dV) are in the same order as the effect of changes in external flows (dD = -dB). Similar responses may be encountered in overdesigned simple distillation columns operating close

to minimum reflux, but is not due to overdesign in this case. It is well known from simple distillation that an effect of a change in internal flows have a much faster effect than a change in external flows (eg. Skogestad and Morari, 1988b). In region II the change in operating lines have somewhat less effect than in region I, but the slow secondary effect due to changes in compositions is dominating the overall effect. Thus both for top and bottom composition we find that the high-frequency dynamics is only slightly dependent on operating point, while the low-frequency dynamics in the top varies significantly with operating point. For control purposes the high-frequency dynamics (initial response) is most important, and from the open-loop dynamics we would therefor not expect big differences in the control properties between different operating points.

For the other configurations we observe similar effects, that is small difference in the high-frequency dynamics between the different operating points. The only exception is for the (L/D)(V/B)-configuration where the gains from (V/B) differs significantly also at higher frequencies. This is explained by the difference in (V/B) in the different operating points. The ratio (V/B) will decrease as we increase the entrainer feed. This non-linearity may be compensated for by measuring the flows which is necessary anyway for this configuration.

## 6 The Relative Gain Array

The Relative Gain Array (RGA) was originally proposed by Bristol (1966) as a steady state interaction measure, and has found widespread applications for selecting single loop pairings in decentralized control. One of the main advantages of the RGA is that it depends only on the plant model itself, and does therefor not require any preliminary controller design. This is due to an assumption of perfect control. Another advantage with the RGA is that it is scaling independent.

The RGA may easily be extended to a frequency dependent measure (eg. Bristol, 1978), and will in this case contain more useful information with respect to feedback control. We are primarily interested in the frequency region around the expected closed-loop bandwidth. The definition of the elements in the RGA is given by

$$\lambda_{ij} = \frac{(\partial y_i/\partial u_j)_{u_l \neq j}}{(\partial y_i/\partial u_j)_{u_l \neq i}} = g_{ij}(s)[G^{-1}(s)]_{ji} \tag{8}$$

As the elements in each row and column sums up to unity in the RGA (eg. Grosdidier et.al, 1985), we only have to consider the 1,1 element for the 2x2 case. The 1,1 element for the 2x2 case is given by

$$\lambda_{11} = \frac{1}{1 - \frac{g_{12}(s)g_{21}(s)}{g_{11}(s)g_{22}(s)}} \tag{9}$$

where  $g_{ij}$  denotes the elements of the transfer matrix.

Skogestad et.al. (1990a) successfully used the frequency dependent RGA for selecting control configurations in simple distillation, and Hovd and Skogestad (1990) have proven its usefullness on a more general basis. Besides giving information on interaction among single-loops the RGA also contains useful information on sensitivity to input uncertainty. Another measure that is frequently used to assess controllability is the condition number,

 $\gamma$ . However, the condition number is scaling dependent and the minimized condition number,  $\gamma^*$ , is used instead. Skogestad and Morari (1987a) give the following relationship between the RGA and the minimized condition number for 2x2 plants

$$\|\Lambda\|_1 - \frac{1}{\gamma^*} \le \gamma^* \le \|\Lambda\|_1$$
 (10)

where  $\|\Lambda\|_1$  denotes the 1-norm of the RGA. Thus the difference between  $\|\Lambda\|_1$  and  $\gamma^*$  is at most equal to  $1/\gamma^*$ . Since  $\|\Lambda\|_1$  is much easier to compute it is the preferred quantity to use.

Figure 4. shows the RGA as a function of frequency for the four configurations in the different operating points. From the figure we see that for all configurations the RGA differs widely between the operating points at steady state. For the LV-configuration we have for instance a steady state value of 70 in operating point  $II_B$  and a steady state value of less than 3 in  $I_B$ . From the steady state RGA one would therefor have concluded that the control problem seems to be much more difficult in the optimal region II than in the non-optimal region I. The same conclusion would be reached for the (L/D)(V/B)-configuration, while interesting enough one would reach the opposite conclusion for the DV- and DB- configuration. However, we stress that steady state arguments may be misleading in analysis for control. It is of more interest that the RGA at higher frequencies are similar in all operating points. This is not surprising as we have seen that the high frequency dynamics are similar in the different operating points. At high frequencies all RGA-values reach a value of one due to the decoupling introduced by the flow dynamics.

To compare the configurations we have plotted the RGA-values of the four configurations in the optimal operating point  $II_A$  in Fig.5. The plot shows that the (L/D)(V/B) has a RGA-value equal to one from a frequency of approximately 0.01  $min^{-1}$  and upwards, and seems to be a favorable configuration. The RGA-plot also indicates a relatively simple control problem in operating point  $II_A$  if we use the (L/D)(V/B)-configuration.

We conclude from the RGA-analysis that with a properly tuned controller (reasonably high bandwidth) and a 'good' configuration we would not expect any worse control problems in the optimal region II than in region I. The RGA-values are also comparable to what has been found in simple distillation, and does therefor not indicate a more difficult control problem for the extractive column than for simple distillation columns with high-purity products.

## 7 Disturbance Sensitivity

The Relative Gain Array is independent of disturbances. However, an important issue when analyzing processes for feedback control properties is sensitivity to disturbances. The main reason for applying feedback control in distillation is rejection of disturbances that enters the process. In the literature it has been usual to consider the open-loop disturbance gains at steady state when evaluating sensitivity to disturbances. However, one should also for disturbances put emphasis on the high-frequency behavior. In addition the direction of the disturbance effect should be considered in the multivariable case. Some disturbances may be easier to reject than others due to a good allignment with

the strong input directions of the plant. Skogestad and Morari (1987b) introduced the disturbance condition number which takes the directions into account, as does also the Relative Disturbance Gain (RDG) introduced by Stanley et.al. (1985). For a particular disturbance  $z_k$  the RDG,  $\beta_{ik}$  is defined for each loop i as the ratio of the change in  $u_i$  needed for perfect disturbance rejection in all outputs to the change in  $u_i$  needed for perfect disturbance rejection in the corresponding output  $y_i$  when all other inputs are kept constant.

