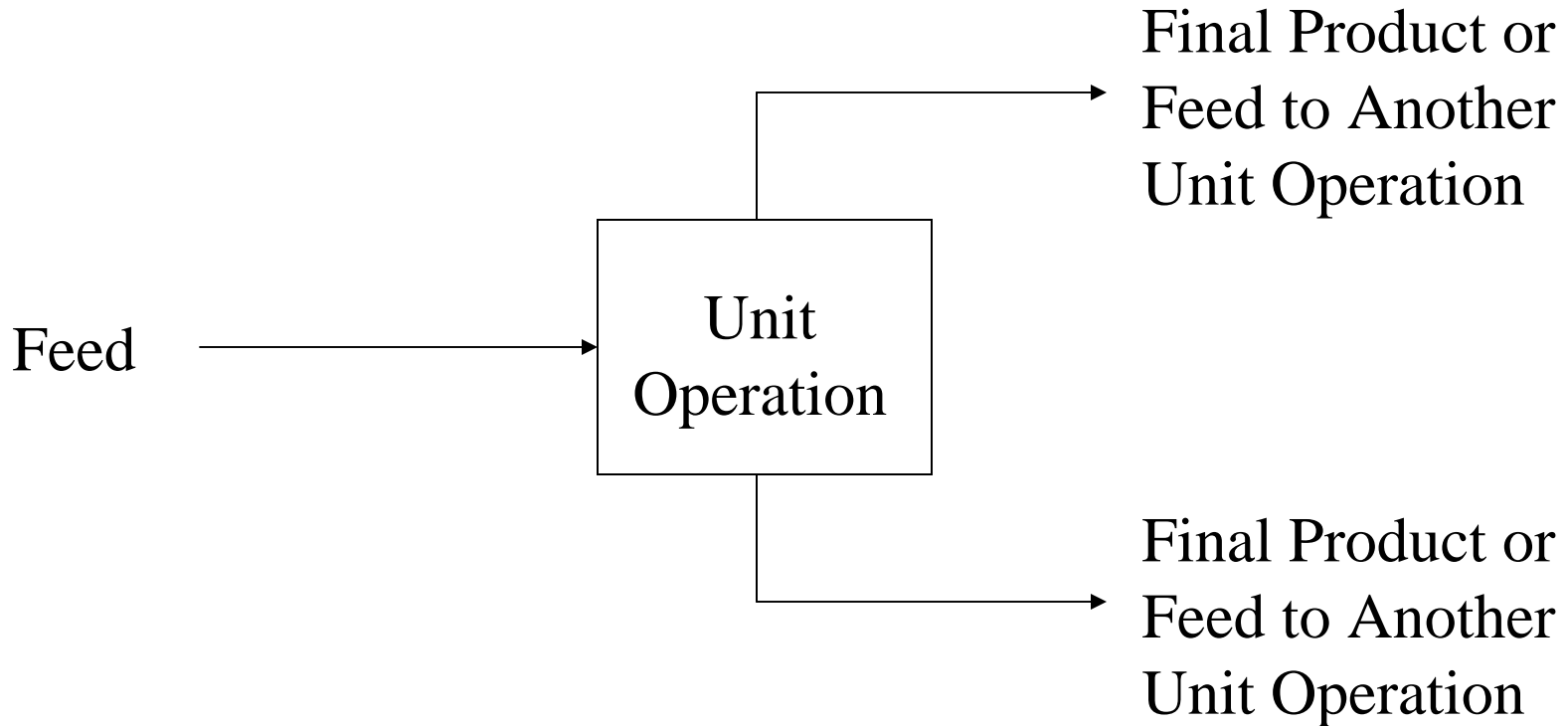


Class 11

Synthesis of Separation Trains

Separation Equipment



Multi-component separations are complex as they involve decisions on which unit operations to select and how to sequence their use

Industrial Methods for Separation

- (1) The creation by heat transfer, shaft work, or pressure reduction of a 2nd phase that is immiscible with the feed phase (no other chemicals are added to the feed mixture; separation by using an energy separating agent) (ESA)**
- (2) The introduction into the system of a 2nd fluid phase (2nd phase is added to the separation unit in the form of a solvent as a mass transfer separating agent - selectively dissolves or alters volatility of certain mixture species) (MSA)**
- (3) The addition of a solid phase on which adsorption can occur (selectively adsorbs certain species; subsequent treatment needed to regenerate adsorbent) (MSA)**
- (4) The placement of a membrane barrier (not limited by thermodynamic equilibrium) (ESA)**

Table 7.1 Common Industrial Separation Methods

Separation Method	Phase Condition of Feed	Separating Agent(s)	Developed or Added Phase	Separation Property
Flash	L and/or V	Pressure reduction or heat transfer ESA	V or L	Volatility
Distillation (ordinary)	L and/or V	Heat transfer or shaft work ESA	V or L	Volatility
Gas absorption	V	Liquid absorbent MSA	L	Volatility
Stripping	L	Vapor stripping agent MSA	V	Volatility
Extractive distillation	L and/or V	Liquid solvent and heat transfer MSA	L and V	Volatility
Azeotropic distillation	L and/or V	Liquid entrainer and heat transfer MSA	L and V	Volatility
Liquid-liquid extraction	L	Liquid solvent MSA	Second L	Solubility
Crystallization	L	Heat transfer ESA	S	Solubility or melting point
Gas adsorption	V	Solid adsorbent MSA	S	Adsorbability
Liquid adsorption	L	Solid adsorbent MSA	S	Adsorbability
Membrane	L or V	Membrane ESA	Membrane	Permeability and/or solubility
Supercritical extraction	L or V	Supercritical solvent MSA	Supercritical fluid	Solubility
Leaching	S	Liquid solvent MSA	L	Solubility
Drying	S and L	Heat transfer ESA	V	Volatility
Desublimation	V	Heat transfer ESA	S	Volatility

Separation Factor

$$SF = \frac{C_1^I / C_2^I}{C_1^{II} / C_2^{II}} = \frac{C_1^I}{C_1^{II}} \cdot \frac{C_2^{II}}{C_2^I} = \frac{C_1^I}{C_1^{II}} \bigg/ \frac{C_2^{II}}{C_2^I}$$

C = mole fraction, mass fraction, or concentration

components 1 & 2
phases I & II

for most applications, SF limited
by thermodynamic equilibrium

by convention, components assigned so that $SF > 1$

for VLE, use y's and x's

$$SF = \frac{y_1}{x_1} \bigg/ \frac{y_2}{x_2} = \frac{K_1}{K_2} = \alpha_{1,2}$$

K = distribution coefficient

relative
volatility

- **ESA operations more economically feasible at lower SF than MSA**
- **distillation always considered first if feed is liquid or L/V mix**

