SEPARATION OPERATIONS 1 (0905451)

04 – MULTISTAGE CASCADES AND HYBRID SYSTEMS

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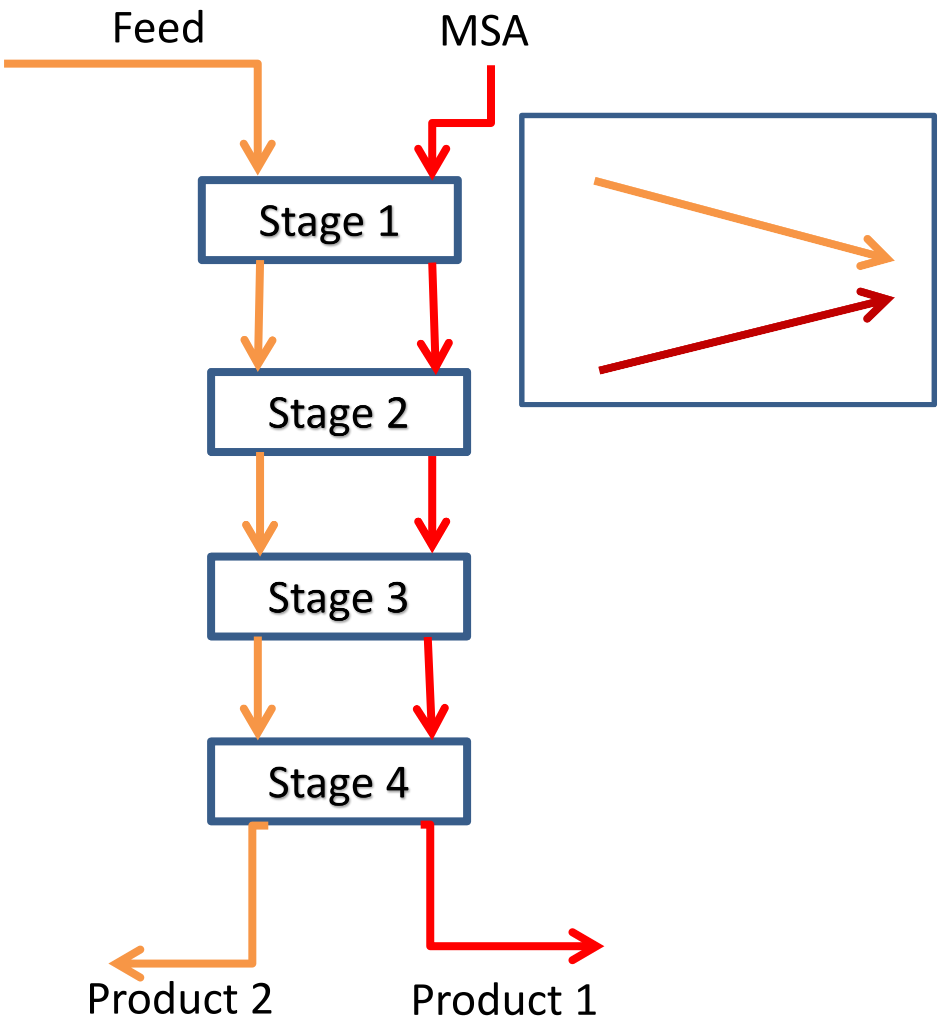
Outline

- Cascade Configurations
- Single-Section and Two-Section Cascades
- Single-Section Liquid-Liquid Extraction Cascades
- Two-Section Distillation Cascades
- Distillation Column Tips