 $\beta_{ik} = \frac{(\partial u_i/\partial z_k)_{y_i}}{(\partial u_i/\partial z_k)_{y_i,u_l \neq i}} \tag{11}$ 

Hovd and Skogestad (1990) suggested a measure, the Closed-Loop Disturbance Gain (CLDG),  $\delta_{ik}$ , based on the RDG but which also takes into account the disturbance gain,  $g_{dik}$ ,

$$\delta_{ik} = \beta_{ik} g_{dik} \tag{12}$$

A matrix of CLDG's may be computed from

$$\Delta = \{\delta_{ik}\} = G_{diag}G^{-1}G_d \tag{13}$$

where  $G_{diag}$  are the diagonal elements of G. Hovd and Skogestad (1990) found that this measure enters nicely into the relation bewteen control set-off and disturbances while the RGA enters in a similar way into the relation between set-off and setpoint changes.

$$e_i \approx -\lambda_{ji} \frac{1}{g_{ji}c_i} r_j + \delta_{ik} \frac{1}{g_{ii}c_i} z_k; \quad \omega < \omega_B$$
 (14)

This equation gives a good approximation within the bandwidth of the closed loop system. In this work we will use the closed-loop disturbance gain as a function of frequency to measure sensitivity to disturbances for the different configurations and operating points. We consider disturbances in the feed rate F, its composition  $z_F$ , the entrainer feed rate E, and its composition  $z_{E1}$  and  $z_{E2}$ . All disturbances are scaled with respect to maximum expected disturbance size. We expect up to 30 % change in the feed rate F and entrainer feed rate E, up to 10 mole % in the azeotropic feed composition  $z_F$ , and up to 0.10 mol % in the entrainer impurities  $z_{E1}$  and  $z_{E2}$ .

We find that for the AHT-column we study in this paper the worst-case disturbances are disturbances in the feeds F and E. Due to limited space we will only present results for these disturbances here. We do however find similar results for all the disturbances encountered. All disturbances are taken into account in the controller design that follows later.

Figure 6 shows the Closed-Loop Disturbance Gains for disturbances in F and E as a function of frequency for the LV-configuration. Figure 7 shows the same measures for the (L/D)(V/B)-configuration. As for the RGA we see that there is a significant difference between the different operating points at low frequencies, while the responses are more similar at higher frequencies. There is however still a difference in the interesting frequency region (approx. 0.01 to  $1 \ min^{-1}$ ). The bottom composition  $r_{12}$  is most sensitive both to disturbances in E and F. The operating point  $V_{min}$  seems to be the best operating point with regards to disturbance sensitivity both at low and high frequencies.

For comparison consider the <u>open-loop</u> disturbance gain of feed flow F on top composition  $y_D$  for the LV-configuration in Fig.8. We see that the open-loop disturbance sensitivity gets worse as we get close to region II. From an open-loop analysis one would therefor incorrectly conclude that it easier to operate as entrainer consumption is increased.

In order to compare the configurations consider Fig.9. which shows the CLDG for disturbances in F and E in operating point  $II_A$  using the four configurations. We observe a significant difference between the configurations, and the (L/D)(V/B)- and DV-configuration seems to be have the best disturbance rejection properties.

We conclude from the CLDG analysis that there is some difference between the operating points with regards to disturbance sensitivity, but the difference is smaller at higher frequencies than at steady state. The operating point  $V_{min}$  (between region I and II) seems to be the best with respect to disturbance rejection.

### 8 Controller Design

The analysis presented above gives an idea of what kind of control performance we may expect in the different operating points using different configurations. However, the ultimate test of achieveable performance is of course design of optimal controllers for a chosen objective. In this work we use the structured singular value, mu, (Doyle, 1982) as a design objective. That is, we design for robust performance. The controllers are optimized with respect to setpoint changes as well as disturbances. We limit the structure of the controller to be two single-loop PI- controllers. This is due both to simplicity in computations and to the fact that this is what usually will be preferred in the industry. Single loop PI-controllers have been successfully designed for simple distillation columns (Skogestad et.al, 1990a) and seems to be close to the optimum (Skogestad and Lundstrom, 1990).

The uncertainty weight we use on each input in this work is given by

$$w_I(s) = 0.2 \frac{5s+1}{0.5s+1} \tag{15}$$

This corresponds to one minute deadtime and 20 % uncertainty in each input and is the same uncertainty description as used by Skogestad et.al. (1990a) for simple distillation columns.

The performance weight was adjusted to give robust performance in at least one operating point and for the AHT- column we arrived at

$$w_P(s) = 0.45 \frac{15s + 1}{15s} \tag{16}$$

This corresponds to a maximum closed-loop time constant of about 30 min. for setpoint changes and a maximum amplification of 2.2 of high-frequency disturbances. This performance weight corresponds to somehat less tight control than what Skogestad et.al (1990a) obtained for simple distillation columns (closed loop time constant 20 min.). They did however only optimize with respect to set point changes.

The same scalings was used for the disturbances as the ones used in the analysis. The outputs are naturally scaled by the logarithm, ie. a magnitude of 1 corresponds to a change in  $y_D$  of 0.002 and a change in  $r_{12}$  of 0.005.

The results of the optimization in terms of mu-values for robust performance are given in Table 3.. A mu-value less than one implies that the performance criteria is fullfilled for any model uncertainty within the uncertainty-weight that was used. The results shows that we can only guarantee robust performance (for the weights used) with the (L/D)(V/B)- configuration in the operating points  $V_{min}$  and  $II_A$ . This may be explained by the good disturbance rejection capabilities and low RGA-values observed close to the closed-loop bandwdith for this configuration in the two operating points. The (L/D)(V/B)-configuration does however get close to a mu-value of one in the operating points  $I_A$  and  $I_B$  as well, while  $II_B$  seems to be the worst operating point for this configuration. The LV-configuration also seems to work best in  $II_A$  and  $V_{min}$ , but the lowest mu-value is at 1.16. For the DV-configuration there is a clear trend that the control gets worse as we use less entrainer. From the results it seems to be opposite for the DB-configuration, that is control gets easier as we approach region II. These results demonstrate once again how important it is to choose a 'good' control configuration. Different configurations will give different conclusions with respect to operability of the different operating points.

In Fig.10 we have plotted the nominal closed-loop gains  $|g_{ii}c_i|$  in both loops together with the RGA and all closed-loop disturbance gains for the (L/D)(V/B)-configuration in operating point  $II_A$ . The controller is the mu-optimal designed above and the control parameters are given in Table 4. From the figure we see that in both loops the closed-loop gain stays above both the RGA and the CLDG's up to the bandwidth. This is necessary to keep the performance specification. One interesting thing to see from Fig.10. is that control of the bottom composition is most difficult. This is also as expected from the Closed-Loop Disturbance Gain analysis which showed that the bottom composition was most sensitive to disturbances. The bandwidth in this loop is much higher than for the top composition loop which is far less sensitive to disturbances. The difference in bandwidth between the two loops is also advantageous from an interaction point of view. (The two loops have their main effect in different frequency regions, and so we avoid possible problems with high interactions due to non-perfect control (Balchen, 1988)) The distance between the closed-loop gain and the RGA and CLDG's give the offset  $e_i$  according to Eq.14.

