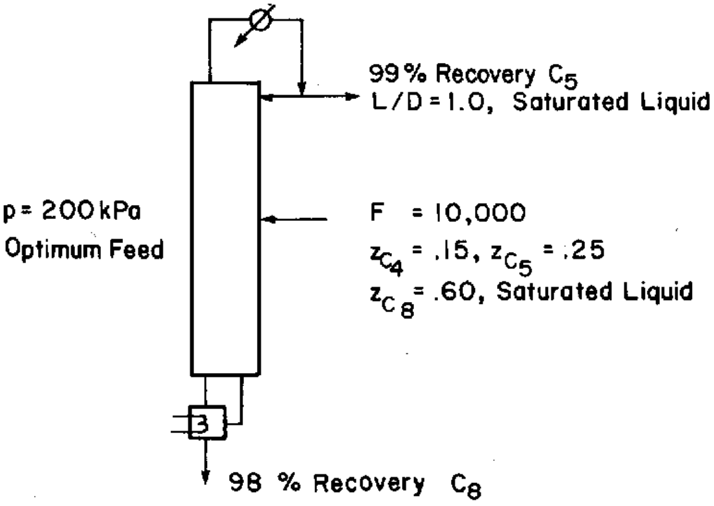
Remember that convergence is easy only when the non-keys are all heavy or are all light. If this is not true, other calculational methods that are not stage-by-stage methods should be used. An example is the matrix method discussed later.

Example 8-1. Analytical Stage-by-Stage Calculation

A distillation column with a partial reboiler and a total condenser is separating nC_4, nC_5, and nC_8. Operating conditions are: Feed: 10,000 kg moles/hr, saturated liquid feed. $z_{C4} = 0.15$, $z_{C5} = 0.25$, $z_{C8} = 0.60$ (mole fractions). Wanted: 99% recovery of nC_5 in the distillate and 98% recovery of nC_8 in the bottoms. Reflux is a saturated liquid and external reflux ratio $L_0/D = 1.0$. Pressure = 200 kPa. Find the optimum feed location and the total number of stages. Use constant molal overflow even though it is not strictly valid. Use the DePriester charts to calculate K values. Assume that all the nC_4 is in the distillate. Thus $x_{C4,bot} = 0$. If $x_{C4,bot\ calc} < 5 \times 10^{-4}$, do not repeat the calculation.

Solution

A. Define. Problem is sketched in the figure.



Find outlet concentrations, feed stage, and total number of stages.

B. Explore. Since this is a multicomponent system, the calculation must be by trial and error. We will assume that all of the LNK (C_4) is in the distillate. Then do external balances. Step off stages from top *down*. Thus, use dew-point calculation,

$$\sum_{i=1}^{3} x_i = \sum_{i=1}^{3} \frac{y_i}{K_i} = 1.0$$

and

$$K_{ref,new} = K_{ref,old} \left(\sum_{i=1}^{3} \frac{y_i}{K_i}\right)$$

to calculate temperatures on each stage. For feed stage, use approximate test that if

$$\left(\frac{y_{LK,j+1}}{y_{HK,j+1}}\right)_{top\ op\ eq} > \left(\frac{y_{LK,j+1}}{y_{HK,j+1}}\right)_{bot\ op\ eq}$$

then stage j is the optimum feed stage.

Done when

$$x_{C8,k+1} \geq x_{C8,bot} \quad \text{and} \quad x_{C5,k+1} \leq x_{C5,bot}$$

Then check for optimum feed stage. Finally, check that initial guess was correct.

C. Plan. (Preliminary calculations will be done here.)

External Balances:

Assume $x_{C5,bot} = 0$, which gives $Fz_{C4} = Dx_{C4,dist} = 1500$. From the fractional recoveries:

$$Dx_{C5,dist} = 0.99(Fz_{C5}) = 2475$$

$$Dx_{C8,dist} = (1 - 0.98)(Fz_{C6}) = 120$$

$$D = \sum_{i=1}^{3}(Dx_i) = 4095 \text{ kg moles/hr}$$

Then mole fractions are $x_{i,dist} = Dx_{i,dist}/D$

$$x_{C4,dist} = 0.366, \quad x_{C5,dist} = 0.605, \quad x_{C8,dist} = 0.029$$

Fractional recoveries for the bottoms are

$$Bx_{C5,bot} = (1 - 0.99)(Fz_{C5}) = 25$$

$$Bx_{C8,bot} = (0.98)(Fz_{C8}) = 5880$$

$$B = \Sigma(Bx_{i,bot}) = 5905 \text{ kg moles/hr}$$

Mole fractions: $x_{C4,bot} = 0, \quad x_{C5,bot} = 0.004, \quad x_{C8,bot} = 0.996$