Separation Factor

ideal solutions and ideal gas law applies

$$SF = \frac{\frac{y_1}{x_1}}{\frac{y_2}{x_2}} = \alpha_{1,2} = \frac{P_1^s}{P_2^s} \quad \text{ratio of vapor pressures (ESA)}$$

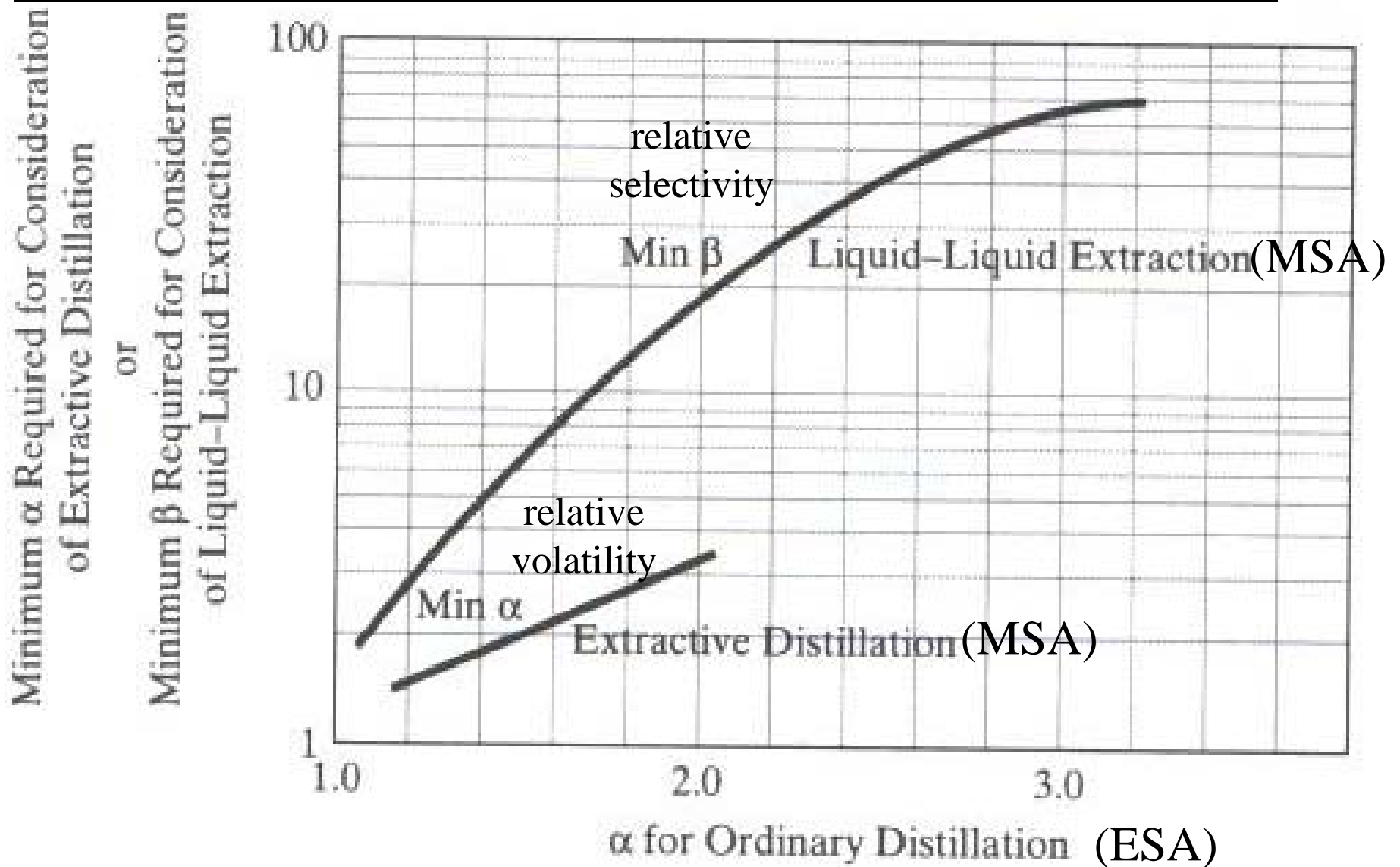
nonideal liquid solutions (azeotropic and extractive distillation) (MSA)

$$SF = \frac{\frac{y_1}{x_1}}{\frac{y_2}{x_2}} = \alpha_{1,2} = \frac{\gamma_1^L P_1^s}{\gamma_2^L P_2^s} \quad \begin{array}{l} \text{(non-ideal liquid sol'n, near ambient P)} \\ \text{modified Raoult's law;} \\ \text{include liquid activity coefficients} \end{array}$$

two immiscible liquid phases (liquid-liquid extraction) (MSA)

$$SF = \beta_{1,2} = \frac{\frac{\gamma_1^{II}}{\gamma_1^I}}{\frac{\gamma_2^{II}}{\gamma_2^I}} \quad \begin{array}{l} \beta \text{ relative selectivity} \\ \text{phase II, MSA-rich phase} \\ \text{component 1, more selective for MSA-} \\ \text{rich phase than component 2} \end{array}$$

MSAs typically require higher α to be economical relative to ESAs



MSA's vs ESA's

Selection criteria for MSA's:

- ease of recovery
- high SF values
- to form azeotropes with components in feed

Single-stage separations rarely meet purity requirements
Counter-current flow cascades of stages achieve high separations

Use single-stage separations (flash, partial condenser) for
high SF value or
only rough separation req'd

$1.05 \leq SF \leq 1.10$ distillation economical choice

if MSA enhances SF to high value, may be more economical
absorption/stripping require $SF \geq 10$

