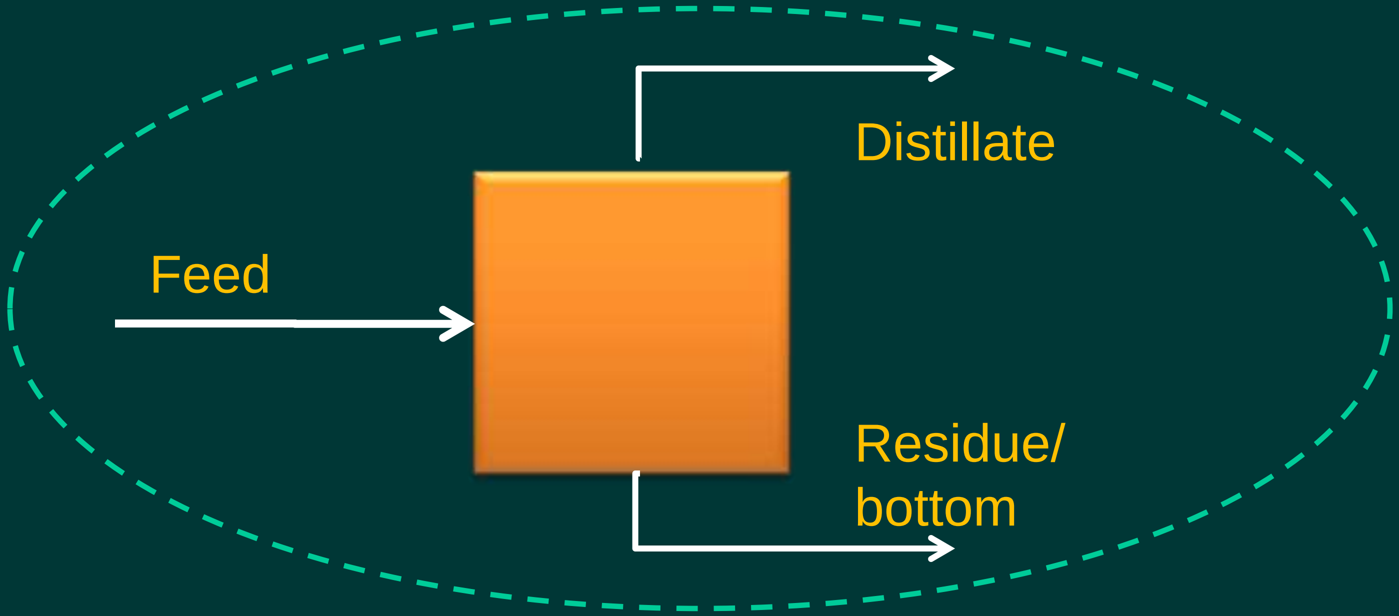
DISTILASI

Distilasi

- Proses pemisahan dua komponen atau lebih berdasarkan perbedaan titik didihnya atau volatilitas
- Pemisahan tepat terjadi pasa saat kondisi setimbang atau equilibrium



Vapor-liquid equilibrium (VLE)