At top of column, $y_{i,1} = x_{i,dist} = x_{i,0}$

Equilibrium Calculation:

Pick T_1 for which $K_{C5} = 1.0$. Calc $K_{C5,new} = (K_{C5})(\sum \frac{y_{i,1}}{K_{i,1}})$. Find T_1 from $K_{C5\,New}$. Repeat to convergence. With T_1 determined, $x_{i,1} = K_i(T_1)y_{i,1}$.

Top Operating Equation:

$$y_{i,j+1} = \frac{L}{V} x_{i,j} + (1 - \frac{L}{V})x_{i,dist}$$

where $\frac{L}{V} = \frac{L/D}{1 + L/D} = \frac{1}{2}$ since reflux is a saturated liquid.

Calculate all $y_{i,2}$. Then calculate T_2 and $x_{i,2}$ from equilibrium. Calculate $y_{i,3}$ with top operating line. Also use bottom operating line,

$$y_{i,j+1} = \frac{\bar{L}}{\bar{V}} x_{i,j} - (\frac{\bar{L}}{\bar{V}} - 1)x_{i,bot}$$

where $\bar{L} = L + F = (\frac{L}{D})D + F = 14{,}095$

since feed is a saturated liquid, and

$$\bar{V} = V = L + D = (\frac{L}{D} + 1)D = 8190$$

Thus $\bar{L}/\bar{V} = 1.721$.

Check whether $(\frac{y_{C5,3}}{y_{C8,3}})_{\text{top op}} > (\frac{y_{C5,3}}{y_{C8,3}})_{\text{bot op}}$. If not, use rectifying operating equation values and do equilibrium calculation for stage 3. If test is satisfied, use bottom operating equation values (stage 2 is feed), and go to equilibrium for stage 3.

Repeat for each stage. Once in bottom of column, use bottom operating equation only.

Stop when $x_{C8,j+1} \geq 0.996$ and $x_{C5,j+1} \leq 0.004$. Finally, check initial assumption.

D. Do It. *Equilibrium Stage 1.* Pick $K_{C5} = 1.0$. Then $T_1 = 58.2\,^\circ C$ from DePriester chart. $K_{C5} = 2.87$, $K_{C8} = 0.065$, and

$$K_{C5,\text{new}} = 1.0(\frac{0.366}{2.87} + \frac{0.605}{1.0} + \frac{0.029}{0.065}) = 1.18$$

which gives $T_{1,\text{new}} = 65\,^\circ C$ and $K_{C4} = 3.25$, $K_{C8} = 0.082$. Continuing,

$$K_{C5,\text{new}} = 1.18(\frac{0.366}{3.25} + \frac{0.605}{1.18} + \frac{0.029}{0.082}) = 1.155$$

or $T_{1,\text{new}} = 64.3$ and $K_{C4} = 3.22$, $K_{C8} = 0.081$.

Check: $\sum(\frac{y_i}{K_i}) = 0.114 + 0.524 + 0.358 = 0.995$ √

Top operating equation is

$$y_{i,2} = \frac{L}{V} x_{i,1} + (1 - \frac{L}{V})x_{i,\text{dist}}$$

Thus, for C_4, $y_{C4,2} = (1/2)(0.114) + (1/2)(0.366) = 0.24$ and $y_{C5,2} = 0.564$, $y_{C8,2} = 0.1935$.

Note that $\sum_{i=1}^{3} y_{i,2} = 0.998$ which is close enough to 1.0. A check on the bottom operating equation is not required, since $y_{C8,\text{bot op}} < 0$.

Stage 2. $T_2 > 64.3\,^\circ C$ since we are going down the column. Dew-point calculation is similar to first stage calculation. The result is $T = 99\,^\circ C$, and the following x values are obtained:

$$x_{C4,2} = 0.039,\ \ x_{CB,2} = 0.225,\ \ x_{C8,2} = 0.744,\ \ \sum x_i = 1.008$$

Now we can use both operating equations to check for feed stage.

