



SEPARATION OPERATIONS 1 (0905451)

01 – INTRODUCTION

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Outline

- Books

- Industrial Chemical Processes

- Basic Separation Process Techniques

 - Common Separation Operations Based on Phase Creation

 - Common Separation Operations Based on Phase Addition

 - Common Separation Operations Based on Barriers

 - Separation Operations by Applied Field or Gradient

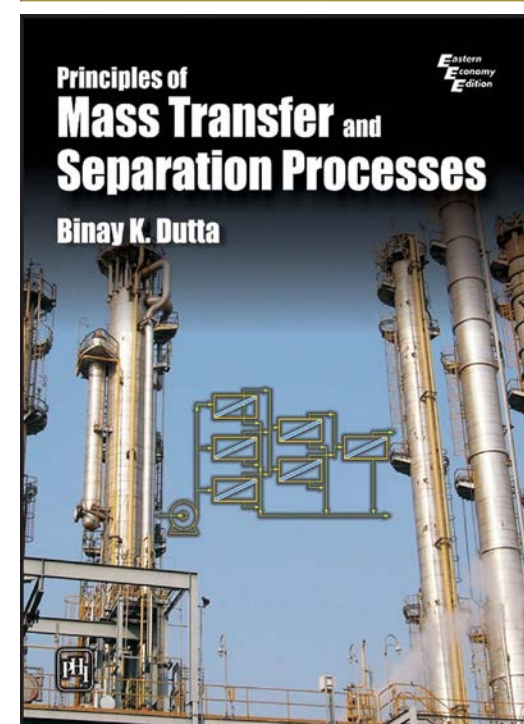
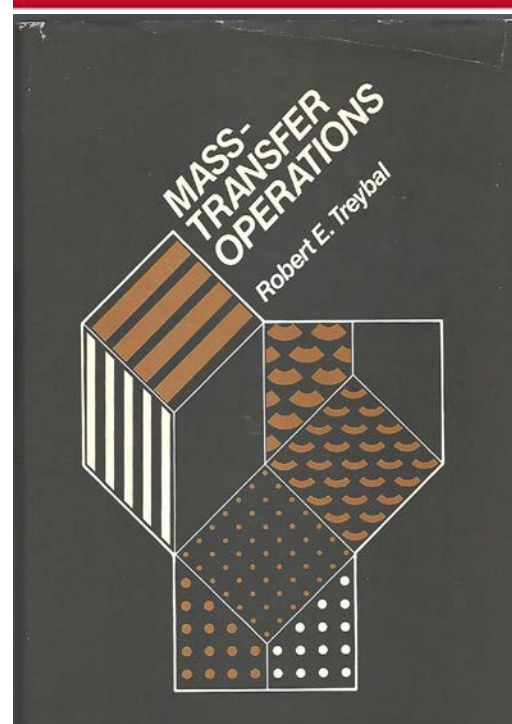
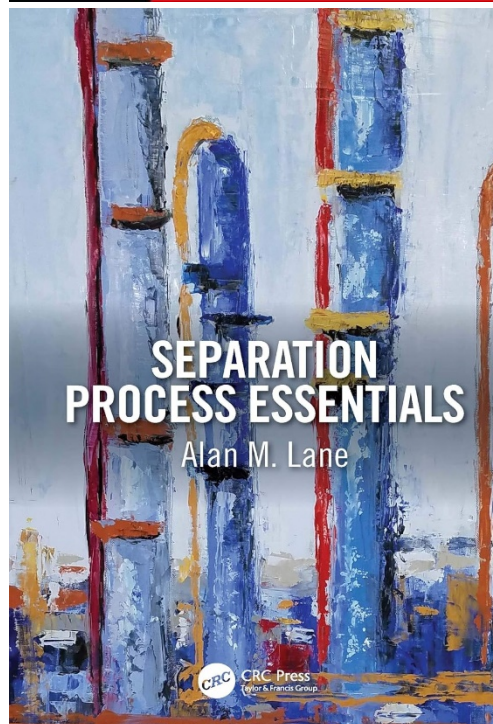
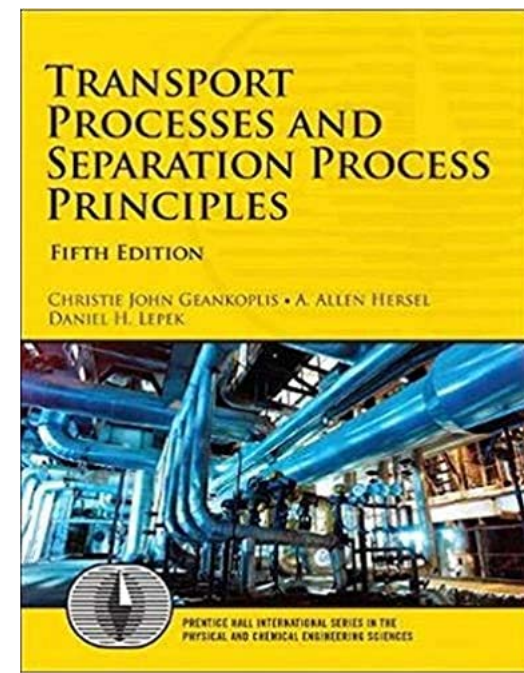
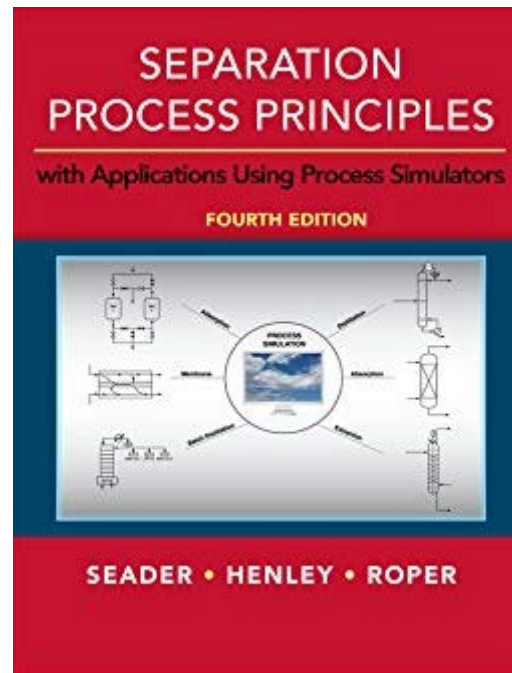
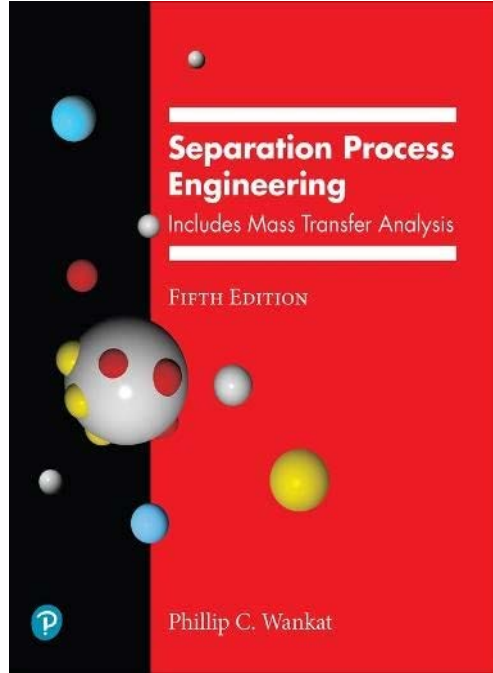
- Factors Influencing Selection of Separation Operations