For comparison we optimized controllers also for the Ethanol-Water-Ethylene glycol (EWE) column. Data for the column are given in Table 2. We used the same uncertainty weight as for the AHT-column, but the performance requirements were somewhat looser

$$w_P(s) = 0.40 \frac{20s + 1}{20s} \tag{17}$$

Results of the optimization for the EWE-column are given in Table 5. The results are very similar to those for the AHT-column, and robust performance can only be guaranteed in operating points  $V_{min}$ ,  $II_A$  and  $II_B$  with the (L/D)(V/B)-configuration.

## 9 Use of Entrainer Feed in Control

So far we have not considered the entrainer as a third degree of freedom in control. The main reason for this is that we did not want to complicate matters too much compared to

simple distillation. Linearly we do not need this extra degree of freedom. However, there are two reasons why one may want to use the entrainer actively also in control. First of all it may be needed to ensure optimal operation under varying operating conditions. Second we must avoid that the entrainer feed goes below the minimum needed for the desired separation. The entrainer may be used in a feedback or feedfoward manner to accomplish this.

In order to get an idea of in what cases we need to change the entrainer feed in order to stay in the optimal operating point we need to know how different disturbances affect the optimality curve in Fig.2. It is obvious that the entrainer rate simply should be scaled by a factor  $(E/F)^*$ , where \* denotes nominal value, for disturbances in F. This may be done in a feedforward manner. It is however not clear what happens to the optimality curve for disturbances in feed and entrainer composition. As the separation in the recovery column is easy we do not expect large disturbances in the entrainer composition and thereby not in the optimality curve. Figure 11 shows the optimality curve for different azeotropic feed compositions. We see that the curve moves almost only in a vertical direction, that is the value of  $E_{min}$  is almost constant while  $V_{min}$  varies. We may therefor conclude that in order to stay close to the optimum and thereby also avoid  $E_{min}$  under changing operating conditions we do only have to change the entrainer feed rate for disturbances in the azeotropic feedrate. This may be implemented in a feedforward loop, and this is what we suggest in this paper. We do however realize that it is impossible to avoid "drift" with feedforward control, and that one therefor maybe should consider using the entrainer feed in a feedback mannner. It is not obvious how this should be done without limiting the flexibility of the column. One possible approach is to determine a composition that varies only slightly except close to  $E_{min}$ .

### 10 Nonlinear Simulations

Figure 12 shows the response to a setpoint change in  $y_D$  from 0.998 to 0.9964 ( $\Delta y_D^s = -0.8$ ) in operating point  $II_A$  using the (L/D)(V/B)-configuration. The controller tunings are the ones obtained from the robust controller design and are given in Table 4. The simulations include one minute deadtime and 20 % uncertainty in the inputs. Figure 13 shows the response to a setpoint change in the bottom composition  $r_{12}$  from 0.005 to 0.001 ( $\Delta r_{12}^s = -0.8$ ) in operating point  $II_A$  with the same controller. We see that the bandwidth for the bottom loop is significantly higher than for the top loop. The simulations demonstrate that setpoint changes are easily handled by the control system, and that the interaction between the control loops is small.

Figure 14 shows the response to a 30 % increase in azeotropic feed rate F with feedforward action in entrainer feed rate. The same uncertainty was included in the controller as above, and we also assumed one minute deadtime and 20 % uncertainty in the entrainer feed rate change, ie. we increased the entrainer feed rate with 24 % after one minute. Without the feedforward action E would go below  $E_{min}$  and the system would go closed-loop unstable. The simulation demonstrate the fact that it is the bottom composition which is most difficult to control. We do however get an acceptable maximum set-off also in this composition.

# 11 Online Location of Optimal Operating Point

We discuss here briefly how the optimal operating point may be located on a column that is under operation. We assume that the starting point is in region I, ie. in the non-optimal region. We know that as we decrease the entrainer feed rate in region I we will simultanously decrease the boilup untill we reach minimum boilup  $V_{min}$ , provided both compositions are kept constant (see Fig.2). By doing a ramp decrease in E with both compositions under feedback control we will therefor observe a decrease in boilup untill we pass  $V_{min}$ . The ramp change should not be done too fast due to the lag that will be in the control. If the ramp change is done too fast we may go far beyond  $V_{min}$  before we actually observe an increase in boilup V. By doing a properly slow ramp change we may continue the ramp untill we observe an increase in boilup. This will give a fairly accurate location of  $V_{min}$ . From this point one may do small stepchanges in E untill one reaches the desired trade-off between entrainer consumption and boilup.

Fig.15 shows non-linear simulation results of a ramp decrease in E from operating point  $I_B$  using the (L/D)(V/B)-configuration. The entrainer feed was decreased with a rate of 0.001 kmol/min. The controller given in Table 4. was used to control compositions. We see that the boilup starts to increase at a value of E around 0.56. The steady state minimum boilup is at E equal to 0.57. Thus we are able to determine the point of minimum boilup quite accurately by the proposed method.

#### 12 Discussion

We have in this paper only studied two columns, both with heavy boiling entrainer, and one should therefor be careful about making general conclusions. However, the main results are explained by the fact that the initial response of the columns are similar in all operating points and also to what is observed in simple distillation. The effects of the non-ideal thermodynamics are slow and does therefor not have a strong effect on the high-frequency dynamics which are most important for control. We expect this to be true also for other columns of the same class.

It is clear for the columns studied that it is the bottom product composition that is most sensitive to disturbances and also limiting in control. However, the ratio  $r_{12}$  is also sensitive to inputs (eg. L and V), which implies that it may be kept more pure for small expenses in terms of boilup and reflux. For instance one could decrease the ratio  $r_{12}$  from 0.005 to 0.001 in operating point  $II_A$  of the AHT-column by only increasing the boilup with 2.5 %. This is not a result of column overdesign as one might expect, as a decrase in number of trays would be expensive in terms of increased boilup. If crucial, we would recommend that the specification for the bottom product is set low enough to never complicate the separation in the recovery column.

All results presented indicate that the control problem in the optimal region II is not more difficult than in region I. These results are however based on reasonably tight control. Tight control will again depend on the choice of a 'good' configuration and measurements without too long delay. The results in this paper indicate that the ratio configuration (L/D)(V/B) will be the best configuration choice. This is similar to what is

found in simple distillation (Skogestad et.al, 1990a). The measurement problem has not been treated in this paper and may be a more difficult problem in azeotropic distillation than in simple distillation. This has been pointed out by several authors (eg. Gilles et.al., 1980), and is due to the more difficult temperature profile encountered in these columns. The measurement problem should be studied separately.