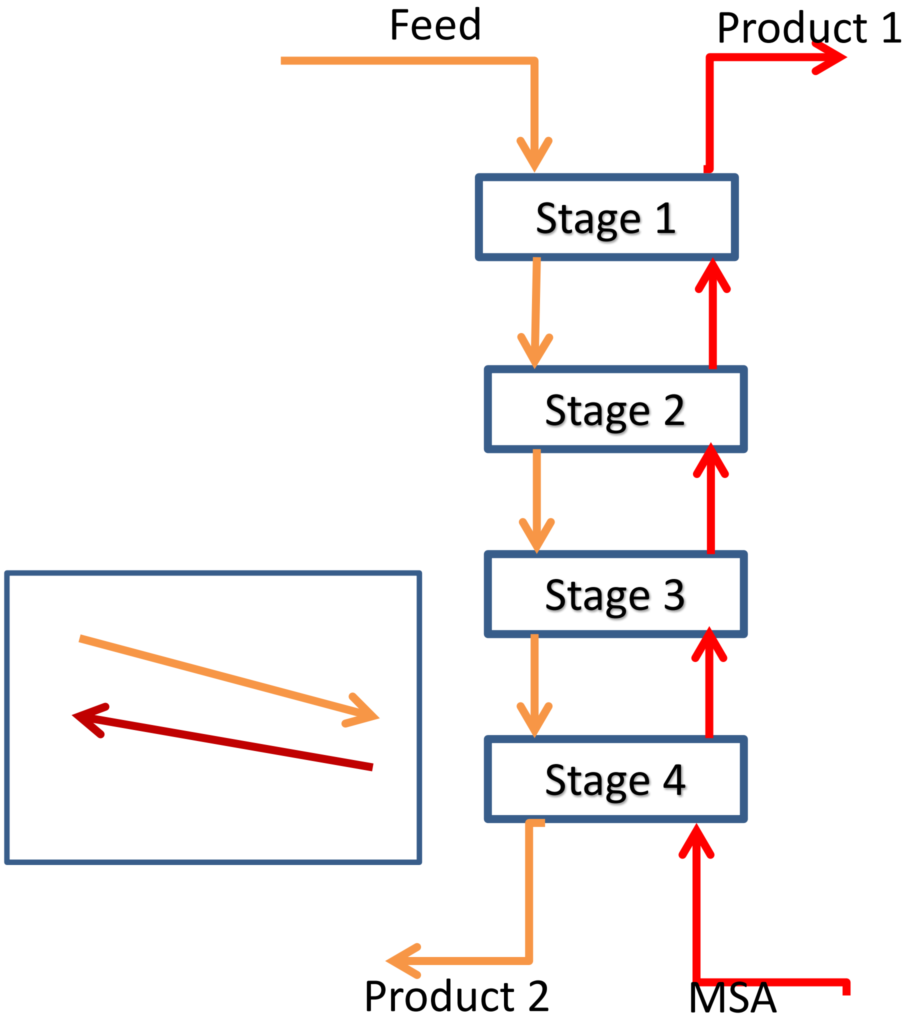
Cascade Configurations

- Separation of a mixture by a single equilibrium stage is not sufficient to achieve a desired **purity** and **recovery** of a particular component in the feed.
- Hence, multiple equilibrium stages are required.
- Common configurations of multiple stages in separation cascades
  - Cocurrent
  - Crosscurrent
  - Countercurrent
- Hybrid separation systems containing two or more different types of separation operations can sometimes save energy.

Cocurrent and Countercurrent Cascade



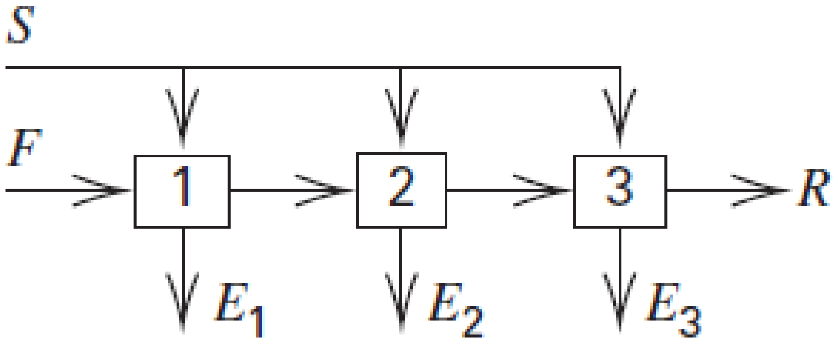
Feed
MSA
Stage 1
Stage 2
Stage 3
Stage 4
Product 2
Product 1



Feed
Product 1
Stage 1
Stage 2
Stage 3
Stage 4
Product 2
MSA

Crosscurrent Cascades

- Not as efficient as the countercurrent cascade.
- Suited for batch processing, particularly liquid–liquid extraction.
- The MSA, S, is divided into fractions that are fed individually to each stage of the process.



Single-Section and Two-Section Cascades

- single-section cascades configured and designed to achieve a desired percent recovery of just one component, called the key component, from a feed stream that enters at one end and leaves at the other.
- When it is desired to achieve a percent purity or percent recovery of two feed components, called key components, a two-section cascade is necessary.

