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we consider a reactor where two reactants A and B form product C: $A + B \rightarrow C$. Since there are three components, we call this system a *ternary example*.

Two kinetic cases will be considered. In the first we assume the reaction rate is so large that the limiting reactant B is completely consumed in the reactor, i.e., there is 100 percent per pass conversion of B. The reactor effluent contains only excess reactant A and product C, so the separation section deals with these two components and recycles A back to the reaction section. In industrial processes, this type of system is typically encountered with extremely hazardous reactants, which we want to be completely consumed in the reactor.

In the second case, which is more general for industrial processes, the reaction rate is not large, so complete one-pass conversion of one reactant would require an excessively large reactor. Economics dictate that reactant concentrations must be significant and recycling of reactants is required. Now the separation section must recover both reactants for recycle.

2.7.1 Complete one-pass reactant conversion

Figure 2.11 shows the ternary process where no B is in the reactor effluent. The size of the reactor and concentrations in the reactor are arbitrary because the consumption of B is independent of these variables. We assume that the separation section consists of a single distillation column. If A is more volatile than C, the overhead product from the column is recycled back to the reactor. If the volatilities are reversed, the bottoms from the column is the recycle stream. Figure 2.11 illustrates the first case.

Two control structures are shown in Fig. 2.11*a* and *b*. In both, the composition of component A in the product stream $x_{B,A}$ is controlled by manipulating vapor boilup in the column. This prevents component A from leaving the system. Except for this small amount of A impurity in the product, all A that enters the system must be consumed in the reactor. This illustrates the point we made in Sec. 2.2 about the need to change conditions in the reactor so that the additional reactant is consumed and will not accumulate.

In the first control structure (Fig. 2.11*a*), both fresh reactant feeds are flow-controlled into the system, with one of the reactants ratioed to the other. This type of control structure is seen quite frequently because we want to set production rate with a reactant feed flow and we know that a stoichiometric ratio of reactants is needed. Unfortunately this strategy does *not* work! It is not possible to feed exactly the stoichiometric amounts of the two reactants. Inaccuracies in flow measurement prevent this from occurring in practice with real instru-

control structure that does not work

From Tyreus & Luyben Ind. Eng. Chem. Res 1993, 32, 1154-1162

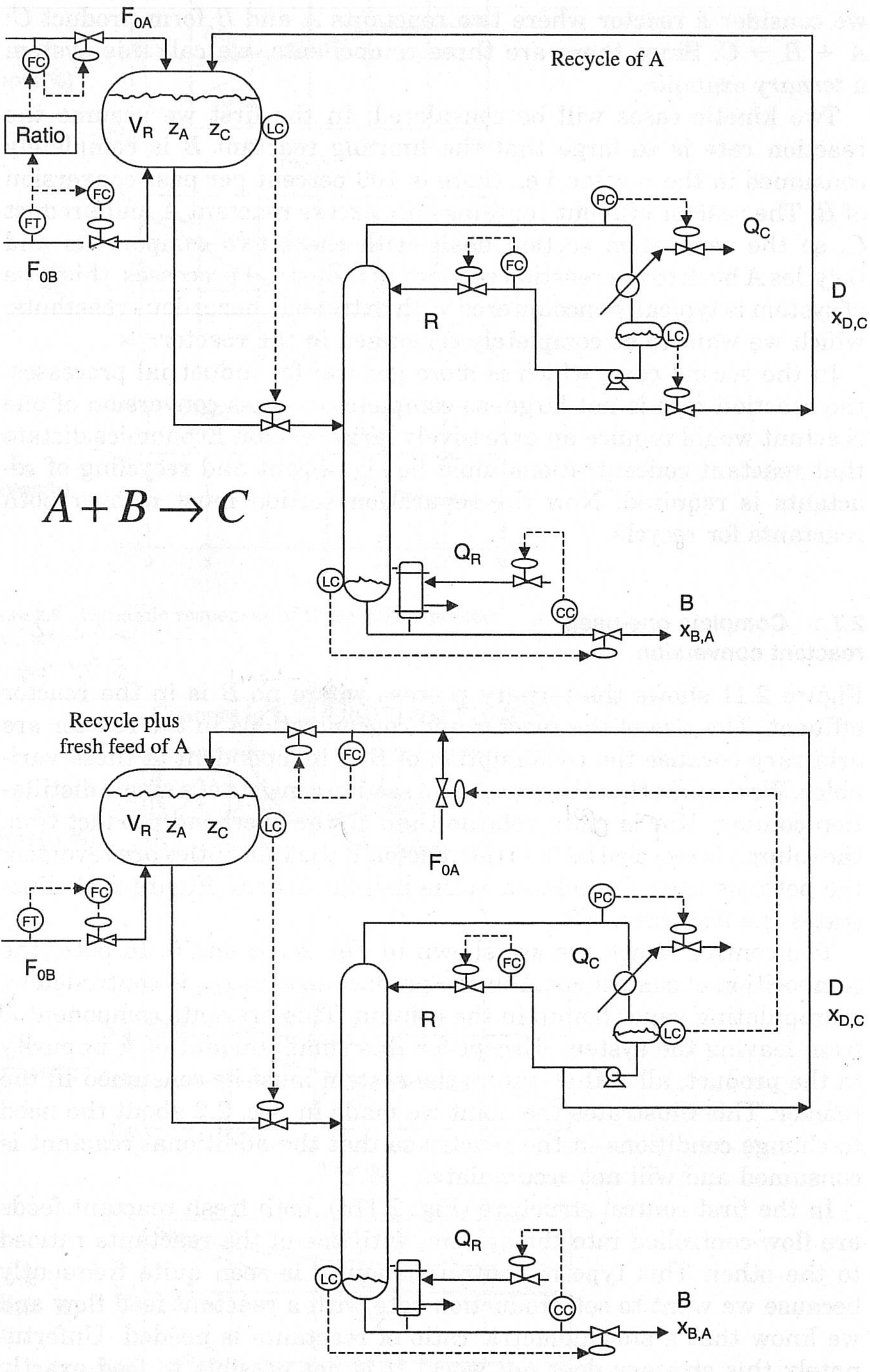
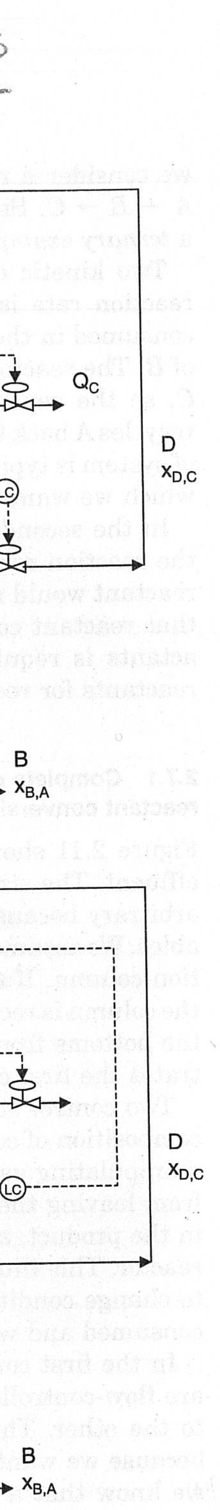


