OPTIMAL STARTUP PROCEDURES FOR BATCH DISTILLATION

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Abstract - Operation of a conventional batch distillation column can be conveniently described in three parts: 1) startup period, 2) production period and 3) shutdown period. For standard separation processes, the production period is the most time consuming. However, for difficult separations, such as for high purity or azeotropic separations, the startup time may also be significant. In this paper, we present results for optimal operation of a number of different separations taking the startup time into consideration. It is generally found that the startup time is significant compared to the total operating time for difficult separations (high purities and recoveries) and when the combined column and condenser holdup is large. It is also found that the exact value of the startup time is of limited significance unless it is very different from the optimal value. Alternative ways of reducing the duration of the startup period are discussed.

INTRODUCTION

Operation of a conventional batch distillation column can be conveniently described in three parts: 1) startup period (normally under total reflux), 2) production period and 3) shutdown period. For standard separation processes, the production period is the most time consuming. However, for difficult separations, such as for high purity or azeotropic separations, the startup time may also be significant as shown in this paper. The importance of the startup period relative to the total separation has only been studied by few authors. Nad and Spiegel (1987) presented experimental results for the separation of 3 components into two main cuts, two off-cuts and a residue where the startup period was more than 1/4 of the total operating time. Holland and Liapis (1983) presented simulation results for the separation of a five component mixture where the startup time was more than 50% of the total operating time. Luyben (1971) presented simulation results for a number of cases where the startup time varied from negligible up to 50% of the total operating time.

The purpose of this paper is to discuss various aspects of startup procedures for batch distillation. We will discuss under which conditions the optimal startup time is significant compared to the optimal total operating time. Also, the influence of a correct startup time on the optimal operating time is considered.

To the best of our knowledge, no studies of optimal startup procedures have been presented in the literature so far. In the second part of the paper we discuss alternative ways of reducing the startup time by either using partial backmixing equipment or by adding light material to the condenser drum initially.

DYNAMIC MODEL

The dynamic model used in this paper for a binary mixture is valid under the following assumptions: 1) Staged batch distillation column, 2) perfect mixing and equilibrium on all trays, 3) negligible vapour holdup, 4) constant stage pressures and tray efficiencies, 5) constant vapour flows, 6) total condensation with no subcooling in the condenser, 7) constant relative volatility, 8) constant molar condenser drum holdup and 9) constant molar liquid holdup on all trays. It is assumed that the vapour flow $V$ can be manipulated directly. Note that the reflux ratio $R$ used here is the internal reflux ratio $R = L/V$.

SIGNIFICANCE OF STARTUP TIME FOR OPTIMAL OPERATION

In this section we will discuss under which conditions the optimal startup time is significant compared to the optimal total operating time. We will consider a binary mixture separated into two or three fractions (see Figure 1):

**Two fractions.** After the initial startup period under total reflux ($t_{startup}$), there is a production period (time $t_1$) where a light product with mole fraction $x_A$ and amount $H_A$ is accumulated.

**Three fractions.** After the first production period ($t_1$), there is a second production period (time $t_2$) where an intermediate off-cut is produced (with composition $x_{off}$ and amount $H_{off}$) and collected in a second accumulator tank.

In both cases, the heavy product is the residual material in the reboiler with mole fraction $z_R$ and amount $H_R$. Note that this residual also includes the holdup in the condenser and the column section which is

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assumed to be drained to the reboiler at the end of the batch. (If the condenser holdup was large, one would probably want to drain this to the accumulator instead).

In the following we will consider an operating policy with constant reflux ratios $R_1$ and $R_2$ in each of the two production periods. For a given separation the optimal startup time $t^{opt}$, the times to reach the specifications for the product cuts, $t_1$ and $t_2$, and the corresponding constant reflux ratios $R_1$ and $R_2$ can be found by minimising the total operating time $t_{tot}$.