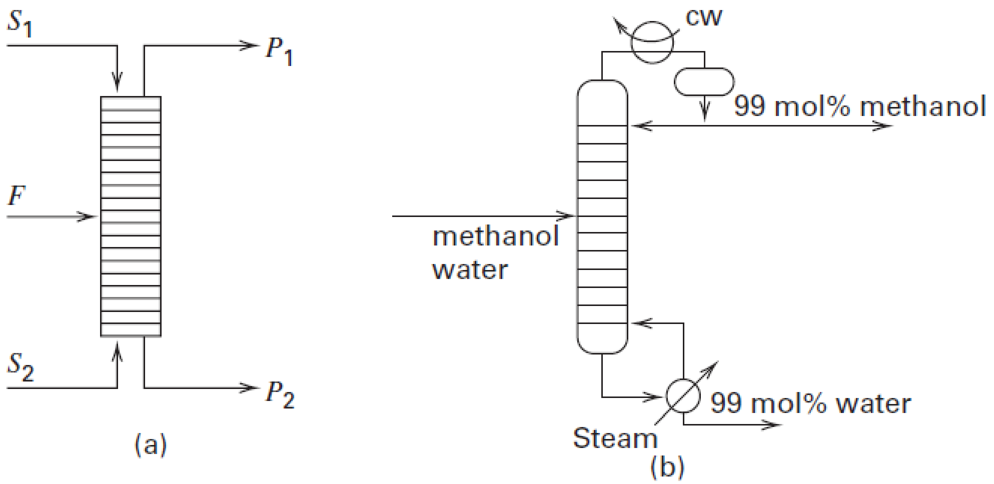


Figure 5.3 Two-section cascades: (a) liquid–liquid extraction with two solvents; (b) distillation.