Top op eq: $$y_{i,3} = \frac{L}{V}\, x_{i,2} + \left(1 - \frac{L}{V}\right) x_{i,\text{dist}}$$

$$y_{C4,3} = 0.2025,\quad y_{C5,3} = 0.415,\quad y_{C8,3} = 0.3865,\quad \sum y_i = 1.004$$

Bottom op eq: $$y_{i,3} = \frac{\overline{L}}{\overline{V}}\, x_{i,2} - \left(\frac{\overline{L}}{\overline{V}} - 1\right) x_{i,\text{bot}}$$

$$y_{C4,3} = 0.067,\ \ y_{C5,3} = 0.384,\ \ y_{C8,3} = 0.562,\ \sum y_{i,3} = 1.01$$

Then: $$\left(\frac{y_{C5,3}}{y_{C8,3}}\right)_{\text{top op}} = 1.07 > \left(\frac{y_{C5,3}}{y_{C8,3}}\right)_{\text{bot op}} = 0.683$$

Use stage 2 as the feed. Then for the equilibrium calculation for stage 3, use $y_{i,3}$ values calculated with the bottom operating line.

Stage 3. Equilibrium Calculation. As first guess, try $134\,^\circ C$ (pick this by approximately balancing C_5 and C_8 to get $\sum x_i = 1.0$). Final result is $T_3 = 132\,^\circ C$ and

$$x_{C4,3} = 0.0066,\quad x_{C5,3} = 0.0853,\quad x_{C8,3} = 0.906,\quad \sum x_i = 0.998$$

Now use the bottom operating equation because we are in the bottom of the column.

$$y_{i,4} = \frac{\overline{L}}{\overline{V}}\, x_{i,3} - \left(\frac{\overline{L}}{\overline{V}} - 1\right) x_{i,\text{bot}}$$

The results are: $y_{C4,4} = 0.0114$, $y_{C5,4} = 0.1439$, $y_{C8,4} = 0.8410$, $\sum y_i = 0.9963$

Stage 4. Equilibrium result is $T_4 = 146\,^\circ C$ and

$x_{C4} = 0.00088, \quad x_{C5} = 0.0257, \quad x_{C8} = 0.967, \quad \sum x_i = .993$

The bottom operating equation gives

$y_{C4,5} = 0.0015, \quad y_{C5,5} = 0.0413, \quad y_{C8,5} = 0.9461$

Stage 5. Equilibrium result is $T_5 = 149\,^\circ C$ and

$x_{C4,5} = 0.00115, \quad x_{C5,5} = 0.007, \quad x_{C8,5} = 0.9959, \quad \sum x_i = 1.003$

This is close to the specified condition.

The bottom operating equation gives

$y_{C4,6} = 0.000198, \quad y_{C5,6} = 0.00916, \quad y_{C8,6} = 0.9958$

Stage 6. Equilibrium calculation gives $T = 153^\circ$ and

$x_{C4} = 0.0000147, \quad x_{C5} = 0.0015, \quad x_{C8} = 0.9997, \quad \sum x_i = 1.001$

which is more separation than was required. Because of the accuracy of the DePriester charts this last number, x_{C8}, is not extremely accurate. Thus, for very tight separations you need *good* equilibrium data.

However, 6 stages (including partial reboiler) are sufficient. You could recheck the feed stage, but this is probably okay. The ratios are quite different, and stage 1 is not a possible feed stage ($y_{HK,bot\ op} < 0$). The initial assumption looks reasonable, since $x_{C4,6}$ is quite small.

