

# A SIMPLE ALGORITHM FOR CALCULATION OF PHASE SEPARATION

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### SYMMETRIC SOLUTIONS

Most undergraduate [1, 2] and graduate [3, 4] texts in chemical engineering thermodynamics illustrate the separation of a single liquid phase into two liquid phases for a binary system by graphs of the change of the Gibbs energy upon mixing,  $\Delta_m G$ , versus composition,  $x_1$ , at constant temperature,  $T$ , and pressure,  $P$ . The first graph, Figure 1, is often for a symmetric, non-ideal liquid solution obeying the one-constant Margules model [1]

$$\begin{aligned} (\Delta_m G/RT) &\equiv \phi \equiv G_m - x_1 G_1 - x_2 G_2 \\ &\equiv x_1 \ln x_1 + x_2 \ln x_2 + Ax_1 x_2 \end{aligned} \quad (1)$$

where  $G_1$  and  $G_2$  are the pure component molar Gibbs energies,  $G_m$  the molar Gibbs energy of the liquid mixture taken as a single phase, and  $A$ , which depends only on temperature, indicates the deviation from an ideal solution (where  $A=0$ ). In terms of the excess Gibbs energy,  $G^E$ , and activity coefficients,  $\gamma_i$ , Eq. (1) is

$$(G^E/RT) \equiv Ax_1 x_2 \equiv x_1 \ln \gamma_1 + x_2 \ln \gamma_2 \quad (2)$$

where

$$\ln \gamma_1 = Ax_2^2 \quad \text{and} \quad \ln \gamma_2 = Ax_1^2$$

Single-phase stability [5] is observed as long as

$$(\partial^2 G_m / \partial x_1^2)_{P,T} \geq 0 \quad \text{or} \quad [\partial^2 (\Delta_m G/RT) / \partial x_1^2]_{P,T} \geq 0$$

For our symmetric binary solution

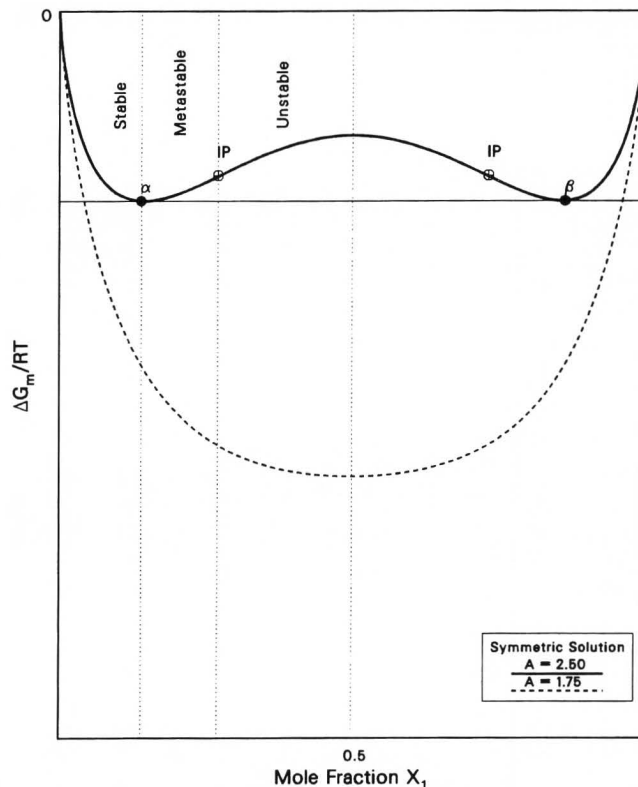
$$\phi' \equiv [\partial (\Delta_m G/RT) / \partial x_1]_{P,T} = \ln(x_1/x_2) + A(x_2 - x_1) \quad (3)$$

and

$$\phi'' \equiv [\partial^2 (\Delta_m G/RT) / \partial x_1^2]_{P,T} = (x_2 x_1)^{-1} - 2A \quad (4)$$

Thus, Figure 1 provides single-phase stability over the entire composition range for  $A \leq 2$  because  $(x_2 x_1)^{-1}$  reaches its minimum value of four at  $x_1 = 1/2$ ,

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**FIGURE 1.** The dimensionless change in the Gibbs energy upon mixing,  $\Delta_m G/RT$ , versus composition  $x_1$  at a constant temperature and pressure for a symmetric solution. IP = inflection point.