Single-Section Liquid-Liquid Extraction Cascades

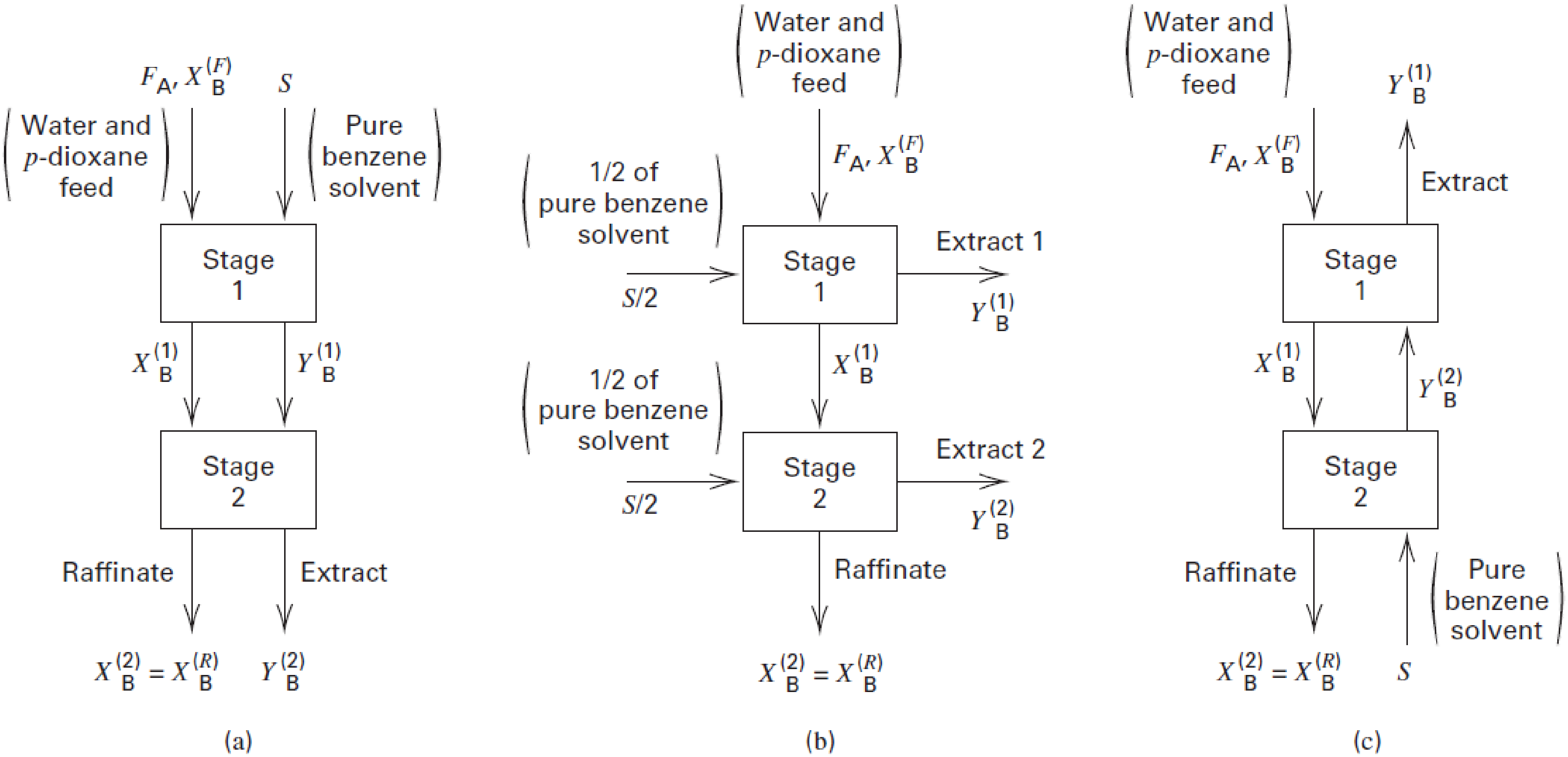


Figure 5.4 Two-stage arrangements: (a) cocurrent cascade; (b) crosscurrent cascade; and (c) countercurrent cascade.

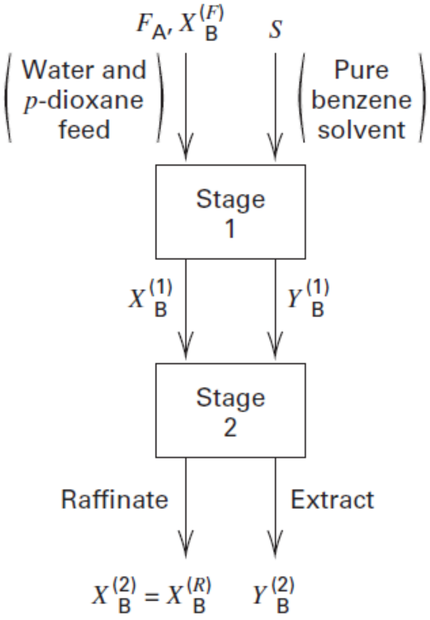
Single-Section Liquid-Liquid Extraction Cascades: Cocurrent Cascade

- Cocurrent cascade has no merit unless required residence times are so long that equilibrium is not achieved in a single stage and one or more extra stages are needed to provide additional residence time.
- Long residence times may be needed to achieve sufficient contacting between feed and solvent, or to accommodate slow mass transfer of solute from feed to solvent.
- Regardless of the number of cocurrent equilibrium stages, N, the fraction of unextracted B in the carrier exiting the terminal stage is

$$\text{Fraction of B unextracted} = \frac{X_B^{(N)}}{X_B^{(F)}} = \frac{1}{1+\epsilon}$$

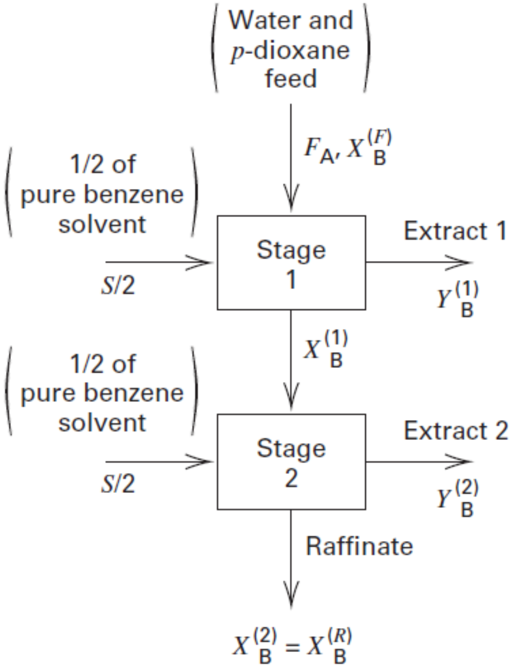
$$\text{Fraction of B unextracted} = \frac{X_B^{(1)}}{X_B^{(F)}} = \frac{1}{1+\epsilon}$$

