

# PERANCANGAN SISTEM/ JARINGAN PEMISAH & RECYCLE

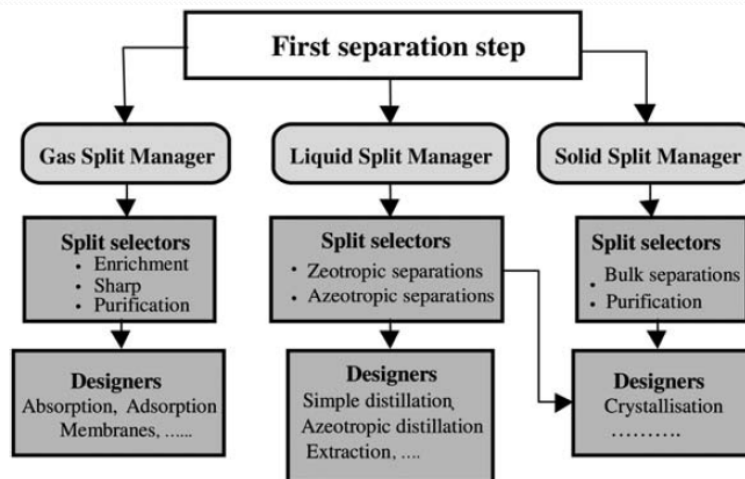
## PERANCANGAN PROSES KIMIA (*CHEMICAL PROCESS DESIGN*)

Section 2 oleh: Dr. Istadi, ST, MT

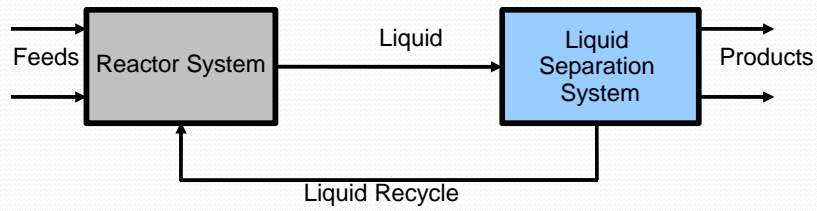
Kode Mata Kuliah : TKK 345  
Beban : 3 SKS

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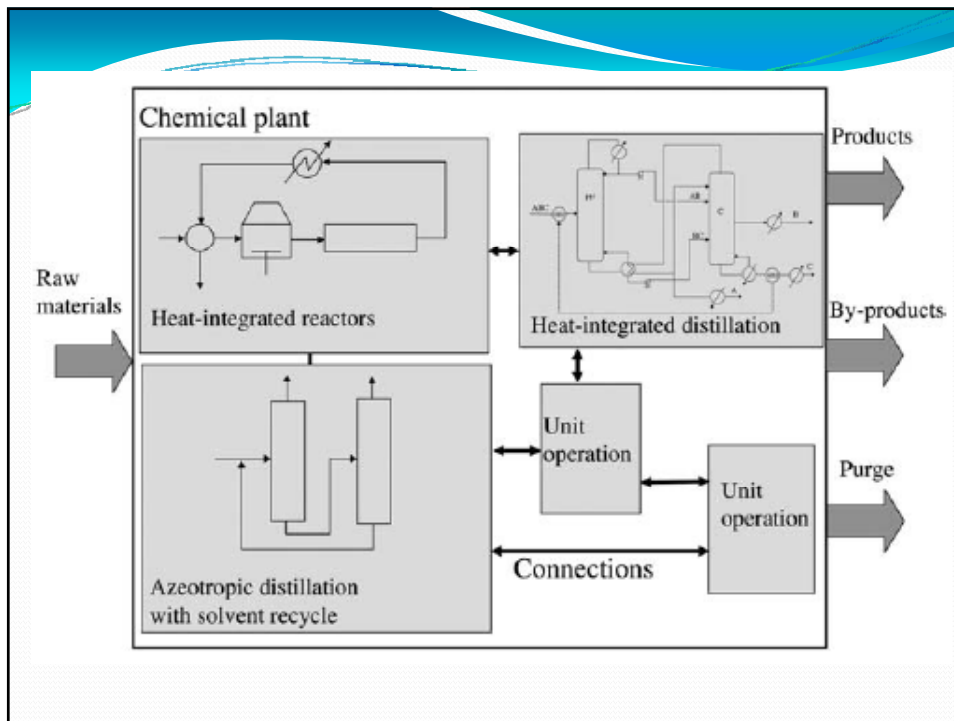
## Separation synthesis hierarchy



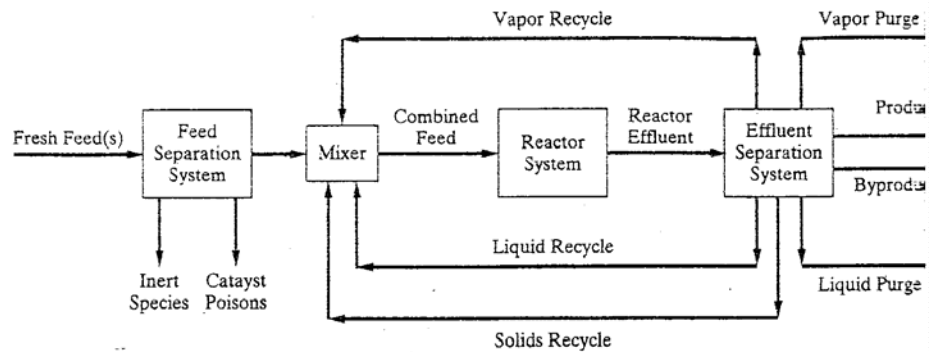
## Basic Configuration of Chemical Process