the vertical line of symmetry. However, when  $A > 2$ , we observe a pair of symmetric inflection points (IP) at  $(x_2 x_1)^{-1} = 2A$  plus a pair of symmetric minima at

$$\ln(x_1/x_2) = A(x_1 - x_2)$$

Between the minima and inflection points the solution (as a single phase) is termed *metastable* whereas between the inflection points it violates material stability [5] and so is termed *unstable*. Physically, a single-phase liquid is stable between the pure-component end points and the minima (labeled  $\alpha$  and  $\beta$ ) whereas two liquid phases of composition

$$x_1^\alpha \quad \text{and} \quad x_1^\beta$$

are stable for any overall system composition  $z_1$  falling between the minima.

Numerical determination of

$$x_1^\alpha (= 0.5 - \epsilon) \quad \text{and} \quad x_1^\beta (= 0.5 + \epsilon)$$

is a simple task for symmetric solutions because by Eq. (3)

$$\ln [(0.5 + \epsilon)/(0.5 - \epsilon)] = 2\epsilon A \quad (5)$$

Given a value of  $A$ , Eq. (5) should be solved for  $\epsilon$  by trial; a useful first approximation is

$$\epsilon^2 \cong \left( \frac{5}{24} \right) \left( \sqrt{1 + \frac{18}{5} (A - 2)} - 1 \right) \quad (6)$$

based upon the series expansion of

$$\ln (1 - y) \cong - \sum_{n=1}^{\infty} (y^n/n)$$

which is exact as  $\epsilon$  approaches zero. At  $A = 2.3$ , Eq. (6) provides  $\epsilon$  within 2.5%. However, when  $A = 3.0$ ,  $\epsilon$  by Eq. (6) is 13.8% too high and the limiting value of  $\epsilon = 0.5$  is reached by Eq. (6) for  $A = 3.067$ .

For the symmetric solution, upper and lower critical solution temperatures (CST) occur at  $A = 2$ . If

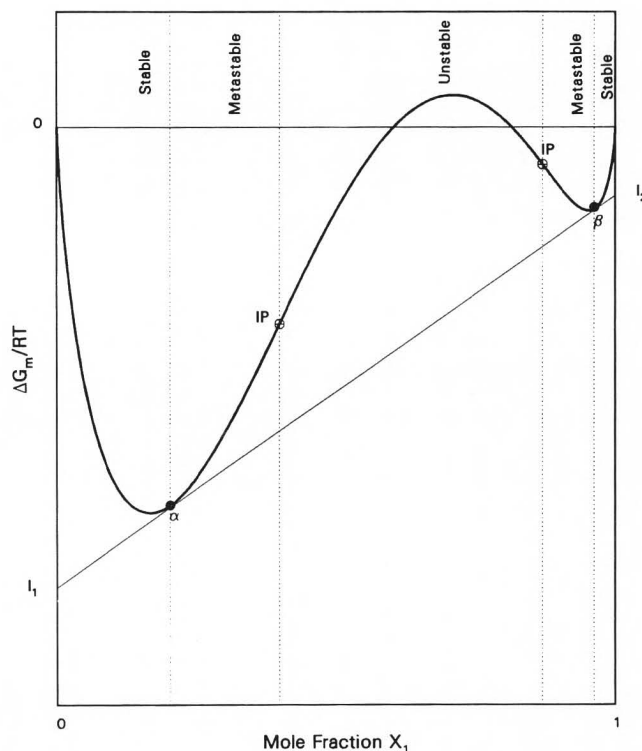
$$(dA/dT) > 0 \quad \text{at} \quad A = 2$$

then we have a lower CST; conversely when



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**FIGURE 2.** Similar to Figure 1 but for an unsymmetric solution showing two minima. The intercepts  $I_i$  equal  $(\bar{G}_i - G_i)/RT$ , the difference between the chemical potential in the solution of component  $i$  and that of pure  $i$ .

$$(dA/dT) < 0 \quad \text{at} \quad A = 2$$

we have an upper CST.