$$\epsilon = \frac{K'_{D_B} S}{F_A} = K'_{D_B} \times \frac{S}{F_A}$$



Single-Section Liquid-Liquid Extraction Cascades: Crosscurrent Cascade

- The feed progresses through each stage, beginning with Stage 1.
- The total solvent flow rate, S, is divided into equal fractions, each of which is sent to a consecutive stage.
- S is replaced by S/N so that $\mathcal{E}$ is replaced by $\mathcal{E}/N$.
- the final raffinate solute mass ratio, corresponding to the fraction of B in the feed that is not extracted is



$$\text{Fraction of B unextracted} = \frac{X_B^{(N)}}{X_B^{(F)}} = \frac{X_B^{(R)}}{X_B^{(F)}} = \frac{1}{\left(1 + \epsilon / N\right)^N}$$

- A crosscurrent cascade decreases the value of X_B in each successive stage.
- For an infinite number of equilibrium stages, *N*:

$$\text{Fraction of B unextracted} = \frac{X_B^{(\infty)}}{X_B^{(F)}} = \frac{1}{\exp(\epsilon)}$$

- Even for an infinite number of stages, the solute concentration in the raffinate cannot be reduced to zero i.e., impossible to completely extract all solute from the feed carrier.

Single-Section Liquid-Liquid Extraction Cascades: Countercurrent Cascade

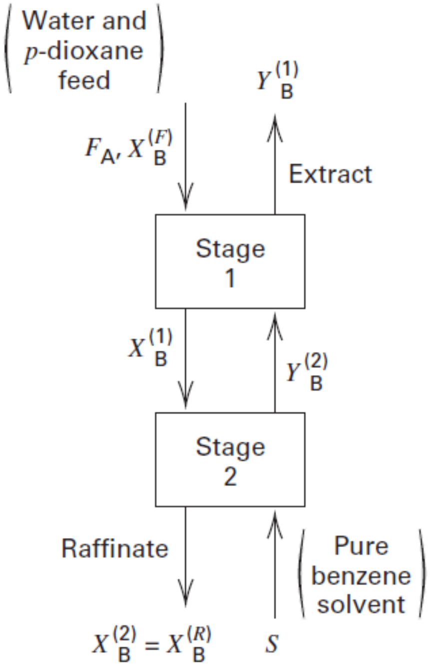
- The feed, a carrier-rich liquid phase, initially rich in solute, passes through the cascade countercurrently to the solvent-rich phase, which is initially solute-poor.
- For a two-equilibrium-stage system, the material-balance and equilibrium equations for solute B for each stage are:

$$\text{Stage 1:} \quad X_B^{(F)}F_A + Y_B^{(2)}S = X_B^{(1)}F_A + Y_B^{(1)}S$$

$$\text{Stage 2:} \quad X_B^{(1)}F_A = X_B^{(2)}F_A + Y_B^{(2)}S$$

- Combining material balances and definition of the distribution coefficients to eliminate the intermediate mass ratios:

$$\text{Fraction of B unextracted} = \frac{X_B^{(2)}}{X_B^{(F)}} = \frac{X_B^{(R)}}{X_B^{(F)}} = \frac{1}{1 + \epsilon + \epsilon^2}$$



- Generalize to an N-stage cascade:

$$\text{Fraction of B unextracted} = \frac{X_B^{(R)}}{X_B^{(F)}} = \frac{1}{\sum_{n=0}^{N} \epsilon^n} = \frac{\epsilon - 1}{\epsilon^{N+1} - 1}$$

- Can we extract 100% of solute? For an infinite number of stages:

$$\frac{X_B^{(\infty)}}{X_B^{(F)}} = \begin{cases} 0, & 1 \leq \epsilon \leq \infty \\ 1 - \epsilon, & \epsilon \leq 1 \end{cases}$$

- complete extraction can be achieved with a countercurrent cascade of an infinite *N* if the extraction factor $\mathcal{E} > 1$.
- The countercurrent arrangement is preferred for a continuous process because:
  - This arrangement results in a higher degree of extraction for a given amount of solvent and number of equilibrium stages.

EXAMPLE 5.1 Liquid–Liquid Extraction with Different Cascade Arrangements.