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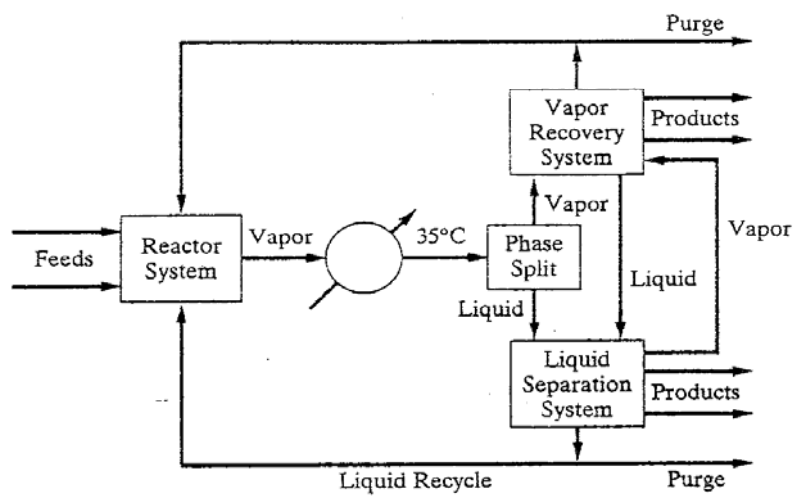


## General Flowsheet for a Separation Process



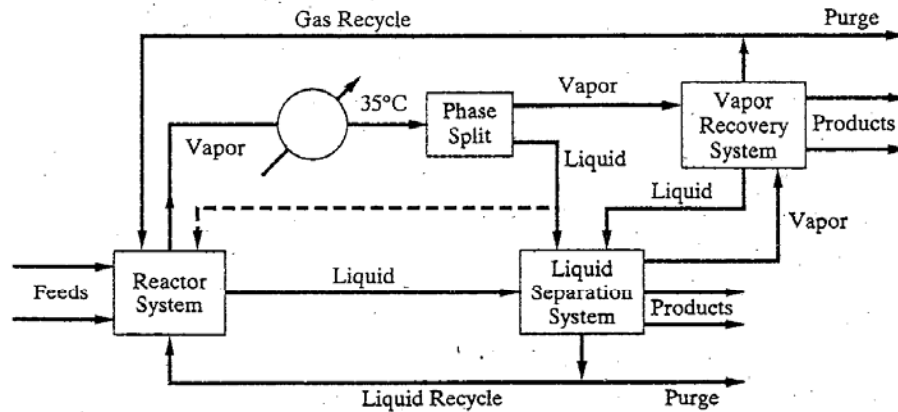
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## Separation of Vapor Reactor Effluents



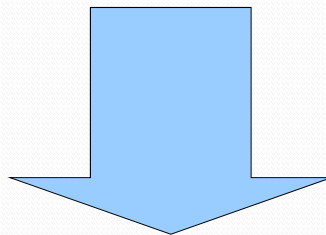
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## Separation of Vapor/Liquid Reactor Effluents



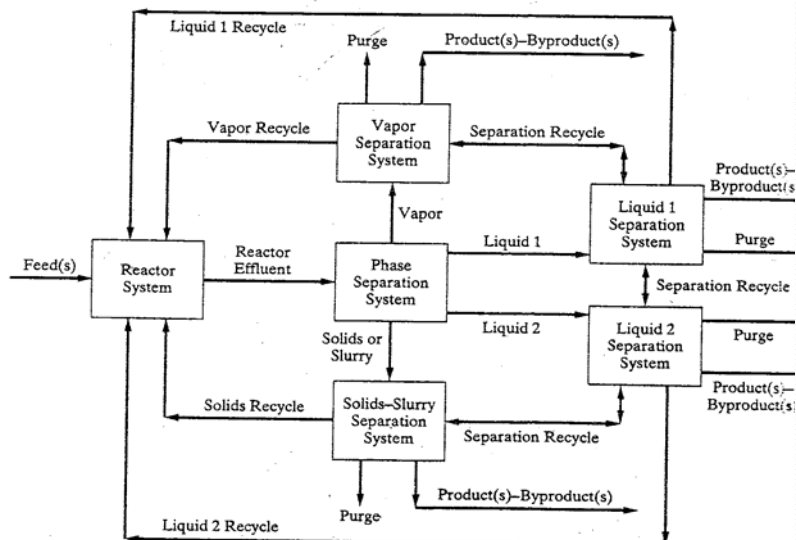
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- Each exiting phase is either:
  - recycled to the reactor
  - purged from the system
  - sent to separate vapor, liquid, or slurry separation systems



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## Separate Separation Systems with Reactor-system and Separation-System Recycles



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## Phase Separation of Reactor Effluent