#### TANGENT LINE METHOD

The equilibrium tie-line between the coexisting liquid phases  $\alpha$  and  $\beta$  of Figure 1 is horizontal because the solution is symmetric. Figure 2 illustrates the tie-line for the general case of an unsymmetric solution. This tie-line must be tangent to the curve at both  $\alpha$  and  $\beta$  in order to have equality of the chemical potential of species  $i$ ,

$$\hat{\mu}_i^\alpha = \hat{\mu}_i^\beta$$

The method of tangent intercepts [3] provides the intercepts  $I_1$  and  $I_2$  (Figure 2) in terms of partial molar properties

$$I_1 = (\bar{G}_1 - G_1)/RT \quad \text{and} \quad I_2 = (\bar{G}_2 - G_2)/RT \quad (7)$$

For a tangent common to points  $\alpha$  and  $\beta$ ,

$$\hat{\mu}_1^\alpha = \bar{G}_1^\alpha = \bar{G}_1^\beta = \hat{\mu}_1^\beta$$

and likewise for the second component. A minimiza-

... it makes little difference whether we are examining liquid-liquid ... or gas-liquid phase separation such as dew and bubble points ... the "inside-out" algorithm remains efficient for determination of the equilibrium states for any type of phase transition for a binary system.

tion of  $G_m$  (or maximization of system entropy) provides the number of phases that are stable for a given overall composition  $z_1$ . A single phase rich in component 2 is thus stable from

$$x_1 = 0 \quad \text{to} \quad x_1^\alpha$$

two phases are stable for  $z_1$  between

$$x_1^\alpha \quad \text{and} \quad x_1^\beta$$

and a single phase rich in component 1 is stable from

$$x_1^\beta \quad \text{to} \quad x_1 = 1$$

The curve of Figure 2 represents the single-phase  $G_m$  whereas the tangent line represents the two-phase system.

Use of the tangent plane criterion for stability in phase equilibria has enjoyed considerable interest in the past decade [6, 7, 8, 9]. Most of the algorithms are based upon Newton's method and are different from that of the next section.

Because the "curvature,"

$$\left[ \partial^2 (\Delta_m G/RT) / \partial x_1^2 \right]_{P,T}$$

is positive infinite at both pure-component end points, it is obvious that the inflection points of the curve come in pairs. For general, unsymmetric solutions, it is further obvious that the equilibrium points ( $\alpha$ ,  $\beta$ ) do not occur at minima and, indeed as in Figure 3, there may be phase separation with only one minimum. In conclusion, the presence of one or more *pairs* of inflection points are a necessary and sufficient condition for phase separation for an overall composition  $z_1$  bounded by

$$x_1^\alpha \quad \text{and} \quad x_1^\beta$$

Each pair of IP provides a tangent line, which is an equilibrium tie line so long as it does not intersect the curve at a composition outside the two tangent compositions (as in Figure 3). The tangent line for each IP pair has tangent compositions outside the compositions at the IP. However, these tangent compositions do not represent stable equilibrium points when their tangent line cuts the curve at another point as in Fi-

gure 4. Here the interior IP pair is "wasted" and meaningless as far as real fluid behavior is concerned.