Figure 2.11 Ternary process with complete one-pass conversion of reactant *B*. (*a*) Ratio control structure with fixed reactant feed (unworkable); (*b*) reactant makeup control based on component inventory (workable).



reactant *B*. (*a*) Ratio
tant makeup control

mentation. But even if the flow measurements were perfect, the slightest change in fresh feed compositions would cause the same component imbalance problem. Unless the amounts of the two reactants are perfectly balanced, a gradual buildup will occur of whichever component is in excess. This phenomenon may take hours, days, or weeks. The time depends upon the amount of mismatch between *A* and *B* feeding the system.

In the second control structure (Fig. 2.11*b*), which *does* work, the fresh feed makeup of the limiting reactant (F_{0B}) is flow-controlled. The other fresh feed makeup stream (F_{0A}) is brought into the system to control the liquid level in the reflux drum of the distillation column. The inventory in this drum reflects the amount of *A* inside the system. If more *A* is being consumed by reaction than is being fed into the process, the level in the reflux drum will go down. Thus this control structure employs knowledge about the amount of component *A* in the system to regulate this fresh reactant feed makeup to balance exactly the amount of *B* fed into the process.

argument for the correct structure

Notice that the total rate of recycle plus fresh feed of *A* is flow-controlled. There is a flow controller in the recycle loop, which prevents the snowball effect. Sometimes the fresh feed of *A* is added directly into the reflux drum, making the effect of its flow on reflux drum level more obvious. The piping system where it is not added directly to the drum still gives an immediate effect of makeup flow on drum level because the flowrate of the total stream (recycle plus fresh feed) is held constant. If the fresh feed flow increases, flow from the drum decreases, and this immediately begins to raise the drum level.

2.7.2 Incomplete conversion of both reactants

Now let us consider what is the more common situation where both reactants are present in the reactor effluent. The reaction rate in the reactor $\mathcal{R}$ depends upon the holdup in the reactor V_R, the temperature (through the specific reaction rate k), and the concentrations of both reactants (z_A and z_B):

$$\mathcal{R} = kV_R z_A z_B \tag{2.7}$$

An infinite number of operating conditions in the reactor give exactly the same reaction rate but have different reactor compositions. The only requirement is that the product of the two concentrations (z_A times z_B) be constant. For a given reactor size and temperature, we can have any number of different reactor compositions, and these reactor compositions have a strong impact on the separation system. If z_A is large and z_B is small, there must be a large recycle of *A* and a small recycle