In addition, there are constraints on the composition and recovery percentage of light product at $t_1$ and heavy product at $t_2$:

$$t_{tot} = t^{opt}_{startup} + t_1 + t_2$$

$$x_A(t_1) \geq x_A^{spec}$$

$$x_R(t_2) \leq x_R^{spec}$$

$$100\% \cdot \frac{z_A}{x_A^{H}}(t_1) \geq \% \text{ light recovery}$$

$$100\% \cdot \frac{(1-x_R)H_R}{(1-x_F)H_F}(t_2) \geq \% \text{ heavy recovery}$$

Also, there may be upper and lower bounds on times and reflux ratios. However, all the constraints are not necessarily active at the optimal solution (inequalities instead of equalities). The initial compositions are assumed to be equal to the feed composition. The column is run under total reflux during startup ($R_{startup} = 1.0$). The optimisation program DAEOPT (Vassiliadis, 1993) is used with an optimisation accuracy of $10^{-4}$. The purpose of this study is to investigate the relative importance of the startup time for a given separation. We do therefore not consider what is done with the off-cut after the separation, e.g. recycling or separate reprocessing.

Optimal results

Optimal results for several cases are given in Table 1. Cases C1 and C2 contain 0.5% of light component, cases C3 and C4 contain 5% of light component and cases C5, C6 and C7 are equimolar mixtures. The specifications are chosen so as to illustrate the influence of startup time for different types of separations (varying purity and recovery specifications). For all cases the total holdup in the column section $\sum^N H_i$ is 1% of the initial charge. The condenser drum holdup $H_C$ is assumed to be 0.25% of the initial charge for cases C1 and C2 and 1% for the others. A time $t_2$ equal to zero means the constraints are met without the production of an off-cut (C2, C3 and C6). Note that for some cases (C2, C3, C6 and C7), not all the constraints are active (for example C2 where $x_R = 0.0025 < 0.01 = x_R^{spec}$). This will in general be the case when an off-cut is not produced (C2, C3 and C6) since then only two constraints can be independently specified. The approach to equilibrium given in Table 1 is defined as:

$$\% \text{ approach to equilibrium} = \frac{[x_D(t_{tot}^{opt}) - x_F]/[x_D^{spec} - x_F]}{100\%}$$

Let us now consider the results in more detail. For case C1, the startup time is almost 70% of the total operating time. In this case only a very small amount of light component (impurity) is to be recovered from the feed charge leaving a heavy product of high purity. (It should be noted that the startup time is significantly reduced if there is no purity constraint on the light product). For case C2, the startup time is 99.5% of the total operating time. The optimal solution is in fact a cyclic operating policy with one cycle where a condenser drum holdup equal to the desired amount of light product is used and the column is run under total reflux until the desired product is obtained (see Sørensen and Skogestad, 1994).

For case C5, which is an easy equimolar separation with lower product purities, the startup time is only 1% of the total operating time. The results depend of course on the specifications for the product compositions and the percentage of recovery of the components. If a high percentage of recovery with a high purity is desired, the startup time will be longer than if the specifications are less strict. This is illustrated by case C3 and C4 which only differ in the specifications for the products.
Table 1: Optimal results (minimum operating time) for different separations for a piecewise constant reflux policy with one startup period and one or two production periods \((H_F = 10 \text{ kmol} \text{ and } V = 10.0 \text{ kmol/hr for all cases})\) (*: on the lower bound, -: not specified).

<table>
<thead>
<tr>
<th>Cases:</th>
<th>C1</th>
<th>C2</th>
<th>C3</th>
<th>C4</th>
<th>C5</th>
<th>C6</th>
<th>C7</th>
</tr>
</thead>
<tbody>
<tr>
<td>(N)</td>
<td>20</td>
<td>20</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>(a)</td>
<td>4.0</td>
<td>4.0</td>
<td>3.0</td>
<td>3.0</td>
<td>2.0</td>
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<td>2.0</td>
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<tr>
<td>(H_F, \text{ kmol})</td>
<td>0.005</td>
<td>0.005</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>(H_C, \text{ kmol})</td>
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<td>0.025</td>
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<td>0.1</td>
<td>0.1</td>
<td>0.1</td>
<td>0.1</td>
</tr>
<tr>
<td>(x_F)</td>
<td>0.005</td>
<td>0.005</td>
<td>0.05</td>
<td>0.05</td>
<td>0.5</td>
<td>0.5</td>
<td>0.5</td>
</tr>
</tbody>
</table>