#### ALGORITHM FOR CALCULATION OF PHASE EQUILIBRIA

Assume that we have an equation,

$$\phi(x_1)$$

for the curve representing the single-phase system. We first set

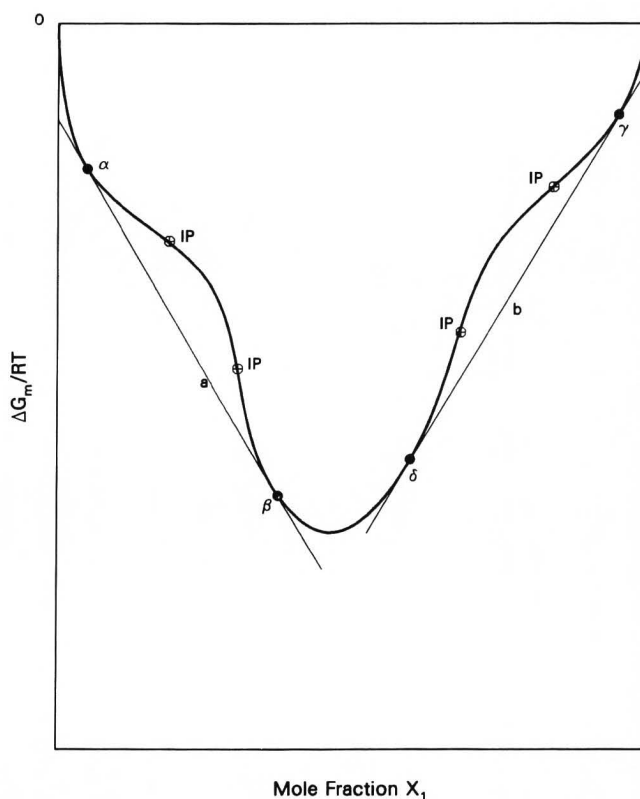
$$\phi''(x_1) = 0$$

and solve for the location of the IP pairs. Imagine for simplicity that only one pair occurs as in Figure 2. To find the tangent line,

$$x_1^\alpha \quad \text{and} \quad x_1^\beta$$

we recognize that the tangent line:

- 1) Must pass through the points  $\alpha$  and  $\beta$ .



**FIGURE 3. An unsymmetric solution exhibiting four inflection points (IP) which produce double phase separation,  $\alpha/\beta$  and  $\delta/\gamma$ , but only a single minimum. Note that the tangent lines *a* and *b* do not cross the curve.**

- 2) Must be tangent to the  $\phi$  curve at  $\alpha$  and at  $\beta$  so that

$$\phi'(x_1^\alpha) = \phi'(x_1^\beta) = m$$

the slope of the tangent line.

- 3) Cannot cut the  $\phi$  curve at any  $x_1$ —that is, must lie entirely below the  $\phi$  curve.

The equation of the tangent line is thus

$$\phi_{TL} = I_2 + mx_1 = I_1 - m(1 - x_1) \quad (8)$$

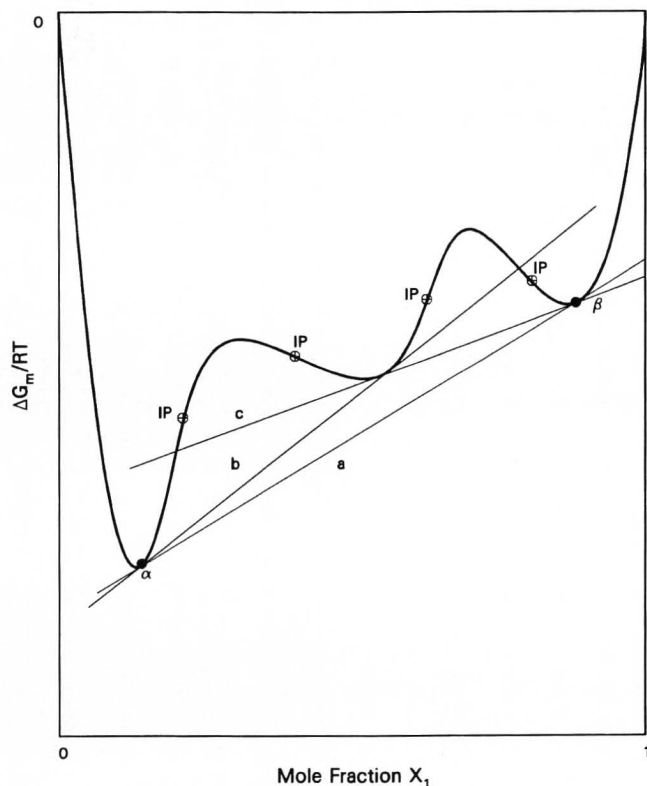
where

$$m = \phi'(x_1^\alpha) = \phi'(x_1^\beta) = [\phi(x_1^\alpha) - \phi(x_1^\beta)] / (x_1^\alpha - x_1^\beta) \quad (9)$$

Our new algorithm is based upon the observations that (1) the equilibrium points  $\alpha$  and  $\beta$  must lie outside the IP pair and (2) a straight line drawn through the IP pair will have a *slope* very near to that of the corresponding tangent line. Rather than seeking

$$x_1^\alpha \quad \text{and} \quad x_1^\beta$$

by approaching from the pure end-points, this al-



**FIGURE 4.** An unsymmetric solution exhibiting four inflection points (IP) which produce only a single phase separation,  $\alpha/\beta$ , but three local minima. Note that the tangent lines b and c cross the curve and so are disallowed in determination of phase equilibria.

gorithm is an “inside-out approach.” The general numerical procedure for this algorithm follows (see Figure 2):