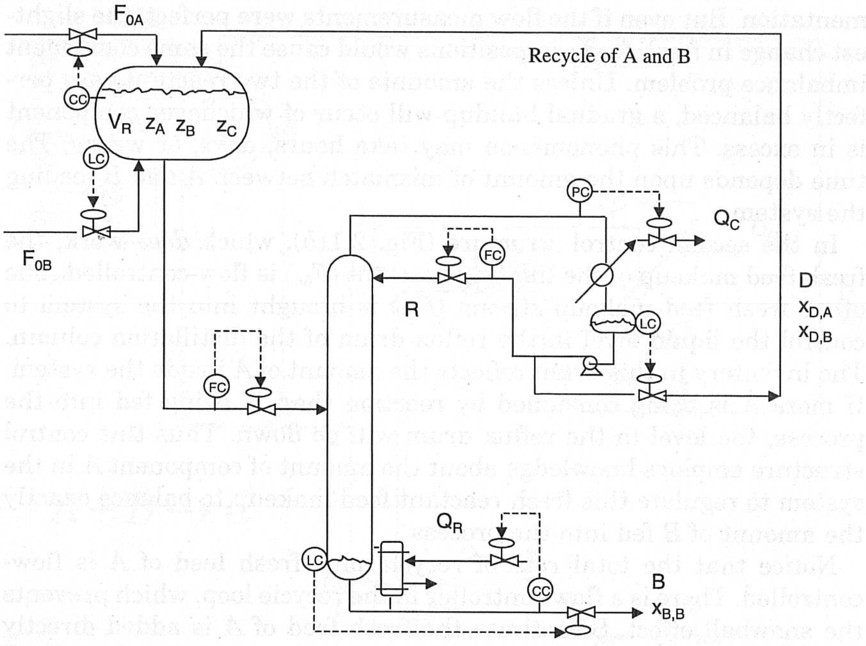


Figure 2.12 Ternary process flowsheet with incomplete conversion of both reactants and one recycle stream.

of B. If the compositions are reversed, the recycle flows are reversed in magnitude. We examine these alternatives later to see how they affect both steady-state economic design and dynamic controllability.

The separation section required to achieve reactant recycle depends upon the relative volatilities of the three components. We consider two cases: (1) the volatility of the product C is heavier or lighter than both of the reactants and (2) the volatility of the product C is intermediate between the reactants. In the first case, we need only one distillation column. In the second, we require two columns if we are limited to a simple two-product configuration.

Single-column case. Let us assume that the relative volatilities are $\alpha_A > \alpha_B > \alpha_C$, so the flowsheet shown in Figure 2.12 is appropriate. Product C is removed from the bottom of the column and contains a small amount of B impurity. It typically has no A because this is the most volatile component. Thus all the A and essentially all the B fed into the process must be consumed in the reactor. The recycle stream is a mixture of mostly B with a modest amount of A and some C. Economics dictate whether this recycle stream should be fairly pure (reducing reactor size but increasing separation costs) or impure.

The control structure shown in Figure 2.12 controls reactor effluent

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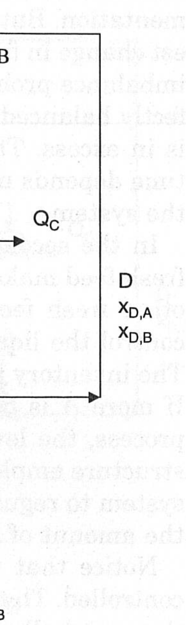

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flow to prevent the snowball effect, controls reactor composition by manipulating fresh feed F_{0A}, and controls reactor level with the fresh feed F_{0B}. Both controlled variables are dominant so we have effective partial control of the reactor. This control strategy works. It satisfies the stoichiometry by adjusting the fresh reactant feed flows.

We might be tempted to control reflux drum level with one of the fresh reactant feeds, as done above. The problem with this is that the material in the drum can contain a little of component *C* mixed with either *A* or *B*. Simply looking at the level doesn't tell us anything about component inventories within the process and which might be in excess. The system can fill up with either. Some measure of the composition of at least one of the reactants is required to make this system work. Compositions in the reactor or the recycle stream indicate an imbalance in the amounts of reactants being fed and being consumed. If direct composition measurement is not possible, inferential methods using multiple trays temperatures in the column are sometimes feasible (Yu and Luyben, 1984).

Two-column case. If the relative volatility of the product *C* is intermediate between the two reactants, a two-column distillation system is typically used. Either the light-out-first (LOF), direct separation sequence, or the heavy-out-first (HOF), indirect separation sequence, can be used. The former is more common because the lightest component only has to be taken overhead once (in the first column) and not twice (as would be the case in the HOF configuration). However there are processes in which the HOF is preferred because it sometimes has the advantage of reducing the exposure of temperature-sensitive components to high base temperatures.

Assuming we use cooling water in both column condensers, the pressure in the first column of the LOF system (with mostly *A*) will be higher than the pressure in the second column (with mostly *B*). The base of the first column contains a mixture of *B* and *C*, and the base temperature can sometimes be too high for thermally sensitive components. Using the HOF system gives a lower pressure in the first column, and even though the base is now mostly *B*, the base temperature is sometimes lower than in the LOF system. In addition, component *B* is being held at high temperature in the base of both columns in the LOF system, and this may be undesirable if *B* is thermally sensitive.

Whatever separation sequence is chosen, the control structures that work well are quite similar. We will choose the HOF system to illustrate this type of process. Figure 2.13 gives a sketch of a ternary process with two recycle streams. The heaviest component *B* is recycled back to the reactor from the base of the first column. The lightest component *A* is recycled back to the reactor from the top of the second column.