- **Reactor effluent:** homogeneous phase or heterogeneous phase
- **Homogeneous phase** ==> change Temperature and/or Pressure ==> to obtain partial separation of heterogeneous mixture
- **Three-phase model** considers the possibility that a vapor may also be present, together with two liquid phases
- **If solids are present** with one or two liquid phases, it is not possible to separate completely the solids from the liquid phase(s).
- Instead, a centrifuge or filter is used to deliver a wet cake of solids

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## LIQUID SEPARATION SYSTEM

- The liquid separation system involves one or more of the following separators:
  - distillation and/or enhanced distillation,
  - stripping,
  - liquid-liquid extraction,
  - and so on, with the unreacted chemicals recovered in a liquid phase and recycled to the reaction operation

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## Separation methods for liquid mixtures

Separation method	Characteristic property	Observation
Simple distillation	Relative volatility $\alpha$	Use heuristics for sequencing. Not feasible if $\alpha < 1.1$
Simple and azeotropic distillations	Vapor-pressure variation	Check thermal stability of components
Stripping, L-L extraction	Boiling point	Use stripping and L-L extraction for thermal sensitive components
Melt crystallization	Freezing point	Differences larger than 20°C
Adsorption chromatography	Polarity	Pay attention to adsorbent regeneration
Membrane permeation	Shape and size	Emerging technology
Azeotropic distillation, Extractive distillation, L-L extraction	Chemical family	MSA selection is the main issue. Recycling of MSA increases the costs
L-L extraction, stripping, adsorption, crystallization	Temperature sensitivity	Recycle of MSA increases the costs

## Heuristic for Liquid Separation System

**Table 3.11** General heuristics for separation sequencing of liquid mixtures.

1. Remove first corrosive, hazardous, fouling, reactive and any troublesome components. Consider also in the first place the removal of light-ends.
2. Deliver high-purity products as top distillate. The same is valid for reactants sent to reactors sensitive to impurities.
3. When separation by distillation is feasible, prefer it in a first attempt.
4. Isolate zeotropic and azeotropic mixtures.
5. Perform difficult zeotropic separations later, but before azeotropic separations. Examine other options, such as extractive distillation, L-L extraction, crystallization, adsorption, or molecular sieves.
6. Examine the separation of azeotropic mixtures last.
7. Remove the components in order of decreasing percentage of the feed. This operation will reduce the cost of the next separation.
8. Favor 50/50 splits.

## Factors for Separation Selection

- Phase condition of the feed
- **Separation Factor** (SF)

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

- Reason for Separation
  - purification
  - removal of undesirable components
  - recovery

## Heuristic of Separations

- **Heuristic 9:** Separate liquid mixtures using distillation, stripping, enhanced (extractive, azeotropic, reactive) distillation, liquid-liquid extraction, crystallization, and/or absorption
- **Heuristic 10:** Attempt to condense or partially condense vapor mixtures with cooling water or a refrigerant. Then use Heuristic 9
- **Heuristic 11:** Separate vapor mixtures using partial condensation, cryogenic distillation, absorption, adsorption, membrane separation and/or desublimation

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## Vapor Recovery and Gas - Separation System

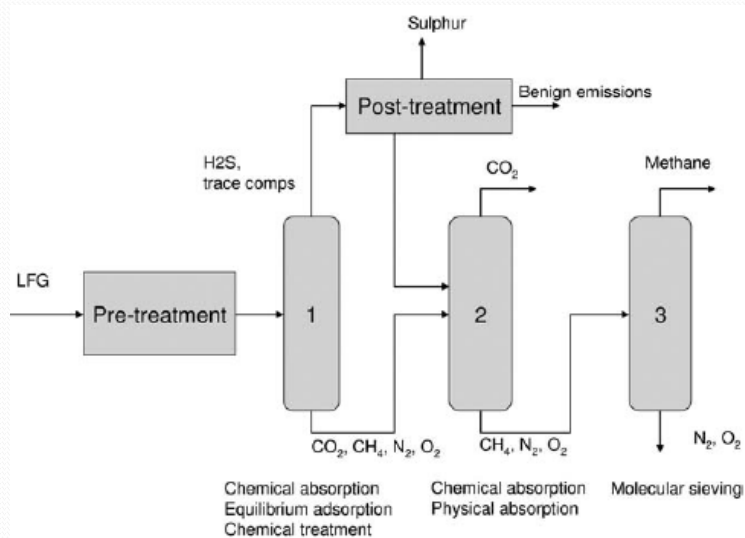
- **on the purge**, if significant amount of product is being lost,
- **on the gas - recycle stream**, if impurities could affect the reactor operation,
- **on the vapor stream after flash**, if both items 1 and 2 are valid,
- does not use vapor recovery if neither item 1 nor 2 are important.

## Method used in Vapor Recovery and Gas Separation

Table 3.2 Methods used in vapor recovery and gas separations.