- 1) Set  $\phi''(x_1) = 0$  and solve for the IP pairs,

$$x_1^{a_0} \quad \text{and} \quad x_1^{b_0}$$

- 2) Approximate  $m$  by

$$m_0 = [\phi(x_1^{a_0}) - \phi(x_1^{b_0})] / (x_1^{a_0} - x_1^{b_0})$$

- 3) Solve the equation

$$\phi'(x_1) = m_0$$

for the roots

$$x_1^{a_1} \quad \text{and} \quad x_1^{b_1}$$

- 4) Approximate  $m$  by

$$m_1 = [\phi(x_1^{a_1}) - \phi(x_1^{b_1})] / (x_1^{a_1} - x_1^{b_1})$$

- 5) Repeat step (3) to provide

$$x_1^{a_2} \quad \text{and} \quad x_1^{b_2}$$

and continue to repeat steps (2) and (3) until

$$(x_1^{a_n} - x_1^{a_{n-1}}) \leq 10^{-4} \geq (x_1^{b_n} - x_1^{b_{n-1}})$$

for example, as a convergence criterion. Then

$$x_1^{a_n} \approx x_1^\alpha \quad \text{and} \quad x_1^{b_n} \approx x_1^\beta$$

with

$$m \approx m_n = [\phi(x_1^{a_n}) - \phi(x_1^{b_n})] / (x_1^{a_n} - x_1^{b_n})$$

#### NUMERICAL EXAMPLES FOR UNSYMMETRIC SOLUTIONS

We now show two numerical examples of the above algorithm as applied to liquid solutions qualitatively similar to Figures 2 and 3, respectively. All the calculations in this article were performed with a simple hand calculator.

The qualitative nature of both Figures 2 and 3 is captured by the simple mathematical model

$$\phi = x_1 \ln x_1 + x_2 \ln x_2 + Bx_1x_2(1 + x_1) \quad (10)$$

where  $B$  is only a function of  $T$ . This empirical equation, which is of the two-constant Margules form ( $A_{12} = B$ ,  $A_{21} = 2B$ ), may be differentiated to yield

$$\phi' = \ln(x_1/x_2) + B(1 - 3x_1^2) \quad (11)$$

and

$$\phi'' = (x_1x_2)^{-1} - 6Bx_1 \quad (12)$$

When set to zero, Eq. (11) provides the location of extrema of  $\phi$  whereas Eq. (12) provides the location of IP pairs. It can be shown that no IP pairs exist for  $B \leq (9/8)$  whereas only a single pair exists for  $B > (9/8)$ . For  $(9/8) < B < 1.484774$  only a single minimum of  $\phi$  exists as in Figure 3. However, when  $B > 1.484774$ , two minima and one maximum occurs as in Figure 2.

**Case I:** Let  $B = (7/4)$  to correspond qualitatively to Figure 2. Eq. (11) provides a minima at  $x_1 = 0.1676$  and at 0.9537 whereas Eq. (12) yields IP at  $x_1 = 0.3976$  and 0.8758. Following the steps of the new algorithm at the end of the previous section

$$x_1^{a_0} = 0.3976 \quad \text{and} \quad x_1^{b_0} = 0.8758$$

so that

$$m_0 = \frac{[-8.6223 - (-1.8142)]10^{-2}}{(0.3976 - 0.8758)} = 0.14237$$

the first estimate of the slope of the tangent line. Eq. (11) is now solved for  $\phi' = m_0$  to obtain the roots

$$x_1^{a_1} = 0.1973 \quad \text{and} \quad x_1^{b_1} = 0.9631$$

Then

$$m_1 = \frac{[-16.4800 - (-3.5874)]10^{-2}}{(0.1973 - 0.9631)} = 0.16835$$

the second estimate of the slope of the tangent line. Eq. (11) now provides the roots

$$x_1^{a_2} = 0.2036 \quad \text{and} \quad x_1^{b_2} = 0.9645$$

Then

$$m_2 = \frac{[-16.3820 - (-3.5657)]10^{-2}}{(0.2036 - 0.9645)} = 0.16844$$

Eq. (11) yields

$$x_1^{a_3} = 0.2036 \quad \text{and} \quad x_1^{b_3} = 0.9645$$

so that our convergence criterion has been met. Thus

$$x_1^\alpha = 0.2036 \quad \text{and} \quad x_1^\beta = 0.9645$$

are the equilibrium points. Note their position relative to the minima.