The existence of a minimum entrainer feed for a given separation may cause problems during operation. If the azeotropic feed rate is increased with more than 10 % in operating point  $II_A$  of the AHT-column without adjusting the entrainer feed, the column will go closed-loop unstable as the specified separation would be infeasible. In this paper we propose using a feed forward loop from azeotropic to entrainer feed to avoid  $E_{min}$ . However, this solution is sensitive to uncertainties in flow measurements. A better solution may be to use the entrainer in a feedback scheme in addition to feedforward action. The problem will be to select a measurement that will not limit the flexibility of the column. The control problem would also be made more complicated as we get a 3x3 control system.

The problem with minimum entrainer feed may also be reduced by selecting a less optimal operating point. For the AHT-column the operating point  $V_{min}$  is not far from the economic optimum but may take significantly larger disturbances in F without the entrainer going below  $E_{min}$ . This operating point does also have good control properties as seen from the analysis and control design.

Another potential problem which we have not discussed in the paper is region III where we have severe right half plane zeros. If this region is entered dynamically during operation one would get into diffculties with closed-loop instability. The probability of entering this region is hard to predict analytically as it is a high order non-linear dynamic problem. One would have to apply some kind of Lyapunov function in order to prove the regions of attraction. Due to the high order and complexity of the problem this is a too difficult problem to be handled in this paper. However, we have not in any simulations been able to perterb the column into a region where we observe problems with inversed sign of gains. Thus, our experience indicate that region III should not pose any problems in practical operation.

The paper has not treated the control of the entrainer recovery column and neither the coupling between the two columns. However, the separation in the recovery column will usually be similar to simple distillation. The control of this column do therefor not provide a new control problem compared to simple ideal binary distillation columns. The coupling between the two columns should not be a difficult problem either. A tank of some size for the entrainer feed will be necessary due to variations in the entrainer feed rate, and this will dampen the effect of changes in entrainer flow and composition from the recovery column.

The use of a heavy entrainer which we have studied in this paper is the most widespread application in industry today. However, it is clear that other entrainers may be more favorable both from a steady state point of view (Laroche et.al, 1990) and also from a control point of view. One kind of entrainer that may be advantageous is an entrainer which is the intermediate boiler. In this case we have two possible sequences of separation: 1) Light component and entrainer in the top of the extractive column. 2) Heavy component and entrainer in the bottom of the extractive column. In both these cases the impurities from the extractive column will enter the entrainer product in the recovery

column. This entrainer product will be fed back to the bottom of the extractive column in case 1) and to the top in case 2). This implies that the impurities in both cases are fed back to a section where they are easy to separate. Due to this the entrainer impurities should be easy to handle in the extractive column, and relatively high impurities may be optimal. Disturbances in the entrainer impurities should from the same argument be easy to reject with feedback control, and the performance specification on the ratio (eg.  $R_{12}$  may be loosened thereby making control easier.

#### 13 Conclusions

In this paper we have studied the control properties both in the optimal operating point (low entrainer feed) and in less optimal operating points for two extractive columns with heavy entrainer. The analysis of the dynamic model does not indicate a more difficult control problem in the optimal operating point than in other operating points. The robust controller design supports the results of the preliminary analysis. We obtain controller performance in the optimal region of operation which is comparable to what is achieved for simple distillation columns. The results depend strongly on the choice of a "good" control configuration, and for the columns studied we find the ratio configuration (L/D)(V/B) to be a best choice. The results does also depend on relatively tight control as the low-frequency characteristics indicate a more difficult control problem if the bandwidth is significantly lower than what is used in this work. The results are mainly explained by the fact that the initial response in an extractive distillation column is similar to what is found in simple distillation. The effect of non-ideal thermodynamics is slow. Due to this we expect the results obtained to be valid for most extractive columns of the type studied in this work.

The excistence of a minimum entrainer feed rate for a desired separation may pose problems in the operation of extractive columns. In this work we have proposed a feed-forward scheme to avoid  $E_{min}$ . This may however not be satisfactory, and one should therefor consider using the entrainer in a feedback scheme.

#### NOMENCLATURE

B - Bottom flow [kmol/min].

 $c_i$  - controller transfer function, loop i.

G - process transfer matrix.

 $g_i$  - process transfer function, loop i.

 $G_d$  - disturbance gain matrix.

 $g_{dik}$  - disturbance gain from disturbance k to output i.

D - Distillate flow [kmol/min].

E - Entrainer feed flow [kmol/min].

 $e_i$  - control off-set in loop i.

F - Azeotropic feed flow [kmol/min].

L - Reflux flow [kmol/min]

 $M_i$  - Liquid holdup on tray i.

N - Number of theoretical trays

NF - Feedtray location for azeotropic feed.

NE - Feedtray location for entrainer feed.

 $r_{12}$  - ratio of light to intermediate component in bottom product.

 $r_j$  - set point change in output j.

V - boilup [kmol/min]

 $V_T$  - condensation rate [kmol/min]

 $w_P$  - Performance weight.

 $w_I$  - Uncertainty weight.

zk - Disturbance k.

 $y_D$  - fraction of light component in distillate.

 $z_F$  - Fraction of light component in azeotropic feed.

 $z_{E1}$  - Fraction of light component in entrainer feed.

 $z_{E2}$  - Fraction of intermediate component in entrainer feed.

#### Greek symbols

 $\beta_{ik}$  - Relative Disturbance Gain form disturbance k to output i.

 $\delta_{ik}$  - Closed Loop Disturbance Gain from disturbance k to output i.

 $\gamma^*$  - Minimized condition number.

 $\lambda_{ij}$  - ij'th element of the Relative Gain Array.

 $\mu$  (mu) - structured singular value

 $\tau_L$  - Hydraulic time constant for each tray [min].

 $\omega_B$  - Closed-loop bandwidth  $[min^{-1}]$ .

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Table 1. Data for Acetone-Heptane-Toluene Column.

Reboiler is tray no.1.

Feed and Entrainer are saturated liquids

Total condenser with saturated reflux

Liquid holdups are  $M_i/F = 0.5$  min., including reboiler and condenser

Constant molar flows

Equilibrium calculated by Van Laar activity coefficient model

Table 2. Data for Ethanol-Water-Ethylene glycol Column.

$$F$$
  $z_F$   $E$   $z_{E1}$   $z_{E2}$   $N$   $NF$   $NE$   $y_D$   $r_{12}$   $1.0$   $0.87$  \*)  $1.e-7$   $1.e-4$   $50$   $17$   $48$   $0.998$   $0.0001$ 

Reboiler is tray no.1.