Method	Characteristic property	Condition	Observation
Condensation	Boiling points Relative volatility	Difference in boiling points $>40^{\circ}\text{C}$ or $\alpha_{ij} > 7$	Optimize pressure and temperature
Cryogenic distillation	Boiling points Relative volatility	$\alpha_{ij} > 2$	Large-scale processes Remove first freezable components
Physical absorption	Solubility	$K_i > 4$	Optimize $P$ and $T$ Recycle the solvent
Chemical absorption	Reactive function as acid or base groups	Reversible process	Optimize the solvent ratio
Molecular sieving	Size/shape	Significant differences	Remove first fouling components
Equilibrium adsorption	Adsorption coefficient	Favorable adsorption	Remove first fouling components
Membrane permeation	Perselectivity	Perselectivity greater than 15	Remove first fouling components
Catalytic oxidation	Chemical family	Impurities below 10% of the flammability point	Danger of dioxine, not for halogenated organics
Catalytic hydrogenation	Chemical family	Components containing double bound	Develop selective catalyst
Chemical treatment	Chemical family	Selective reaction	Dry treatment preferred Recovery of chemical agent

## Separation sequence and methods for landfill gas treatment.



## Split Sequencing : Enrichment

- Gas - separation manager makes use of three selectors: enrichment, sharp separation, and purification
- Enrichment consists of a significant increase in the concentration of one or several species in the desired stream, although by this operation neither high recovery nor purity is achieved.
- Condensation, physical absorption, membrane permeation, cryogenic distillation, and adsorption are convenient separation techniques.

## Split Sequencing : sharp separation

- Sharp separation consists of obtaining splitting of the mixture into products with a high recovery of target components.
- The sharpness is defined as the ratio of key component concentrations in products. This should be better than 10.
- Potential techniques are: physical absorption, cryogenic distillation, molecular sieving, as well as equilibrium adsorption when the molar fraction of the adsorbate is less than 0.1.
- Chemical absorption may also be applicable when the component concentration is low.

## Split Sequencing : purification

- Purification deals with the removal of impurities with the goal of achieving very high concentration of the dominant component.
- The initial concentration of impurity in the mixture should be lower than 2000 ppm, while the final concentration of impurity in the product should be less than 100 ppm.
- Suitable separation methods are equilibrium adsorption, molecular - sieve adsorption, chemical absorption and catalytic conversion

## General heuristics for separation sequencing of liquid mixtures.

1. Remove first corrosive, hazardous, fouling, reactive and any troublesome components. Consider also in the first place the removal of light-ends.
2. Deliver high-purity products as top distillate. The same is valid for reactants sent to reactors sensitive to impurities.
3. When separation by distillation is feasible, prefer it in a first attempt.
4. Isolate zeotropic and azeotropic mixtures.
5. Perform difficult zeotropic separations later, but before azeotropic separations. Examine other options, such as extractive distillation, L-L extraction, crystallization, adsorption, or molecular sieves.
6. Examine the separation of azeotropic mixtures last.
7. Remove the components in order of decreasing percentage of the feed. This operation will reduce the cost of the next separation.
8. Favor 50/50 splits.

## Sequencing of Ordinary Distillation Columns

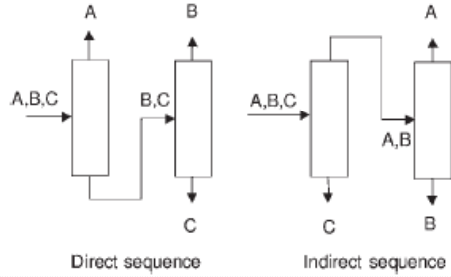
- The relative volatility between the two selected key components for the separation in each column is  $>1.05$
- The reboiler duty is not excessive. (low relative volatility  $\implies$  high duty reboiler)
- The tower pressure does not cause the mixture to approach its critical temperature
- The overhead vapor can be at least partially condensed at the column pressure to provide reflux without excessive refrigeration requirements
- The bottoms temperature for the tower pressure is not so high that chemical decomposition occurs
- Azeotropes do not prevent the desired operation
- Column pressure drop is tolerable, particularly if operation is under vacuum

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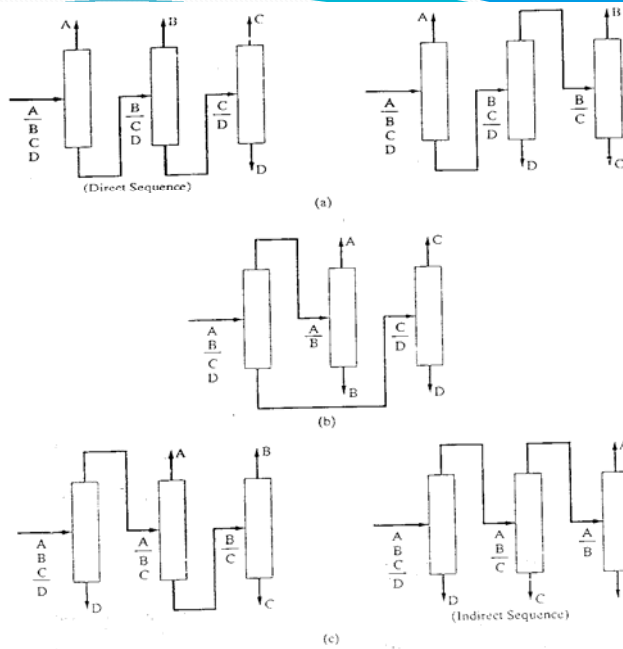
## Heuristics for separation sequencing of zeotropic mixture