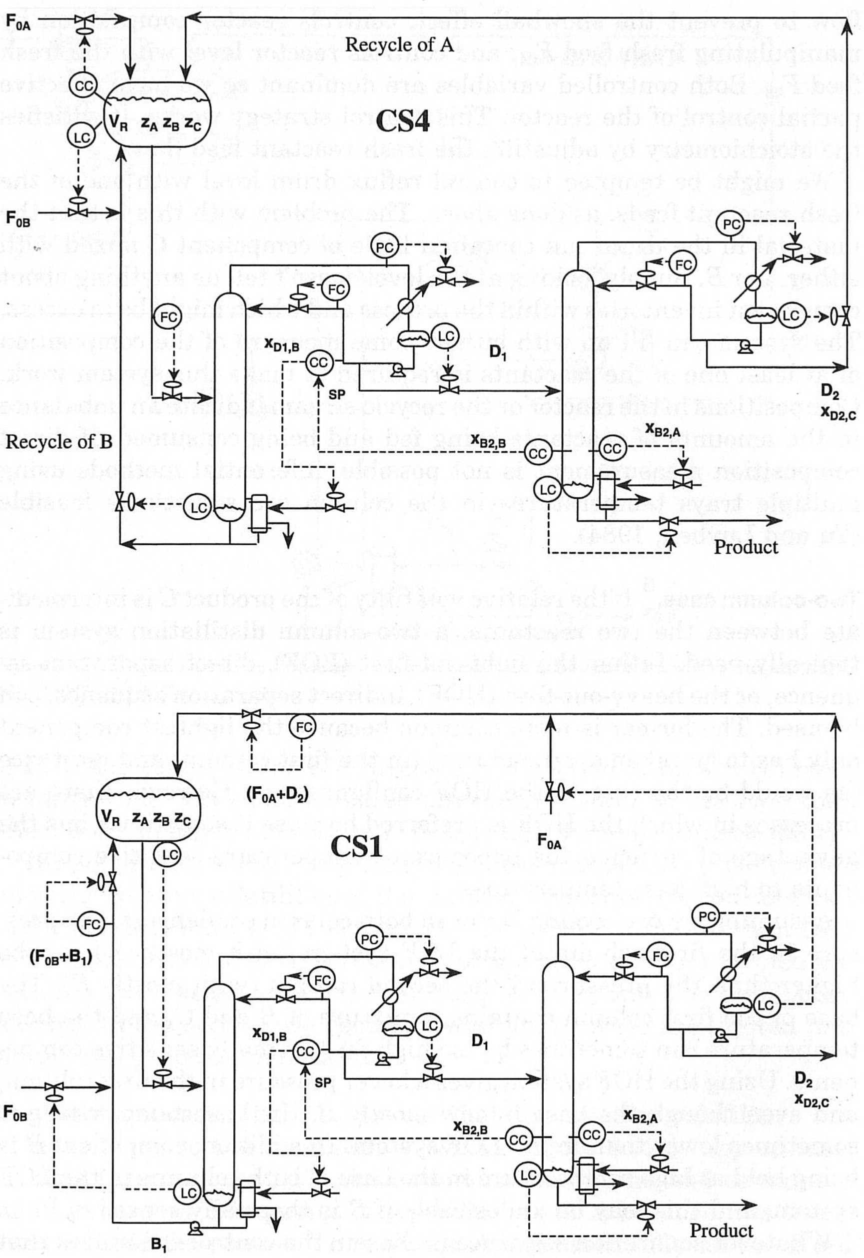

Figure 2.13 Ternary process flowsheet with incomplete conversion and two recycle streams (heavy-out-first sequence). (*a*) Control structure CS4: reactor composition and level control (workable); (*b*) control structure CS1: reactant makeup control based on component inventories (workable).

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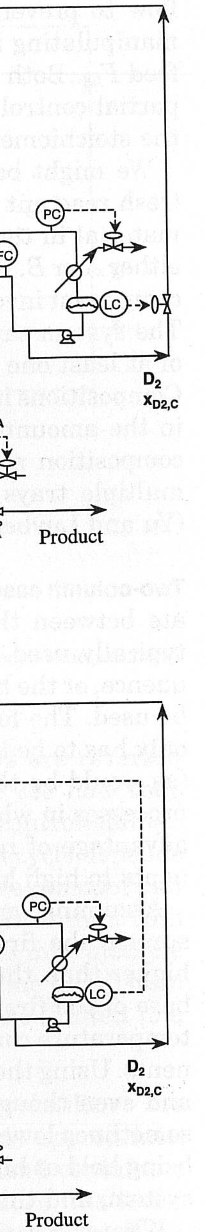

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Figure 2.13*a* and *b* shows two control structures that work (CS4 and CS1). Both of these provide a mechanism for adjusting the fresh feed reactant flowrates so that the overall stoichiometry can be satisfied. In CS4 this is accomplished by measuring reactor composition. In CS1 it is accomplished by deducing the amounts of the reactants in the process from two levels in the two recycle loops.

In both strategies the control of the separation section is similar:

1. In both columns, reflux flows are fixed (or ratioed to feedrates) and pressures are controlled by condenser cooling.
2. The impurity of A ($x_{B2,A}$) in the product stream B_2 from the second column is controlled by vapor boilup in the second column.
3. The impurity of B ($x_{B2,B}$) in the product stream B_2 from the second column is controlled by vapor boilup in the first column through a composition-composition cascade control system. Any B that goes overhead in the first column comes out the bottom of the second column. So the first column must be operated to prevent B from going overhead. The impurity of B in the first column distillate ($x_{D1,B}$) is controlled by a composition controller that manipulates the vapor boilup in the first column. The setpoint of this composition controller is changed by a second composition controller looking at the impurity of B in the product stream ($x_{B2,B}$).