Feed and Entrainer are saturated liquids

Total condenser with saturated reflux

Liquid holdups are  $M_i/F = 0.5$  min., including reboiler and condenser

Constant molar flows

Equilibrium calculated by Van Laar activity coefficient model

Pressure P=1 atm.

\*) Operating point:  $II_B$   $II_A$   $V_{min}$   $I_A$ E: 0.35 0.75 1.00 2.00

Table 3. Optimal mu-values obtained in each operating point for Acetone-Heptane-Toluene Column.

Table 4. PI-settings for AHT-column with (L/D)(V/B)-configuration in operating point  $II_A$ ;  $C(s) = k \frac{1+\tau_{IS}}{\tau_{IS}}$ . Parameters are for scaled compositions (Eq. 5 and 6)

 $k_y$   $k_{r12}$   $\tau_{Iy}$   $\tau_{Ir12}$  0.152 1.243 4.101 10.273

Table 5. Optimal mu-values obtained in each operating point for Ethanol-Water-Ethylene glycol Column.

	$I_A$	$V_{min}$	$II_A$	$II_{B}$
LV	1.18	1.13	1.14	1.50
DV	1.38	1.40	1.52	1.69
(L/D)(V/B)	1.11	0.99	0.99	0.99
DB	1.52	1.42	1.37	1.14

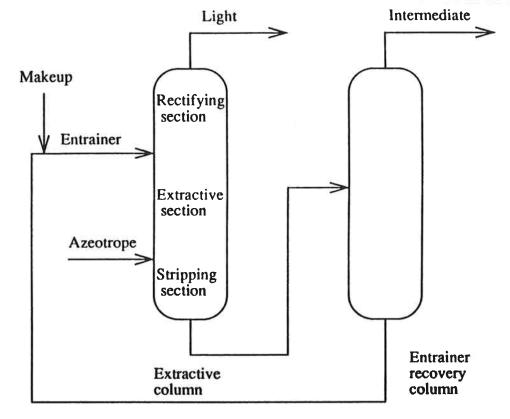


Figure 1. Separation sequence for the case of minimum boiling binary azeotrope and heavy entrainer.

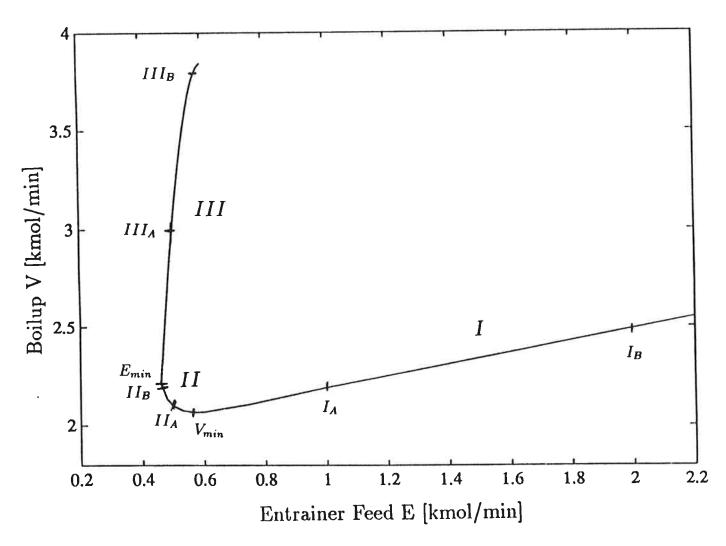


Figure 2. Set of operating points for the Acetone-Heptane-Toluene Column.

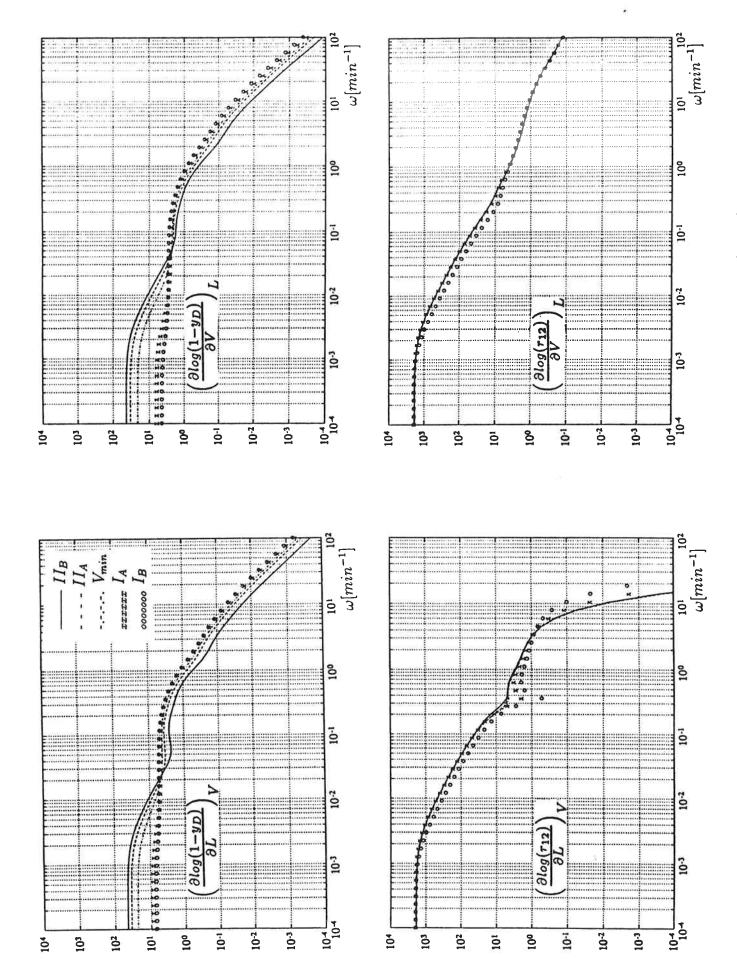


Figure 3. Open-loop gains for the LV-configuration in different operating points.