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1. Perform difficult separations the last, but before separations of azeotropes.
  2. Remove firstly the lightest component one by one as overhead products.
  3. Remove components in order of decreasing percentage of the feed.  
This operation will reduce the cost of the next separation.
  4. Favor near 50/50 splits.
-

# DIRECT AND INDIRECT SEQUENCES



No.	Type	First split	Second split	Third split
1	Direct	A/BCD	B/CD	C/D
2	Equal split	AB/CD	A/B	C/D
3	Indirect	ABC/D	AB/C	A/B
4	Direct/indirect	A/BCD	BC/D	B/C
5	Indirect/direct	ABC/D	A/BC	B/C

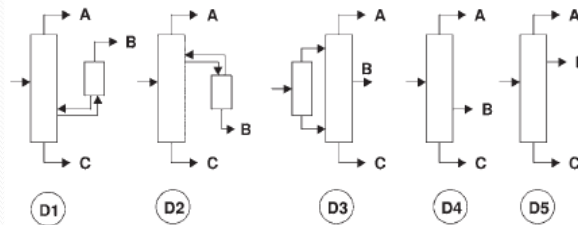


## Heuristics for Determining Favorable Sequence ==> Economic

- Remove thermally unstable, corrosive, or chemically reactive components early in the sequence
- Remove final products one by one as distillates
- Sequence separation points to remove, early in the sequence, those components of greatest molar percentage in the feed
- Sequence separation points in the order of decreasing relative volatility so that the most difficult splits are made in the absence of other components
- Sequence separation points to leave last those separations that give the highest purity products
- Sequence separation points that favor near equimolar amounts of distillate and bottoms in each column

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## Types of Complex Columns



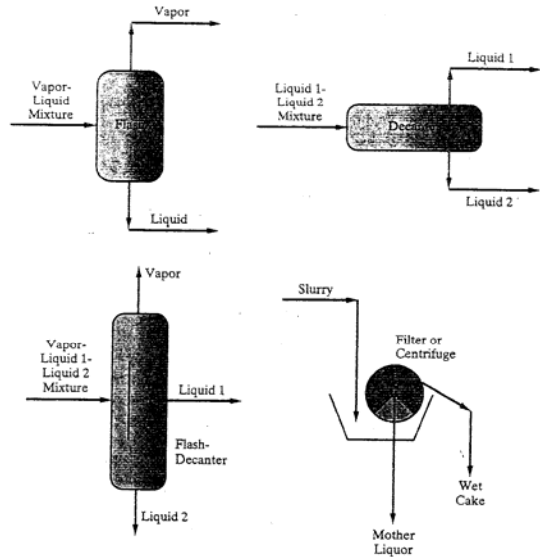
1. **Side - stream rectifier**: A and C from main column, B as top product of side rectifier.
2. **Side - stream stripper**: A and C as before, but B as bottoms of side stripper.
3. **Prefractionator**: separate AB and BC mixtures in first column by sloppy separation, then take pure components A, B, and C in a side - stream second column.
4. **Side - stream low position**: take B as side stream below the feed.
5. **Side - stream high position**: take B as side stream above the feed.

## Phase Conditions of The Feed as Criterion

- **Vapor feed:**
  - Partial condensation
  - Distillation under cryogenic conditions
  - Gas absorption
  - Gas adsorption
  - Gas permeation with a membrane
  - desublimation

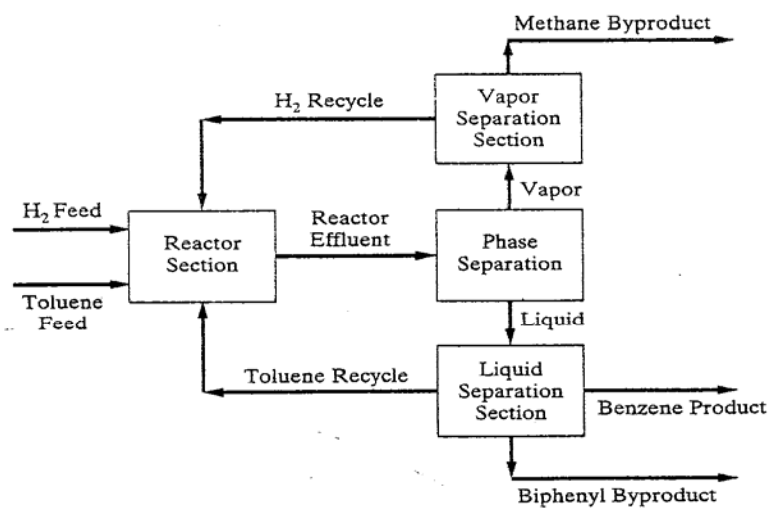
- **Liquid Feed:**
  - Flash
  - Distillation
  - Stripping
  - Extractive distillation
  - Azeotropic distillation
  - Liquid-liquid extraction
  - Crystallization
  - Liquid adsorption
  - Dialysis, reverse osmosis, ultrafiltration, etc
  - Supercritical extraction

## Various Phase-Separation Devices



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## Example: hydrodealkylation of toluene to benzene



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## Slurries, wet cake, dry solids