Ethylene glycol is catalytically dehydrated to *p*-dioxane (a cyclic diether) by the reaction $2HOCH_2CH_2HO \rightarrow H_2CCH_2OCH_2CH_2O + 2H_2O$.Water and *p*-dioxane have normal boiling points of 100°C and 101.1°C, respectively, which precludes using distillation to separate them. Liquid–liquid extraction at 25°C using benzene as a solvent is preferable. A feed of 4,536 kg/h of a 25wt% solution of *p*-dioxane in water is to be separated continuously with 6,804 kg/h of benzene.

Assume that benzene and water are mutually insoluble. Use a constant, average literature value for partitioning of p-dioxane between water and benzene. Determine the effect of the number and arrangement of stages on the percent extraction of p-dioxane. The process flow diagram is given in Figure 5.5.

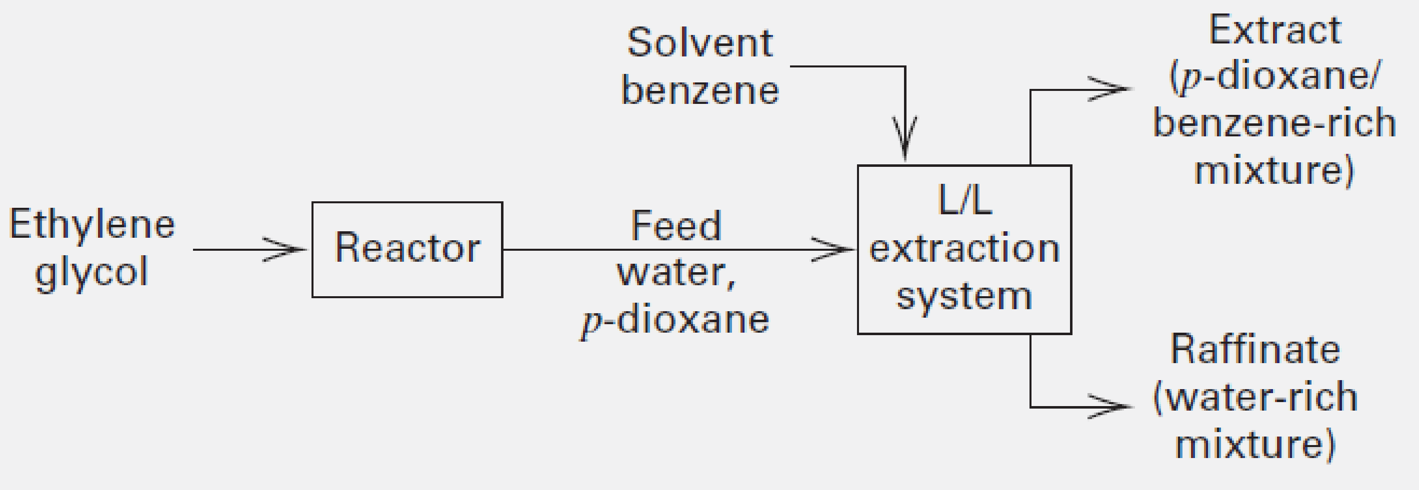


Figure 5.5 Process Flow Diagram for Example 5.1.

Solution

Three arrangements of equilibrium stages are examined: (a) cocurrent, (b) crosscurrent, and (c) countercurrent. Because water and benzene are assumed mutually insoluble, and the partitioning is assumed constant, (5-6), (5-8), and (5-15) can be used to estimate $X_{\mathrm{B}}^{(R)}/X_{\mathrm{B}}^{(F)}$, the fraction of *p*-dioxane not extracted, as a function of N. From the equilibrium data of Berdt and Lynch [1], the distribution coefficient for *p*-dioxane, varies from 1.0 to 1.4 as a function of concentration. Assume a constant value of 1.2. From the given data, $S = 6{,}804$ kg/h of benzene, $F_A = 4{,}536(0.75) = 3{,}402$ kg/h of water, and $X_{\mathrm{B}}^{(F)} = 0.25/0.75 = 1/3$. From (5-2), the extraction factor is $\mathcal{E} = 1.2(6{,}804)/3{,}402 = 2.4$.

Single equilibrium stage:

Cocurrent, crosscurrent, and countercurrent arrangements give identical results for a single stage. By (5-6), the fraction of *p*-dioxane remaining unextracted in the raffinate is,

$$X_{\mathrm{B}}^{(1)}/X_{\mathrm{B}}^{(F)} = 1/(1 + 2.4) = 0.294$$

The corresponding fractional extraction into the solvent is

$$1 - X_{\mathrm{B}}^{(R)}/X_{\mathrm{B}}^{(F)} = 1 - 0.294 = 0.706 \text{ or } 70.6\%$$

More than one equilibrium stage:

(a) Cocurrent: For any number of equilibrium stages, extraction is still only 70.6%.