- Scale-up of Common Separation Operations

- Alternative Separation Sequences

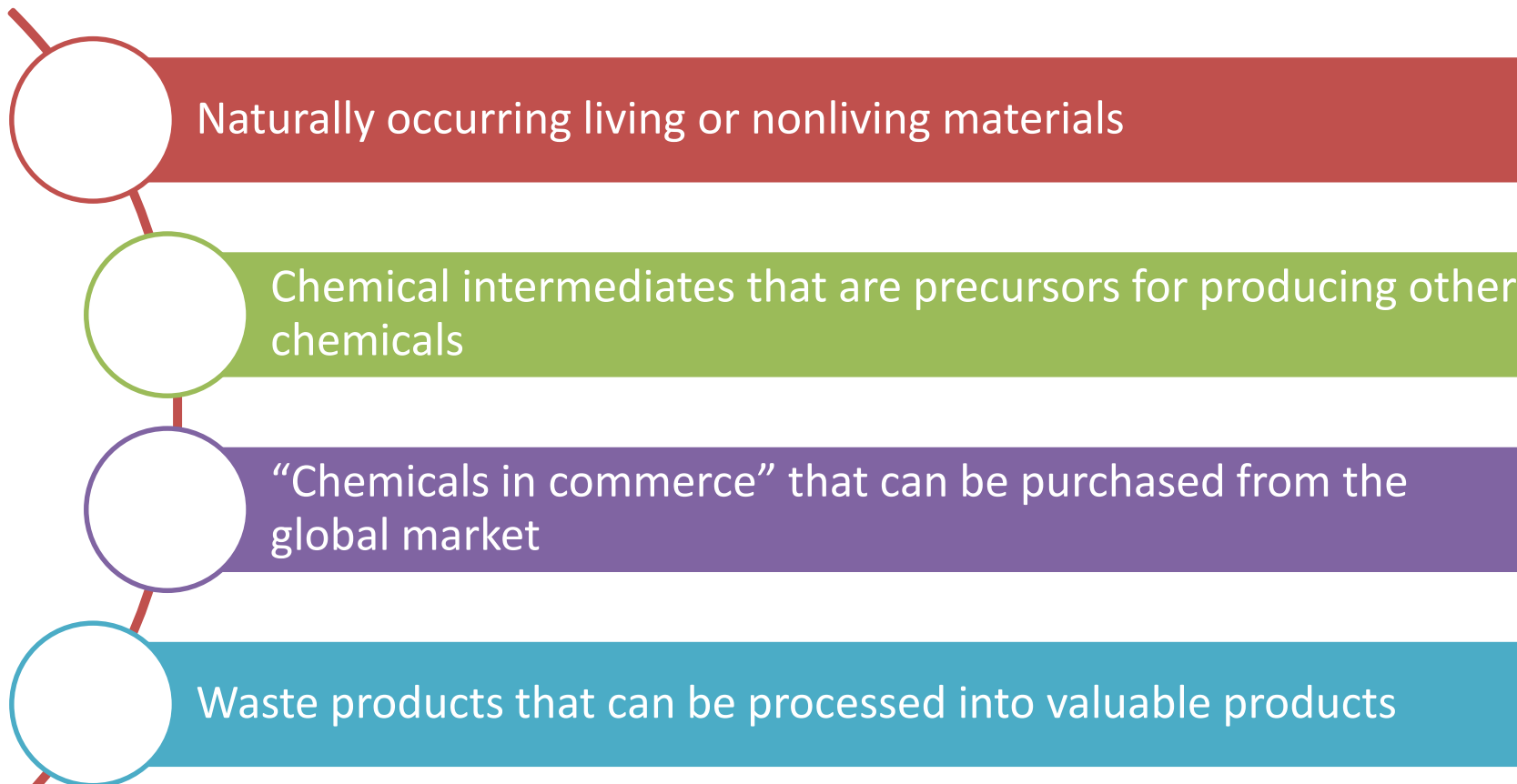
- Purity, Recovery and Units





Industrial Chemical Processes

- Chemical companies manufacture products that differ from those in the feedstocks.



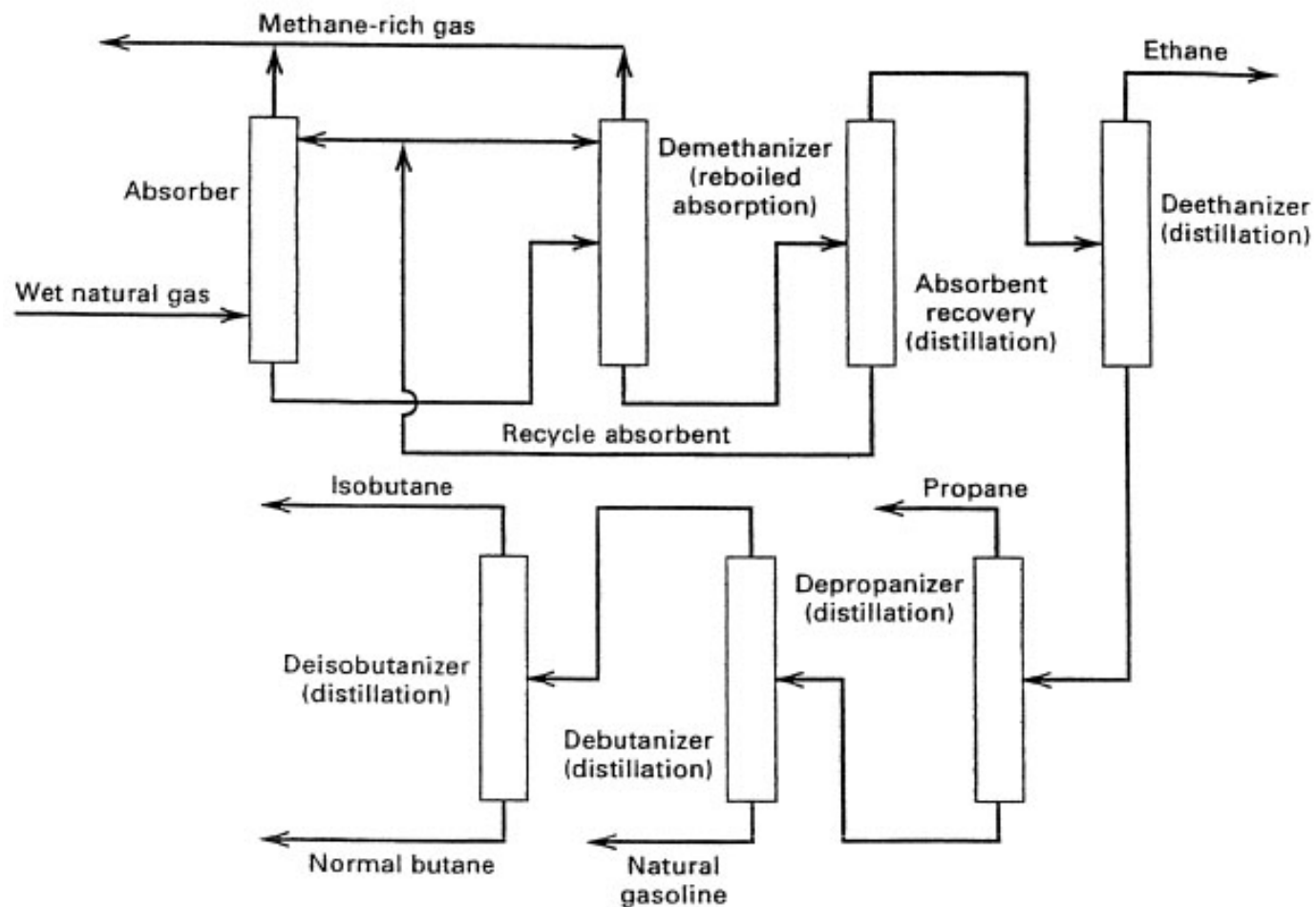


Figure 1.3 Process for recovery of light hydrocarbons from casinghead gas.

