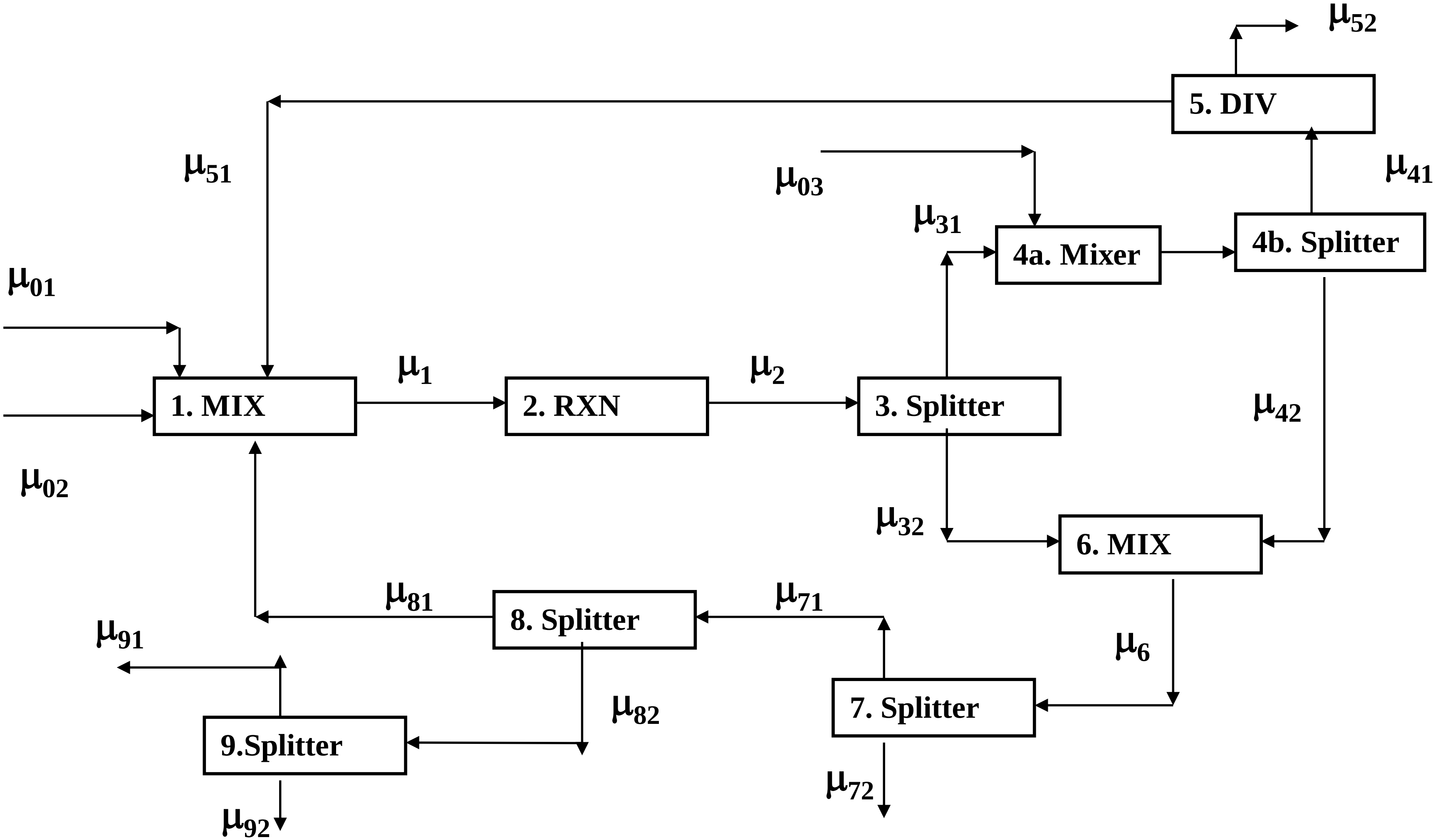
Lecture 5

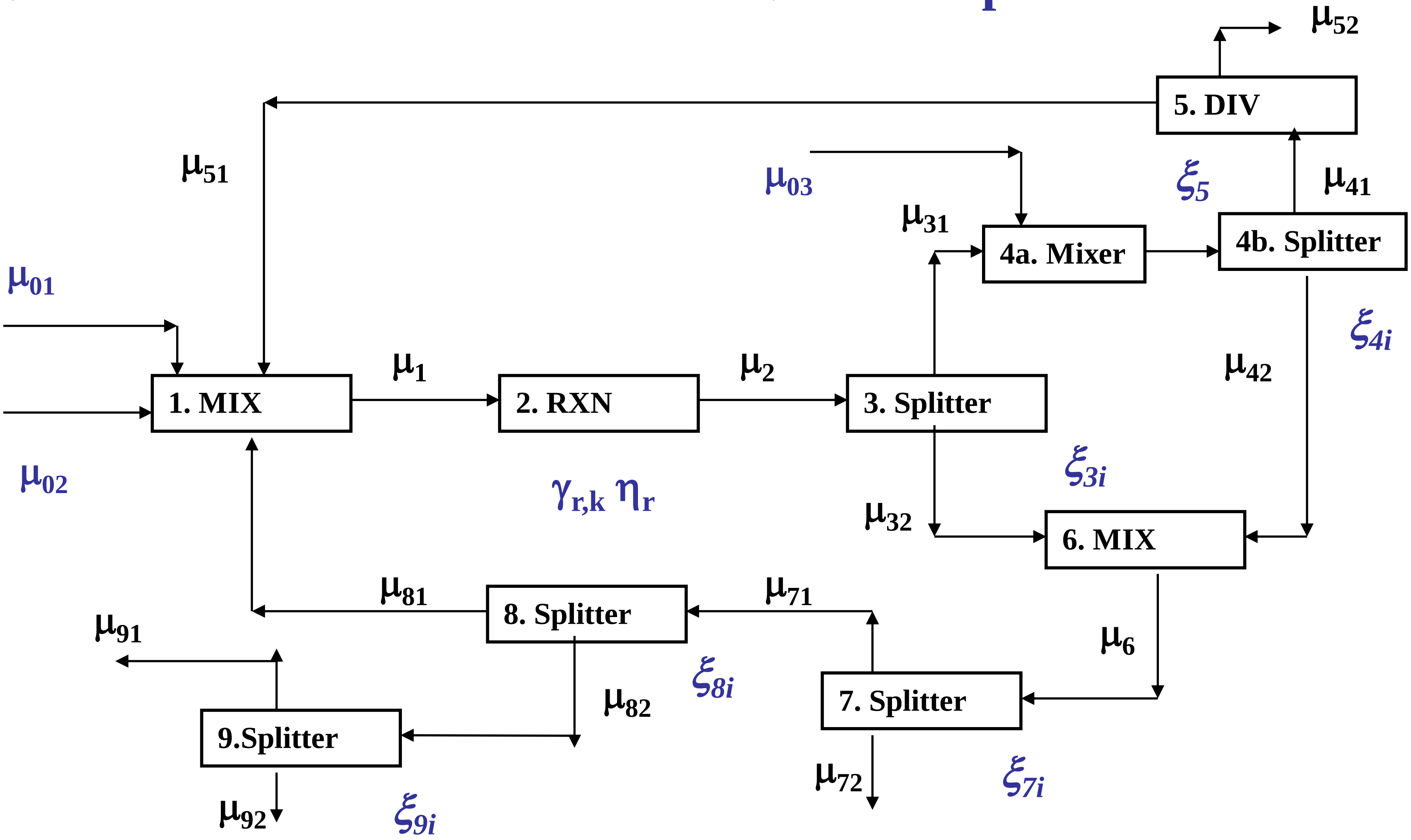
Reactor & separation process design & analysis

3b. Redraw flowsheet for MB-model: Redrawn flowsheet



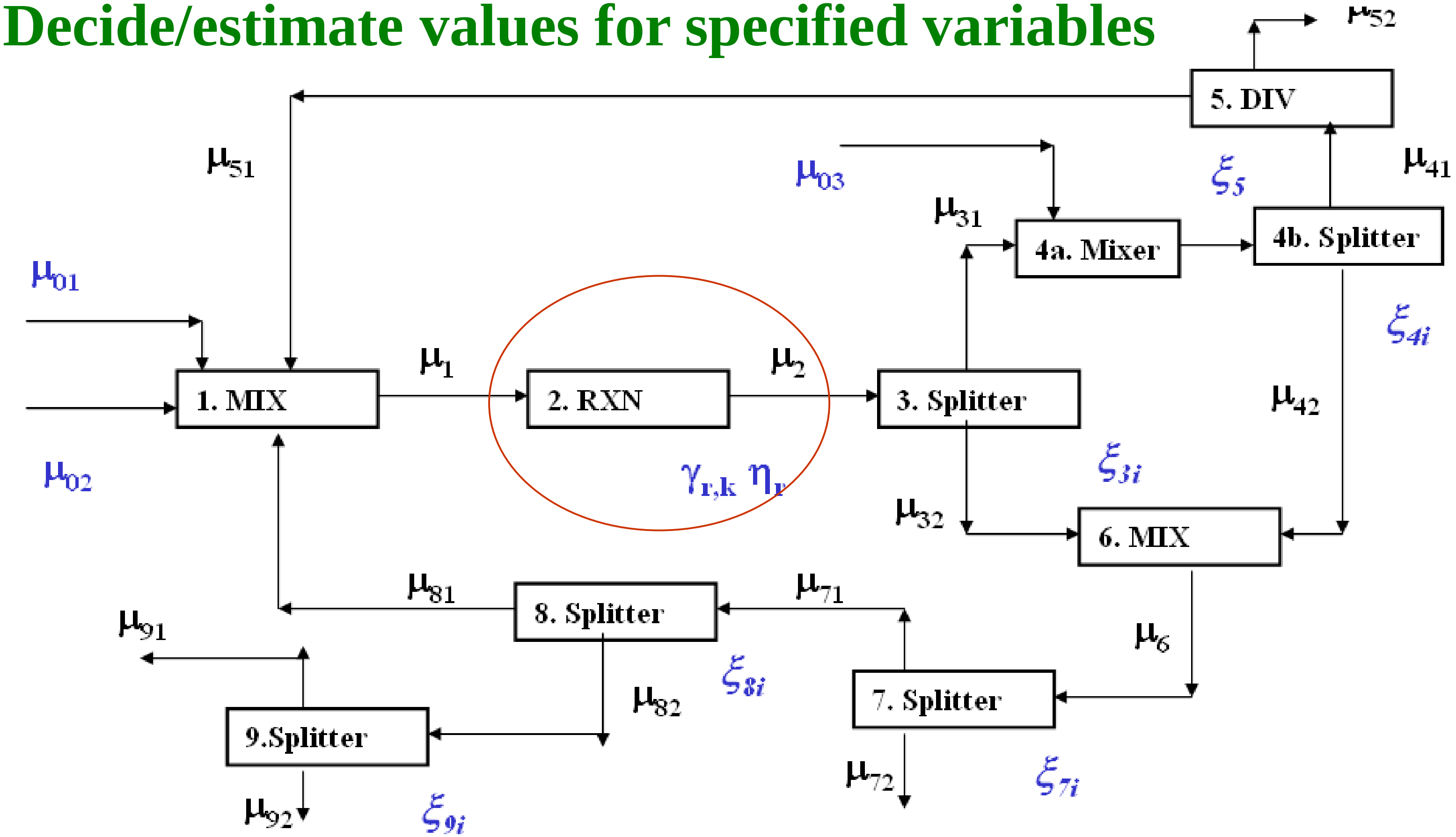
Use only mixers, reactors, dividers and splitters!

3c. Redraw flowsheet for MB-model: Locate specified variables



If all the variables marked in blue are known, then all other variables representing the flowhseet (MB-model) can be calculated!

4a. Decide/estimate values for specified variables

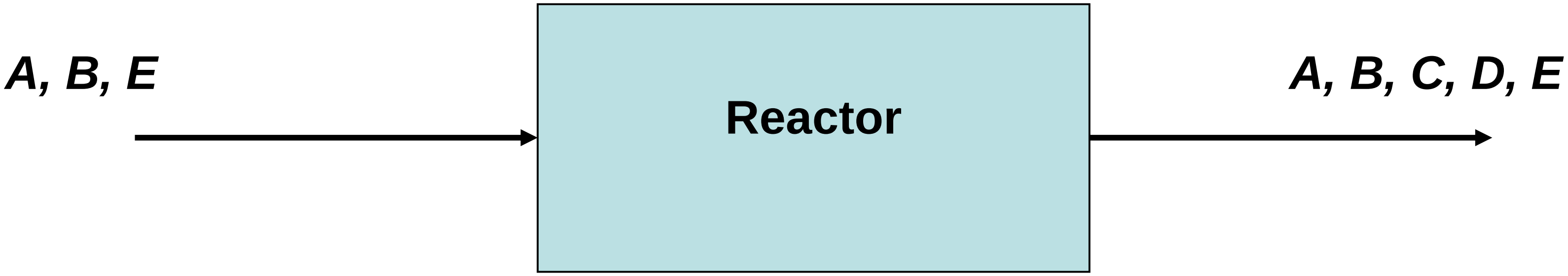


$EL + W \rightarrow EA$ 7% conversion/pass EL to EA (η_1)

$PL + W \rightarrow EA$ 0.7% conversion/pass PL to IPA (η_2)

$2EA \leftrightarrow DEE + W$ Equilibrium controlled, $K(T, P) = 0.2$

Reactor Design & Analysis



A, B, E

Reactor

A, B, C, D, E

A + B = C + D

Design Issues: Type of reactor; number of reactor; reactor design parameters (temperature, pressure, conversion, kinetic-equilibrium, volume, residence time, ….)