(b) Crosscurrent: For any number of equilibrium stages, (5-8) applies. For two stages, assuming equal flow of solvent to each stage,

$$X_{\mathrm{B}}^{(2)}/X_{\mathrm{B}}^{(F)} = \frac{1}{(1+\mathcal{E}/2)^2} = 1/(1+2.4/2)^2 = 0.207$$

and extraction is 79.3%. Results for increasing values of N are in Figure 5.6.

(c) Countercurrent: For any number of stages, (5-15) applies. For example, for two stages,

$$X_{\mathrm{B}}^{(2)}/X_{\mathrm{B}}^{(F)} = \frac{1}{1+\mathcal{E}+\mathcal{E}^2} = \frac{1}{1+2.4+2.42^2} = 0.109$$

and extraction is 89.1%. Results for other discrete values o N are shown in Figure 5.6, where a probability-scale ordinat is convenient because for the countercurrent case with $\mathcal{E} > 1$ 100% extraction is approached as N approaches ∞. For th crosscurrent arrangement, the maximum extraction from (5.9) i 90.9%, while for five stages, the countercurrent cascade achieve 99.2% extraction.

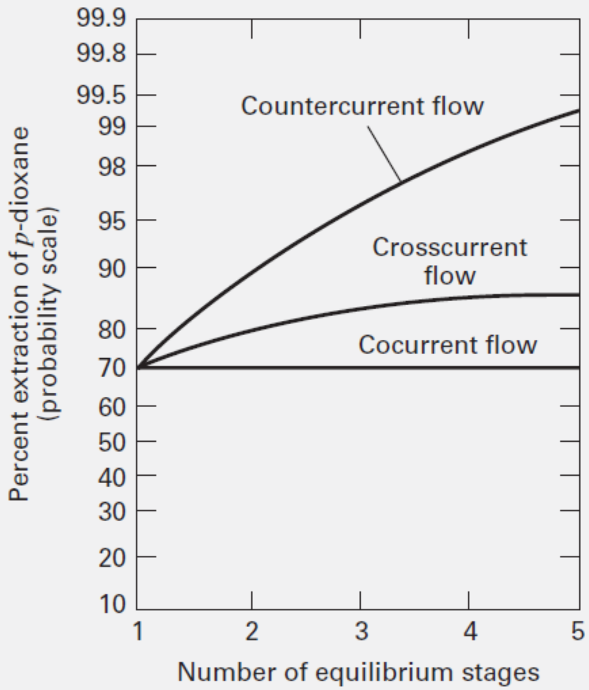
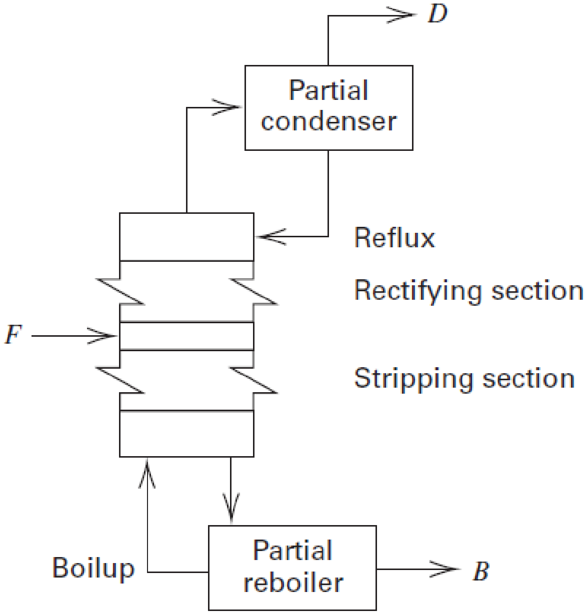
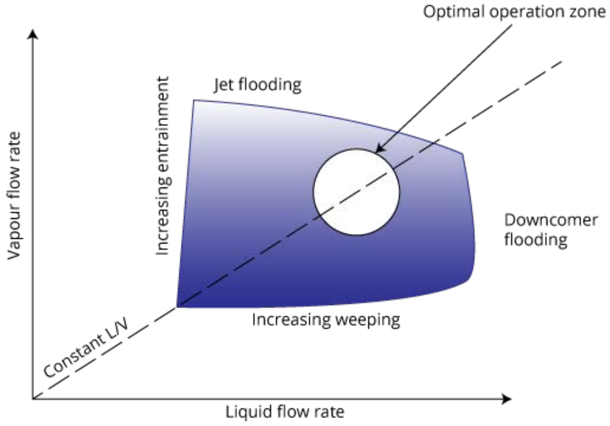


Figure 5.6 Effect of multiple equilibrium stages on extractio efficiency.