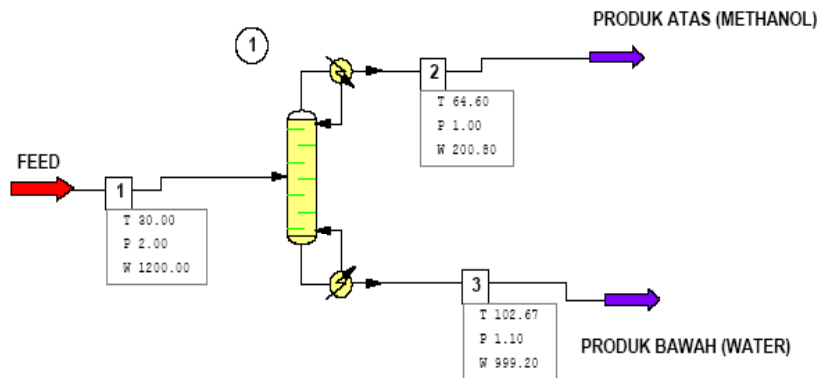
- Filtration
- Centrifugation → obtain a wet cake
- The separated into a vapor and a dry solid by drying

## Enhanced/ Azeotropic Distillation

- Extractive Distillation
- Chemically Enhanced Distillation
- Pressure - Swing Distillation

# Shortcut Column untuk Prediksi Jumlah Stage dan Feed Stage

SHORTCUT COLUMN  
(setelah jumlah tray ketemu baru ke distilasi jenis lainnya untuk simulasi)



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# Shortcut Column Specification

- Shortcut Column (SHOR) - ID: 1

Select mode: 2 Design: FUG with Fenske feed tray location

Select condenser type: 0 Total

Column pressure: 1 atm

Column pressure drop: 0.1 atm

Number of stages: 19.3493

Reflux ratio:

R/Rmin: 1.4

Case Study

Number of points:

Lower bound R/Rmin:

Upper bound R/Rmin:

Key Component Specifications

Light key component: 1 Methanol

Light key split: 0.999

Heavy key component: 2 Water

Heavy key split: 0.001

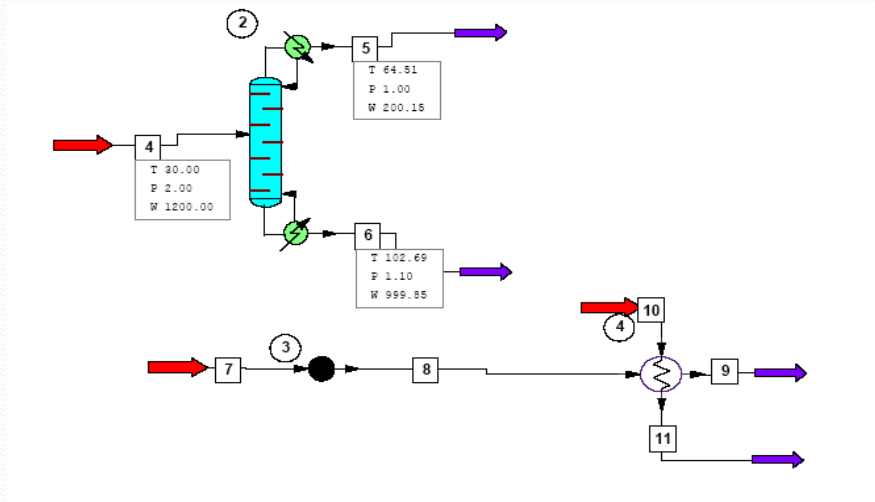
Calculated Results

Condenser duty	-1048.17	MJ/h	Reflux ratio, minimum	2.65379
Reboiler duty	1371.16	MJ/h	Reflux ratio, calculated	3.7153
Minimum stages	10.8479			
Feed stage	10.1746			

Help Cancel OK

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# Plate Column from Shortcut Column