Basic Steps in Reactor Design & Analysis

* **Write reactions**

* **Find data for the reaction (conversion, kinetic model, catalyst, …)**

* **Write simple models for CSTR, Plug-Flow & Recyle-Reactors**

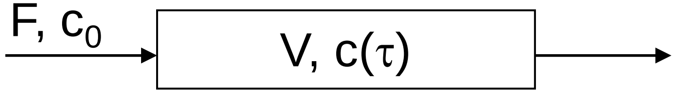
  - **Reaction rate model**
  - **Determine required reactor volume**
  - **Residence time**
  - **Use kinetic or equilibrium model to fix T, P**

* **Verify through simulation**

Basic Steps in Reactor Design & Analysis

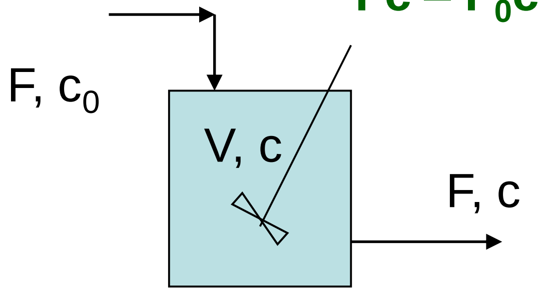
- * **Write reactions** $A + B = 2C$
- ***Write simple models for CSTR, Plug-Flow & Recyle-Reactors**

$d(Fc)/dV = r(c);\ c(0) = c_0$

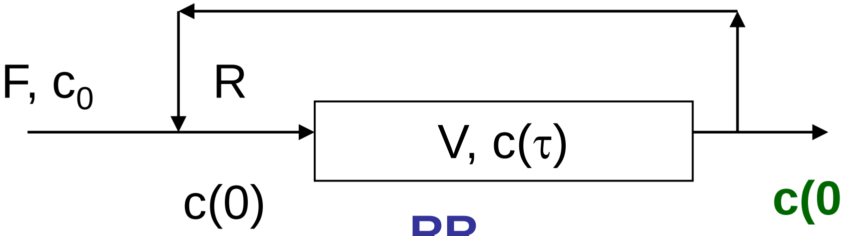


PFR

$Fc - F_0c_0 = Vr(c)$



CSTR



RR

$d(Fc)/dV = r(c)$

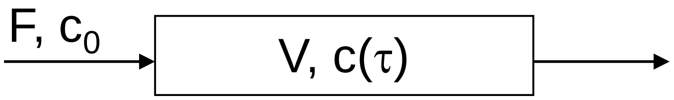
$c(0) = (RF_Vc_V + F_0c_0)/(RF_V+1)$

- ***Find data for the reaction** **(conversion, kinetic model, catalyst, …)**
- * **Verify through simulation**

Basic Steps in Reactor Design & Analysis: models

$d(Fc)/dV = r(c);\ c(0) = c_0$

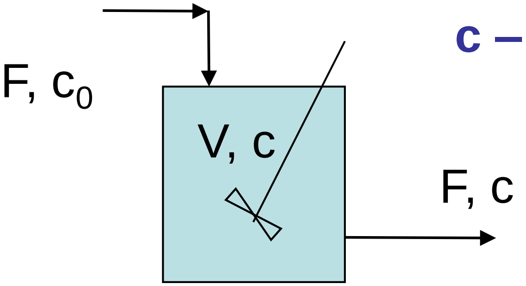
$dc/d\tau = r(c)$



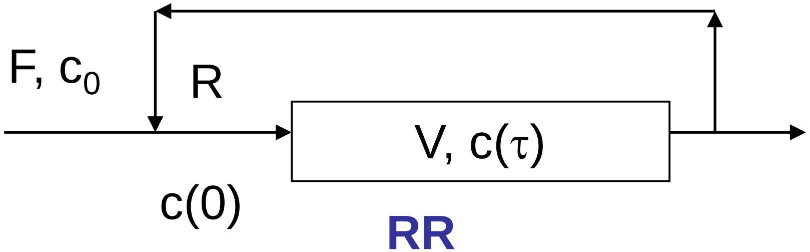
PFR

$Fc - F_0c_0 = Vr(c)$

$c - c_0 = \tau\ r(c)$



CSTR



RR

$d(Fc)/dV = r(c)$

$c(0) = (RF_Vc_V + F_0c_0)/(RF_V+1)$

$(R+1)dc/d\tau = r(c)$

$c(0) = (Rc_V + c_0)/(R+1)$

Note: $\tau = V/F$

Basic Steps in Reactor Design & Analysis: Volume

$d(Fc)/dV = r(c); \; c(0) = c_0$

$dc/d\tau = r(c)$

PFR

$Fc - F_0 c_0 = Vr(c)$

$c - c_0 = \tau \, r(c)$

CSTR

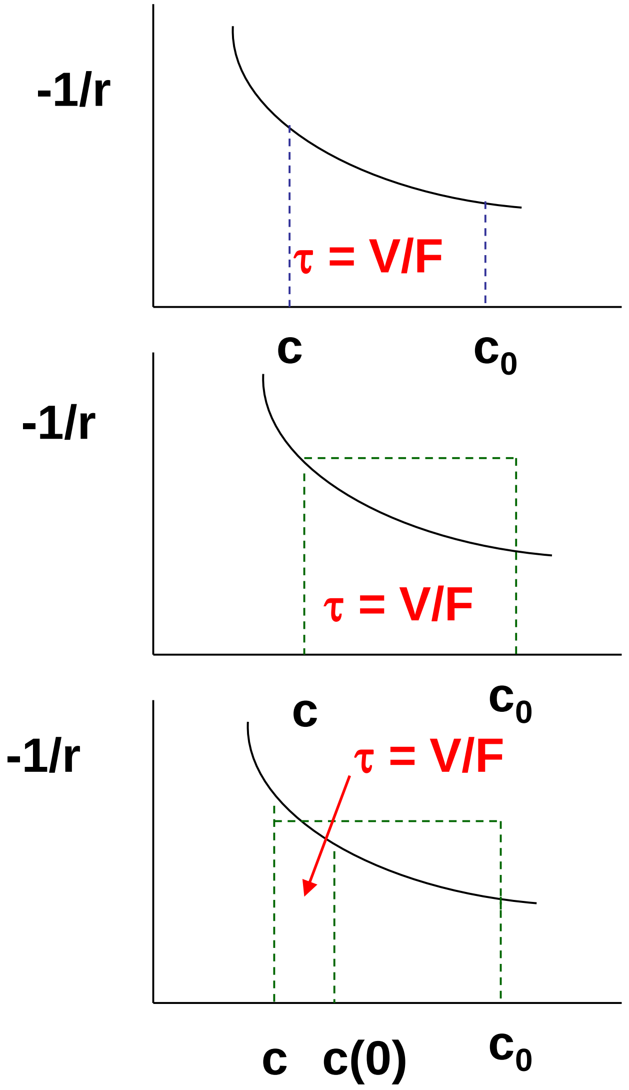
$d(Fc)/dV = r(c)$

$c(0) = (RF_V c_V + F_0 c_0)/(RF_V + 1)$

$(R+1) dc/d\tau = r(c)$

$c(0) = (Rc_V + c_0)/(R+1)$

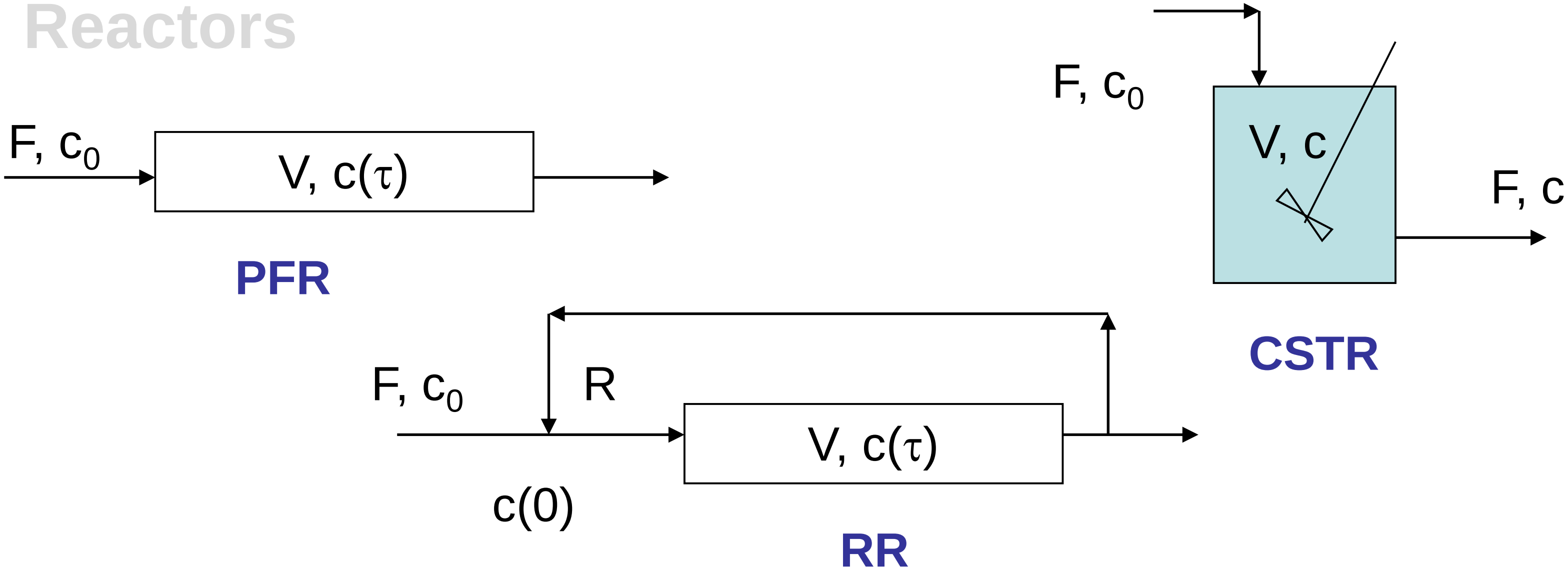
RR



Basic Steps in Reactor Design & Analysis

* Write reactions $A + B = 2C$

*Write simple models for CSTR, Plug-Flow & Recyle-Reactors



***Find data for the reaction** **(conversion, kinetic model, catalyst, …)**
– Depending on the values of F, V, τ, conversion, decide the type of reactor (CSTR, PFR, RR, or network)

* **Verify through simulation**

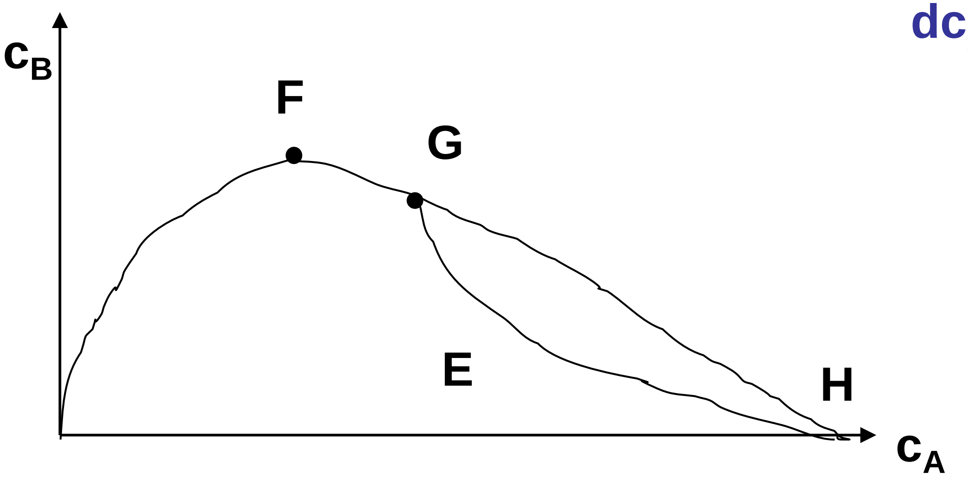
Basic Steps in Reactor Design & Analysis

* **What happens when there are multiple reactions?**

A = B = C series

or, A + B = C; 2A = D parallel

Concept of Attainable region helps to select the reactors and the optimal conversion at the highest selectivity