Two-Section Distillation Cascades

- If the feed to a distillation column is a binary zeotropic mixture, high purity bottoms and distillate can be achieved.
- **Distillate,** *D*, the product drawn from the top of the column.
- **Reflux**, *R*, the liquid that recirculates back to the top stage to provide contact with the vapor rising from stage-to-stage up the column.
- **Rectifying section** are the Stages above the feed entry.
- **Bottoms**, *B*, the product drawn from the bottom of the column.
- **Boilup** the vapor that flows back to and up from the bottom stage to provide vapor to contact the liquid flowing from stage-to-stage down the column.
- **stripping section** are the Stages below the feed entry.

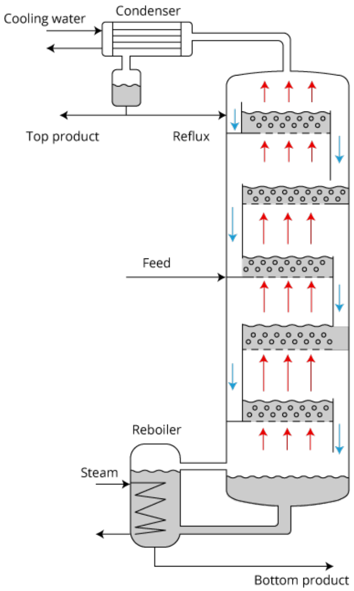




https://chemengvirtual.uwaterloo.ca/distillation-lab/components/distillation-column.html

Distillation Column Tips

- Increasing the reflux ratio decreases the number of stages required to achieve a given separation.
- More stages increases the capital cost of a column while increasing the reflux increases the energy cost because more liquid has to be vaporized in the reboiler.
- Each stage in the column is assumed to be an equilibrium stage for which the vapor exiting the stage and flowing upward to the next stage is in physical equilibrium with the liquid exiting the stage and flowing downward to the next stage below.
- Stages in the column are **adiabatic**, while the condenser and reboiler are **diabatic** stages, with heat transfer to the system in the reboiler and from the system in the condenser.
- Trays are spaced sufficiently to allow exiting vapor to disentrain from exiting liquid.
- For mixtures of close-boiling components, physical equilibrium between the vapor and liquid flows leaving a tray is closely approached. Thus, each tray may ideally correspond to one equilibrium stage. However, the efficiency of trays for wide-boiling mixtures can be significantly less than 100% and more than a single tray may be needed to achieve separation equivalent to one equilibrium stage.
- Most common tray spacing is 600 mm (24”).



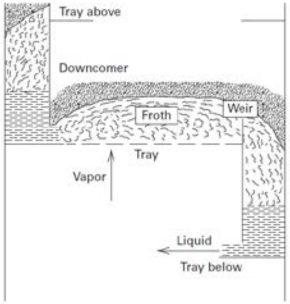
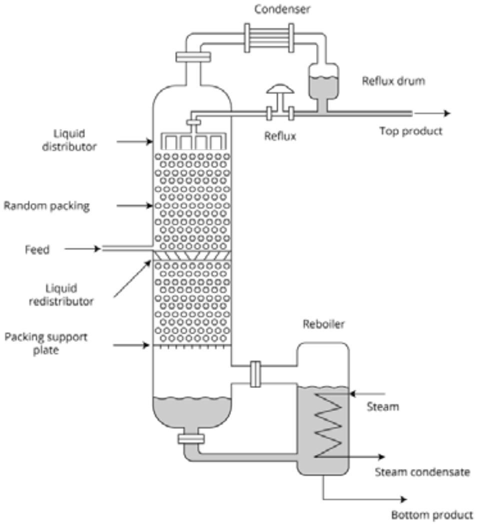


Figure 5.8 Typical vapor-liquid contacting tray.



https://epcmholdings.com/distillation-columns-plates-and-packing/