Basic Separation Process Techniques

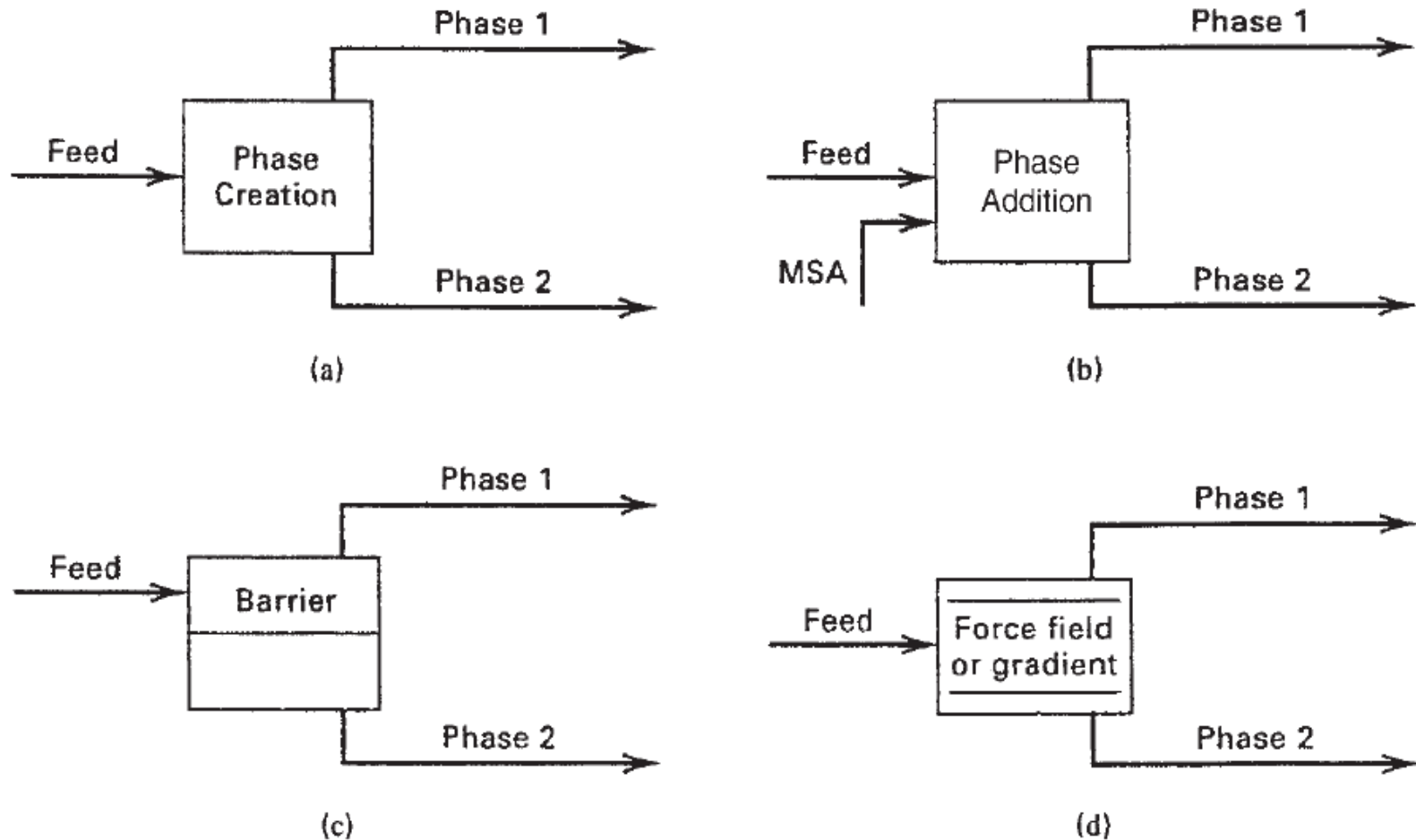
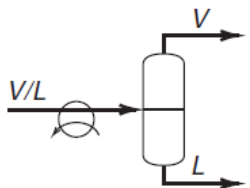
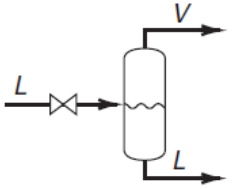
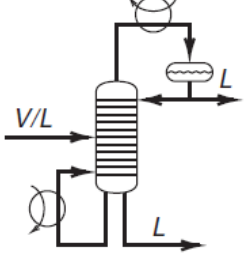


Figure 1.6 Basic separation process techniques: (a) separation by phase creation; (b) separation by phase addition; (c) separation by barrier; (d) separation by external force field or gradient.

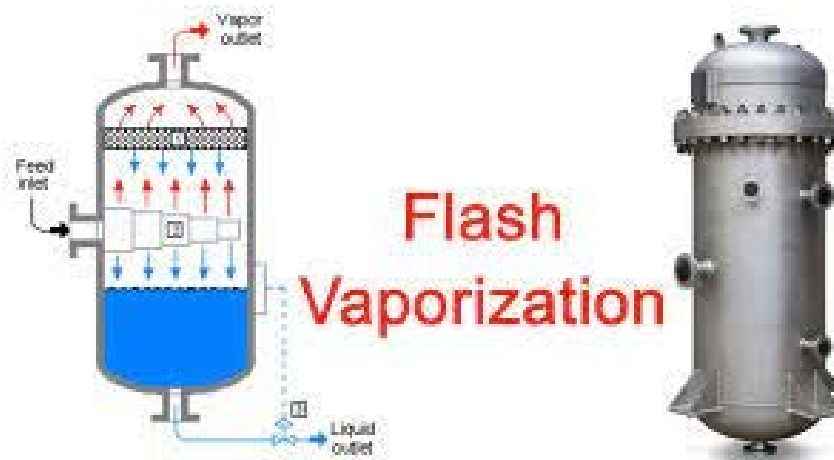
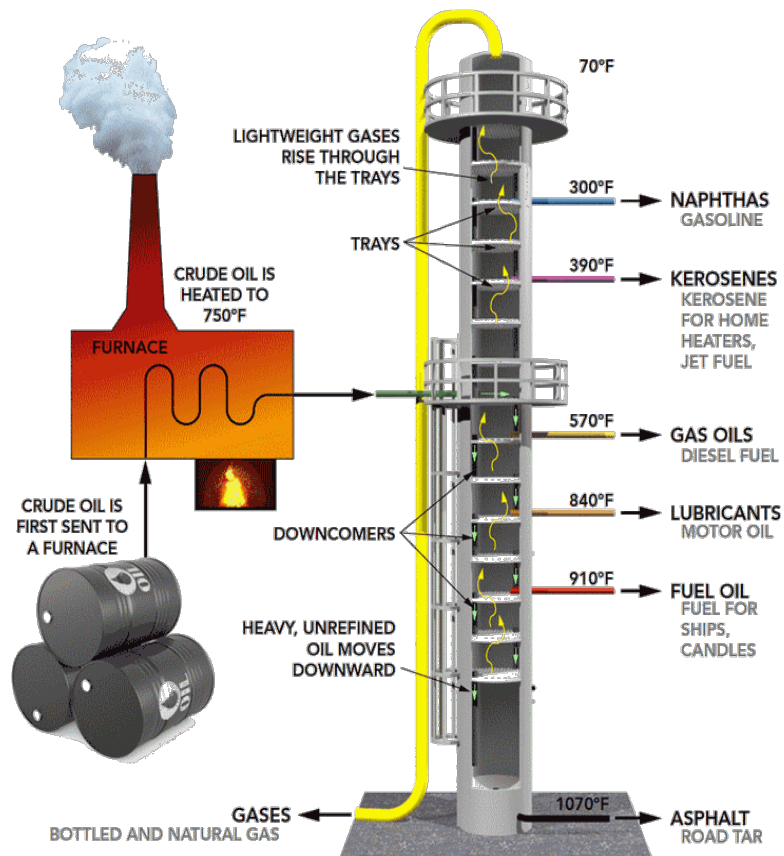