$dc_A/d\tau = r_A$ $dc_B/d\tau = r_B$

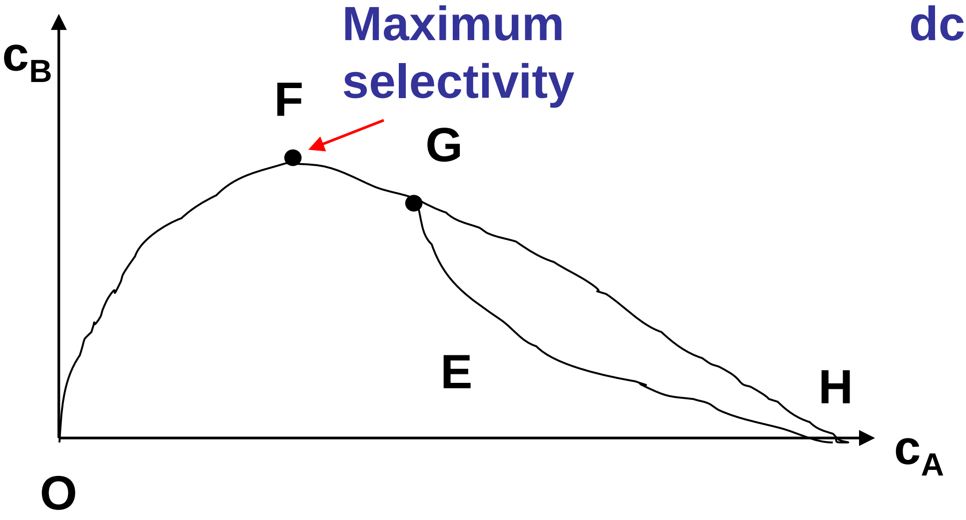
$dc_B/dc_A = r_B/r_A$ for PFR

$(c_B-c_{B0})/(c_A-c_{A0}) = r_B/r_A$ for CSTR

For known r_A & r_B, solve for c_B at different values of c_A to obtain the plot of c_B vs c_A

Basic Steps in Reactor Design & Analysis – Multipe Reactions

Concept of Attainable region helps to select the reactors and the optimal conversion at the highest selectivity



$dc_A/d\tau = r_A$

$dc_B/d\tau = r_B$

$dc_B/dc_A = r_B/r_A$ for PFR

$(c_B - c_{B0})/(c_A - c_{A0}) = r_B/r_A$ for CSTR

For known r_A & r_B, solve for c_B at different values of c_A to obtain the plot of c_B vs c_A

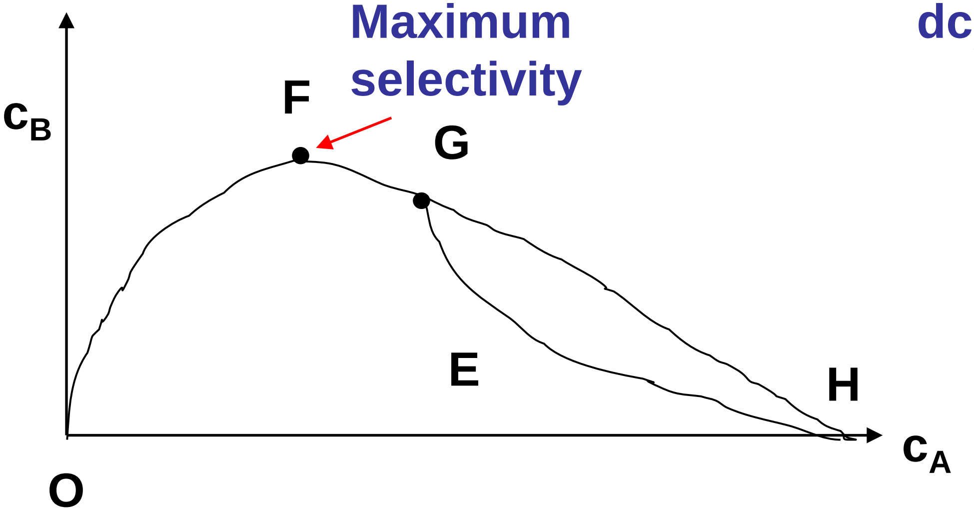
Convex region OFGH

Non-convex region OFGEH

Reactor with non-convex attainable region does not achieve the reaction that is possible or attainable

Basic Steps in Reactor Design & Analysis – Multipe Reactions

Concept of Attainable region helps to select the reactors and the optimal conversion at the highest selectivity



Maximum selectivity

Convex region OFGH

Non-convex region OFGEH

$dc_A/d\tau = r_A$ $dc_B/d\tau = r_B$

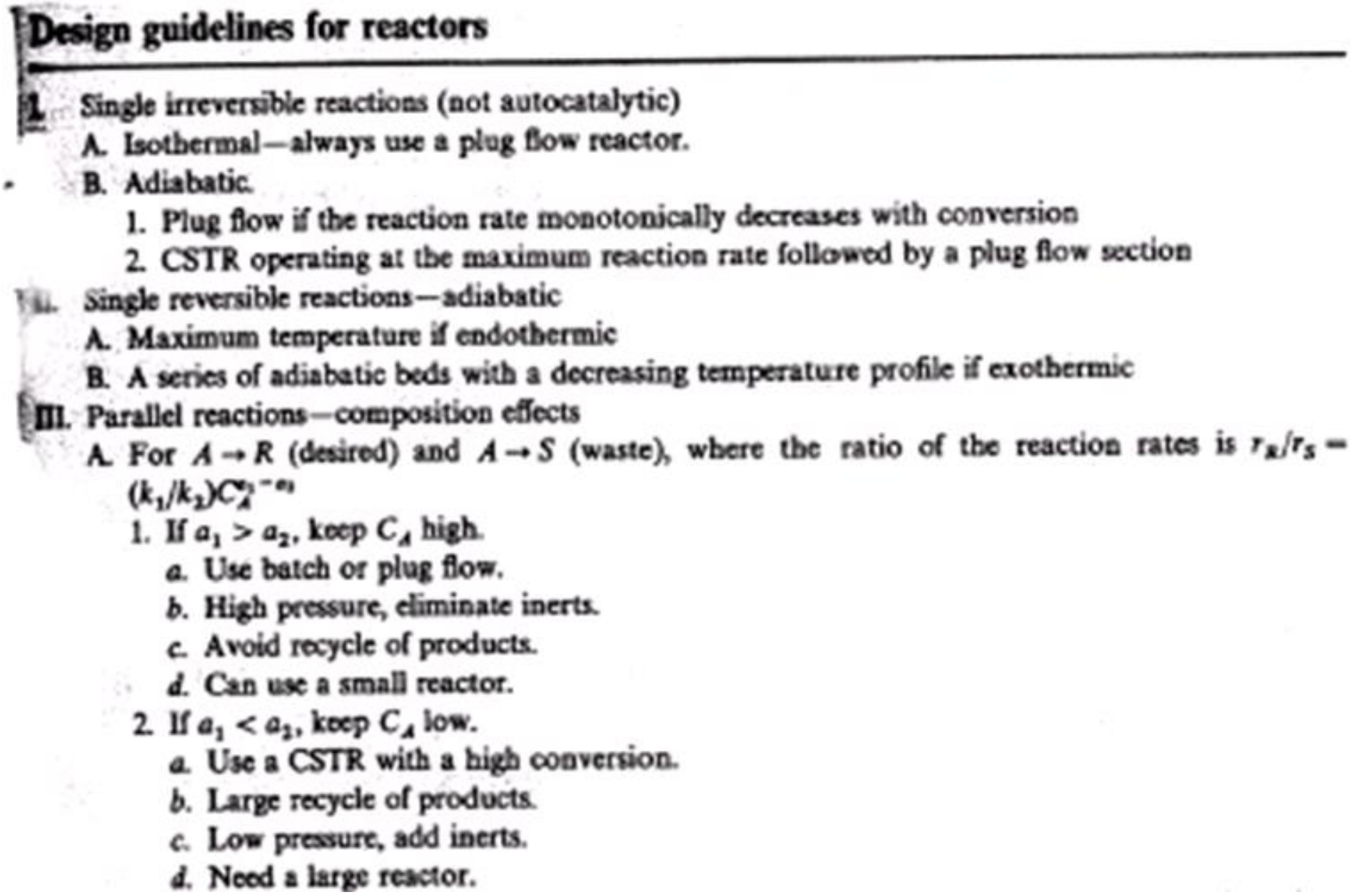
$dc_B/dc_A = r_B/r_A$ for PFR

$(c_B - c_{B0})/(c_A - c_{A0}) = r_B/r_A$ for CSTR

For known r_A & r_B, solve for c_B at different values of c_A to obtain the plot of c_B vs c_A

Reactor with non-convex attainable region (AR) does not achieve the reaction that is possible or attainable – Maximum AR must be convex, reaction vectors cannot point outward from the boundary

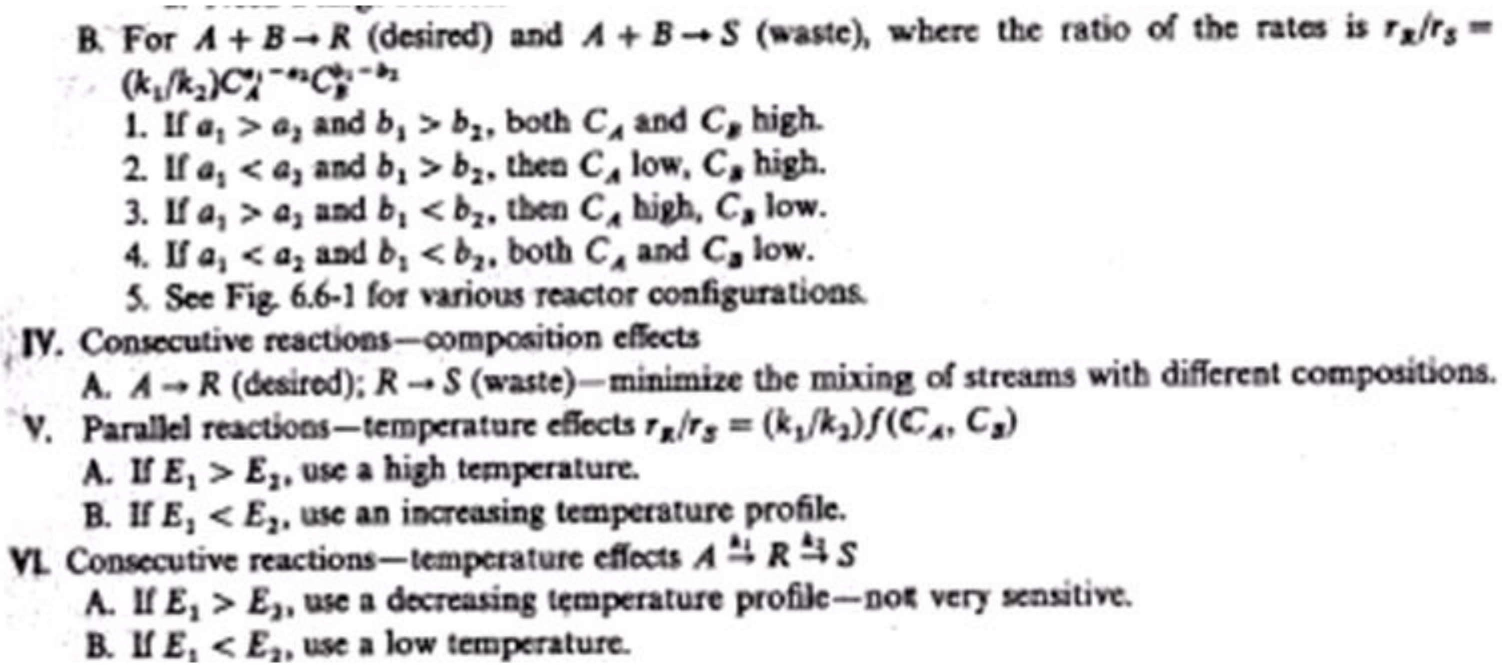
Design Guidelines for Reactors - 1



Design guidelines for reactors

- I. Single irreversible reactions (not autocatalytic)
  - A. Isothermal—always use a plug flow reactor.
  - B. Adiabatic.
    - 1. Plug flow if the reaction rate monotonically decreases with conversion
    - 2. CSTR operating at the maximum reaction rate followed by a plug flow section
- II. Single reversible reactions—adiabatic
  - A. Maximum temperature if endothermic
  - B. A series of adiabatic beds with a decreasing temperature profile if exothermic
- III. Parallel reactions—composition effects
  - A. For $A \rightarrow R$ (desired) and $A \rightarrow S$ (waste), where the ratio of the reaction rates is $r_R/r_S = (k_1/k_2)C_A^{a_1-a_2}$
    - 1. If $a_1 > a_2$, keep C_A high.
      - *a.* Use batch or plug flow.
      - *b.* High pressure, eliminate inerts.
      - *c.* Avoid recycle of products.
      - *d.* Can use a small reactor.
    - 2. If $a_1 < a_2$, keep C_A low.
      - *a.* Use a CSTR with a high conversion.
      - *b.* Large recycle of products.
      - *c.* Low pressure, add inerts.
      - *d.* Need a large reactor.