# Plate Column Specification

TOWER Distillation Column - ID: 2

General | Specifications | Convergence | Cost Estimation 1 | Cost Estimation 2

**General Model Parameters**

Condenser type: 0 Total or no condenser

Subcooled temp: [ ] C

Top pressure: 1 atm

Cond press drop: [ ] atm

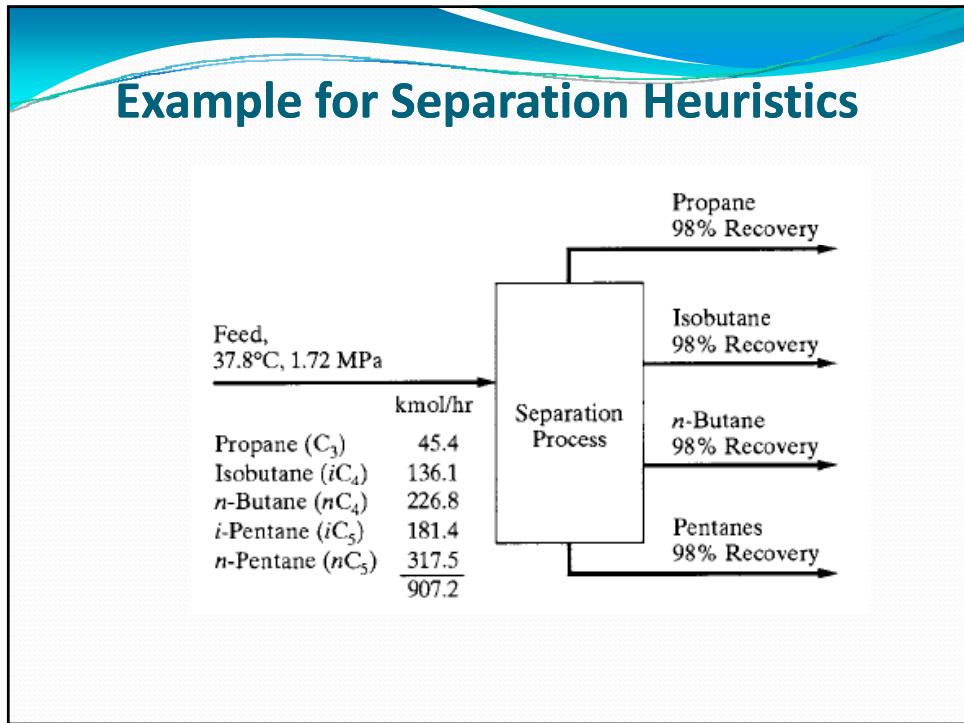
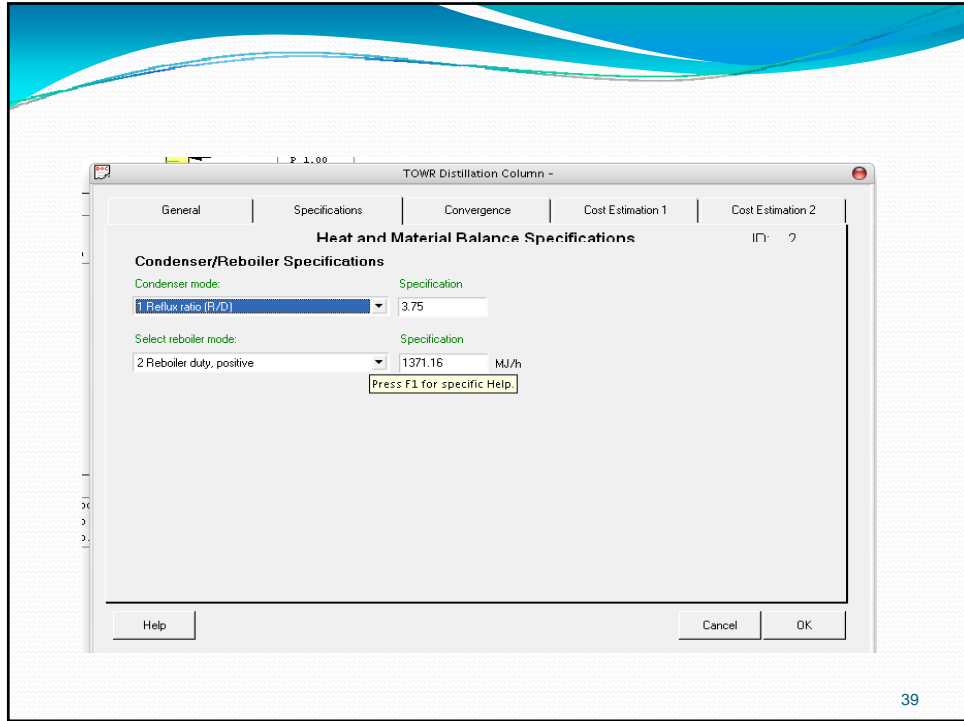
Colm press drop: 0.1 atm

No. of stages: 20

Feed stages:

Feed tray for stream: 4 11

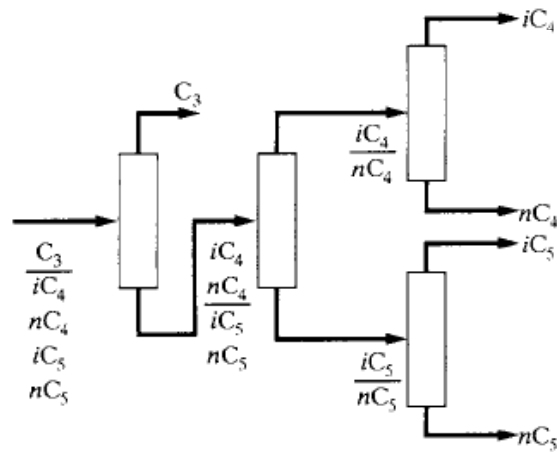
Buttons: Help, Cancel, OK

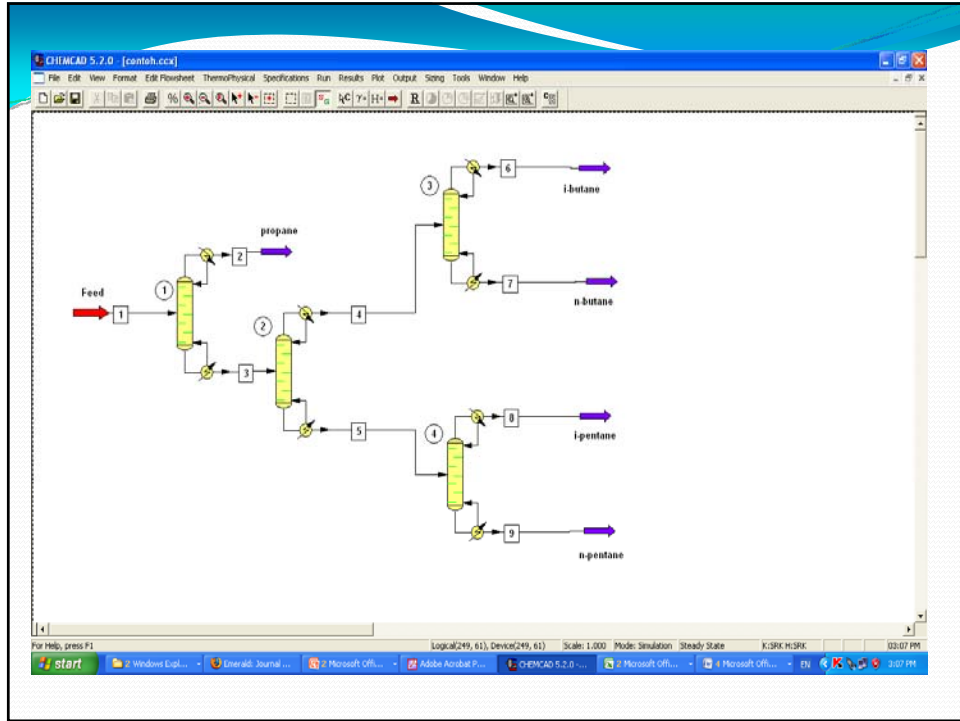


## Relative Volatility Data

Component pair	Approximate $\alpha$ 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

## Solution based on heuristics





**- Shortcut Column (SHOR) -** ID: 1

Select mode: [2 Design: FUG with Fenske feed tray location]

Select condenser type: [0 Total]

Column pressure: [1.7] MPa

Column pressure drop: [0.1] MPa

Number of stages: [52.7212]

Reflux ratio: [ ]

R/Rmin: [1.4]

Case Study

Number of points: [ ]

Lower bound R/Rmin: [ ]

Upper bound R/Rmin: [ ]

Key Component Specifications

Light key component: [1 Propane]

Heavy key component: [2 I-Butane]

Light key split: [0.00]

Heavy key split: [1e-005]

Calculated Results

Condenser duty: [-3887.22] MJ/h

Reboiler duty: [18750.4] MJ/h

Minimum stages: [30.3757]

Feed stage: [39.5867]

Reflux ratio, minimum: [4.20531]

Reflux ratio, calculated: [5.88743]

Buttons: Help, Cancel, OK

**Shortcut Column (SHOR)** ID: 2

Select mode: 2 Design: FUG with Fenske feed tray location

Select condenser type: 0 Total

Column pressure: 1.5 MPa

Column pressure drop: 0.1 MPa

Number of stages: 59.874

Reflux ratio: [ ]

R/Rmin: 1.4

Case Study

Number of points: [ ]

Lower bound R/Rmin: [ ]

Upper bound R/Rmin: [ ]

Key Component Specifications

Light key component: 3 N-Butane

Heavy key component: 4 I-Pentane

Light key split: 0.98

Heavy key split: 1e-005

Calculated Results

Condenser duty: -29127.3 MJ/h

Reboiler duty: 28619 MJ/h

Minimum stages: 33.9563

Feed stage: 44.923

Reflux ratio, minimum: 3.16089

Reflux ratio, calculated: 4.42524

Buttons: Help, Cancel, OK

**Shortcut Column (SHOR)** ID: 3

Select mode: 2 Design: FUG with Fenske feed tray location

Select condenser type: 0 Total

Column pressure: 1.4 MPa

Column pressure drop: 0.1 MPa

Number of stages: 132.656

Reflux ratio: [ ]

R/Rmin: 1.5

Case Study

Number of points: [ ]

Lower bound R/Rmin: [ ]

Upper bound R/Rmin: [ ]

Key Component Specifications

Light key component: 2 I-Butane

Heavy key component: 3 N-Butane

Light key split: 0.98

Heavy key split: 1e-005

Calculated Results

Condenser duty: -39514.5 MJ/h

Reboiler duty: 39452.7 MJ/h

Minimum stages: 83.7598

Feed stage: 99.2218

Reflux ratio, minimum: 12.8169

Reflux ratio, calculated: 19.2254

Buttons: Help, Cancel, OK

**Shortcut Column (SHOR)** ID: 4

Select mode: 2 Design: FUG with Fenske feed tray location

Select condenser type: 0 Total

Column pressure: 1.4 MPa

Column pressure drop: 0.1 MPa

Number of stages: 238.235

Reflux ratio:

R/Rmin: 1.4

Case Study

Number of points:

Lower bound R/Rmin:

Upper bound R/Rmin:

Key Component Specifications

Light key component: 4 I-Pentane

Light key split: 0.98

Heavy key component: 5 N-Pentane

Heavy key split: 1e-005

Calculated Results

Condenser duty: -103433 MJ/h

Reboiler duty: 102836 MJ/h

Minimum stages: 143.424

Feed stage: 177.989

Reflux ratio, minimum: 23.6164

Reflux ratio, calculated: 33.0629

Help Cancel OK