Common Separation Operations Based on Phase Creation

Table 1.1 Common Separation Operations Based on Phase Creation

Separation Operation	Symbol	Feed Phase	Created Phase	Separating Agent(s)
(1) Partial condensation or vaporization		Vapor and/or liquid	Liquid or vapor	Heat transfer (ESA)
(2) Flash vaporization		Liquid	Vapor	Pressure reduction
(3) Distillation		Vapor and/or liquid	Vapor and liquid	Heat transfer (ESA) and sometimes shaft work (ESA)





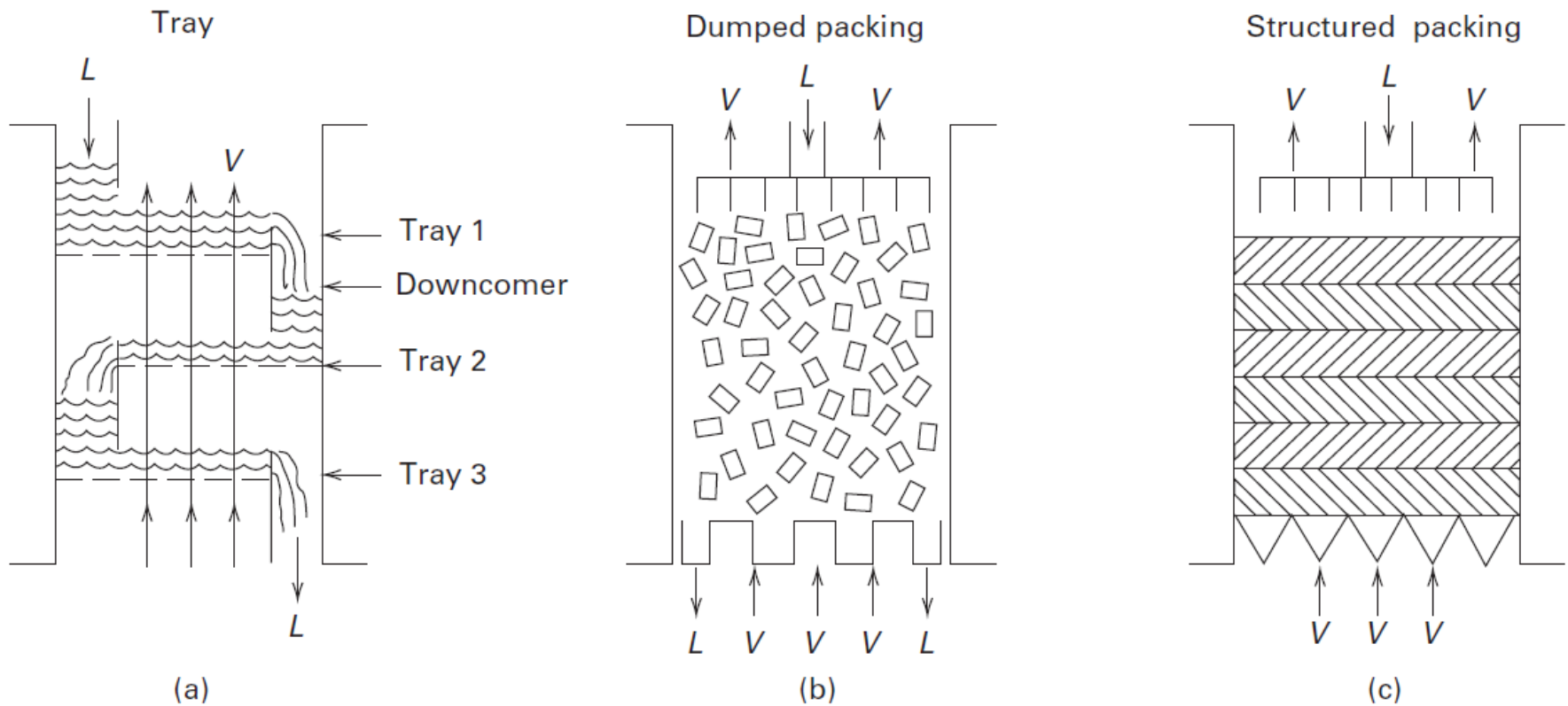
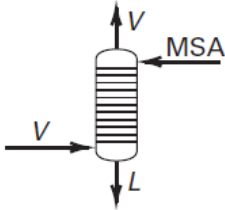
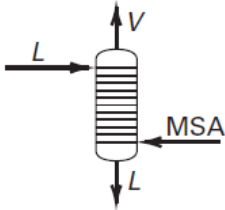
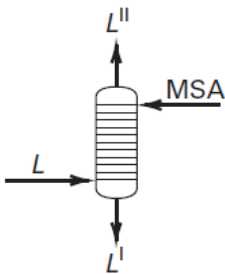
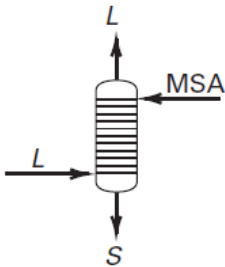


Figure 1.7 Phase-contacting methods in distillation columns.