**Case II:** Let  $B = (4/3)$  to correspond qualitatively to Figure 3 on the right side of the minimum. Now there is but one minimum at  $x_1 = 0.2546$  plus the IP pair at  $x_1 = 0.5000$  and 0.8090 where  $\phi = -0.193147$  and  $-0.114967$ , respectively. The first approximation of the tangent slope is thus

$$m_0 = \frac{(-0.114967 + 0.193147)}{(0.8090 - 0.5000)} = 0.25299$$

From Eq. (11),

$$x_1^{a_1} = 0.3696 \quad \text{and} \quad x_1^{b_1} = 0.8891$$

where

$$\phi = -0.233290 \quad \text{and} \quad -0.10040$$

respectively. Then,

$$m_1 = 0.25645$$

where

$$x_1^{a_2} = 0.3722 \quad \text{and} \quad x_1^{b_2} = 0.8902$$

with

$$\phi = -0.232600 \quad \text{and} \quad -0.099756$$

respectively. Then,

$$m_2 = 0.25646$$

where

$$x_1^{a_3} = 0.3722 \quad \text{and} \quad x_1^{b_3} = 0.8902$$

are our equilibrium values of

$$x_1^\alpha \quad \text{and} \quad x_1^\beta$$

respectively.

## SUMMARY

The new algorithm also provides more rapid convergence for more complicated forms of  $\phi$  requiring the use of a digital computer. Examples are where  $\phi$  is given by the van Laar equation, general two-constant forms of the Margules equation, or from equations of state (EOS) with mixture combining rules (MCR). Further, it makes little difference as to whether we are examining liquid-liquid phase separation or gas-liquid phase separation such as dew and bubble points (with  $\phi$  from EOS/MCR). This "inside-out" algorithm remains efficient for determination of the equilibrium states for any type of phase transition for a binary system.

Cases of more than one pair of inflection points, Figures 3 and 4, are solved easily by the present algorithm provided an override computer program is used to first calculate the slope of line through the paired inflection points. In the case of Figure 3, these slopes are of opposite sign so two separate regions of liquid immiscibility exist. The equilibrium points ( $\alpha$ ,  $\beta$ ) and ( $\delta$ ,  $\gamma$ ) may then be found by double application of the present algorithm as illustrated by Case II for ( $\delta$ ,  $\gamma$ ) on the right side of Figure 3. With Figure 4, the override program would see that the slopes of lines through the paired IP are the same. It would then check the slope from the two outermost IP. Knowing this slope to be between the original two in magnitude



is a strong indication that the final equilibrium tangent is **a** rather than **b** or **c** of Figure 4. These comments are only guidelines for Figures 3 and 4—see [6], Figure 5, for a different case involving double pairs of IP. The only rule is that no true equilibrium tangent may cut the Gibbs' curve at any composition.

We are presently working on procedures to extend this algorithm to ternary and higher component systems.

## ACKNOWLEDGEMENT

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## REVIEW: Reactor Engineering

Continued from page 7.

California Professional Engineers exam. Some involve new technologies (semiconductor processing, biotechnology) and some require numerical solutions. In several chapters excellent problems on critiques of journal articles are given.

A series of accompanying interactive programs for personal computers is available on floppy disks, though they must be purchased separately from the University of Michigan. These are interesting problems that can be used as homework assignments since they provide the student a coded grade. Students find the programs to be both fun and helpful for learning reactor design.

A few aspects of the book could be improved. As done in essentially all reactor design texts, fractional conversion is used as a dependent variable and solutions start with an integrated form of the design equation. A more general approach, which is more easily extended to multiple reactions and complicated reactors, would be to use flow rates and number of moles as dependent variables and start with the differential form of the design equation. The energy balances in Chapter 8 are complicated by using variable heat capacities and symbols for several types of heat capacities. In Chapter 6, the rate of reaction is incorrectly shown as being proportional to the square of the total site concentration on the catalyst surface. Also, as done in many texts, more significant figures are given in the solutions to the example problems than are justified by the data presented.

In summary, this is an excellent undergraduate text for reactor design and it will likely be adopted by a large number of departments. It could also be used as a graduate text if supplemented. □

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