Degree of Sharpness of Separation

Recovery Factor

$$FR_i = \frac{n_i^I}{n_i^F}$$

product stream rich in i

feed

Motivation for Separation

purification: one species or a group of species

ESA concern: higher temperatures causing decomposition
of some components

removal: undesirable components

Concern: loss of valuable components

recovery: for subsequent processing

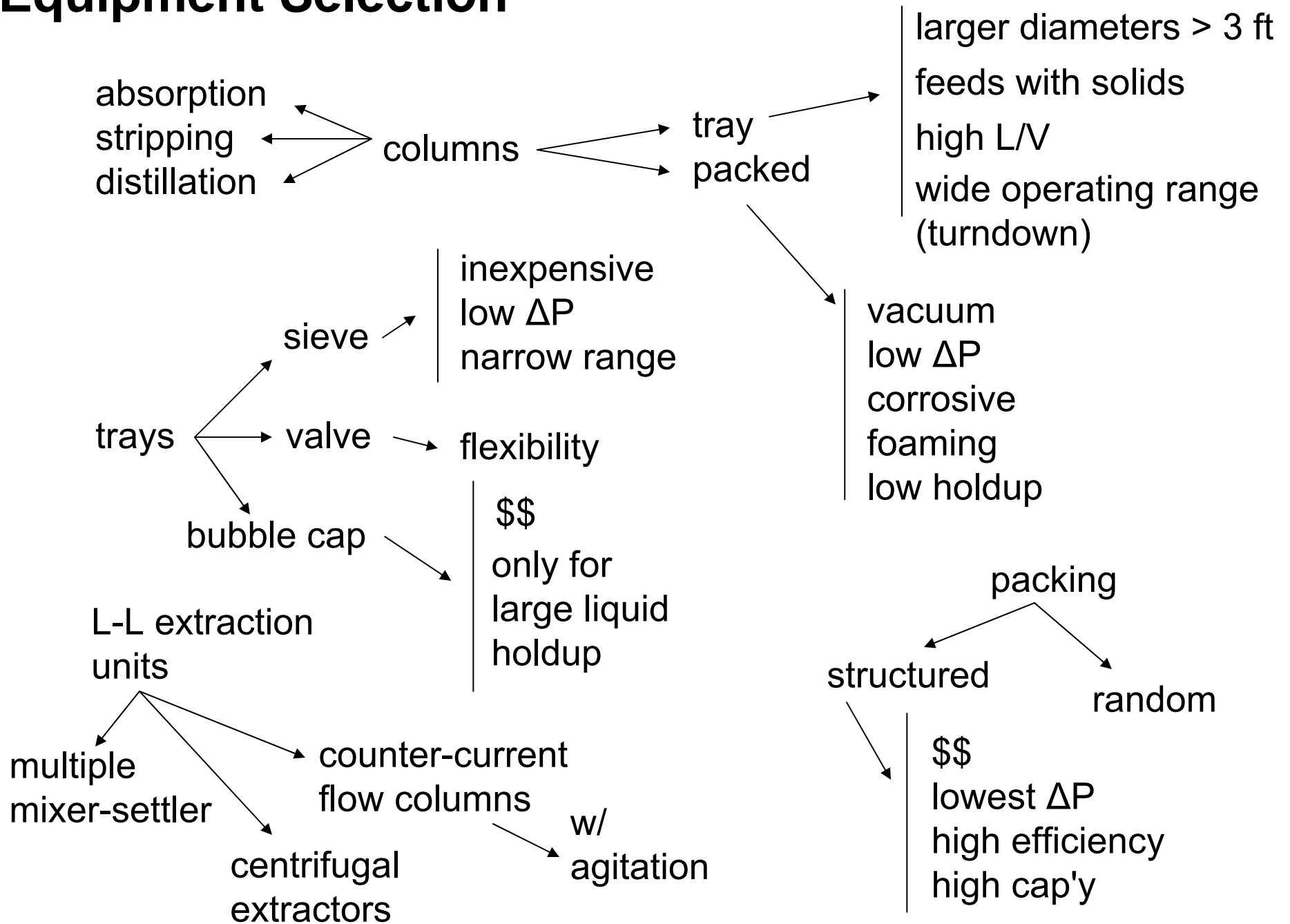
high degree of separation may be unnecessary

solid separation:

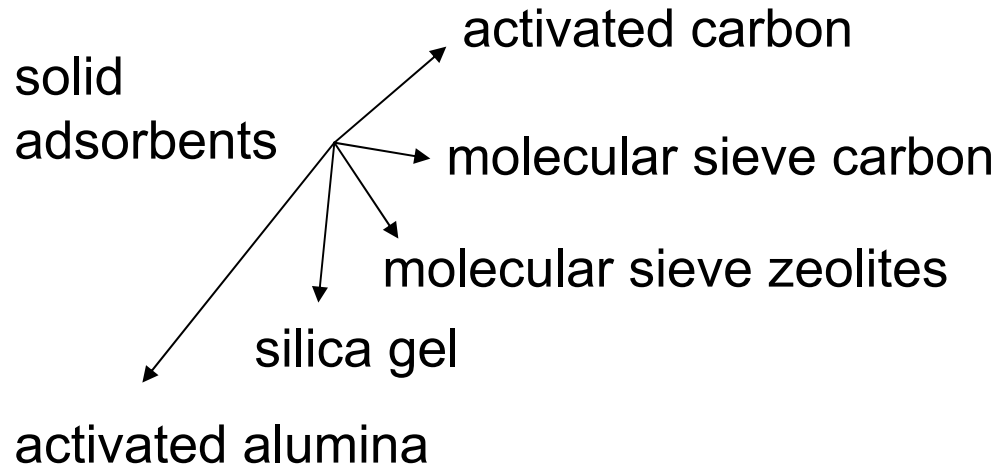
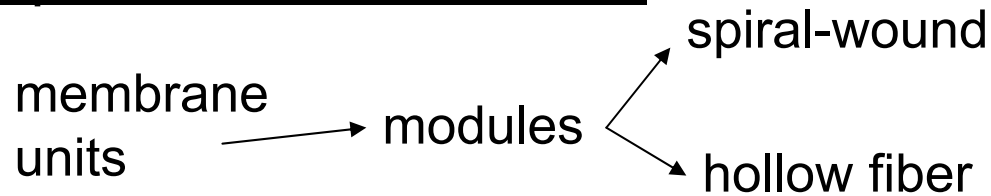
when b.p.'s close, but m.p.'s separated
avoid when separating small amt of 1 component
from others that solidify

membrane separations: separation factor defined by
mass transfer rates or permeabilities

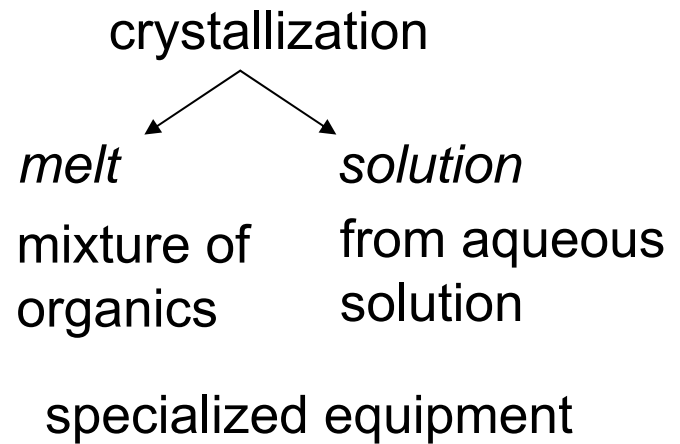
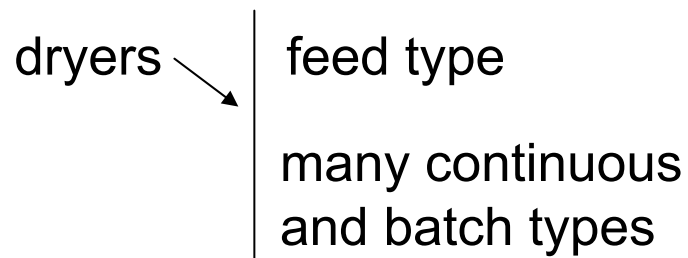
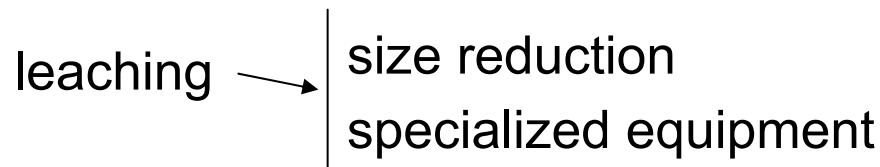
Equipment Selection