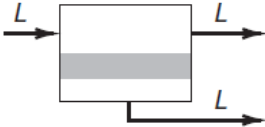
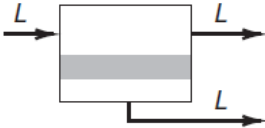

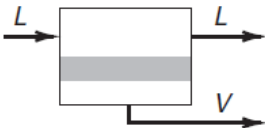
Common Separation Operations Based on Phase Addition

Table 1.2 Common Separation Operations Based on Phase Addition

Separation Operation	Symbol	Feed Phase	Added Phase	Separating Agent(s)
(1) Absorption		Vapor	Liquid	Liquid absorbent (MSA)
(2) Stripping		Liquid	Vapor	Stripping vapor (MSA)
(3) Liquid–liquid extraction		Liquid	Liquid	Liquid solvent (MSA)
(4) Adsorption		Vapor or liquid	Solid	Solid adsorbent (MSA)

Common Separation Operations Based on Barriers

Table 1.3 Common Separation Operations Based on Barriers

Separation Operation	Symbol	Feed Phase	Barrier	Separating Agent(s)
(1) Dialysis		Liquid	Microporous membrane	Pressure (ESA)
(2) Reverse osmosis		Liquid	Microporous membrane	Pressure (ESA)
(3) Gas permeation		Vapor	Nonporous membrane	Pressure (ESA)
(4) Pervaporation		Liquid	Nonporous membrane	Pressure and heat transfer (ESA)



Separation Operations by Applied Field or Gradient

Table 1.4 Separation Operations by Applied Field or Gradient

Separation Operation	Initial or Feed Phase	Force Field or Gradient	Industrial Example ^a
Centrifugation (1)	Vapor	Centrifugal force field	Separation of uranium isotopes (Vol. 23, pp. 531–532)
Thermal diffusion (2)	Vapor or liquid	Thermal gradient	Separation of chlorine isotopes (Vol. 7, p. 684)
Electrolysis (3)	Liquid	Electrical force field	Concentration of heavy water (Vol. 7, p. 550)
Electrodialysis (4)	Liquid	Electrical force field and membrane	Desalinization of sea water (Vol. 24, pp. 353–359)
Electrophoresis (5)	Liquid	Electrical force field	Recovery of hemicelluloses (Vol. 4, p. 551)
Field-flow fractionation (6)	Liquid	Laminar flow in force field	

^aCitations refer to volume and page(s) of *Kirk–Othmer Encyclopedia of Chemical Technology*, 3rd ed., John Wiley and Sons, New York (1978–1984).



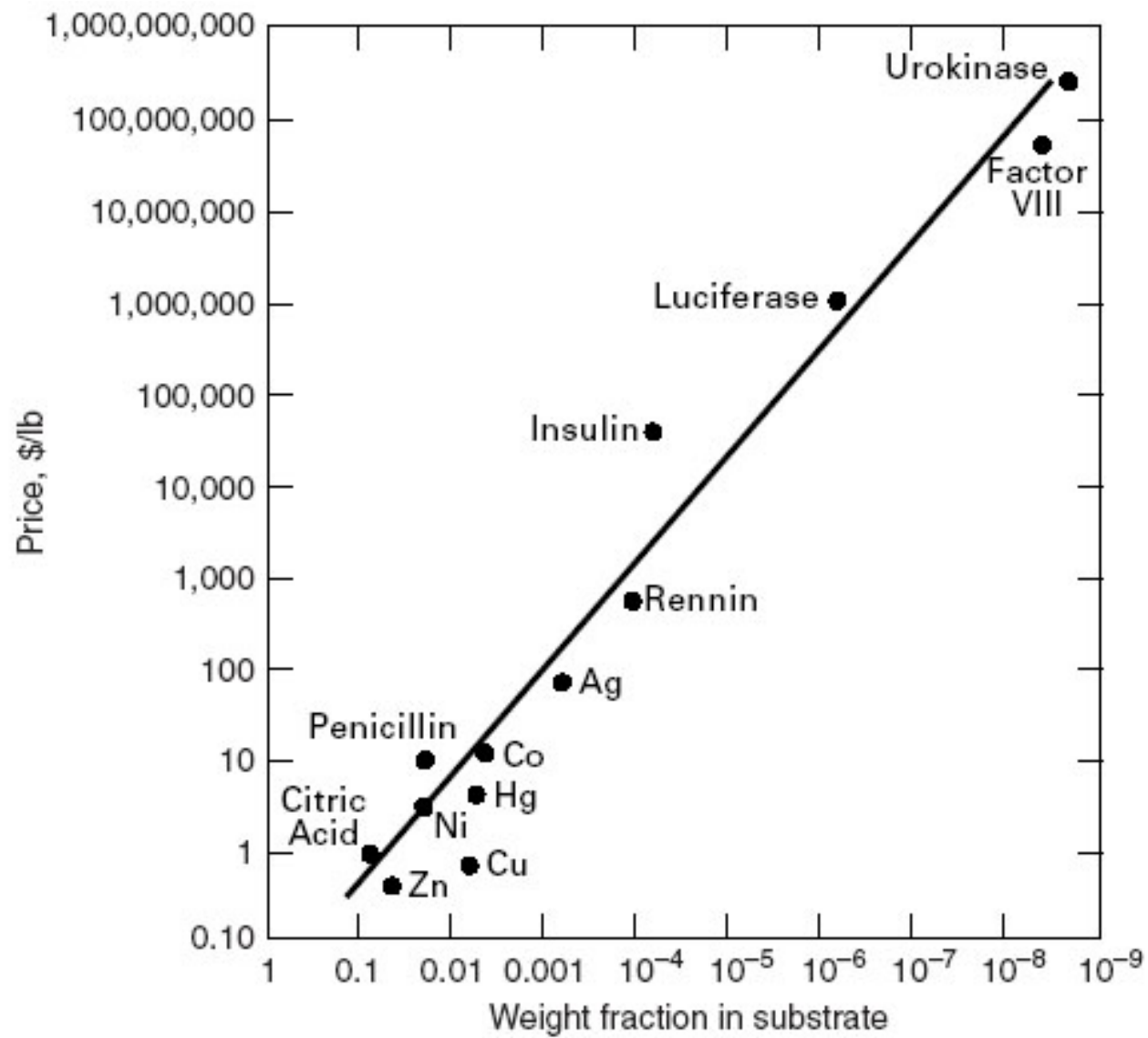


Figure 1.10 Effect of concentration of product in feed material on price [9].