Control structure CS4 (Fig. 2.13*a*) controls reactor effluent flow, brings fresh A in to hold reactor composition z_A, and brings fresh B in to control reactor level. In both columns, the base levels are controlled by manipulating bottoms flowrates and the reflux drum levels are controlled by manipulating distillate flowrates.

Control structure CS1 (Fig. 2.13*b*) controls the flowrates of the two total light and heavy recycle streams; i.e., the sum of the fresh feed and recycle of A ($F_{0A} + D_2$) is flow-controlled and the sum of the fresh feed and recycle of B ($F_{0B} + B_1$) is flow-controlled. The fresh reactant A feed controls the level in the reflux drum of the second column, which reflects this component's inventory within the process. Similarly, the fresh reactant B feed controls the level in the base of the first column.

Both of these control structures have the slight disadvantage of lacking a single direct handle to set production rate, i.e., a one-to-one relationship with product flow. Desired throughput must be achieved by changing the setpoint of the reactor concentration controller, the reactor level controller, the reactor effluent flow controller, and/or the recycle flow controllers (one or both). Structure CS4 has another disadvantage since it requires a composition measurement, which can be very expensive and unreliable in many systems.

We could easily propose many other control structures for this pro-

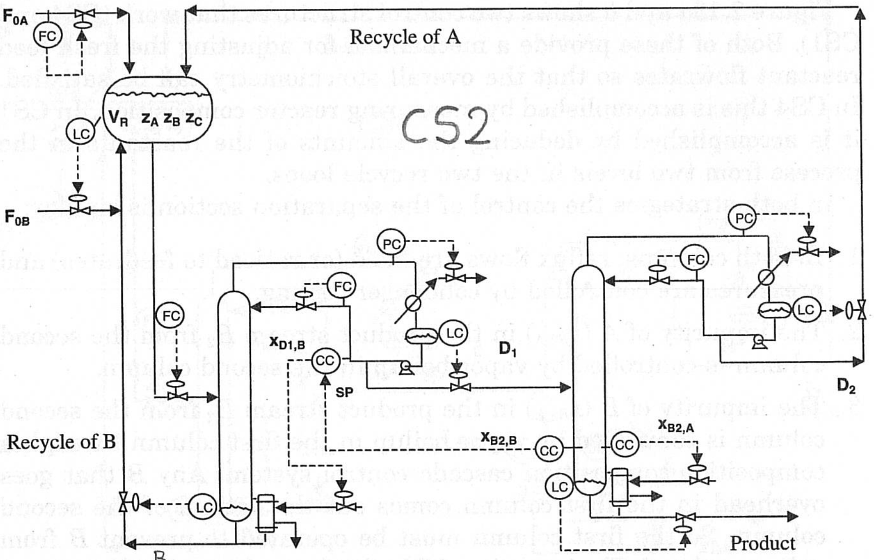

Figure 2.14 Ternary process flowsheet with incomplete conversion and two recycle streams (heavy-out-first sequence): control structure CS2 with fixed flow of one reactant (unworkable).

cess, but most *do not work* in these types of systems. Schemes where one of the reactant fresh feeds is simply flow-controlled into the process do not work unless the per-pass conversion of this limiting component is quite high; i.e., the concentration of this component in the reactor effluent is very small. An analysis of this problem is given in Luyben et al. (1996).

For example, consider the control system shown in Figure 2.14. Here there is a direct handle on production: the flow of fresh A into the system. However, this scheme does not work. Figure 2.15*a* illustrates that the system is able to handle a very small (2 percent) change in fresh feed flow. But if the change in fresh feed flow is increased to 5 percent, the system fills up with A and shuts down after 150 hours (see Fig. 2.15*b*). If the increase is +10 percent (Fig. 2.15*c*), the system shuts down in 70 hours. Thus this control structure can handle only very small disturbances. The imbalance in chemical components and the long time period over which the problem occurs highlight the importance of these phenomena in the plantwide control problem.

2.7.3 Stability analysis

To gain some understanding of what is happening in the results shown in Fig. 2.15 and to explain why the process shuts down, it is useful to

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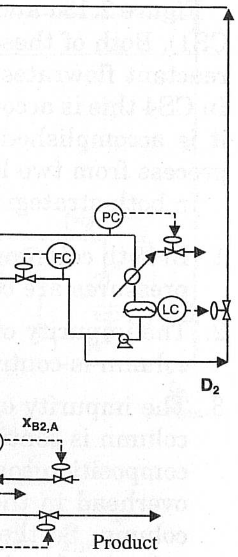