Specifications:

<table>
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<th>Specifications:</th>
<th>(x_A^{\text{spec}})</th>
<th>(x_R^{\text{spec}})</th>
<th>(x_F^{\text{spec}})</th>
<th>% light recovery</th>
<th>% heavy recovery</th>
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<td></td>
<td>0.500</td>
<td>0.990</td>
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<td>0.950</td>
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<td></td>
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<tr>
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<td>0.0001</td>
<td>0.01</td>
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<tr>
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<td>99.00</td>
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<td></td>
<td>0.0043</td>
<td>-</td>
<td>-</td>
<td>0.277</td>
<td>0.500</td>
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<td></td>
<td>0.886</td>
<td>0.556</td>
<td>0.183</td>
<td>0.162</td>
<td>0.031</td>
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<td></td>
<td>0.009</td>
<td>0.003</td>
<td>2.285</td>
<td>0.0008</td>
<td>1.652</td>
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<td></td>
<td>0.01*</td>
<td>0.01*</td>
<td>0.983</td>
<td>0.342</td>
<td>0.761</td>
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<td>0.387</td>
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<td>0.0</td>
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<td>2.468</td>
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<td>0.9927</td>
<td>0.9841</td>
<td>0.9609</td>
<td>0.8246</td>
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<tr>
<td></td>
<td>0.9995</td>
<td>0.9995</td>
<td>0.9995</td>
<td>0.9998</td>
<td>0.9995</td>
</tr>
</tbody>
</table>

We see from Table 1 that the approach to equilibrium at the end of the startup time as defined by Eq. 3, varies considerably between the cases (99.9 % for case C1 to 65.0 % for case C5). Thus, the approach to equilibrium during startup will, as the startup time, depend on the mixture and the specifications.

In summary, the startup time will be significant compared to the total operating time for difficult separations (high purities and recoveries). It will also be significant when the combined column and condenser holdup is large. The last conclusion regarding holdup follows from the fact that the equilibrium time increases proportionally with holdup whereas the total operating time increases much less than proportionally.

**Optimal results with pre-specified startup time**

In the previous section we found that for difficult separations the column is run closer to steady state than for easier separations. The question is now how sensitive the results are to the exact value of the startup time. To this effect, we preselect the startup time equal to an equilibrium time \(t_{eq}\) defined as the time when the distillate composition has reached 98% of its steady state value:

\[
[x_D(t_{eq}) - x_F]/[x_D^{\text{eq}} - x_F] = \gamma
\]

where \(\gamma = 0.98\) (see Sørensen, 1994) and compute the corresponding optimal total operating time \(t_{tot}^{eq}\). The results are found by first running a simulation under total reflux to find the steady state distillate composition \(x_D^{eq}\) and thereby the startup time \(t_{eq}\). In the new optimisations, the startup time is pre-specified to this value and \(t_{eq}\) is found.\(t_{eq}\). These values are compared with the previous optimal values in Table 2, \(t_{opt}\) and \(t_{tot}^{opt}\). Interestingly, although the startup time is quite different \(t_{eq}/t_{tot}^{eq}\) is very small for all the cases studied (except for case C2 where a cyclic operating policy is better than the reflux policy studied here).