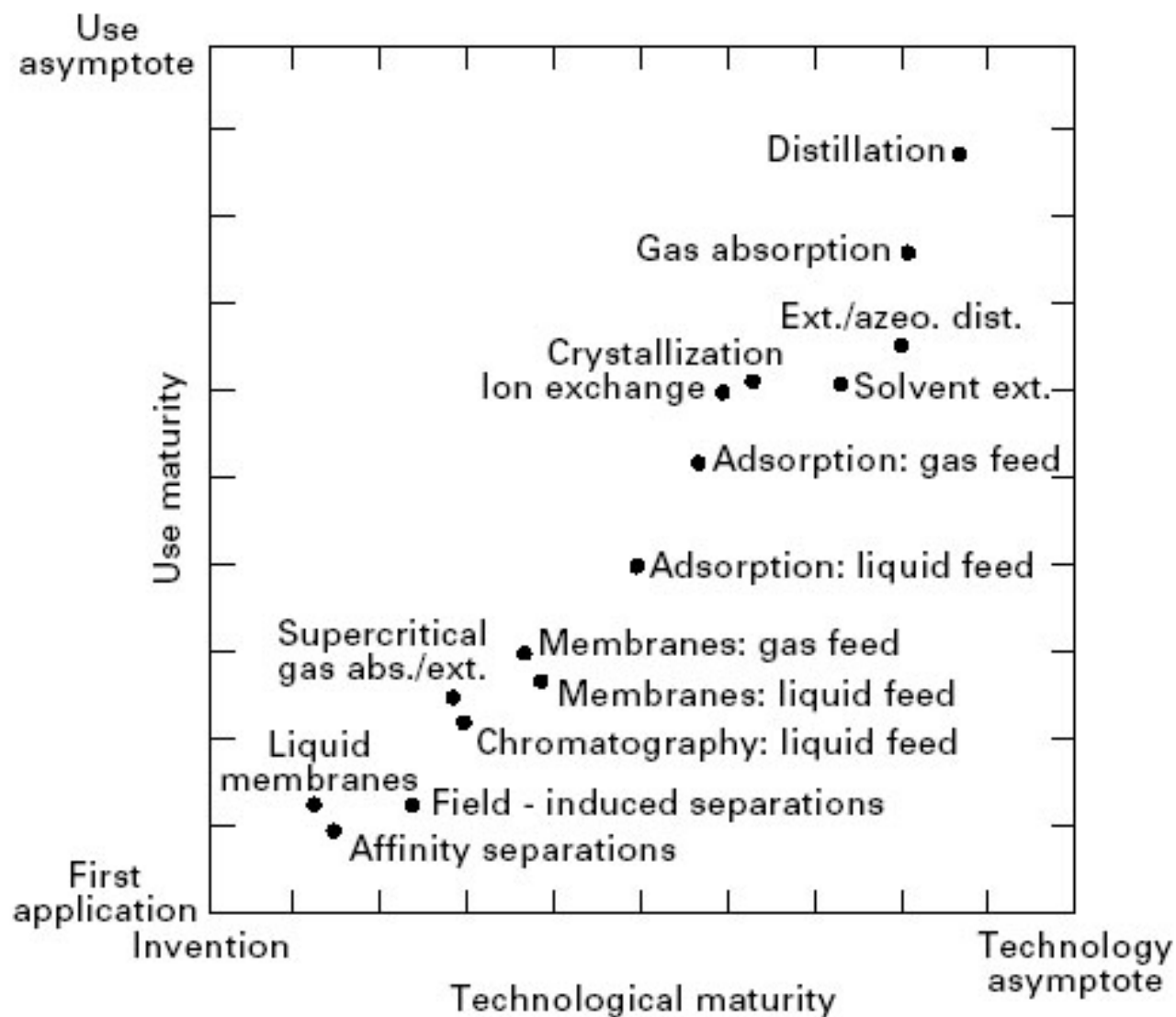


Figure 1.11 Technological and use maturities of separation processes [9].

Factors Influencing Selection of Separation Operations

Table 1.9 Factors That Influence the Selection of Feasible Separation Operations

A. Feed conditions

1. Composition, particularly concentration of species to be recovered or separated
2. Flow rate
3. Temperature
4. Pressure
5. Phase state (solid, liquid, and/or gas)

B. Product conditions

1. Required purities
2. Temperatures
3. Pressures
4. Phase states

C. Property differences that may be exploited

1. Molecular
2. Thermodynamic
3. Transport

D. Characteristics of separation operation

1. Ease of scale-up
 2. Ease of staging
 3. Temperature, pressure, and phase-state requirements
 4. Physical size limitations
 5. Energy requirements
-



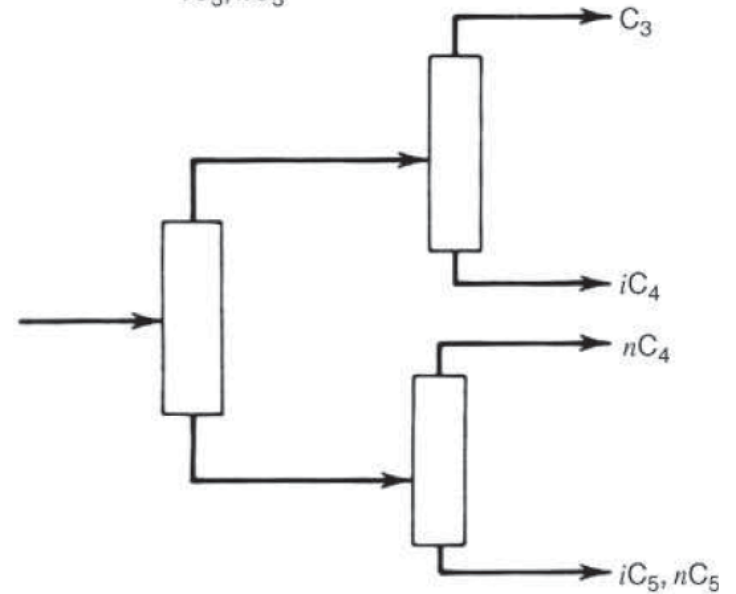
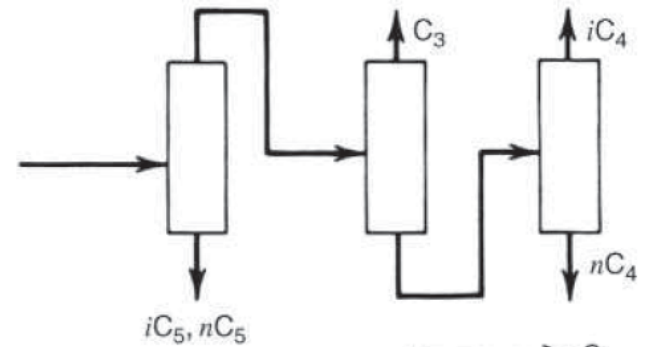
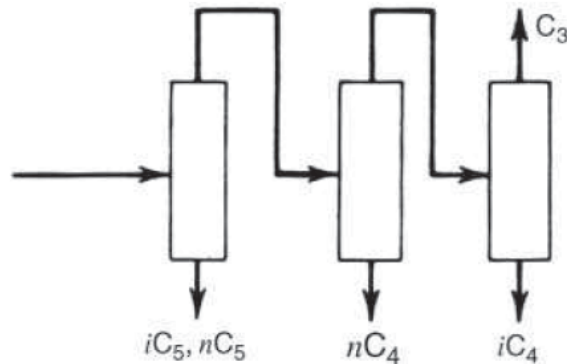
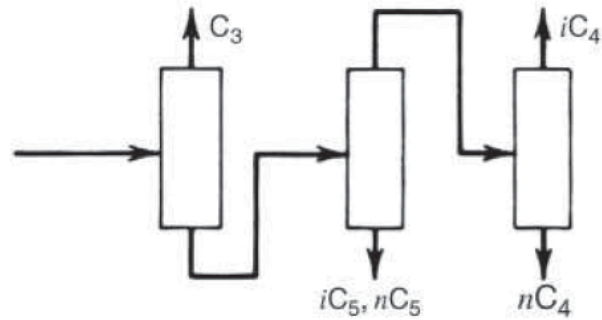
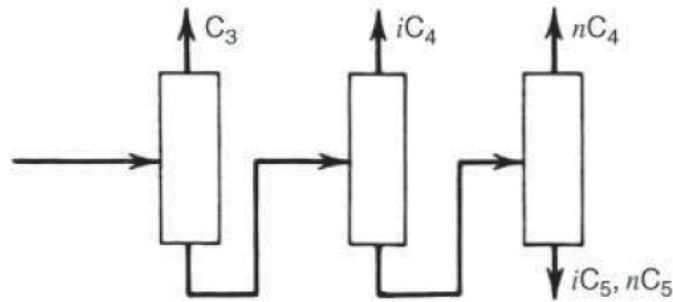
Scale-up of Common Separation Operations