E. Check. As a check we can do the overall mass balances for the column using the observed $x_{LNK,bot}$ value. With 6 equilibrium contacts, we have $x_{C4,bot} = 0.0000147$. We also want 99% recovery of C5 in the distillate and 98% recovery of C8 in the bottoms. From the fractional recoveries,

$$Dx_{C5,dist} = (0.99)\,Fz_{C5} = 2475$$

$$Dx_{C8,dist} = (1 - 0.98)Fz_{C8} = 120$$

$$Bx_{C5,bot} = (1 - 0.99)Fz_{C5} = 25$$

$$Bx_{C8,bot} = (0.98)\,Fz_{C8} = 5880$$

In the bottoms, $\sum x_{i,bot} = 1.0$, which is

$$25/B + 5880/B + 0.0000147 = 1.0.$$

Solving for B, we find $B = 5905/0.9999853 = 5905.868$. From the external mass balance,

$$D = 10{,}000 - 5905.868 = 4094.132 \text{ (not much change)}$$

Then,

$$x_{C5,dist} = \frac{Dx_{C5,dist}}{D} = \frac{2475}{4094.13} = 0.6045$$

$$x_{C8,dist} = \frac{120}{4094.132} = 0.02931$$

$$x_{C4,dist} = 1 - x_{C5,dist} - x_{C5,dist} = 0.3662$$

Since $x_{C4,dist\ calc}$ is very close to $x_{C4,dist\ assumed}$ (difference is 0.0002), another trial is not warranted.

F. Generalize. When a high fractional recovery of light key is required, the guess that all light non-key is in the distillate will be a good guess. Likewise, if a heavy non-key is present, guessing that all the heavy non-key is in the bottoms is a good guess if a high fractional recovery of the heavy key is specified.

This was a relatively easy separation because the volatilities are quite different. If we pick pentane as the reference, we can calculate the relative volatilities of $\alpha_{C8\text{-}C5}$ for several stages, since the temperature and K values have been calculated. Thus we have

$$(\alpha_{C8\text{-}C5})_{stage\ 1} = \left(\frac{K_{C8}}{K_{C5}}\right) = \frac{0.081}{1.155} = 0.070$$

$$(\alpha_{C8\text{-}C5})_{stage\ 2} = \frac{0.25}{2.51} = 0.10$$

$$(\alpha_{C8\text{-}C5})_{stage\ 6} = \frac{0.996}{6.2} = 0.16$$

Note that this relative volatility varies by more than a factor of 2. Thus constant relative volatility is not valid for this problem.

8.4. NONCONSTANT MOLAL OVERFLOW

Several methods can be used if the molal overflow is not constant. First, we could use latent heat units for the operating equations. Then when the equilibrium calculations are done, the known mole fractions ($x_{i,k}$ for bubble-point calculations) are determined from the calculated latent heat fractions $x^*_{i,k}$. The bubble-point calculation is done in mole fraction units, and the $y_{i,j}$ are determined. We convert this value to the latent heat unit fractions y^*_{ik} and then use the operating equations to determine $y^*_{i,k+1}$. We repeat this procedure up the column. Although two additional calculations are required on each stage, only the bubble-point calculations on each stage are trial-and-error. Programming this method for a computer is straightforward.

If the relative volatilities are constant, the calculation is even simpler, since relative volatilities are the same in molal and latent heat units ($\alpha^*_{i\text{-ref}} = \alpha_{i\text{-ref}}$). Thus if we change all mole fractions and flow rates to latent heat units, the entire analysis can be done in latent heat units using $\alpha^*_{i\text{-ref}} = \alpha_{i\text{-ref}}$.

Essentially, the latent heat unit approach includes an abbreviated energy balance that contains only the latent heat of vaporization. A second approach is to include the complete energy balance on every stage as we do the stage-by-stage analysis. At first this sounds appealing, but upon closer examination it is less so. Consider the case where we are stepping off stages from the bottom up. To use an energy balance around, say, stage k, we need the temperature of stage k plus the temperature of the stage above, stage k+1. We would also need the liquid mole fractions, $x_{i,k+1}$, of stage k+1. This is easily seen from the stripping section energy balance using the balance envelope shown in Figure 8-5.

$$\overline{V}H_k + Bh_B = \overline{L}_{k+1} h_{k+1} + Q_R$$

where liquid enthalpy h_{k+1} depends on T_{k+1} and $x_{i,k+1}$. To know $x_{i,k+1}$ and T_{k+1} we must simultaneously solve the equilibrium equations and enthalpy functions on stage k+1 with the mass and energy balances around stage k. This can be done, but I'd prefer not to. And I'm sure you'd prefer not to also. This procedure greatly complicates the trial-and-error calculation on each stage. One of the alternative calculation methods is normally preferred.