**Total operating time as a function of startup time**

For a given prespecified startup time one may compute the optimal total operating time. This relationship is shown graphically in Figure 2 for cases C3, C5 and C7. We see that for all cases, the total operating time increases moderately when the operating time is longer than minimum. However, the optimum is very flat for cases C5 and C7 but not for case C3. For case C3 there is a very large penalty in terms of longer operating time when the startup time is less than the optimal value. Two factors contribute to this: 1) The
Table 2: Optimal results ($t_{eq}^{98\%}$) when the startup time is pre-specified, $t_{startup} = t_{eq}^{98\%}$.

<table>
<thead>
<tr>
<th></th>
<th>C1</th>
<th>C2</th>
<th>C3</th>
<th>C4</th>
<th>C5</th>
<th>C6</th>
<th>C7</th>
</tr>
</thead>
<tbody>
<tr>
<td>$t_{eq}^{98%}$, hr</td>
<td>0.429</td>
<td>0.429</td>
<td>0.179</td>
<td>0.179</td>
<td>0.100</td>
<td>0.100</td>
<td>0.100</td>
</tr>
<tr>
<td>$t_{tot}$, hr</td>
<td>1.287</td>
<td>0.909</td>
<td>2.469</td>
<td>0.863</td>
<td>2.589</td>
<td>4.760</td>
<td>4.218</td>
</tr>
<tr>
<td>$t_{eq}^{98%}$/t_{start-up}</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$t_{eq}^{98%}$/t_{start-up}</td>
<td></td>
<td></td>
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<td></td>
</tr>
<tr>
<td>$t_{eq}^{98%}$/t_{start-up}</td>
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<td></td>
</tr>
<tr>
<td>$t_{eq}^{98%}$/t_{start-up}</td>
<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>$t_{eq}^{98%}$/t_{start-up}</td>
<td></td>
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<tr>
<td>$t_{eq}^{98%}$/t_{start-up}</td>
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<td></td>
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<tr>
<td>$t_{eq}^{98%}$/t_{start-up}</td>
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<tr>
<td>1.004</td>
<td>1.626</td>
<td>1.000</td>
<td>1.001</td>
<td>1.018</td>
<td>1.002</td>
<td>1.010</td>
<td></td>
</tr>
</tbody>
</table>

Figure 2: Total operating time $t_{tot}$ as a function of startup time $t_{startup}$ for cases C3, C5 and C7 (Note that the scaling is different).

Startup time and approach to equilibrium is more important for cases where the tightest specification is for the light product (case C3) and 2) the startup time and approach to equilibrium is less important when an off-cut is produced since there are more degrees of freedom (cases C5 and C7).

**IMPROVEMENTS IN THE STARTUP PROCEDURE**

Obviously a quick composition change has to take place during the startup period in order to reach steady state or a prescribed reflux composition as soon as possible. Changes in the startup procedure and/or the equipment characteristics may be necessary in order to reduce the duration of this period. The following suggested improvements will be considered in the last part of this paper: 1) Partial backmixing equipment and 2) light material in the condenser drum initially.

**Partial backmixing equipment**

González-Velasco et al. (1987) proposed to modify the overhead equipment of the batch distillation unit in order to more closely approach a plug flow behaviour within the condenser. They demonstrated that this behaviour, instead of the normal complete mixing, reduces the inertia to composition changes and permits a more rapid startup of the unit. One of the modifications, *partial backmixing equipment* is illustrated in Figure 3. When the lowest drum has initially filled up during startup, the valve between the drums is closed and total reflux operation begun. The reflux is provided from the lower drum but the condensed vapour stored in the upper drum. As soon as the lower drum is empty, the upper drum is completely full; at this time the valve is opened and liquid transferred from the upper to the lower drum during a period of time which is considered to be negligible. This cycle is repeated until steady state or a prescribed reflux concentration is reached. The authors reported time savings in the startup time of up to 25%.