1. Phase rule and Raoult's law

$$x_A + x_B = 1$$

$$y_A + y_B = 1$$

$$p_A = x_A P_A$$

$$p_A + p_B = P$$

$$P_A . x_A + P_B . x_B = P$$

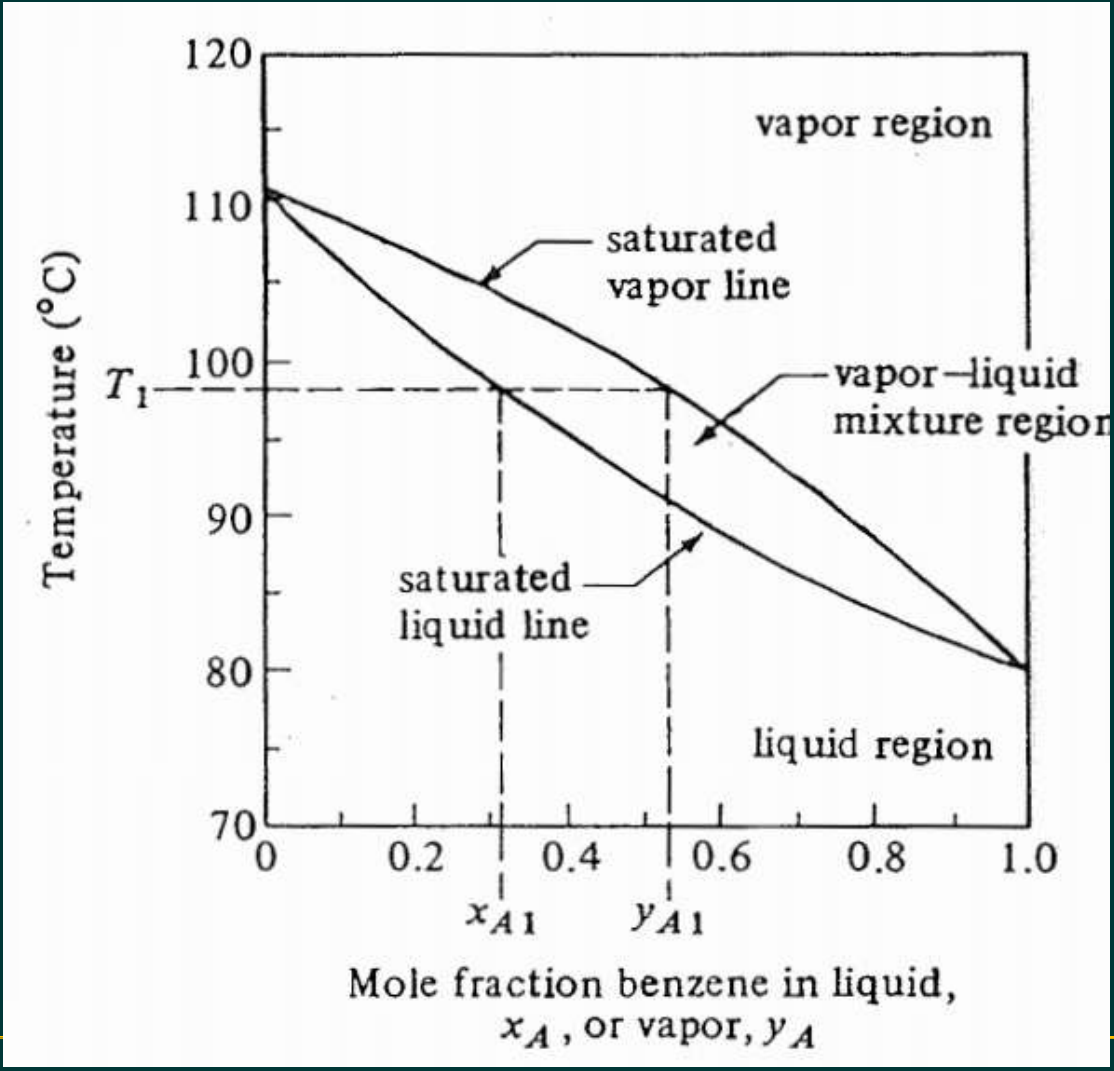
$$P_A . x_A + P_B (1 - x_A) = P$$

$$y_A = \frac{p_A}{P} = \frac{P_A x_A}{P}$$

x = fraksi liquid
y = fraksi vapor
p_A = partial pressure A (atm)
P_A = vapor pressure pure A (atm)

Vapor-liquid equilibrium (VLE)

2. Boiling-point diagram and xy plots



Vapor-liquid equilibrium (VLE)

Contoh Soal

Hitung komposisi vapor dan liquid benzene(A) – toluene(B) dalam kesetimbangan pada suhu 95°C, 1atm(101.32 kPa). Diketahui tekanan uap benzene dan toluene pada kondisi tersebut adalah 155.7 kPa dan 63.3 kPa.

Vapor-liquid equilibrium (VLE)

Penyelesaian

$$P_A.x_A + P_B(1 - x_A) = P$$

155.7 ($\mathbf{x_A}$) + 63.3 - 63.3($\mathbf{x_A}$) = 101.32

→ $\mathbf{x_A}$ = 0.411 → $\mathbf{x_B}$ = 0.589

$$y_A = \frac{P_A x_A}{P} = 0.632$$

$$y_B = 0.368$$

Vapor-liquid equilibrium (VLE)