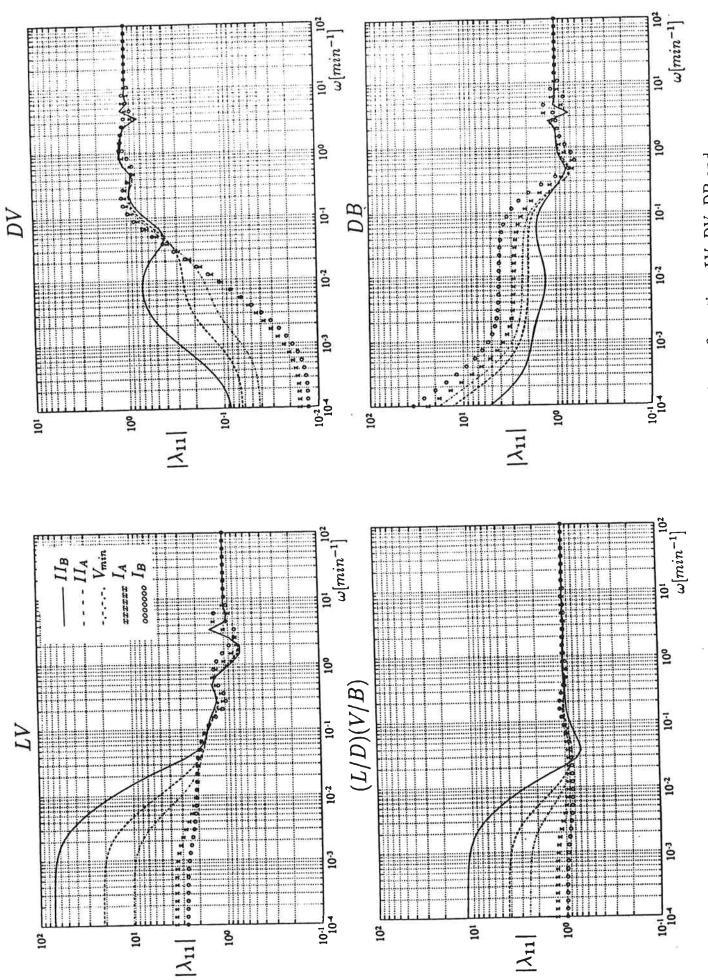


Figure 4. RGA as a function of frequency for the configurations LV, DV, DB and (L/D)(V/B) in different operating points.

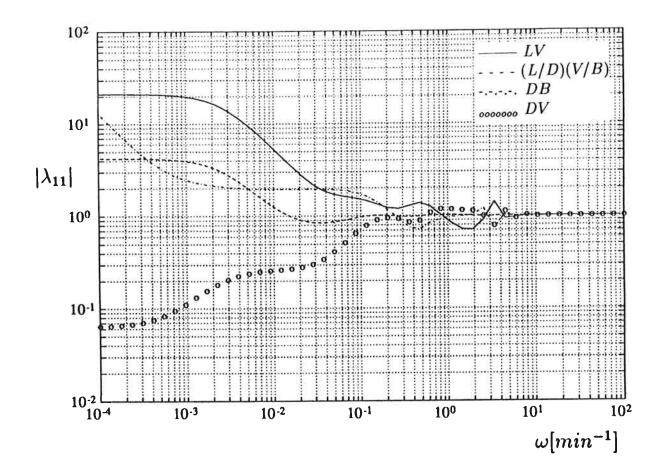


Figure 5. RGA as a function of frequency for the configurations LV, DV, (L/D)(V/B) in operating point  $II_A$ .

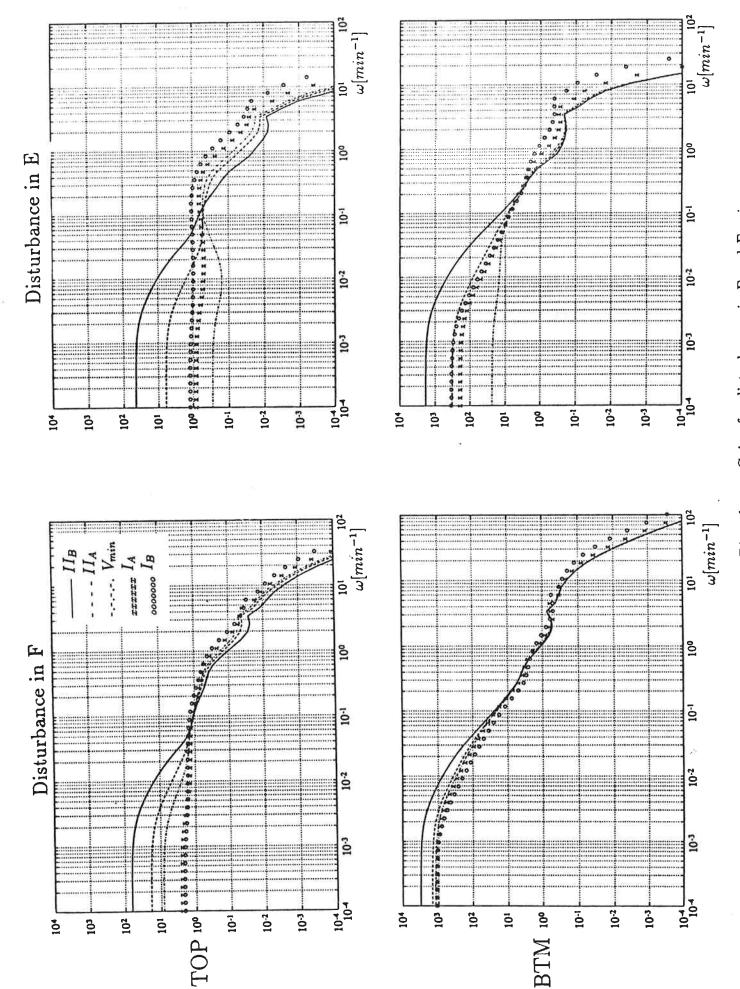


Figure 6. Closed-Loop Disturbance Gains for disturbances in F and E using configuration LV in different operating points.