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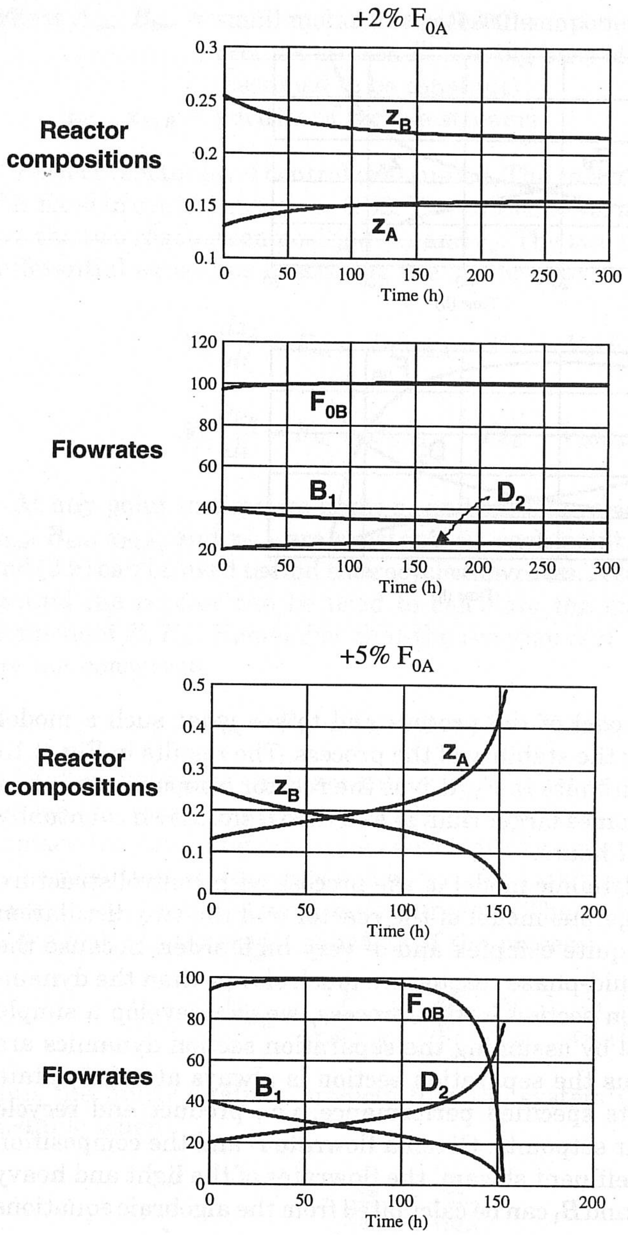

Figure 2.15 Dynamic response of ternary process with CS2 for change in fixed reactant feed rate. (a) 2 percent increase; (b) 5 percent increase; (c) 10 percent increase.

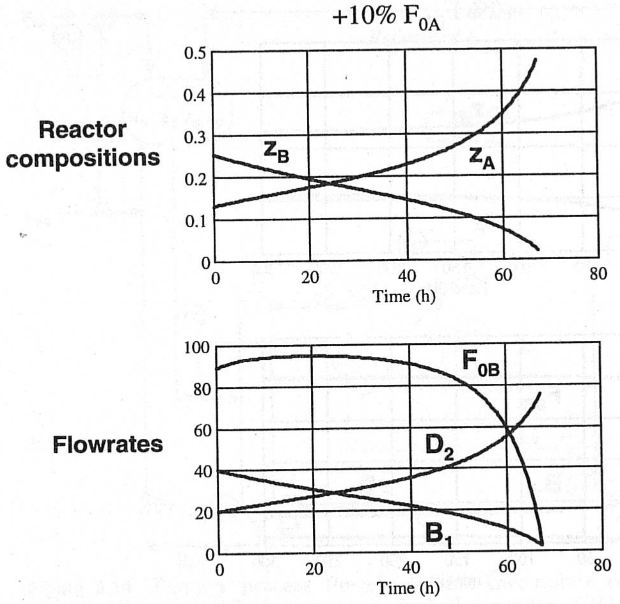

Figure 2.15 *(Continued)*

look at a simple model of the process and to see what such a model predicts concerning the stability of the process. The results in Fig. 2.15 show that the disturbance in F_{0A} drives the reactor compositions into a region where z_A becomes larger than z_B and then a shutdown eventually occurs after several hours.

Let us derive a dynamic model of the process with control structure CS2 included. A rigorous model of the reactor and the two distillation columns would be quite complex and of very high order. Because the dynamics of the liquid-phase reactor are much slower than the dynamics of the separation section in this process, we can develop a simple second-order model by assuming the separation section dynamics are instantaneous. Thus the separation section is always at steady state and is achieving its specified performance, i.e., product and recycle purities are at their setpoints. Given a flowrate F and the composition z_A/z_B of the reactor effluent stream, the flowrates of the light and heavy recycle streams D_2 and B_1 can be calculated from the algebraic equations

$$D_2 = \frac{Fz_A - A_{\text{loss}}}{x_{D2,A}} \tag{2.8}$$

$$B_1 = \frac{Fz_B - B_{\text{loss}}}{x_{B1,B}} \tag{2.9}$$

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where $A_{\text{loss}}, B_{\text{loss}}$ = small molar flowrates of components A and B in product stream B_2 leaving base of second column (assumed to be constant)

$x_{D2,A}, x_{B1,B}$ = purities of recycle streams

Perfect reactor level control is assumed. The reactor effluent flowrate F is fixed in control structure CS2. The two state variables of the system are the two reactor compositions z_A and z_B. The two nonlinear ordinary differential equations describing the system are

$$V_R \frac{dz_A}{dt} = F_{0A} + D_2 x_{D2,A} - F z_A - V_R k z_A z_B \tag{2.10}$$

$$V_R \frac{dz_B}{dt} = F_{0B} + B_1 x_{B1,B} - F z_B - V_R k z_A z_B \tag{2.11}$$