In this section we will compare the equilibrium startup time, defined as the time for 98% approach to equilibrium (Eq. 4), for a number of cases using one condenser drum (conventional equipment) and two condenser drums (partial backmixing equipment). The bottom condenser drum is assumed initially filled ($H_{b1}^2 = H_C$) whereas the top drum is empty ($H_{b2}^2 = 0$). The results are given in Table 3 for different feed compositions and condenser holdups. The condenser drum holdup $H_C$ is 0.5, 5 and 10% of the initial charge respectively. Note that the total drum holdup for the case with two condenser drums is equal to the holdup in the single drum case ($H_{b1}^2 + H_{b2}^2 = H_C$). The difference in equilibrium time is small when $x_F = 0.1$, or when there is a low amount of light component in the feed. Using two drums will actually slightly increase the equilibrium time for low $x_F$ and large $H_C$. The largest time saving (12%) is for the case with the largest feed composition and the largest condenser drum. González-Velasco et al. (1987) reported time savings from 2 to 25% using two drums instead of one. However, they only studied cases where the condenser holdup was 5 or 10% of the initial charge and the feed composition $x_F = 0.5$ to 0.6. It can therefore be concluded that the *partial backmixing equipment* is advantageous only if the feed composition $x_F$ is large and the condenser holdup $H_C$ has to be large too for some reason.
Figure 3: a) Usual equipment with backmixing in the condenser; and b) partial backmixing equipment with two condenser drums.

Table 3: Steady state distillate composition $x_D^{eq}$, equilibrium time $t_{eq}^{eq}$ and % time saving for one and two condenser drums for different feed composition $x_F$ and condenser drum holdup $H_C$. (Parameter values: $N = 10$, $\alpha = 2.0$, $V = 10.0$ kmol/hr, $H_f = 0.05$ kmol, $H_P = 10$ kmol).

<table>
<thead>
<tr>
<th>$x_F$</th>
<th>$H_C$ kmol</th>
<th>composition</th>
<th>one drum $t_{eq}^{eq}$ hr</th>
<th>two drums $t_{eq}^{eq}$ hr</th>
<th>% time saving</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1</td>
<td>0.05</td>
<td>0.9931</td>
<td>0.421</td>
<td>0.418</td>
<td>0.7%</td>
</tr>
<tr>
<td></td>
<td>1.0</td>
<td>0.8566</td>
<td>3.230</td>
<td>3.263</td>
<td>-1.0%</td>
</tr>
<tr>
<td>0.5</td>
<td>0.05</td>
<td>0.9995</td>
<td>0.144</td>
<td>0.143</td>
<td>0.7%</td>
</tr>
<tr>
<td></td>
<td>1.0</td>
<td>0.9994</td>
<td>0.502</td>
<td>0.458</td>
<td>8.8%</td>
</tr>
</tbody>
</table>

Light material in the condenser drum initially

A second possible improvement in the startup procedure is to start the batch with either light product from the previous batch or pure light component in the condenser initially. This way, the reflux will already be at its prescribed value and steady state will be reached quicker. This mode of operation is particularly easy to implement if the light component is water. Luyben (1988) proposed to use the first off-cut from a batch to fill up the condenser drum prior to startup of the next batch. However, no results were given. One drawback with this method is that light component which has already been separated is returned to the column and re-processed. The time saved during startup must therefore compensate for a longer production time if this procedure is to be beneficial.

We here consider the following alternative startup procedures: P1) Normal startup where the condenser is initially empty, P2) The condenser is initially filled with light product from the previous batch ($x_D^{eq} = x_D^{spec}$) and P3) The condenser is initially filled with pure light component, $x_D^{eq} = 1.0$. (This is reasonable if the light component is water.) The feed is initially charged to the reboiler and the tray holdups are assumed negligible. The optimal separation of a given mixture according to the three procedures can be found in terms of minimum operating time. Here, we consider a piecewise constant reflux ratio policy with one startup and one production period subject to constraints on the composition and amount of accumulated product:

$$
t_{\text{startup}} + t_{\text{prod}} R_{\text{prod}} t_{\text{tot}} = t_{\text{startup}} + t_{\text{prod}} , \quad H_A(t_{\text{tot}}) \geq H_{A}^{\text{spec}} + H_{C} , \quad z_A(t_{\text{tot}}) \geq \frac{H_{A}^{\text{spec}} x_{A}^{\text{spec}} + H_{D}^{\text{spec}} x_{D}^{\text{spec}}}{H_{A}^{\text{spec}} + H_{C}^{\text{spec}}}
$$