Table 1.10 Ease of Scale-up of the Most Common Separation Operations

Operation in Decreasing Ease of Scale-up	Ease of Staging	Need for Parallel Units
Distillation	Easy	No need
Absorption	Easy	No need
Extractive and azeotropic distillation	Easy	No need
Liquid–liquid extraction	Easy	Sometimes
Membranes	Repressurization required between stages	Almost always
Adsorption	Easy	Only for regeneration cycle
Crystallization	Not easy	Sometimes
Drying	Not convenient	Sometimes



Alternative Separation Sequences



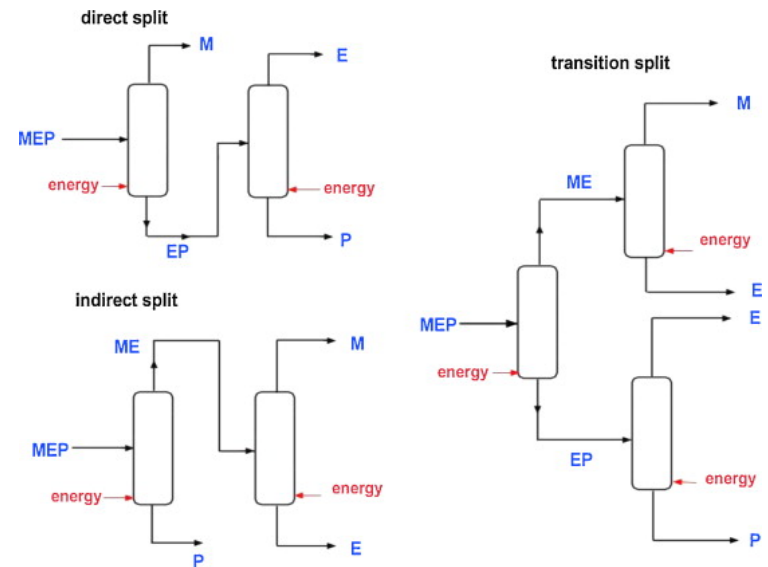
Counting of Alternative Separation Sequences

- An equation for the number of different sequences of ordinary distillation columns, N_s , to produce a number of products, P is:

$$N_s = \frac{[2(P-1)]!}{P!(P-1)!}$$

Table 1.7 Number of Alternative Sequences

Number of Final Products	Number of Columns	Number of Alternative Sequences
2	1	1
3	2	2
4	3	5
5	4	14
6	5	42



Angelo Lucia, Bradley R. McCallum, Energy targeting and minimum energy distillation column sequences, Computers & Chemical Engineering, Volume 34, Issue 6, 2010, Pages 931-942,



Heuristics for Alternative Separation Sequences

■ ■ For the initial selection of a feasible sequence, the following heuristics (plausible but not infallible rules) are useful and easy to apply, and do not require an economic evaluation:

1. Remove unstable, corrosive, or chemically reactive components early in the sequence.
 - A. Materials of construction used in later columns will be less expensive.
 - B. Remove very volatile components early in the sequence so that column pressures can be reduced in later columns.
2. Remove final products one by one, in order of decreasing volatility or increasing boiling point, as overhead distillates.
3. Remove, early in the sequence, those components of greatest molar percentage in the feed. The remaining columns will be smaller in diameter.
4. Make the most difficult separations in the absence of the other components → will usually lower the diameter of the tallest column.
5. Leave later in the sequence those separations that produce final products of the highest purities → will also lower the diameter of the tallest column.
6. Select the sequence that favors near-equimolar amounts of distillate and bottoms in each column → the two sections of the column will tend to have the same diameter.



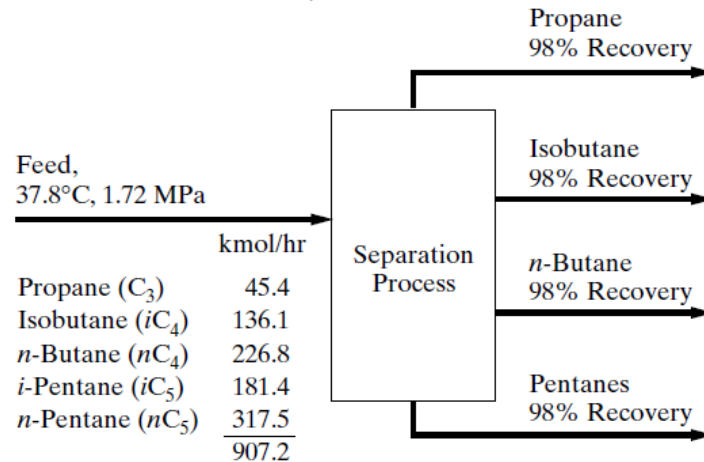
Pitfalls

- ■ Heuristics sometimes conflict with one another so that one clear choice may not be possible.
 - ■ If applicable, Heuristic 1 should always be employed.
 - ■ The most common industrial sequence is that of Heuristic 2.
 - ■ When energy costs are high, Heuristic 6 is favored because of lower utility costs.
 - ■ When one of the separations is particularly difficult, such as the separation of isomers, Heuristic 4 is usually applied.
- ■ For determining an optimal sequence, Seider *et al.* present rigorous methods that do require column designs and economic evaluations. They also consider complex sequences that include separators of different types and complexity.



EXAMPLE 8.2 (2009 Product and Process Design Principles, 3rd ed)

Consider the separation problem shown in Figure 8.12, except that separate $i\text{-C}_5$ and $n\text{-C}_5$ products are also to be obtained with 98% recoveries. Use heuristics to determine a good sequence of ordinary distillation units.

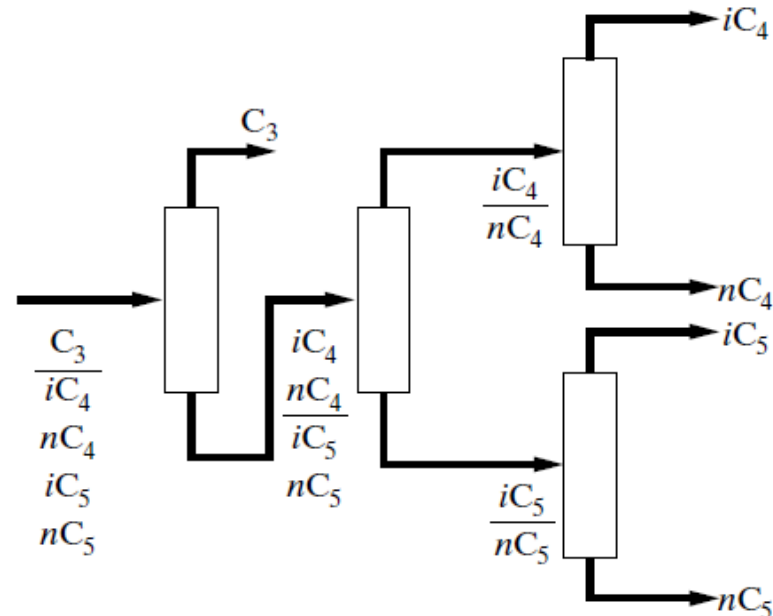


SOLUTION

Approximate relative volatilities for all adjacent pairs are

Component pair	Approximate α at 1 atm
$\text{C}_3/i\text{C}_4$	3.6
$i\text{C}_4/n\text{C}_4$	1.5
$n\text{C}_4/i\text{C}_5$	2.8
$i\text{C}_5/n\text{C}_5$	1.35

For this example, there are wide variations in both relative volatility and molar percentages in the process feed. The choice is Heuristic 4, which dominates over Heuristic 3 and leads to the sequence shown in Figure 8.12b, where the first split is between the pair with the highest relative volatility. This sequence also corresponds to the optimal arrangement.