Equipment Selection cont.



adsorption isotherms of competing solutes
regeneration step
batch/slurry
fixed-bed



Synthesis of Separation Trains

Basic Strategy and Available Methods

distillation-type separation methods usually of choice for liquid feeds

partially vaporized feeds

relative volatility > 1.1

nominal pressure and temperature conditions

other methods considered for

vapor, dry solid or wet solid feeds

feeds not meeting conditions for distillation

if feed is mixed phase, separate phases first by

flash drum, decanter, centrifuge, filter

additional methods:

absorber/stripper

extractive/azeotropic distillation

membranes

crystallization

gas/liquid adsorption

supercritical extraction

leaching

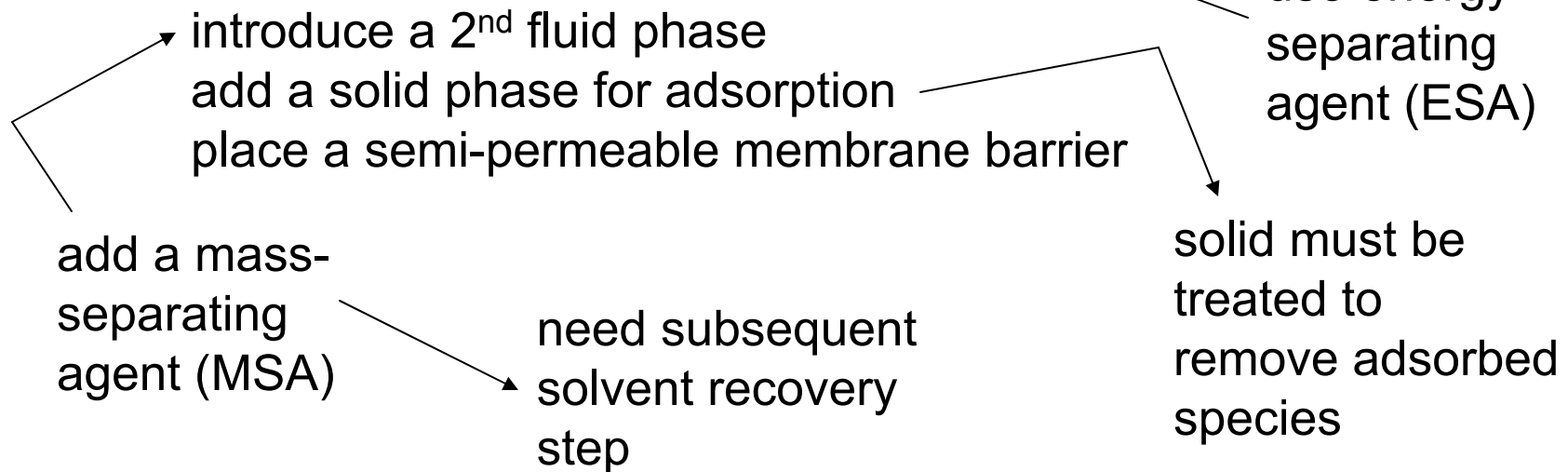
drying

Synthesis of Separation Trains

Basic Strategy and Available Methods

Multicomponent separation by segregating species in different locations:
use of heat transfer, shaft work, or pressure reduction

to create 2nd immiscible phase



mass transfer rates govern all methods
all except membranes limited by equilibrium

Ordinary Distillation

- **Normal distillation separations are the methods of choice for liquid or partially vaporized feeds unless the relative volatility between the two key components is $\alpha_{1,2} < 1.1$ or extreme conditions of T and P are needed**
- **For $1.05 < \alpha_{1,2} < 1.1$, ordinary distillation may still be the most economical choice**
- **Most towers do not exceed 200 ft tall for structural reasons**

• **Types of columns: Trayed (Sieve, Valve, & Bubble Cap) or Packed**

- **Trayed columns if more than 3 ft in diameter; feeds containing solids; high liquid to gas ratios; wide operating range**

Sieve Trays: least expensive, lowest ΔP , narrowest turndown

Valve Trays: higher flexibility (turndown)

Bubble-cap: most expensive; used for large liquid hold-ups

- **Packed columns if operation under vacuum or low ΔP required; corrosive systems; foaming systems; low liquid hold-up desired**

Random Packing: Lowest Cost

**Structured Packing: Expensive; Lowest ΔP ,
Highest Efficiency**

Sequencing of Ordinary Distillation Columns

Ordinary distillation columns indicated when
relative volatility between keys > 1.05
reboiler duty not excessive
column pressure not such to approach critical temperature
condense overhead vapor without refrigeration
bottoms temperature not high to cause decomposition
azeotropes do not prevent desired separation
column pressure drop is practicable, esp. vacuum column