Design Guidelines for Reactors - 2



B. For $A + B \rightarrow R$ (desired) and $A + B \rightarrow S$ (waste), where the ratio of the rates is $r_R/r_S = (k_1/k_2)C_A^{a_1-a_2}C_B^{b_1-b_2}$

1. If $a_1 > a_2$ and $b_1 > b_2$, both C_A and C_B high.
2. If $a_1 < a_2$ and $b_1 > b_2$, then C_A low, C_B high.
3. If $a_1 > a_2$ and $b_1 < b_2$, then C_A high, C_B low.
4. If $a_1 < a_2$ and $b_1 < b_2$, both C_A and C_B low.
5. See Fig. 6.6-1 for various reactor configurations.

IV. Consecutive reactions—composition effects

A. $A \rightarrow R$ (desired); $R \rightarrow S$ (waste)—minimize the mixing of streams with different compositions.

V. Parallel reactions—temperature effects $r_R/r_S = (k_1/k_2)f(C_A, C_B)$

A. If $E_1 > E_2$, use a high temperature.

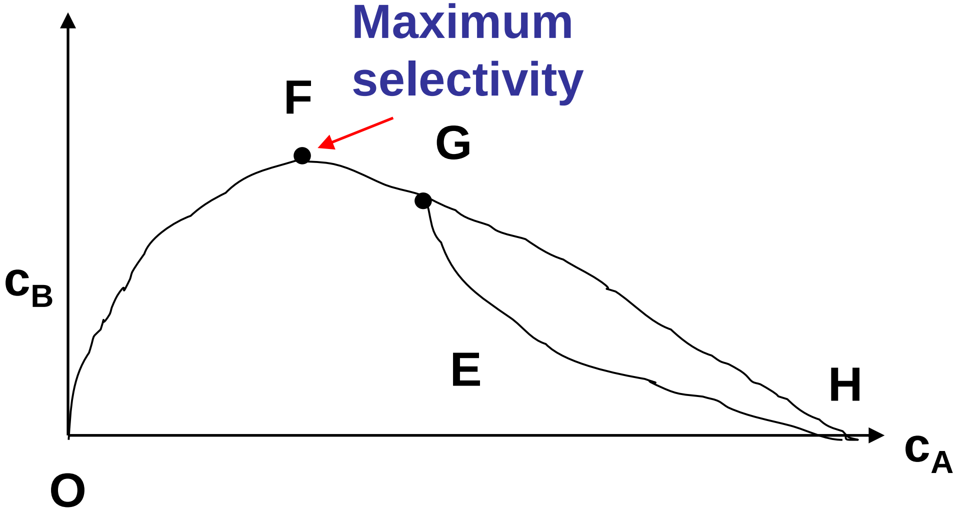
B. If $E_1 < E_2$, use an increasing temperature profile.

VI. Consecutive reactions—temperature effects $A \xrightarrow{k_1} R \xrightarrow{k_2} S$

A. If $E_1 > E_2$, use a decreasing temperature profile—not very sensitive.

B. If $E_1 < E_2$, use a low temperature.

Tutorial Exercise: Analysis with AR-concept



$dc_A/d\tau = r_A$ $dc_B/d\tau = r_B$

$dc_B/dc_A = r_B/r_A$ for PFR

$(c_B - c_{B0})/(c_A - c_{A0}) = r_B/r_A$ for CSTR

For known r_A & r_B, solve for c_B at different values of c_A to obtain the plot of c_B vs c_A

Consider the following reaction scheme

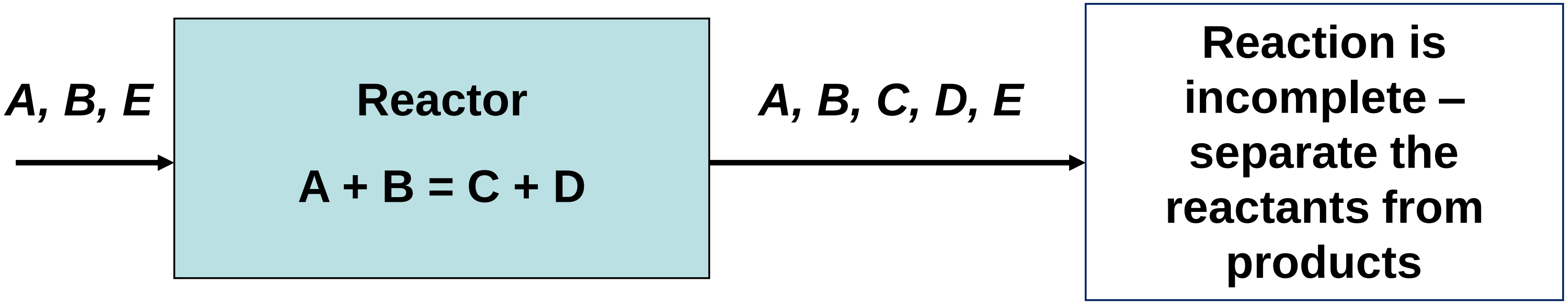
$A \leftrightarrow B \rightarrow C$ *and* $2A \rightarrow D$

$k_{1f} = 0.01; k_{1r} = 5; k_2 = 10, k_3 = 100$

$r_A = -k_{1f}c_A + k_{1r}c_B - k_3(c_A)^2$; $r_B = k_{1f}c_A - k_{1r}c_B - k_2c_B$

Find the reactor composition c_A where the selectivity of B is the highest with a PFR and a CSTR. Then check the residence time – temperature that gives a selectivity as close as the highest. Then work out the remaing design variables

Downstream separation design/analysis



A, B, E

Reactor

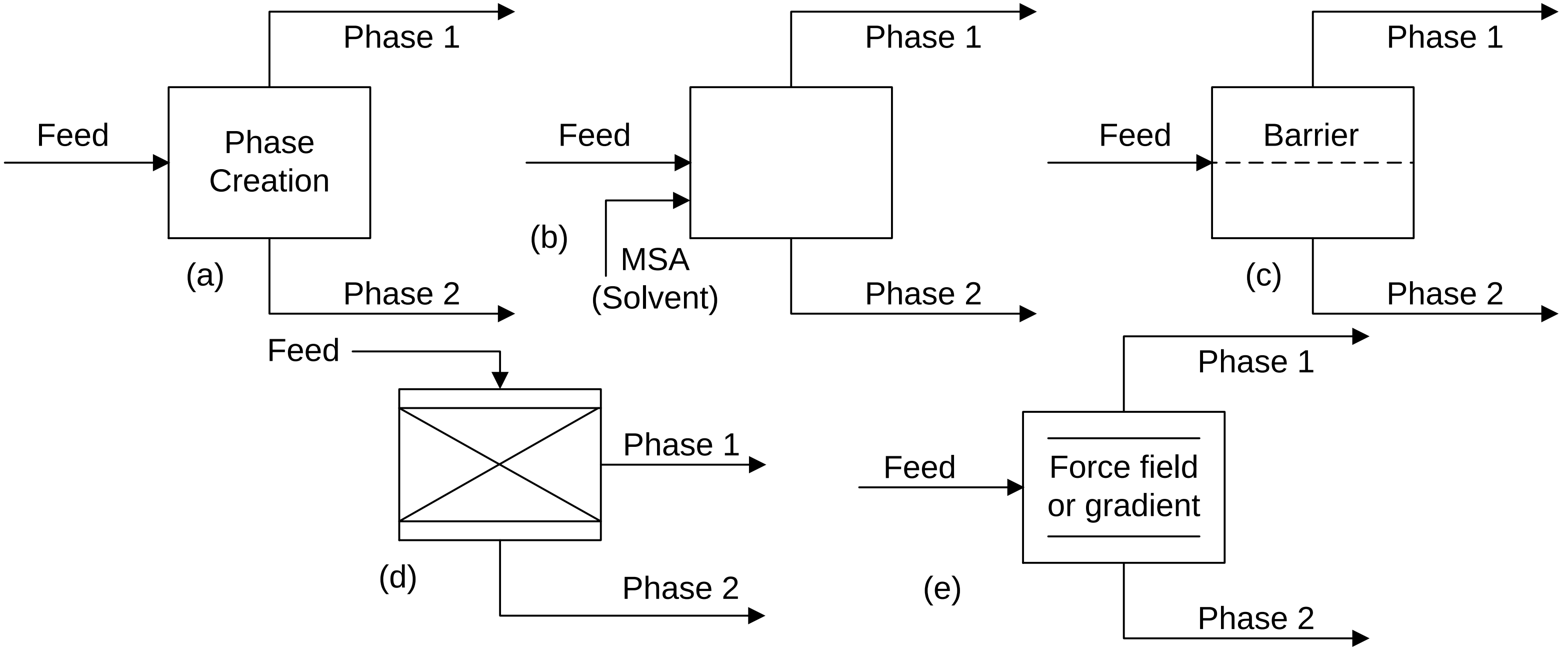
A + B = C + D

A, B, C, D, E

Reaction is incomplete – separate the reactants from products

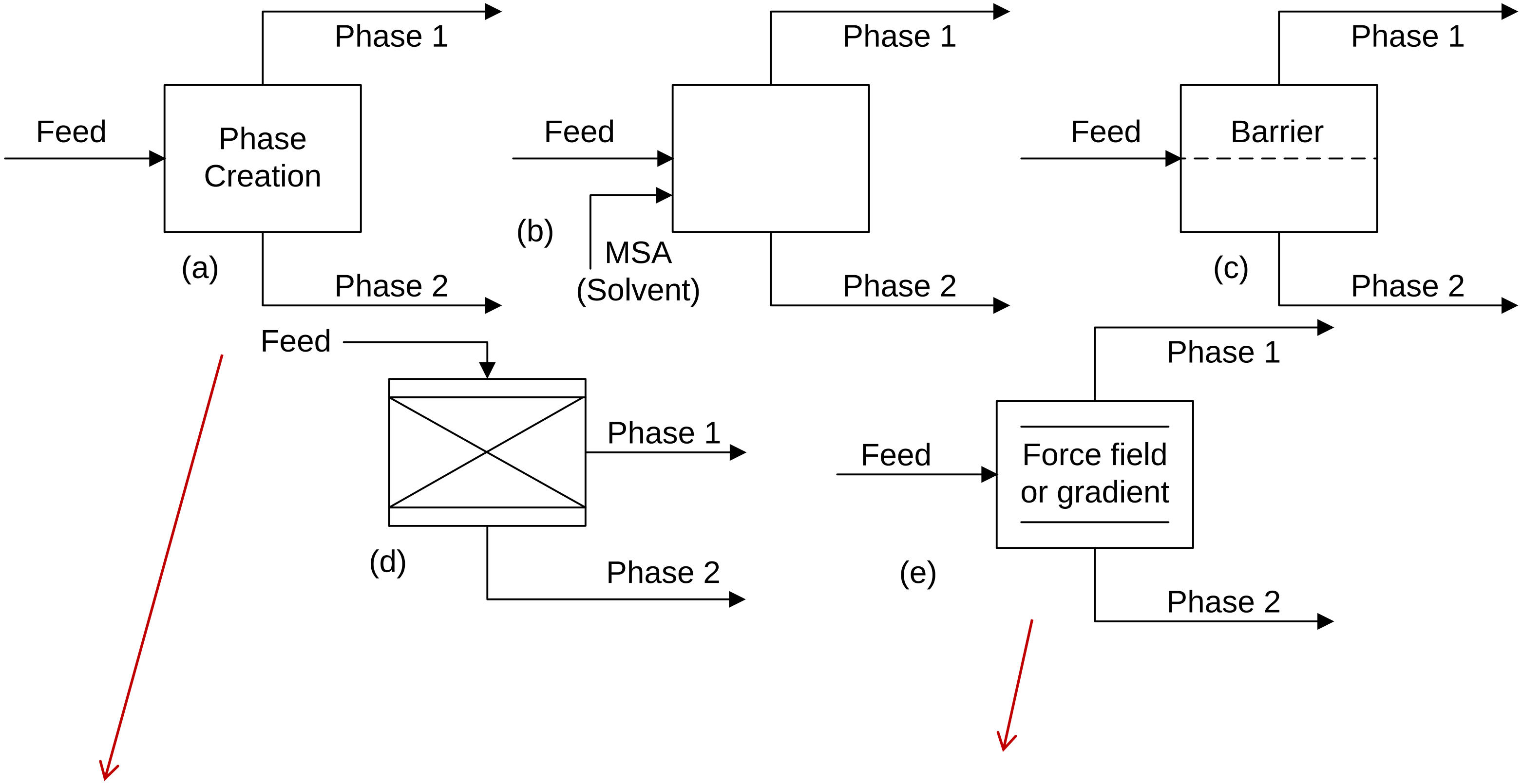
Design Issues: Type of separator; number of separators; separator design parameters (temperature, pressure, separation factor, equilibrium, volume, residence time, ….)