Purity, Recovery and Units

- Separation processes usually consist of more than one operation and may produce more than one product.
- The process is designed to meet product specifications, given as **product purities** and compositions.
- The process strives to do this with high **component recoveries**.
- The product
 - **Gases: Purity as mol%, vol%** is equivalent to mol%.
 - **Liquids:** Mass fractions or **wt%.**, and Vol% common but not easily calculated when the liquid mixture is a nonideal solution.
 - To meet environmental **regulations, the allowable concentration** of an impurity in gas, liquid, or solids streams in **ppm** or **ppb**.
 - Aqueous solutions: **molarity (M:** moles of solute per liter of solution), or **molality (m:** moles of solute per kilogram of solvent); and **normality (N:** number of equivalent weights of solute per liter of solution).
 - Concentrations (*c*) in mixtures can be in units of moles or mass per volume (e.g., **mol/L, g/L, kg/m³, lbmol/ft³, lb/ft³**).
- For some chemical products, an attribute, such as color, may be used in place of purity in terms of composition.



EXAMPLE 1.1 Material balances around a separator

A feed, F , of 100 kmol/h of air containing 21 mol% O_2 (1) and 79 mol% N_2 (2) is to be partially separated by a gas permeation membrane unit, Operation (3) in Table 1.3, according to each of three sets of specifications. Compute the flow rates (n_P and n_R) in kmol/h and compositions in mol% of the two products (retentate, R , and permeate, P). In Figure 1.6(c), Phase 1 is the retentate while Phase 2 is the permeate. The membrane is more permeable to O_2 than to N_2 .

1. *Case 1:* 50% recovery of O_2 to the permeate and 87.5% recovery of N_2 to the retentate.
2. *Case 2:* 50% recovery of O_2 to the permeate and 50 mol% purity of O_2 in the permeate.
3. *Case 3:* 85 mol% purity of N_2 in the retentate and 50 mol% purity of O_2 in the permeate.

Solution

The feed (F) rates of oxygen (1) and nitrogen (2) are

$$n_{1,F} = 0.21(100) = 21 \text{ kmol/h}$$

$$n_{2,F} = 0.79(100) = 79 \text{ kmol/h}$$

Case 1: Because two recoveries are given:

$$n_{1,P} = 0.50(21) = 10.5 \text{ kmol/h}$$

$$n_{2,R} = 0.875(79) = 69.1 \text{ kmol/h}$$

$$n_{1,R} = 21 - 10.5 = 10.5 \text{ kmol/h}$$

$$n_{2,P} = 79 - 69.1 = 9.9 \text{ kmol/h}$$

For the permeate: $n_P = 10.5 + 9.9 = 20.4 \text{ kmol/h}$

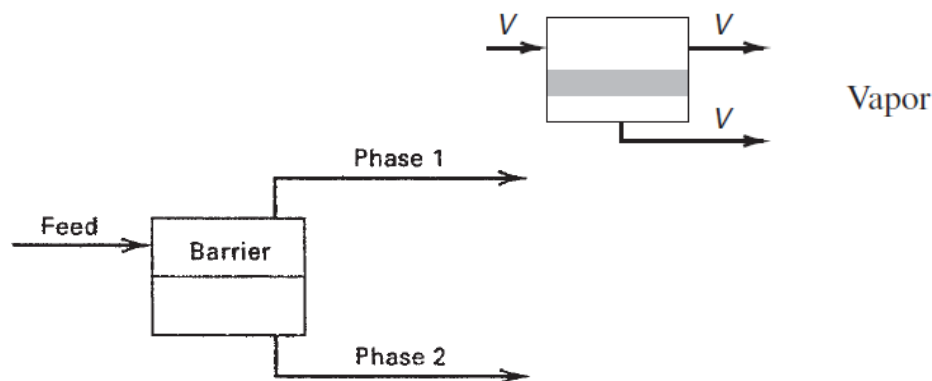
$$\text{mol\% } O_2 = 10.5/20.4 = 0.515 = 51.5\%$$

$$\text{mol\% } N_2 = 100 - 51.5 = 48.5\%$$

For the retentate: $n_R = 69.1 + 10.5 = 79.6 \text{ kmol/h}$

$$\text{mol\% } O_2 = 10.5/79.6 = 0.132 = 13.2\%$$

$$\text{mol\% } N_2 = 100 - 13.2 = 86.8\%$$



Case 2: O₂ recovery is given; its distribution to the products is

$$n_{1,P} = 0.50(21) = 10.5 \text{ kmol/h}$$

$$n_{1,R} = 21 - 10.5 = 10.5 \text{ kmol/h}$$

Using the purity of O₂ in the permeate, the total permeate flow rate is

$$n_P = 10.5/0.5 = 21 \text{ kmol/h}$$

By a total permeate material balance:

$$n_{2,P} = 21 - 10.5 = 10.5 \text{ kmol/h}$$

By an overall N₂ material balance:

$$n_{2,R} = 79 - 10.5 = 68.5 \text{ kmol/h}$$

For the permeate: $n_P = 21 \text{ kmol/h}$

$$\text{mol\% O}_2 = 10.5/21 = 0.50 = 50\%$$

$$\text{mol\% N}_2 = 100 - 50 = 50\%$$

For the retentate: $n_R = 100 - 21 = 79 \text{ kmol/h}$

$$\text{mol\% O}_2 = 10.5/79 = 0.133 = 13.3\%$$

$$\text{mol\% N}_2 = 100 - 13.3 = 86.7\%$$

Case 3: Two material-balance equations, one for each component, can be written:

For nitrogen, with a purity of 0.85 in the retentate and $1.00 - 0.50 = 0.50$ in the permeate,

$$n_{2,F} = 0.85n_R + 0.50n_P = 79 \text{ kmol/h} \quad (1)$$

For oxygen, with a purity of 0.50 in the permeate and $1.00 - 0.85 = 0.15$ in the retentate,

$$n_{1,F} = 0.50n_P + 0.15n_R = 21 \text{ kmol/h} \quad (2)$$

Solving (1) and (2) simultaneously for the total flow rates of the products gives

$$n_P = 17.1 \text{ kmol/h} \text{ and } n_R = 82.9 \text{ kmol/h}$$

Therefore, the component flow rates are

$$n_{1,P} = 0.50(17.1) = 8.6 \text{ kmol/h}$$

$$n_{2,R} = 0.85(82.9) = 70.5 \text{ kmol/h}$$

$$n_{1,R} = 82.9 - 70.5 = 12.4 \text{ kmol/h}$$

$$n_{2,P} = 17.1 - 8.6 = 8.5 \text{ kmol/h}$$

For the permeate: $n_P = 17.1 \text{ kmol/h}$

$$\text{mol\% O}_2 = 8.6/17.1 = 0.503 = 50.3\%$$

$$\text{mol\% N}_2 = 100 - 50.3 = 49.7\%$$

For the retentate: $n_R = 100 - 17.1 = 82.9 \text{ kmol/h}$

$$\text{mol\% O}_2 = 12.4/82.9 = 0.150 = 15.0\%$$

$$\text{mol\% N}_2 = 100 - 15.0 = 85.0\%$$

Solve

1.12

1.19

1.23

Read Chapters 2 and 3. They are a recap of Thermodynamics II and Transport Phenomena II.