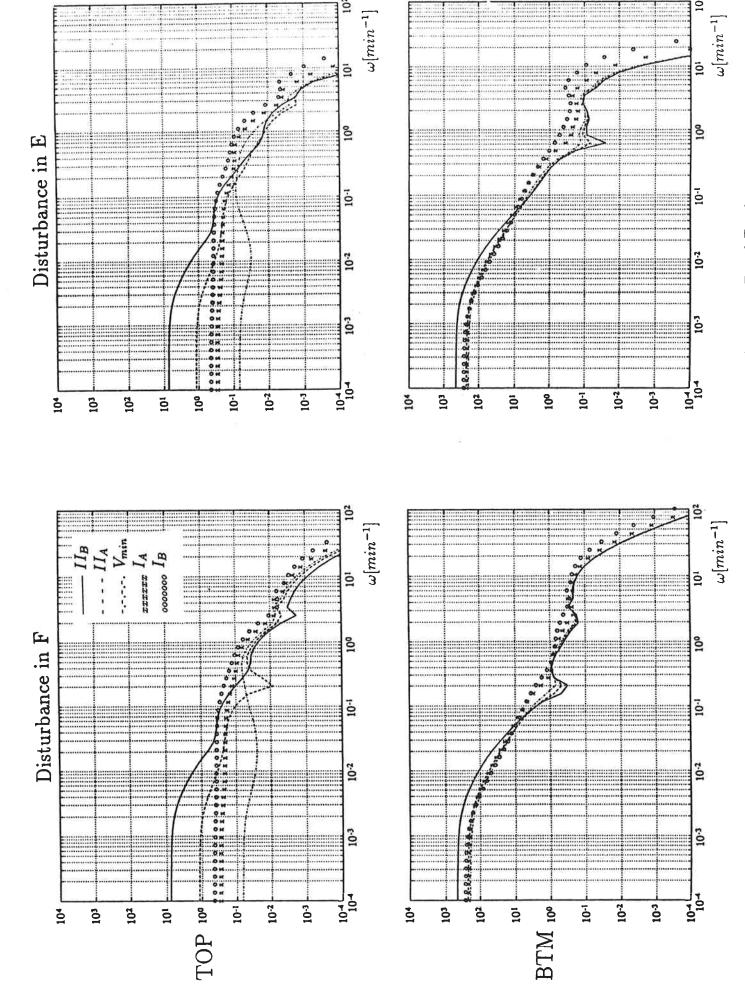


Figure 7. Closed-Loop Disturbance Gains for disturbances in F and E using configuration (L/D)(V/B) in different operating points.

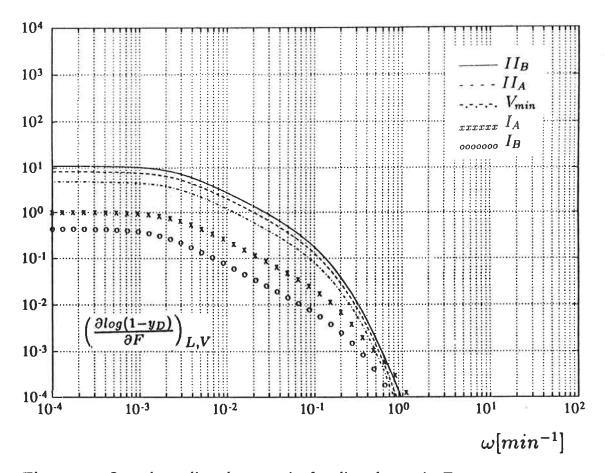
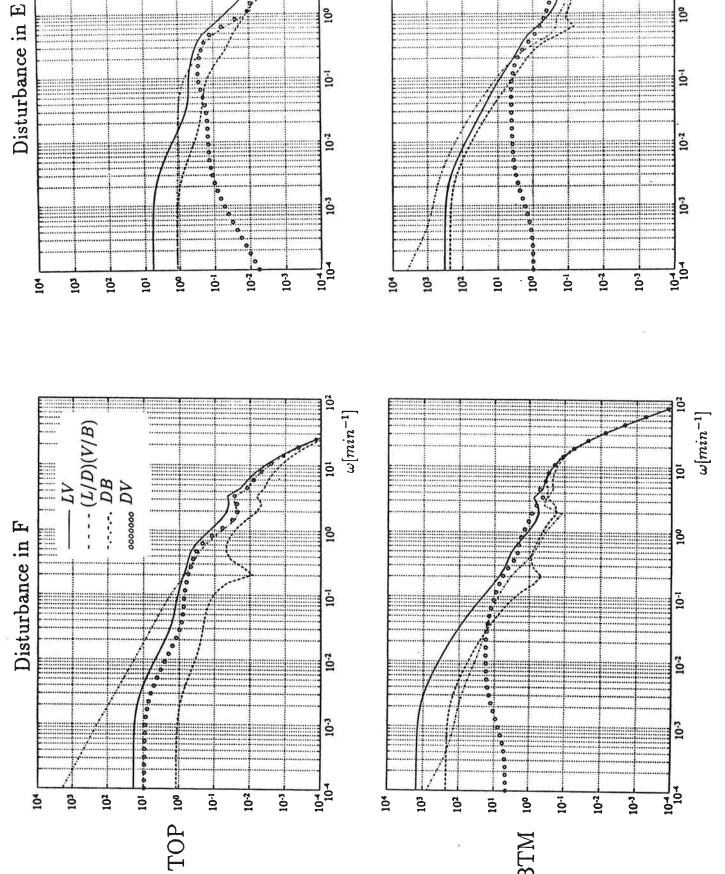


Figure 8. Open-loop disturbance gain for disturbance in F on top composition using the LV-configuration in different operating points.



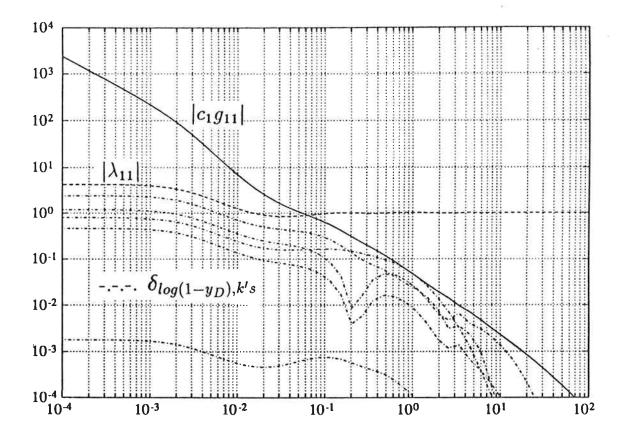
 $\omega[min^{-1}]$ 

001

Figure 9. Closed-Loop Disturbance Gains for disturbances in F and E using con-Aminations IV DV DR and (I INVIVIB) in anomating naint II.

 $\omega[min^{-1}]$ 

100



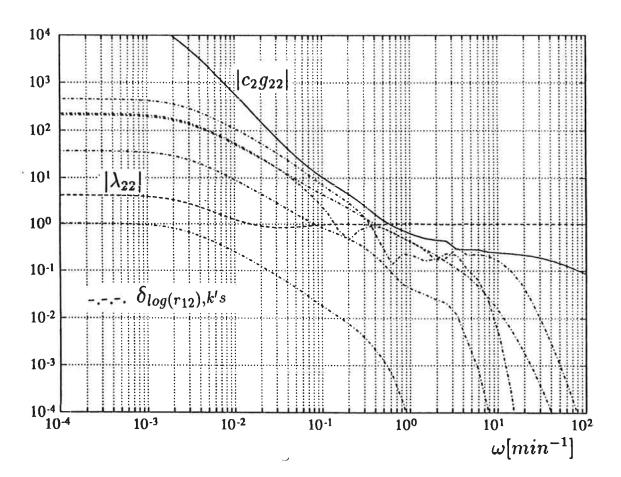


Figure 10. Closed-loop gains, RGA and CLDG's for all disturbances in the two single loops using the (L/D)(V/B)-configuration in operating point  $II_A$ . Controller parameters are given in Table 4.