Mechanisms of Separation



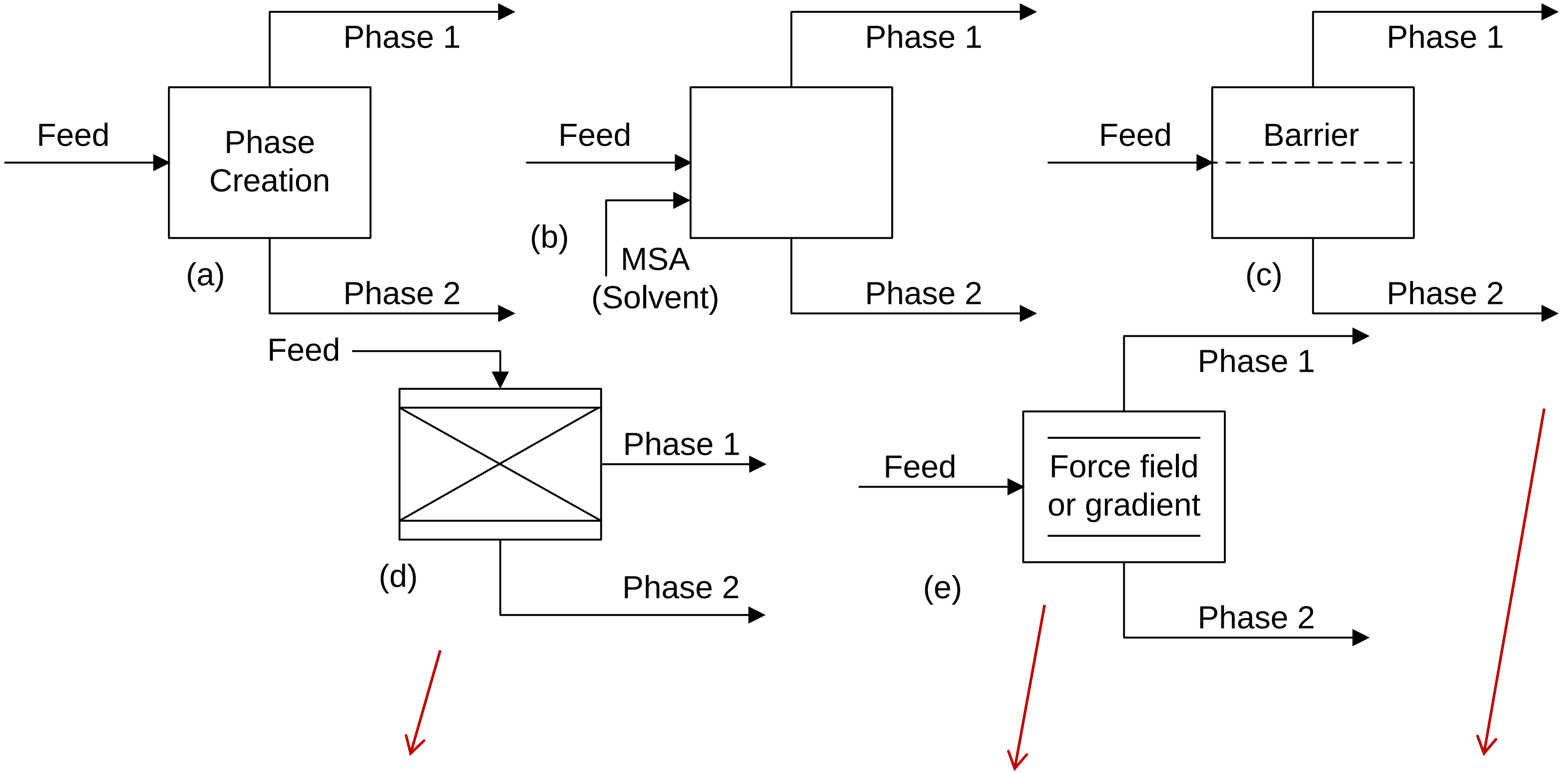
a) Phase Creation; b) Agent-based separation; c) Barrier; d) Solid agent; e) Force field or Gradient

Easy Separations

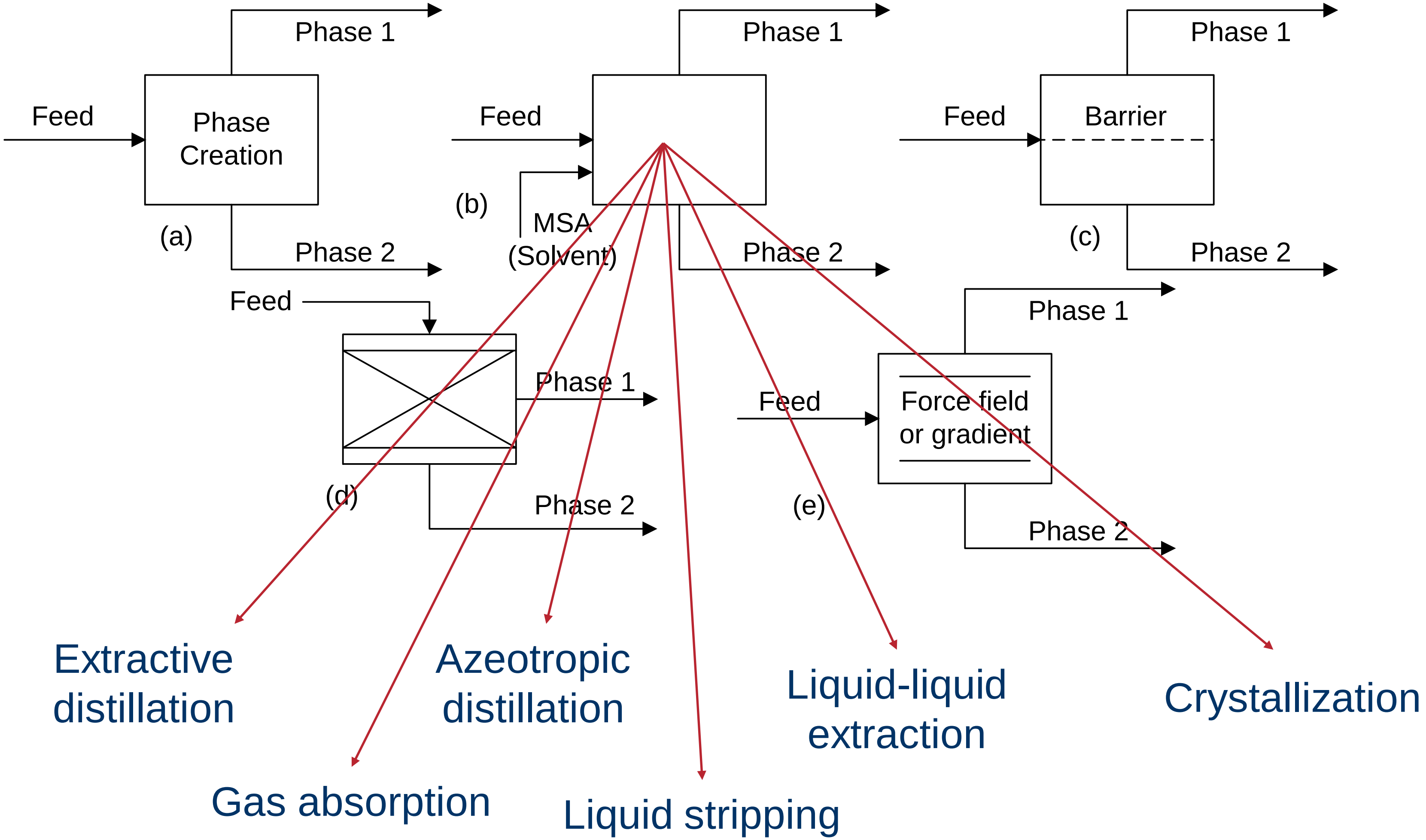


Easy separation: two phases exist at a given T and P and they have different compositions (distillation, evaporation, TP-flash, centrifuge, ...)

Separation difficulties overcome through external medium



External medium aided separation: forces differences in composition in two phases (may not be in phase equilibrium) – filtration, membrane-based separation, adsorption, ion-exchange, ...)



Extractive distillation

Azeotropic distillation

Liquid-liquid extraction

Crystallization

Gas absorption

Liquid stripping

Deciding/selecting the design parameters for simple separations

Design Specifications for single stage separation (VLE)

Isothermal flash (T, P, F, z)

Other problems:

Bubble-point T (P, x)

Dew-point T (T, y)

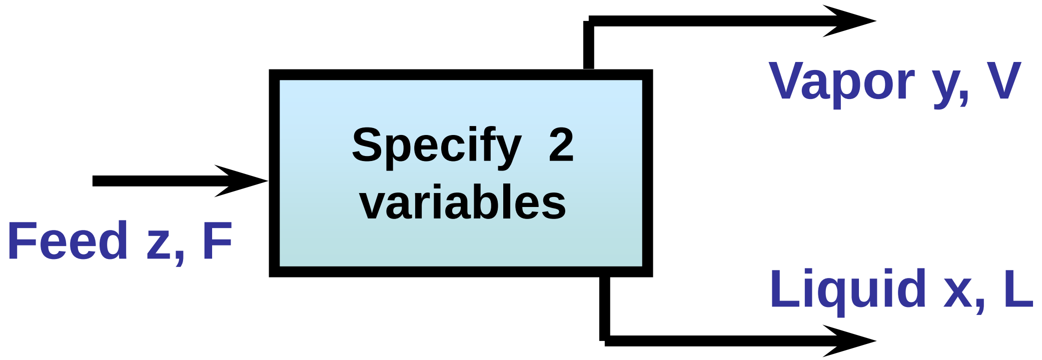
Bubble-point P (T, x)

Dew-point P (T, y)

Adiabatic flash (Q=0, P, F, z)

Non-adiabatic flash (Q, P, F, z)

% Vaporization (V/F, P)



Vapor y, V

Specify 2 variables

Feed z, F

Liquid x, L

Variables in parenthesis
needs to be specified, that is,
these are design decisions

Case 1: ξ_n and P (or T) Fixed ← **Hand calculation**

a. For a specified ξ_n and P (or T), guess T (or P).

b. Calculate K_k, $\alpha_{k/n}$ at specified T.

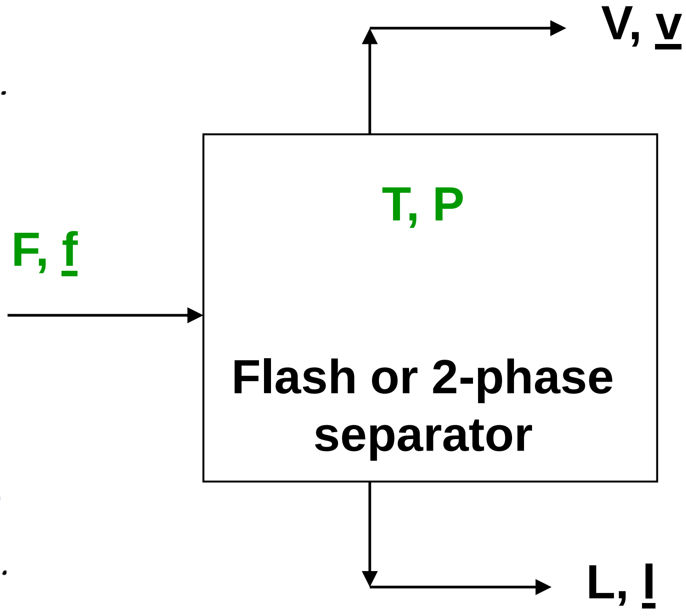
c. Evaluate $\xi_k = \alpha_{k/n}\,\xi_n/(1 + (\alpha_{k/n} - 1)\xi_n)$ for each component k.

d. Reconstruct a mass balance and calculate mole fractions.

$$v_k = \xi_k f_k \qquad y_k = v_k/\sum v_i$$
$$l_k = (1 - \xi_k) f_k \qquad x_k = l_k/\sum l_i$$

e. For T fixed, $P = \dfrac{\bar{\alpha}}{\alpha_{k/n}} P_k^0(T)$.

For P fixed, solve for T from $P_k^0(T) = \alpha_{k/n} P/\alpha$.


V, $\underline{v}$

F, $\underline{f}$

T, P

Flash or 2-phase separator

L, $\underline{l}$

Case 2: T and P Fixed ← **Using software**

a. For a specified T and P, pick a key component n and guess ξ_n.

Follow steps b, c, and d of algorithm for Case 1.

e. If the bubble point equation is satisfied: $\alpha = P\alpha_{k/n}/P_k^0$, stop. Otherwise, reguess ξ_n, and go to step c. (Simple iterative methods, such as the secant algorithm in Chapter 8, can be used to obtain convergence for ξ_n.)