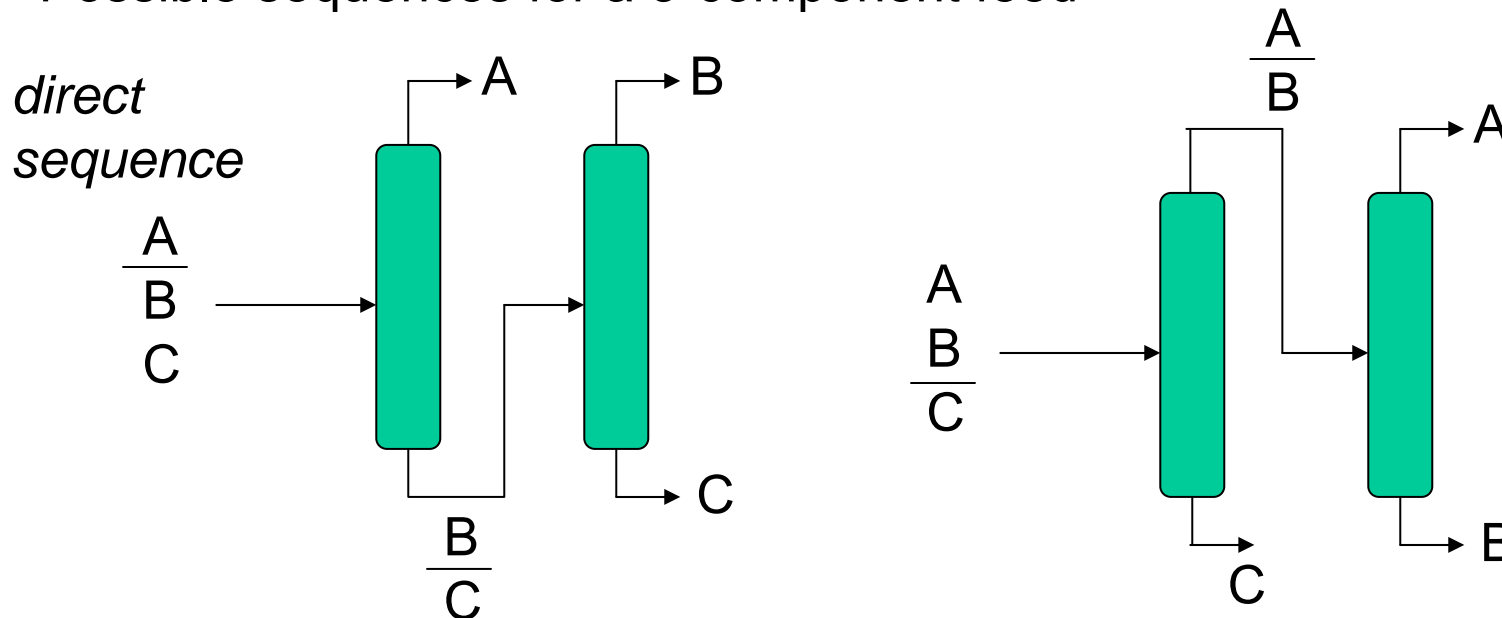
Sequencing of Ordinary Distillation Columns

Sequencing suggested when single feed to each column and two product streams from each column (distillate & bottoms)

Number of possible sequences is combinatorial problem:

$$N_s = \frac{[2 \cdot (P - 1)]!}{P! (P - 1)!} \quad P : \text{no. of product streams}$$

Possible sequences for a 3-component feed



Number of separators required: $P - 1$

Separation of a mixture of benzene (80.1°C BP), toluene (110.8°C BP), & biphenyl (254.9°C BP)

Separation of a mixture of ethylbenzene, p-xylene, m-xylene, & o-xylene; p- & m-: $\Delta T_{BP} = 0.8^\circ\text{C}$

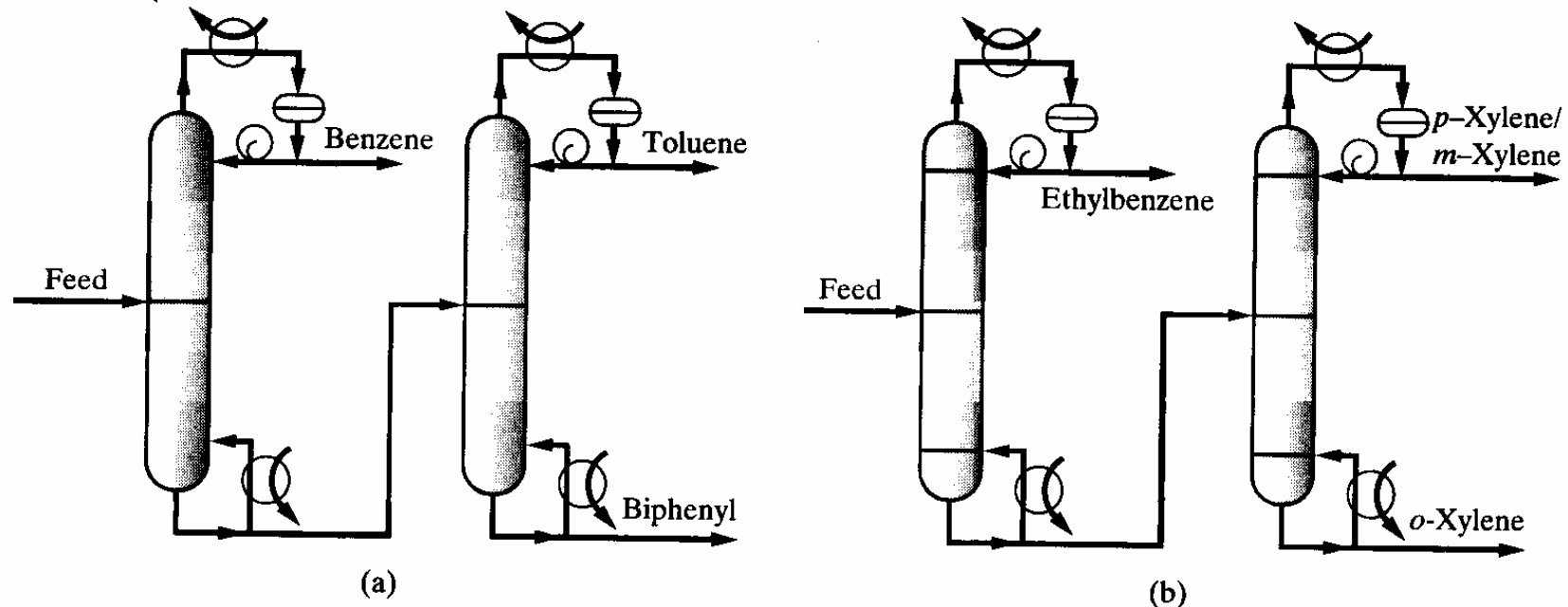


Figure 5.5 Distillation configurations for separation of ternary mixtures: (a) separation of a benzene–toluene–biphenyl mixture; (b) separation of xylene isomers.