where $i$ is the given procedure P1, P2 or P3 and $H_{C}^{\text{spec}}$ is the amount of added material for procedures P2 and P3. ($H_{C} = 0$ for procedure P1.) In words, the problem is to find the optimal time periods $t_{\text{startup}}$ and $t_{\text{prod}}$ and the optimal constant reflux ratio $R_{\text{prod}}$ which minimises the total operating time $t_{\text{tot}}$ subject to constraints on the composition and amount of accumulated product, $x_A$ and $H_A$. (Equivalently we could have specified the compositions $x_A$ and $x_R$.) Note that the startup time is also optimised. For procedures P2 and P3, the amount of light product added initially must also be removed during the course of operation if these procedures are to be beneficial. For procedure P3 this means that the purity specification is increased. This will ensure the same purity and recovery of the heavy product (but will give a more difficult separation).

During startup the column is run under total reflux ($R_{\text{startup}} = 1.0$).

The optimal results for some examples are given in Table 4. It is found that filling the condenser drum with light product from the previous batch (P2), may be beneficial for mixtures with a very low contents of light component. However, the time saved with procedure P2 relative to the normal procedure P1 is small. Filling the condenser with pure light component (P3) yields in all cases a longer operating time than the normal procedure (P1). However, note that the reflux ratio was assumed constant during the production
Table 4: Optimal results for a constant reflux ratio policy for procedures P1, P2 and P3. (Parameter values: N = 10, α = 2.0, V kmol/hr, H_F = 10 kmol, H_j = 1e - 6 kmol).

<table>
<thead>
<tr>
<th>x_F</th>
<th>H_C</th>
<th>x^A</th>
<th>H^A</th>
<th>% light rec.</th>
<th>t^P1</th>
<th>t^P2</th>
<th>t^P3</th>
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<td>0.2</td>
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<tr>
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<td>2.83</td>
<td>2.76</td>
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</tr>
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</table>

The startup times for procedures P2 and P3 are negligible for all the examples, i.e. product removal can be started immediately. Thus although the startup time is reduced in procedures P2 and P3, this advantage is lost when the total operating time is considered.

CONCLUSIONS
In this paper we have discussed various aspects of startup of a batch distillation column. We have compared the optimal startup time with the optimal total operating time for several cases. It was found that the startup time is a considerable part of the total operating time for difficult separations with high purity and recovery specifications. This is as expected. However, the actual duration of the startup time is of limited significance unless it is very different from the optimal value. The correct startup time is most important for separations with high purity constraints for the lightest product since this is the component taken off first. Alternative ways of reducing the duration of the startup period where discussed and it was found that using two condenser drums in series can reduce the startup time for some separations.

NOTATION

-D distillate flow, kmol/hr
-H_A accumulator holdup, kmol
-H_C condenser drum holdup, kmol
-H_F amount of initial feed, kmol
-H_j liquid holdup on tray j, kmol
-H_off amount of off-cut, kmol
-H_R amount of residual, kmol
-L reflux flow, kmol/hr
-N number of trays in column section
-R reflux ratio = L/V
-t time, hr
-t_startup startup time, hr
-t^opt optimal value
-t^ss steady state

Greek letters

-\alpha relative volatility
-\nu mole fraction in distillate
-\nu_{eq} mole fraction in distillate, 98% appr. to equilibrium
-\nu_f mole fraction of feed
-\nu_{off} mole fraction in off-cut
-\nu_R mole fraction in residual

References

Sørensen, E. and S. Skogestad (1994), Optimal operating policies of batch distillation with emphasis on the cyclic operating policy, Proceedings Process Systems Engineering (PSE), Kyongju, Korea, May-June.