At any point in time we know z_A and z_B. The variables F, F_{0A}, k, V_R, A_{loss}, B_{loss}, $x_{D2,A}$, and $x_{B1,B}$ are constant. At each point in time Eqs. (2.8) and (2.9) can be used to find the recycle flowrates. A total molar balance around the reactor can be used to calculate the makeup flowrate of component B, F_{0B}. Remember that the reaction is $A + B \rightarrow C$, so moles are not conserved.

$$F_{0A} + F_{0B} + D_2 + B_1 = F + V_R k z_A z_B \tag{2.12}$$

The two nonlinear ordinary differential equations can be linearized around the steady-state values of the reactor compositions $\bar{z}_A$ and $\bar{z}_B$. Laplace transforming gives the characteristic equation of the system. It is important to remember that we are looking at the closed-loop system with control structure CS2 in place. Therefore Eq. (2.13) is the closed-loop characteristic equation of the process:

$$s^2 + s\left[k\bar{z}_B + \frac{F}{V_R x_{B1,B}}\right] + \frac{kF}{V_R}\left[\frac{\bar{z}_B}{x_{B1,B}} - \frac{\bar{z}_A}{x_{D2,A}}\right] = 0 \tag{2.13}$$

Thus the linear analysis predicts that the system will be closed-loop unstable when

$$\frac{\bar{z}_B}{x_{B1,B}} < \frac{\bar{z}_A}{x_{D2,A}} \tag{2.14}$$

If the two recycle purities are about the same ($x_{B1,B} \cong x_{D2,A}$), which is the case in the numerical example considered earlier in the chapter, the linear analysis predicts that instability will occur when z_A is bigger than z_B. This is exactly what we observed in Fig. 2.15.

The physical reason for this instability is the lack of some mechanism in the process or in the control structure to ensure that the A and B component balances are satisfied in this integrating plantwide process. Both reactant components are prevented from leaving the system by the impurity controllers that are looking at the product stream. Thus essentially all of the reactants fed into the system must be consumed by chemical reaction. And the stoichiometry must be satisfied down to the last molecule: every mole of A requires exactly one mole of B to react with. The flowrates of the fresh feed cannot be controlled in an open-loop fashion anywhere nearly accurately enough to match the molecules of the two reactants exactly. This is why we need some information about the amounts of the two components in the system. This knowledge can be used in a feedback control system to make some adjustments so that the component in excess does not continue to build up in the system.

2.7.4 Modification of CS2

Both of the control structures discussed in Sec. 2.7.2 (CS1 and CS4) work because they detect the inventories of the reactant components A and B in the system and bring in fresh feed streams to balance the consumption of the two components. Structure CS1 does this by using the liquid level in the reflux drum of the second column as an indicator of the amount of A in the system and the liquid level in the base of the first column as an indicator of the amount of B in the system. Structure CS4 uses a composition analyzer to measure directly the concentration of one of the reactants in the reactor. But both of these structures lack a direct handle on production rate.

Control structure CS2 has such a direct handle, but this structure does not work. However, a modification can be made to CS2 that will make it work. The basic idea is to recognize that the separation section acts like an on-line analyzer. Any component B in the reactor effluent gets recycled in B_1. Any component A in the reactor effluent gets recycled in D_2. Therefore, the flowrates of these two streams give a direct indication of the amounts of the two reactants in the system.

Figure 2.16 shows a control scheme in which the ratio of the two recycle flowrates is controlled by adjusting the flowrate of the reactor effluent. The dynamics of the separation system must be considered because a change in the amount of A in the reactor effluent has to work its way through two columns before showing up as a change in the flowrate of D_2. Thus a lag is added to the measurement of B_1 before it is used to calculate the ratio. This control structure works.

In this modified CS2, the feedback adjustment that is made to adjust for any imbalance in the amounts of the two reactants in the system is a change in the reactor effluent flowrate to achieve a constant ratio

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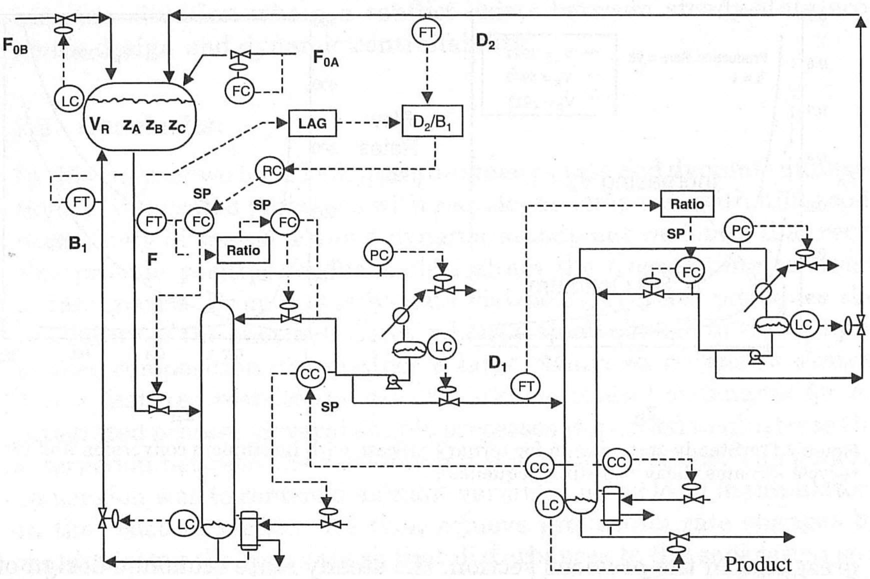


Figure 2.16 Ternary process flowsheet with incomplete conversion and two recycle streams (heavy-out-first sequence): control structure CS2C using separation as analyzer for control of D_2/B_1 ratio (workable).

of the two recycle flowrates. This works because these flowrates give good indications of the concentrations of the two reactants in the reactor. The two columns act like composition analyzers, separating the A and B components from the product C.