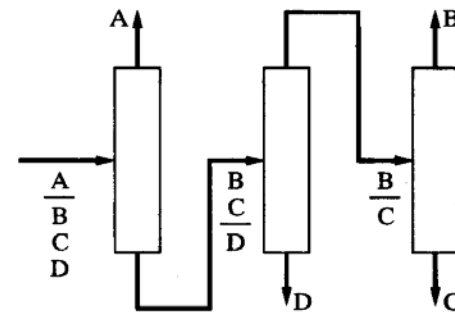
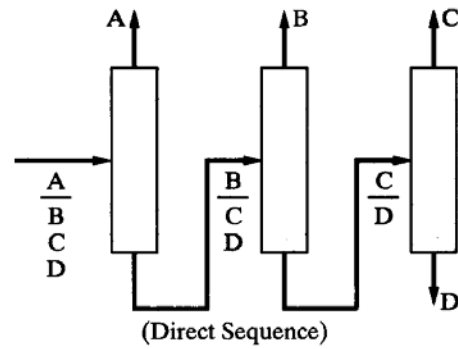
Sequencing Options

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

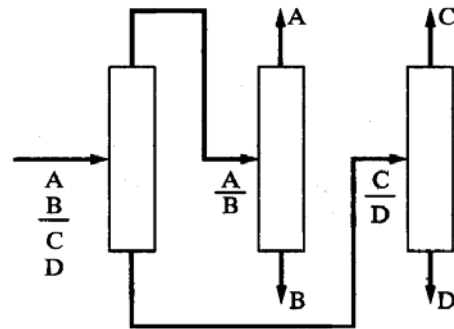
Table 5.2 Number of Possible Sequences for Separation by Ordinary Distillation

Number of Products, P	Number of Separators in the Sequence	Number of Different Sequences, N_s
2	1	1
3	2	2
4	3	5
5	4	14
6	5	42
7	6	132
8	7	429
9	8	1,430
10	9	4,862

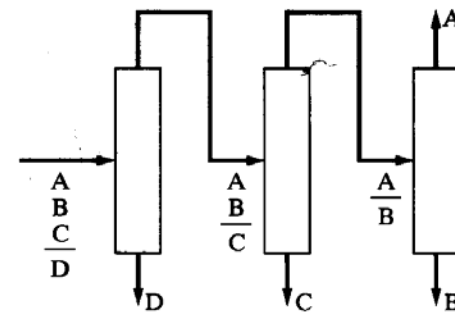
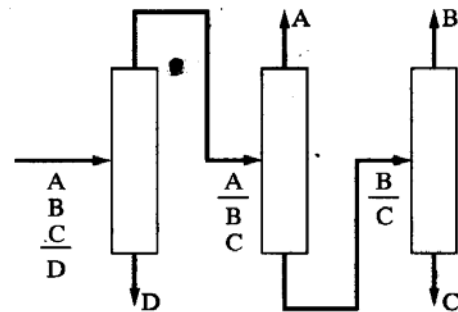
Five sequences for a 4 component feed



(a)



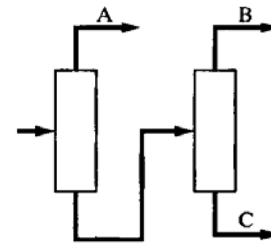
(b)



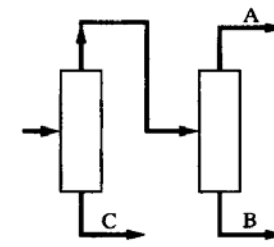
(Indirect Sequence)

(c)

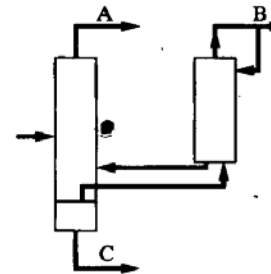
Configurations for Ternary Distillation



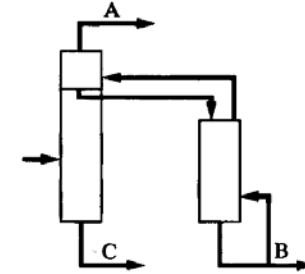
I. Direct Sequence



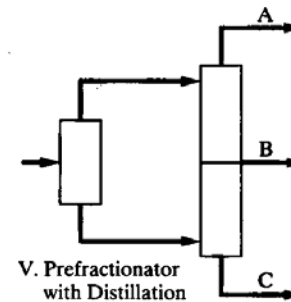
II. Indirect Sequence



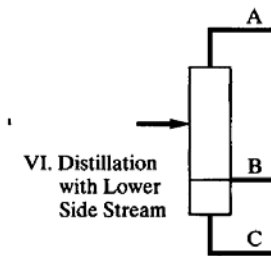
III. Distillation with Vapor Side Stream Rectifier



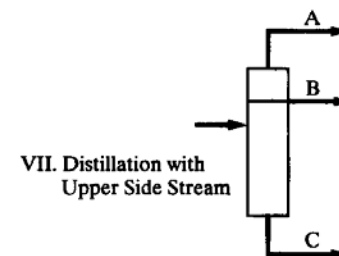
IV. Distillation with Liquid Side Stream Stripper



V. Prefractionator with Distillation



VI. Distillation with Lower Side Stream



VII. Distillation with Upper Side Stream

Sequencing of Ordinary Distillation Columns

Strategy for selecting optimal sequence

for $P = 3$ or 4 , evaluate all sequences (design & cost estimate)
direct sequence often the choice

for $P > 4$, use heuristics to reduce candidate sequences

- (1) remove thermally unstable, corrosive, or chemically reactive components early in the sequence
- (2) remove final products one by one as distillates
- (3) sequence separation points to remove, early in the sequence, those components of greatest molar percentage in the feed
- (4) sequence separation points in order of decreasing relative volatility so most difficult splits are carried out later in the sequence
- (5) leave to last those separations that produce the highest purity products
- (6) sequence separation points that favor near equimolar amounts of distillate and bottoms in each column (high energy costs)

Heuristics often conflict.

Wide variation in both α_{ij} and molar % in the process feed

Using Heuristic 4: (order is decreasing relative volatility)

Approximate relative volatilities for all adjacent pairs are

Component Pair	Approximate α at 1 atm
C_3/iC_4	3.6
iC_4/nC_4	1.5
nC_4/iC_5	2.8
iC_5/nC_5	1.35