Case 3: ϕ and P (or T) Fixed

a. For a specified $\phi = V/F$ and P (or T)

b. Guess T (or P), calculate $\alpha_{k/n}$, K_k and define $\theta = K_n\,\phi/(1 - \phi) = v_n/l_n$

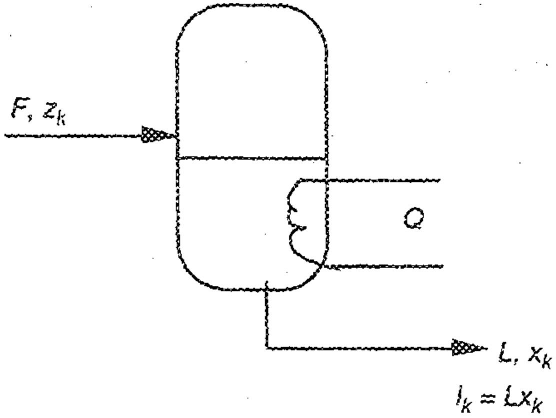
Define $\xi_n = \theta/(1 + \theta)$.

Then follow steps c and d of the previous algorithm.

e. If the bubble point equation is satisfied: $\alpha = P\alpha_{k/n}/P_k^0$, stop. Otherwise, reguess T (or P), and go to step b. (Simple iterative methods, such as the secant algorithm can be used to obtain convergence for ξ_n.)

Specify F, $\underline{f}$ and any two of T, P, V/F, v_k/f_k

See example 3.2



Bubble Point Calculation

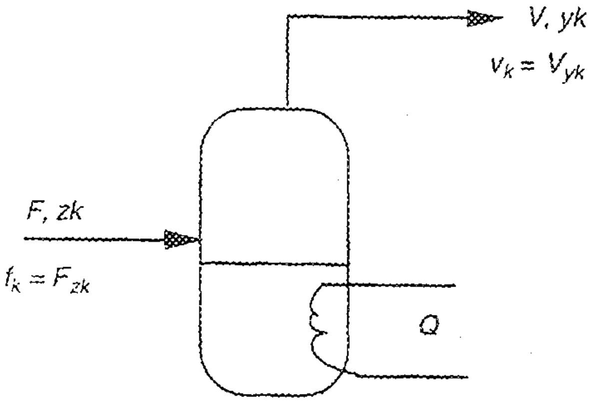
Here $\xi_k = 0$, $\ell_k = f_k$ and $x_k = z_k$

For P fixed, calculate T directly from $P_n^0(T) = P/\bar{\alpha}_n$

For T fixed, calculate P from $P = \bar{\alpha}_n P_n^0(T)$

In both cases, n is chosen as the most abundant component.

Dew Point Calculation



Here $\xi_k = 1$, $v_k = f_k$ and $y_k = z_k$.

For this case, we derive a dew point equation based on: $y_k = z_k$

$$\text{Here } \sum x_k = 1 \Rightarrow K_n \sum \frac{y_k}{K_k} = \sum \frac{y_k}{\alpha_{k/n}} = K_n$$

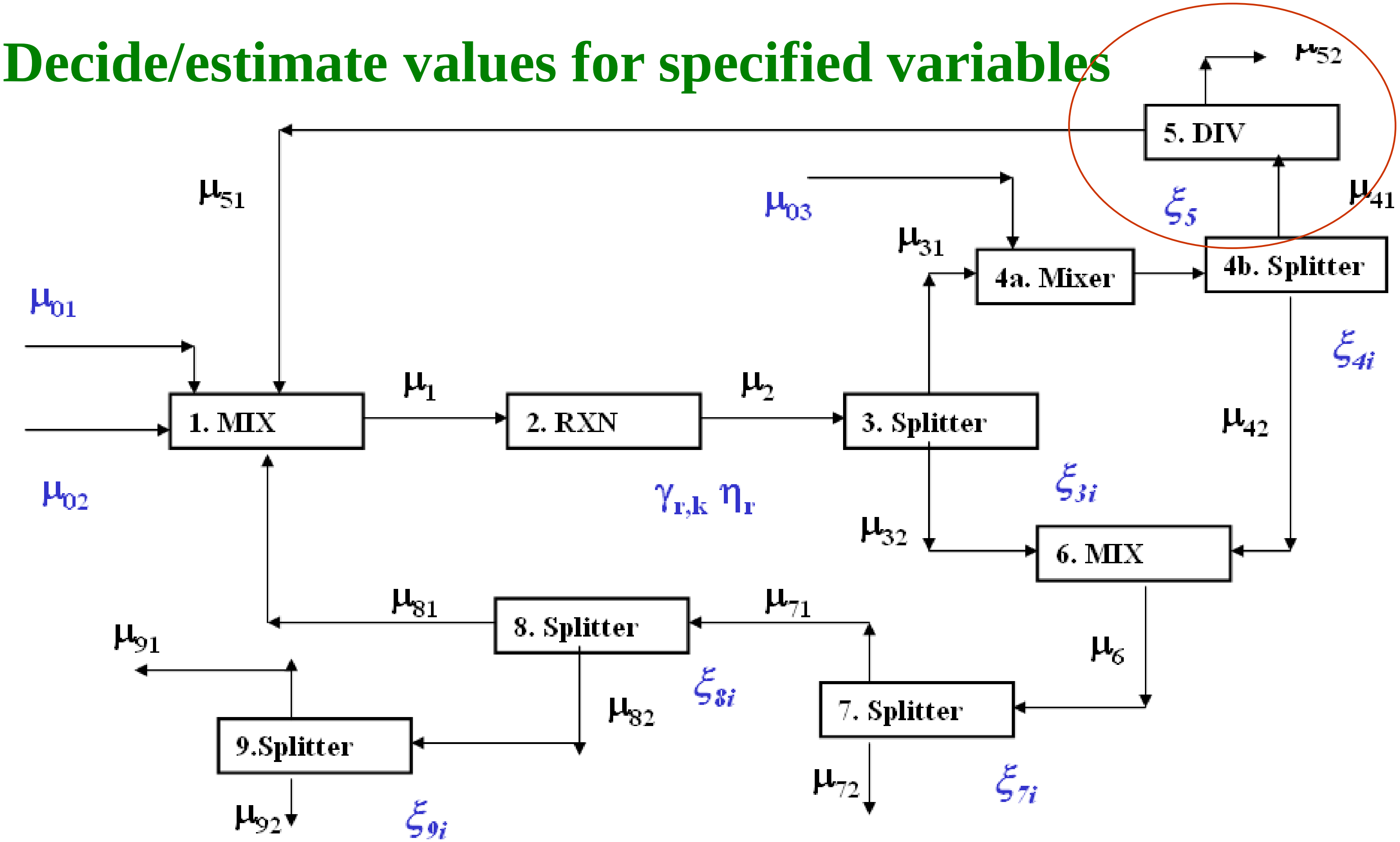
Select as $k = n$ the most abundant vapor component. Then $\sum \frac{y_k}{\alpha_{k/n}} = \frac{P_n^0}{P}$ and:

For T fixed $\quad P = P_n^0(T) / \left(\sum \frac{y_k}{\alpha_{k/n}} \right)$

For P fixed $\quad P_n^0(T) = P \left(\sum \frac{y_k}{\alpha_{k/n}} \right)$ and solve directly for T

Use PROII or ICAS

4a. Decide/estimate values for specified variables



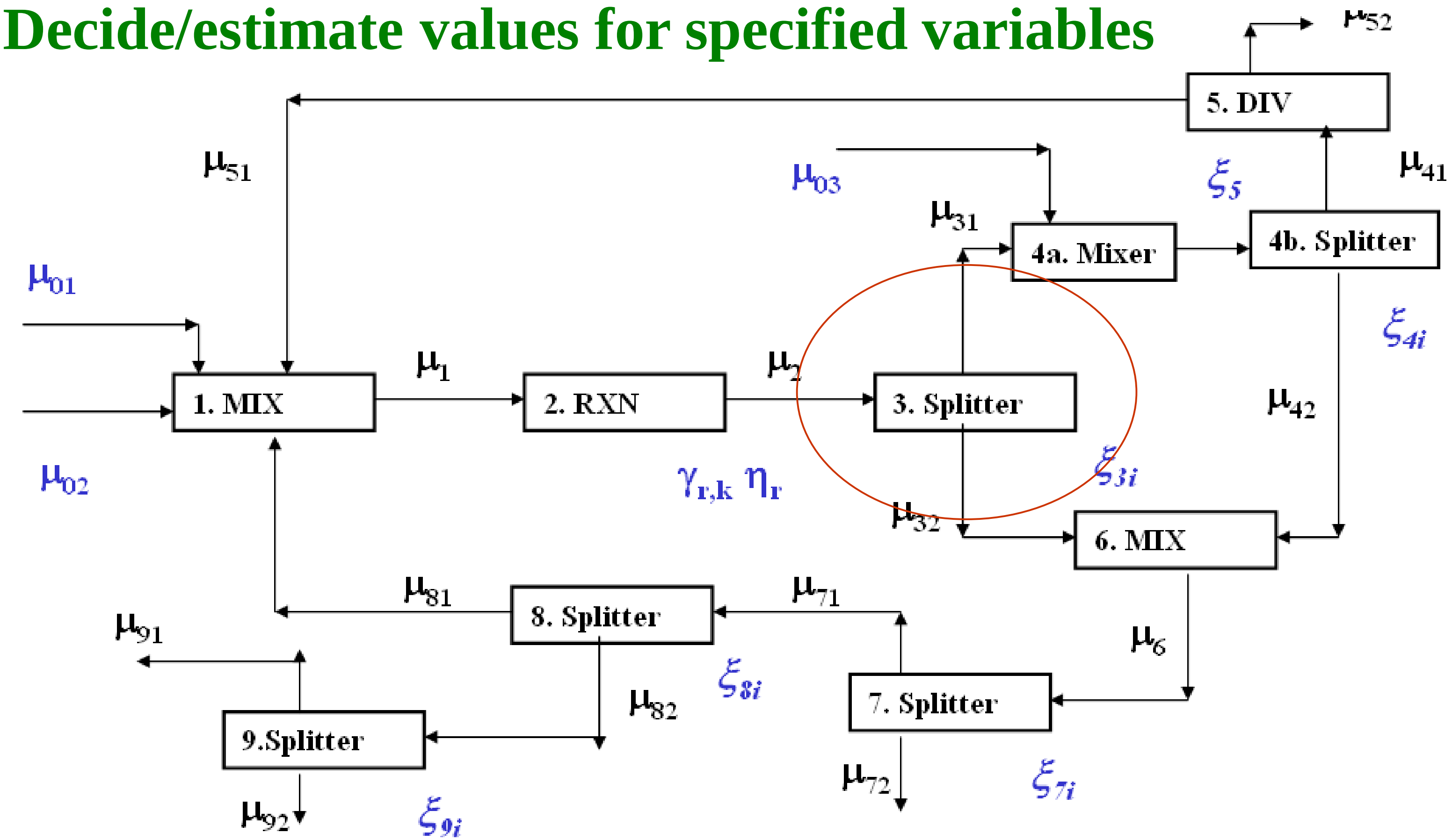
Divider Model

$$\mu_{51} = (1 - \xi_5)\,\mu_{41}$$

$$\mu_{52} = \xi_5\ \mu_{41}$$

- **Value of ξ_5 effects the recycle flow μ_{51}**
- **Select a value between 0 – 0.1 (as an initial estimate**

4b. Decide/estimate values for specified variables



Component Splitter model (for component k)

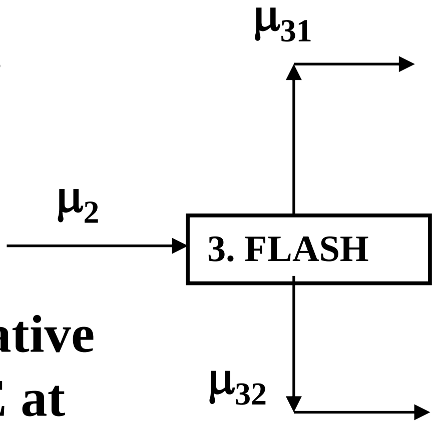
$\mu^k_{31} = \xi_{3k} \mu^k_2$; vapor (or light product)

$\mu^k_{32} = (1 - \xi_{3k})\mu^k_2$; liquid (or heavy product)

- **Use the method given in the book**
- **Perform a quick single flash simulation**

4c. Specification/decisions for a flash unit (ethanol case study)