In the numerical case studied in this chapter, we considered a liquid-phase reactor with dynamics that were slower than the dynamics of the separation system. Suppose we have a process with a vapor-phase reactor whose dynamics are much faster than those of the separation section. Will the modified CS2 control structure work in this process?

Luyben et al. (1996) explored this question in detail by developing a rigorous simulation of such a process. Their results demonstrate that the proposed control structure does provide effective control for processes with fast reactor dynamics. The time constant of the separation section is about 30 minutes. The reactor time constant was reduced to 3 minutes, and control was still good.

2.7.5 Reactor composition trade-offs

As discussed earlier, if the concentration of A (or B) in the reactor is essentially zero, we can flow-control the fresh feed of A (or B) into the system, and large disturbances can be handled. In the numerical case

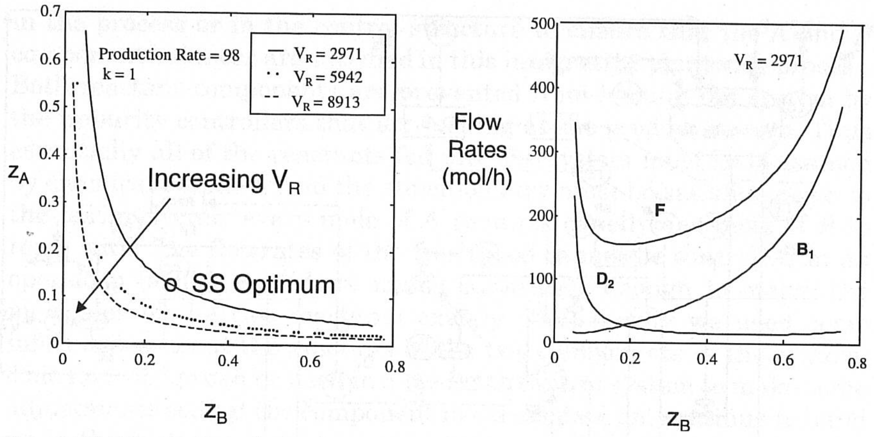


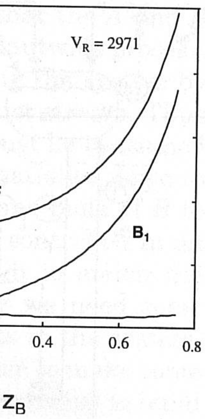
Figure 2.17 Steady-state design for ternary process with incomplete conversion and two recycle streams (heavy-out-first sequence).

presented in the previous section, the steady-state economic design of the process yielded reactor compositions that are $z_A = 0.15$ mole fraction and $z_B = 0.25$ mole fraction. It is cheaper to recycle B than A because B comes out the bottom of the first column and does not have to be vaporized. Component A, on the other hand, must be vaporized twice as it is taken overhead in both columns. Therefore the steady-state separation design favors smaller z_A and larger z_B. But remember that if reactor temperature and holdup are constant (fixed k and V_R), the product of the two concentrations must be fixed to achieve a given production rate of C.

Figure 2.17 illustrates that we must lie somewhere on the hyperbolic line in the $z_A - z_B$ plane. At any position on one of the constant reactor volume lines, the production rate is constant. The concentrations fed to the separation section vary with our choice of location on this curve. For large z_A and small z_B, the recycle of A (D_2) is large. For large z_B and small z_A, the recycle of B (B_1) is large.

Since we are dealing with the product of the two reactant concentrations, making them approximately equal is the best way to minimize reactor holdup. Thus steady-state reactor design favors compositions that are somewhat similar. From a dynamic viewpoint, the system can handle disturbances more easily if the concentrations of the two reactants are very different (very small z_A and large z_B). We saw an indication of this in the ternary process considered earlier. Control structure CS2 worked when the concentration of the limiting reactant was very low, but failed when the concentration of the limiting reactant was in the 0.15 mole fraction region.

So this simple process provides another nice example of the very



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common situation where a conflict exists between steady-state economic design and dynamic controllability.

2.8 Conclusion

In this chapter we have looked at the steady-state and dynamic implications of integrated processes with recycle (as compared with units connected only in series). From a dynamic standpoint we found that recycles provide positive feedback that alters the overall time constant of the process. From a steady-state viewpoint, recycles introduce the possibility of the snowball effect, where a small change in throughput or feed composition can produce a large change in recycle flowrates. These features restrict the set of workable control structures for an integrated process. Several simple processes were used to illustrate the interaction between the reaction and separation sections. The generic conclusion was to control dominant variables using local manipulators in the reaction section. We then achieve production rate changes by manipulating the setpoints so that disturbances to the separation section are minimized, thereby reducing product quality variability. Another point that was highlighted involved the need for the control strategy to account for the chemical component balances, i.e., to keep track of the inventory of components within the system.

2.9 References

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