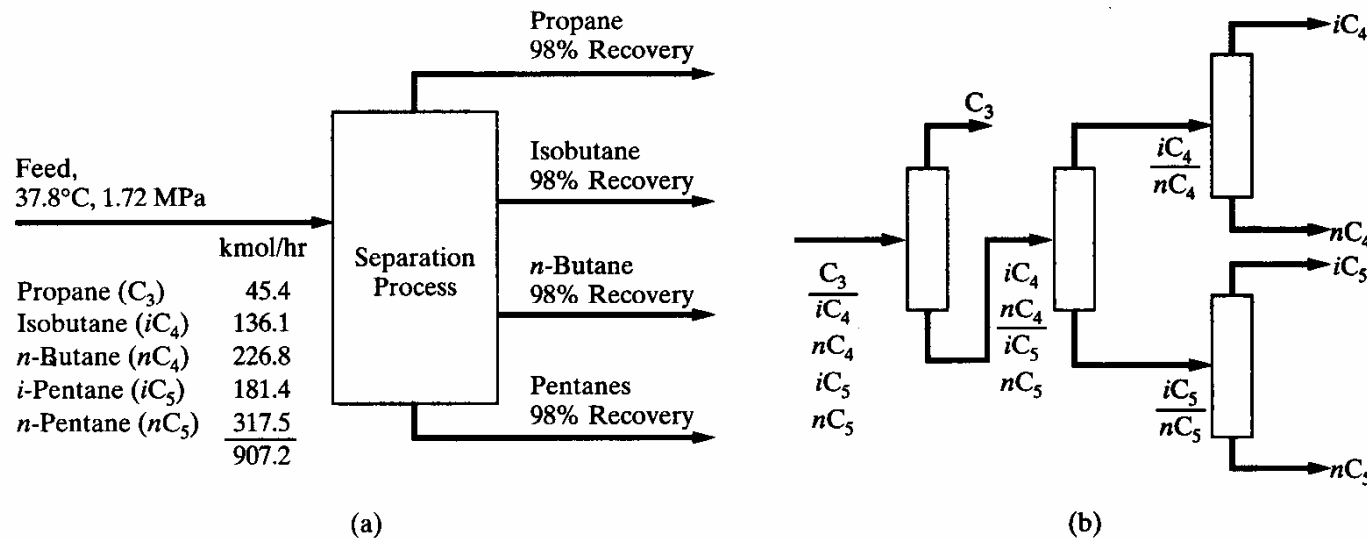


Figure 5.7 Synthesis problem and separation train for Example 5.2: (a) paraffin separation problem; (b) sequence developed from heuristics.

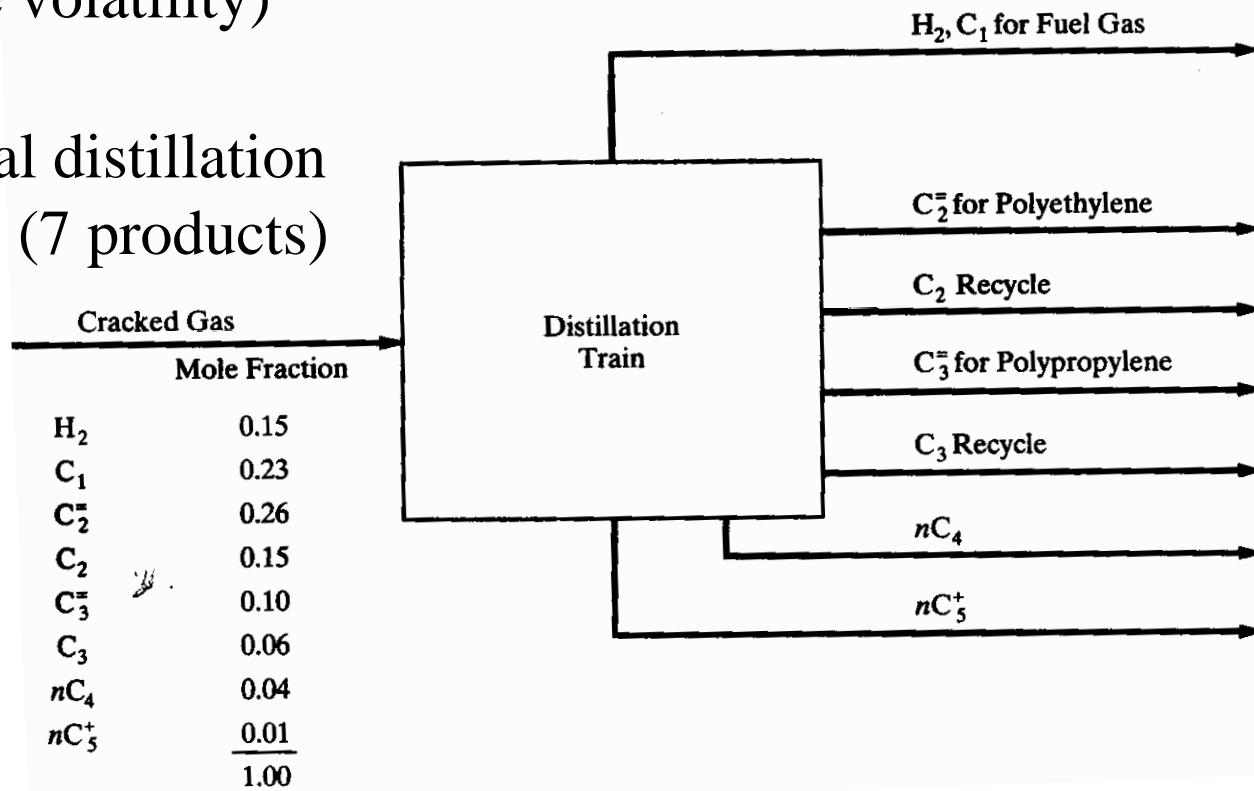
Example: Problem # 7.3, page 296

Thermal Cracking of naphtha yields a gas that is to be separated by a distillation train into the products indicated below. If reasonably sharp separations are to be achieved, determine sequences using heuristics 4 and 2 on page 251.

(relative volatility)

(direct sequence)