2. Calculate by using a vapor pressure model at specified T



Vapor at dew point

1. Assume/decide

P_{flash} = 68.5 bar

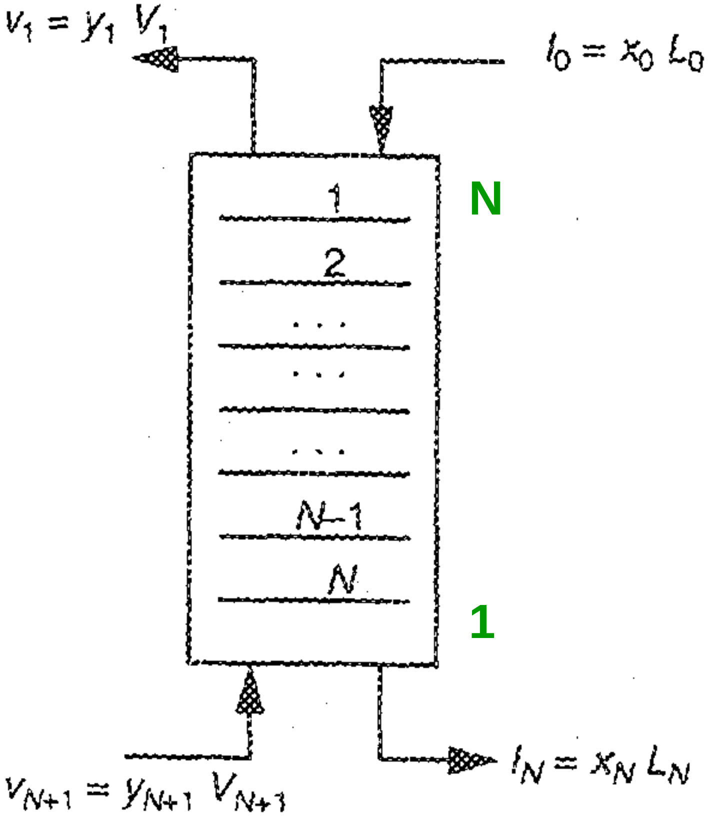
Liquid at bubble point

3. Calculate by relative volatility w.r.t DEE at specified T = P^0_k/P^0_{DEE}

	M	EL	PL	DEE	EA	IPA	W
$P^0(T=310\ K)$	2.1×10^5	5.5×10^4	11360	824	114.5	75.1	47.1
$\alpha_{k/DEE}$	256.1	67.3	13.8	1.0	0.138	0.091	0.057
ξ_{3K}	0.996	0.985	0.932	0.5	0.121	0.083	0.054

4. Calculate Use the formula given in the book ξ_{3K}

Deciding/selecting the design parameters for absorbers



Mass balance model for absorber/stripper has 4 degrees of freedom: P, T, key component recovery and liquid rate

That is, the designer needs to select values for the 4 variables and values of all other variables can be calculated if the component separation (split) factors are known

Absorber and Stripper Preliminary Calculations

Absorption Algorithm

1. Select key component n, fix recovery (typically, $r = 0.99$) fix P and solvent temperature.
2. Calculate L_0 from

$$A_E = \frac{L_0}{V_{N+1}} K_n = 1.4$$

$$L_0 = 1.4 V_{N+1} \boxed{\frac{P_N^0(T)}{P}}$$

Not correct

Note from this expression that L_0 decreases with increasing pressure and decreasing temperature.

3. a. Calculate the number of stages from the Kremser equation:

$$N = \ell n \left(\frac{r v_{N+1}^n + \ell_0^n - A_E v_{N+1}^n}{\ell_0^n - A_E (1 - r) v_{N+1}^n} \right) / \ell n \{A_E\}$$

(Note that if $r = 0.99$ and $\ell_0^n = 0$ then $N = 10$)

b. Prepare the mass balance by calculating absorption factors and aggregate terms for all of the remaining components by:

$$A^k = \frac{L_0}{V_{N+1}} \frac{P}{P_k^0(T)} \quad k \neq n$$

or $$A^k = \frac{1.4}{\alpha_{k/n}}$$

and for β_N^k, β_{N-1}^k with $\beta_N^k = [1 - (A^k)^{N+1}] / (1 - A^k)$

4. Complete the mass balance for all components:

$$v_1^k = \frac{v_{N+1}^k}{\beta_N^k} + \frac{\beta_{N-1}^k}{\beta_N^k}\ell_0^k$$

$$\ell_N^k = \left(1-\frac{\beta_{N-1}^k}{\beta_N^k}\right)\ell_0^k + \left(1-\frac{1}{\beta_N^k}\right)v_{N+1}^k$$

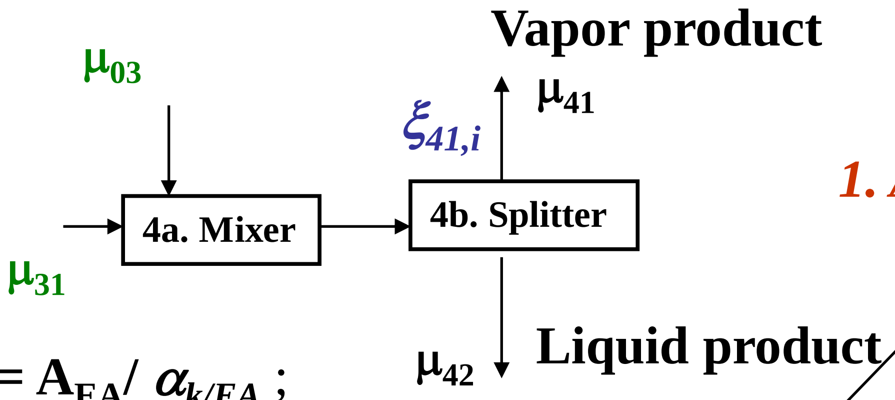
5. If necessary, readjust P or T and return to step 1 under the following conditions
   - a. If the temperature of ℓ_N is too high (check with the bubble point equation), increase L_0. If the final design has significant temperature changes between the top and bottom of the column, use an effective absorption factor calculated with the Edmister equation.
   - b. If too much solvent vaporizes in v_1, increase P or decrease T.
   - c. If too many undesirable components are absorbed, increase T, decrease P, or select a more suitable solvent for absorption.

Note: $\xi_{A1,k} = v_{1,k}/v_{N+1,k} = 1/\beta_{N-1,k} = 1 - \xi_{A2,k}$; **top product**

$\xi_{A2,k} = l_{N,k}/v_{N+1,k} = 1 - (1/\beta_{N,k}) = 1 - \xi_{A1,k}$; **bottom product**

4d. Specification/decisions for an absorber

2. Calculate by relative volatility w.r.t EA at specified T = P^0_k/P^0_{DEE}



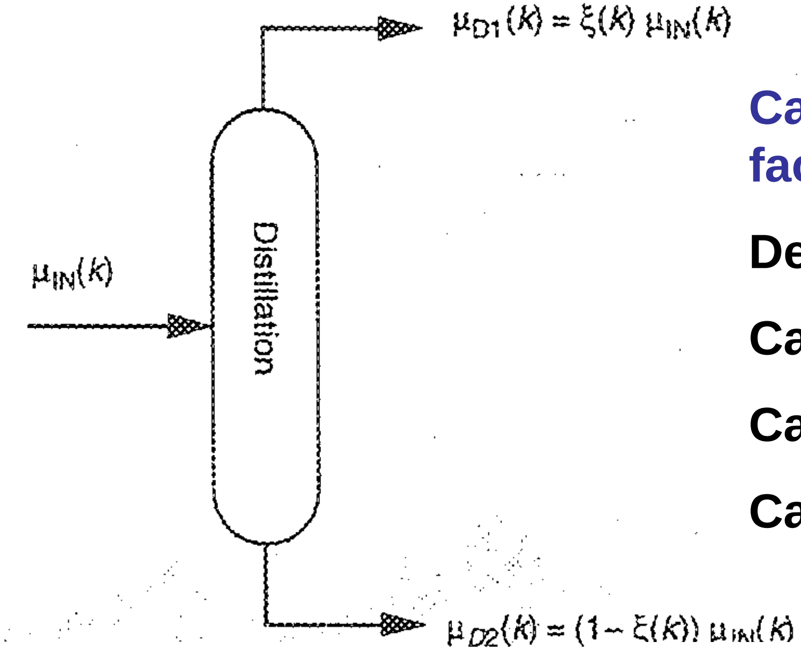
1. Assume/decide

3. Calculate $A_k = A_{EA}/\alpha_{k/EA}$;
then $\beta_{N,k}$ & $\beta_{N-1,k}$ (see page 88)

	M	EL	PL	DEE	EA	IPA	W
$\alpha_{k/EA}$	1854	486.3	99.5	7.24	1.0	0.79	0.41
A_k	0.0054	0.021	0.101	1.38	10	12.66	24.4
$\beta_{N,k}$	1	1.021	1.11	4.17	98.92	153.2	529.1
$\beta_{N-1,k}$	1	1.021	1.10	2.30	9.79	12.02	21.6
$\xi_{42,k}$	0	0.021	0.099	0.760	0.99	0.993	0.998

4. Calculate use the formula given in slide 17 for $\xi_{42,k}$

Deciding/selecting the design parameters for simple distillation



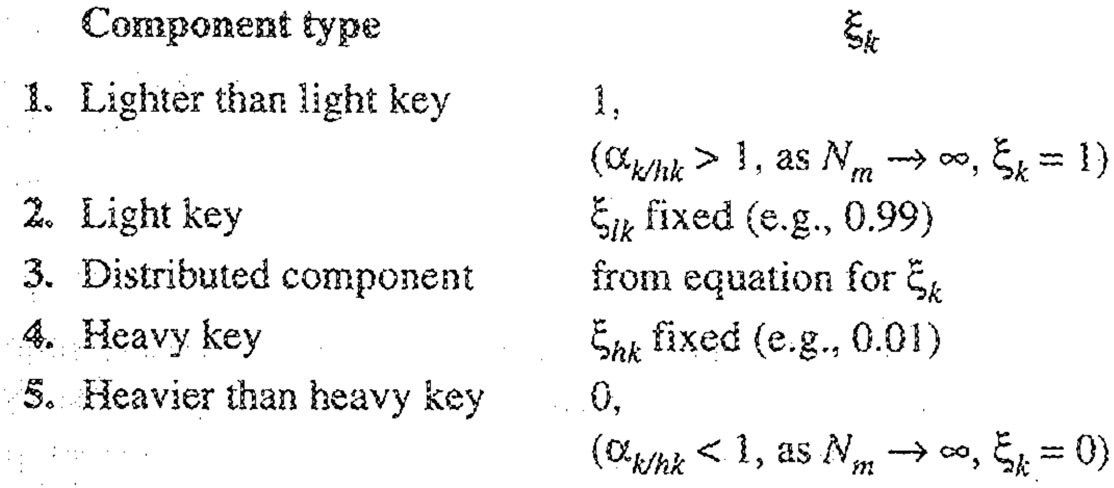
Calculation of component separation factors for distillation columns

Decide on values for $\zeta_k = d_k/f_k$

Calculate $\alpha_{lk/hk}$

Calculate Nm by Fenske Equation

Calculate $\xi_k = (\alpha_k)^{Nm}\,\zeta_{hk} / [1+(\alpha_k)^{Nm}]\zeta_{hk}$



Component type	ξ_k
1. Lighter than light key	1, ($\alpha_{k/hk} > 1$, as $N_m \rightarrow \infty$, $\xi_k = 1$)
2. Light key	ξ_{lk} fixed (e.g., 0.99)
3. Distributed component	from equation for ξ_k
4. Heavy key	ξ_{hk} fixed (e.g., 0.01)
5. Heavier than heavy key	0, ($\alpha_{k/hk} < 1$, as $N_m \rightarrow \infty$, $\xi_k = 0$)

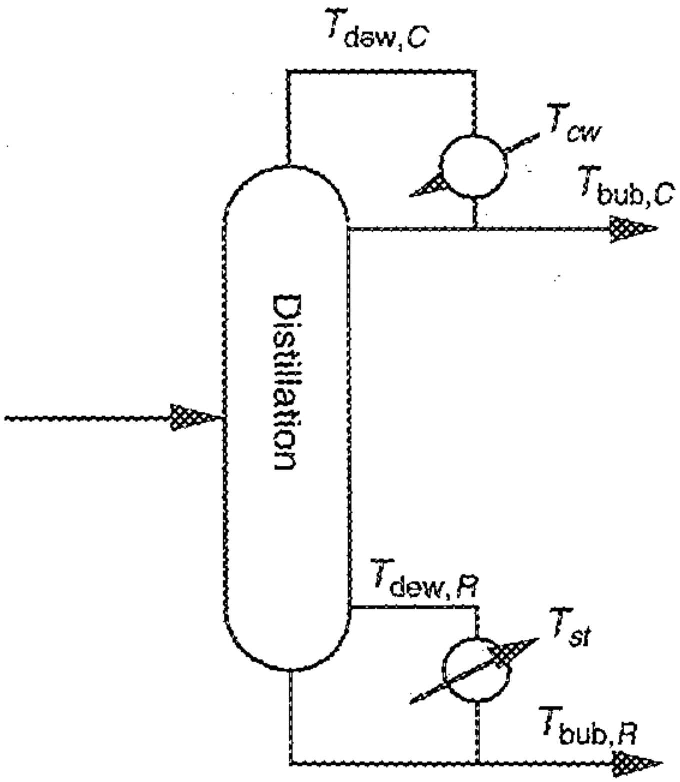


FIGURE 3.13 Setting column pressure and temperature.