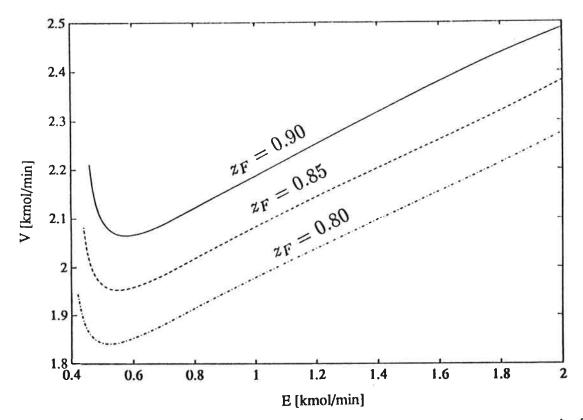


Figure 11. Set of solutions for the AHT-column for different azeotropic feed-compositions. Region III is not shown.

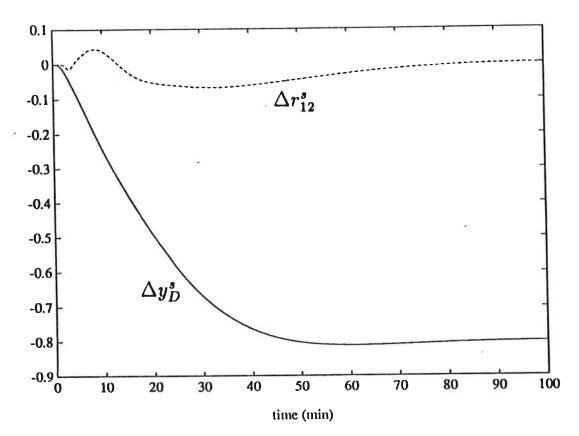


Figure 12. Nonlinear simulation of a set point change in top composition  $y_D$  in operating point  $II_A$  using the (L/D)(V/B)- configuration. Controller tunings from Table 4. The simulation includes input uncertainties as given in Eq.15.

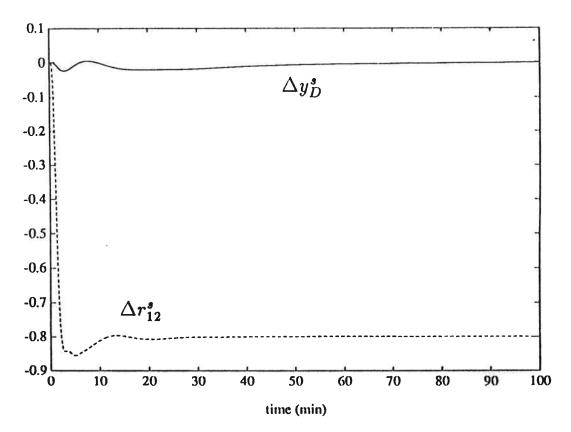


Figure 13. Nonlinear simulation of a set point change in bottom composition  $r_{12}$  in operating point  $II_A$  using the (L/D)(V/B)- configuration. Controller tunings from Table 4. The simulation includes input uncertainties as given in Eq.15.

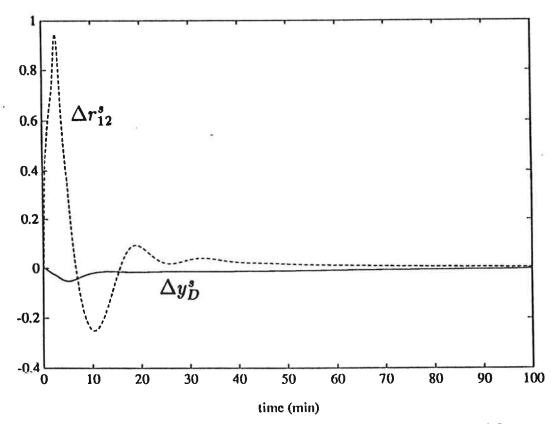


Figure 14. Nonlinear simulation of a 30 % increase in azeotropic feed flow rate F in operating point  $II_A$  using the (L/D)(V/B)- configuration. Controller tunings from Table 4. Feedforward action for entrainer feed is implemented by keeping (E/F) constant. The simulation includes input uncertainties as given in Eq.15.

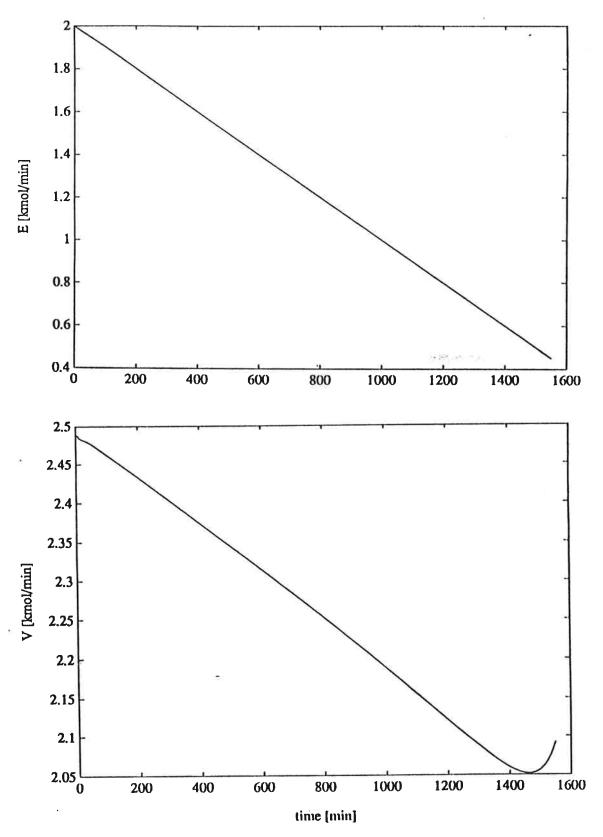


Figure 15. Response in boilup to a ramp decrease in entrainer feed from operating point  $I_B$ . Both compositions under feedback control with the (L/D)(V/B)-configuration. Controller tunings from Table 4. The simulation includes uncertainties as given in Eq.15. .