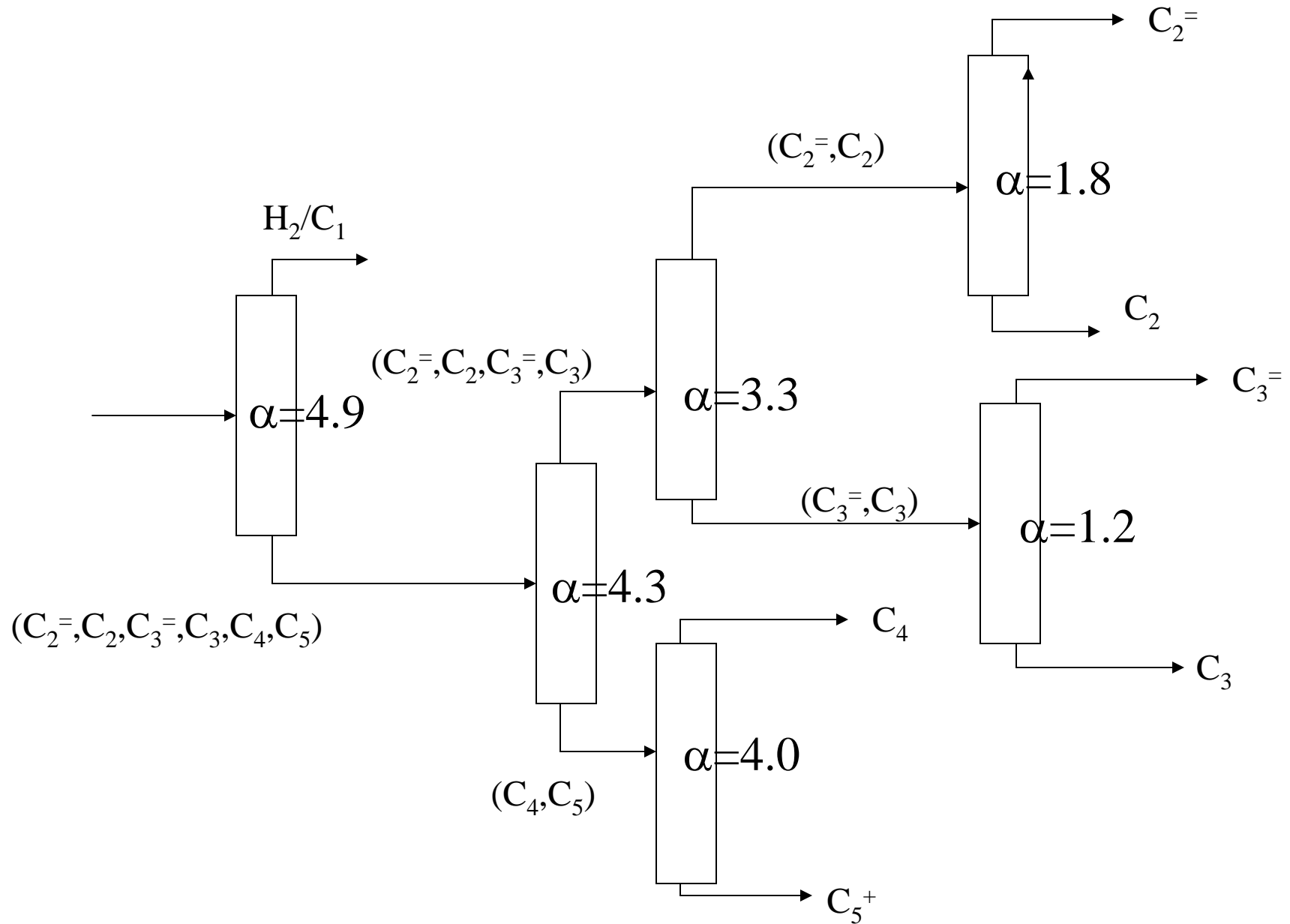
132 normal distillation sequences (7 products)



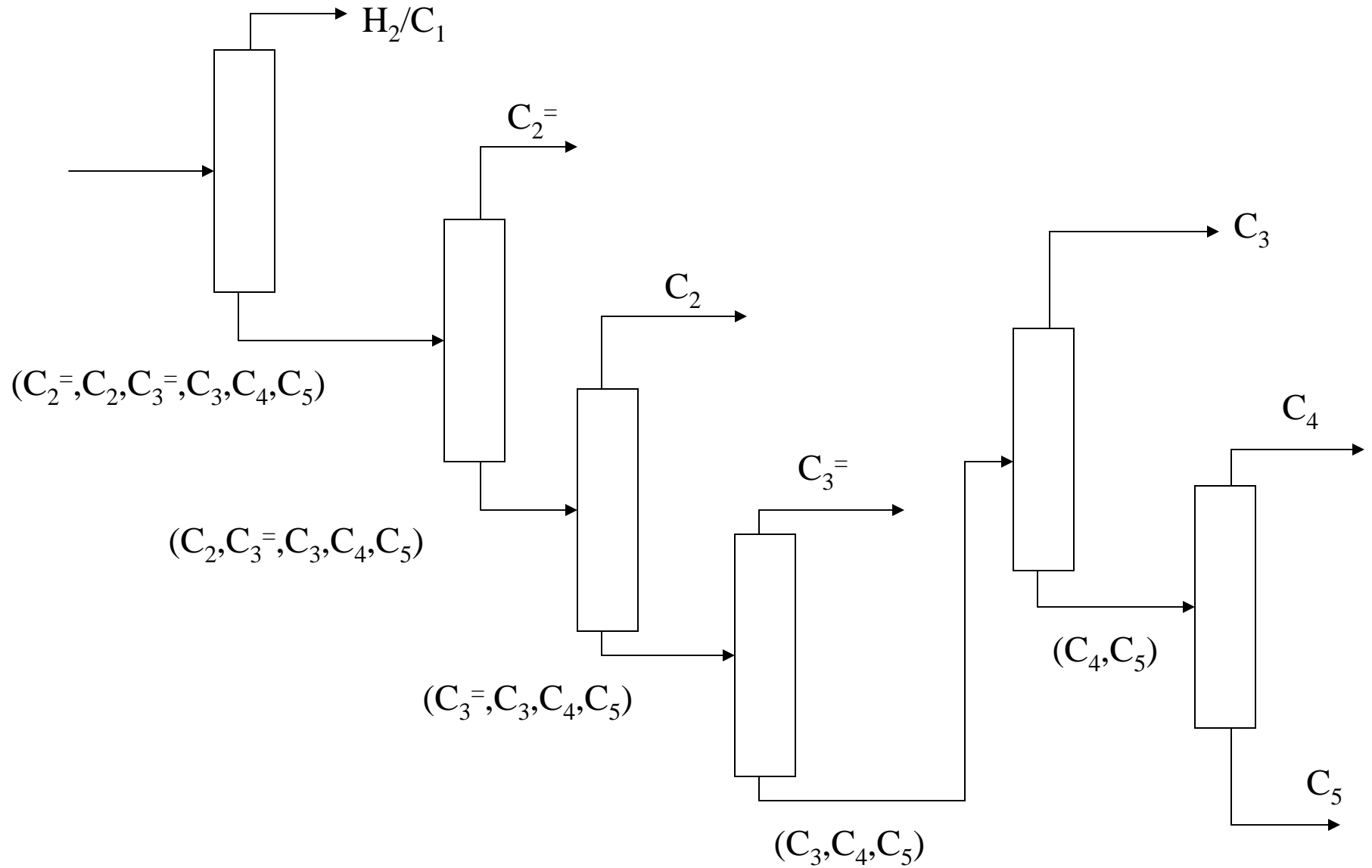
Distribution Coefficients @ 14.7 psia & 20°F

	<u>T_{bp}(°C)</u>	<u>K</u>		<u>α_{ij}</u>	<u>Order</u>
H ₂ /C ₁ (Methane):	-161.5	122		4.9	1
C ₂ ⁼ (Ethylene):	-103.7	25		1.8	5
C ₂ (Ethane):	-88.6	14.3		3.3	4
C ₃ ⁼ (Propylene):	-47.7	4.4		1.2	6
C ₃ (Propane):	-42.07	3.6		4.3	2
C ₄ (n-butane):	-0.6	0.8		4.0	3
C ₅ ⁺ (n-pentane ⁺):	36.07	0.2			

Sequential Separation based on Heuristic 4: Relative Volatility



Sequential Separation based on Heuristic 2: Direct Sequence



Algorithm to determine column pressure and condenser type