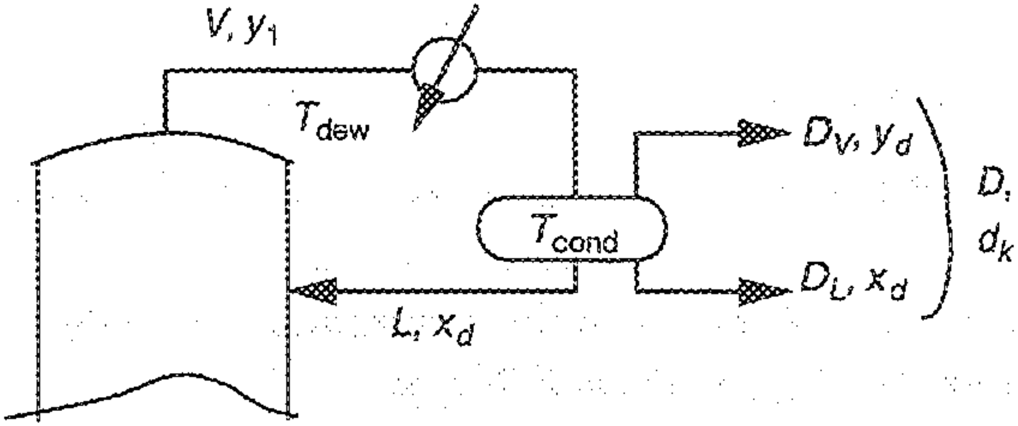


FIGURE 3.14 Partial condenser.

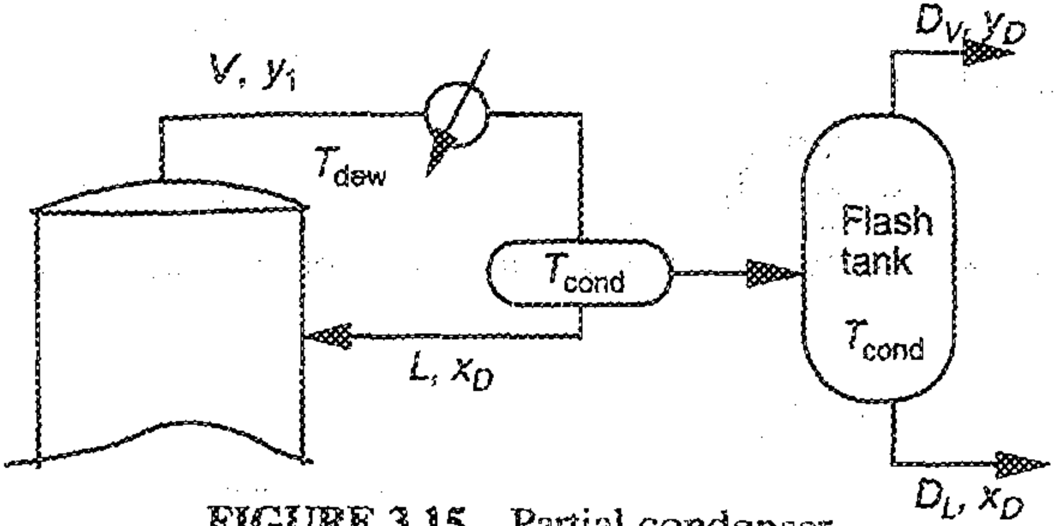


FIGURE 3.15 Partial condenser representation for calculation.

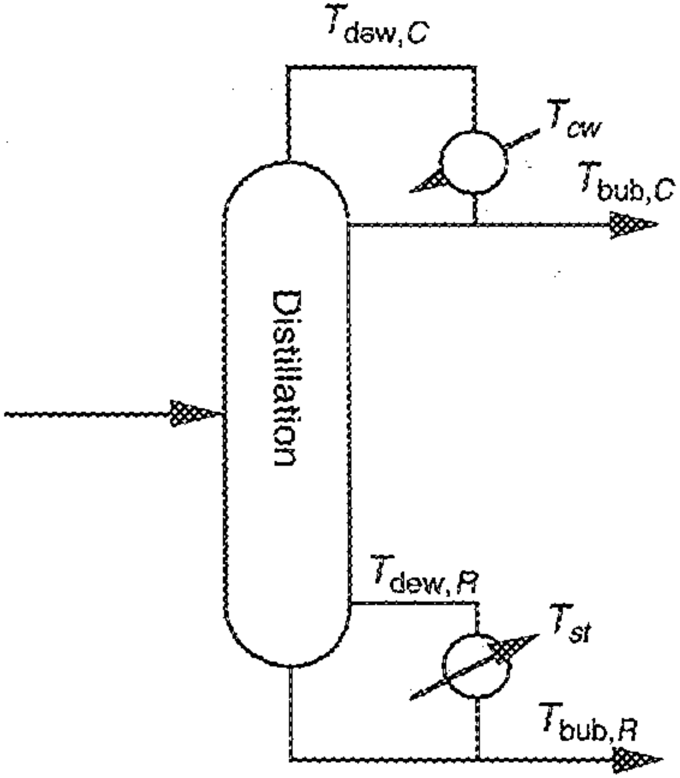
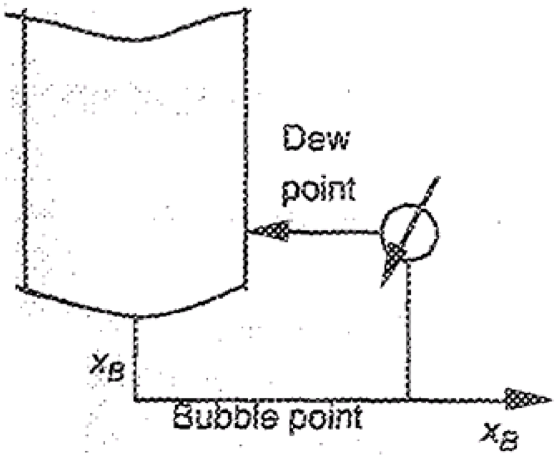
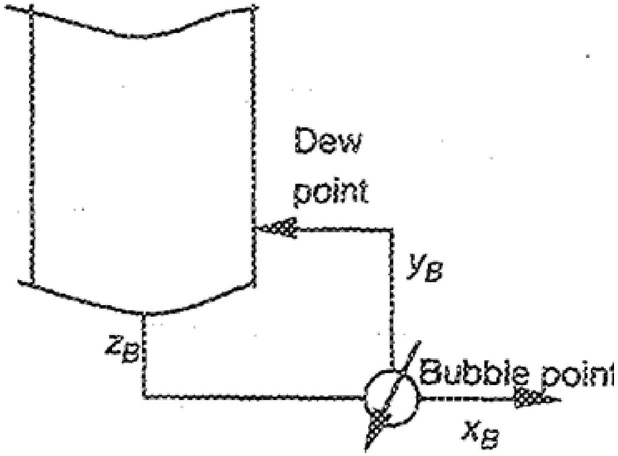


FIGURE 3.13 Setting column pressure and temperature.

FIGURE 3.16 Reboiler configurations.



Total Reboiler



Partial Reboiler

4e. Specification decisions for distillation columns (case study)

	M	EL	PL	DEE	EA	IPA	W	
ξ_k	1.0	1.0	1.0	1.0	0.995	0.96	0.1	7.dist

Recover 99.5% ethanol and remove 90% water

	M	EL	PL	DEE	EA	IPA	W	
ξ_k	1.0	1.0	1.0	0.995	0.005	0.00	0.00	8.dist

Recover 99.5% DEE plus gases at the top and recycle and 99.5% Ethanol at the bottom

---

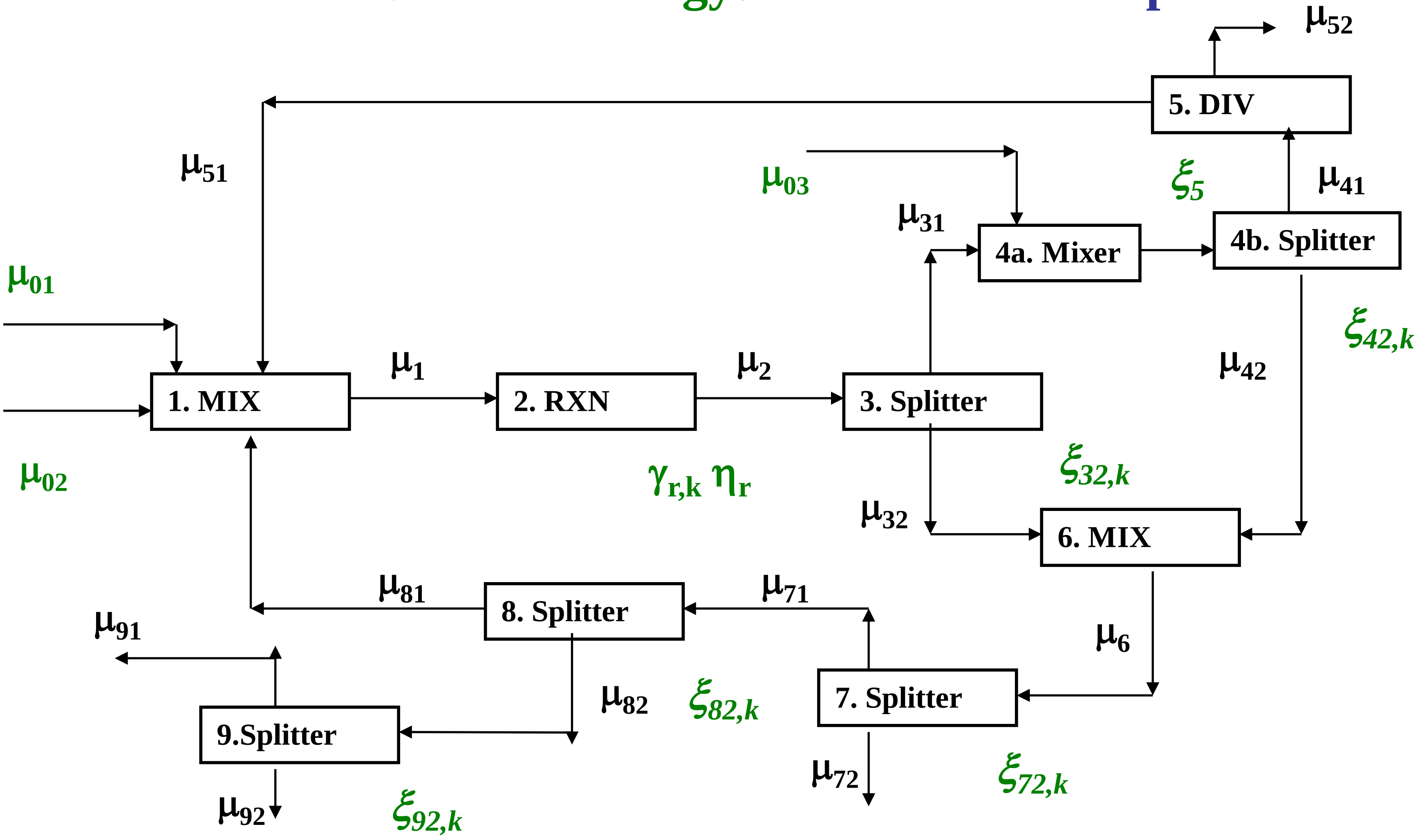
Ethanol-water azeotrope at 85.4% EA & 14.6% W (mole percent)

Recover 99.5 % azeotrope, that is, $\xi_{az} = 0.995$

9.dist

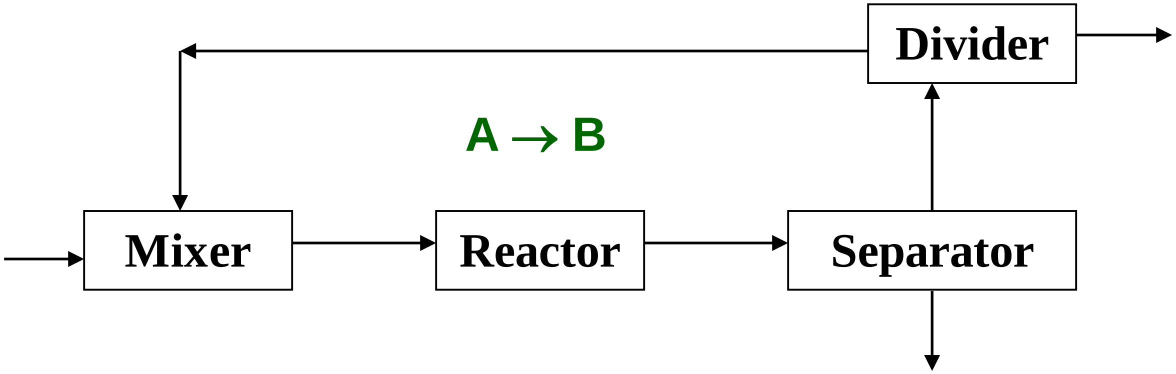
IPA in distillate is 0.1%

5. Determine a calculation strategy: Flowsheet decomposition



How should we solve the MB-model equations? How should we set-up the simulation problem for the simulator? Given, all variables marked in green, calculate all other variables

Design-Analysis Issues with Reactor-Separation-Recyle Systems



$A \rightarrow B$

Assume perfect separation of A from B and C

Conversion of reactants directly related to recycle flow as well as design and operation of the reactor

$D_a - X_A/(1- X_A)[1 - \alpha(1 - X_A)] = 0$

Where, $D_a = kV\rho/F_A \geq 1$

X_A = conversion of A

= (amount of A reacted) / (amount of A entering the reactor)

α is separation factor of A in the separator