Penyelesaian

Titik didih adalah suhu pada saat tekanan total = 1 atm

$$P_B.x_B + P_w(1 - x_B) = P$$

Untuk T = 308.5

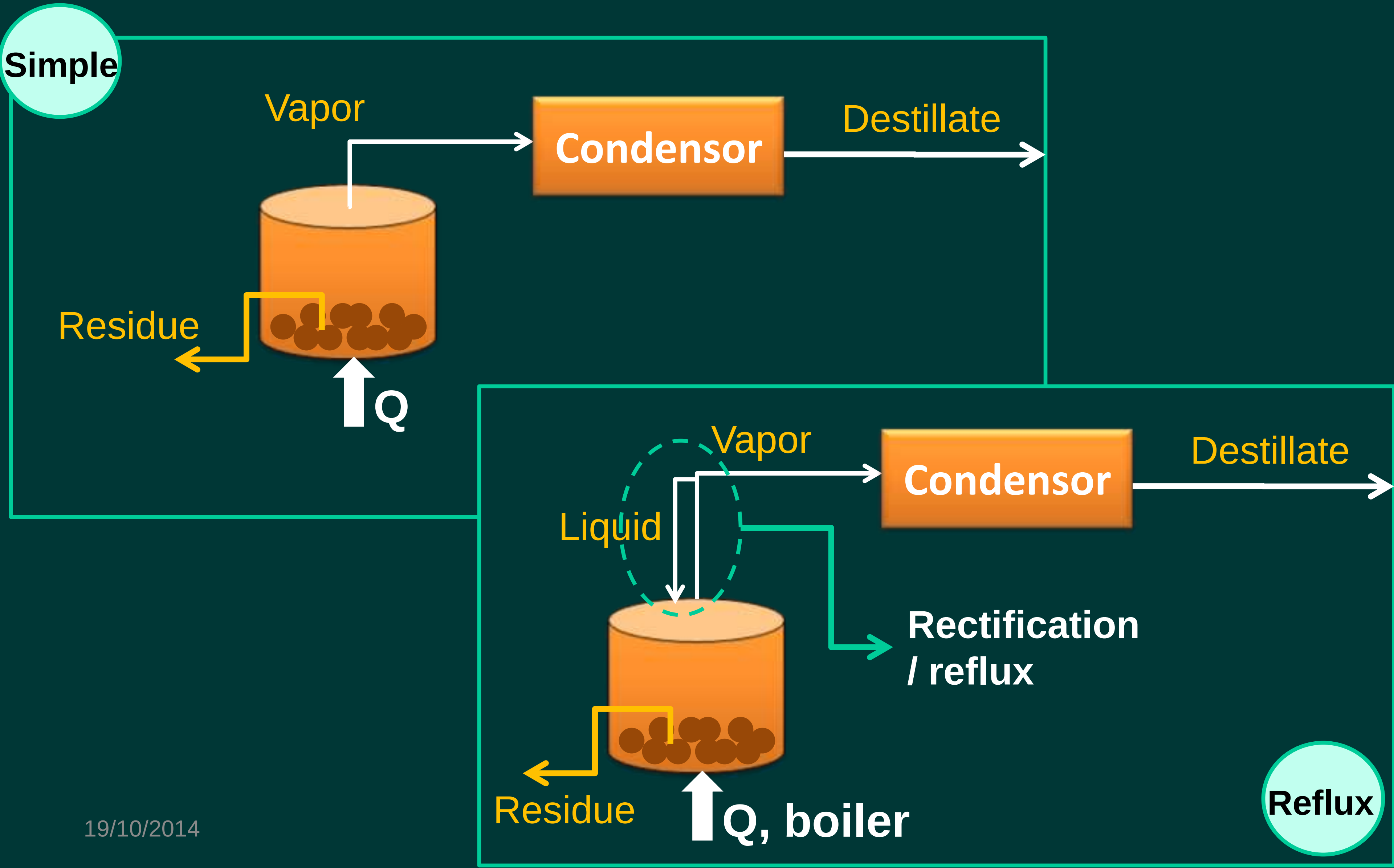
0.043 (0.5) + 0.15(0.5) = 101.32

→ $\mathbf{x_A}$ = 0.411 → $\mathbf{x_B}$ = 0.589

$$y_A = \frac{P_A x_A}{P} = 0.632$$

$$y_B = 0.368$$

Basic concept of distillation methods



Single stage distillation

FLASH Distillation

Campuran liquid akan **menguap** sebagian, setelah terjadi **kesetimbangan**, liquid-vapor **terpisah.**

SIMPLE BATCH Distillation

Komposisi vapor kaya akan komponen volatil. **Komposisi** vapor **berubah** terhadap waktu

SIMPLE STEAM Distillation

Memisahkan *high-boiling component* dari pengotor non-volatil.
Steam menurunkan T_d serta membawa uapnya pada kondensor.

$$\frac{n_B}{n_A} = \frac{P_B}{P_A}$$

Pemisahan *n-heptane - n-octane*, Benzene, Toluene, Xylene

Kondensat berupa campuran komponen dan air *(immisible). Cth: minyak esensial*

Flash distillation

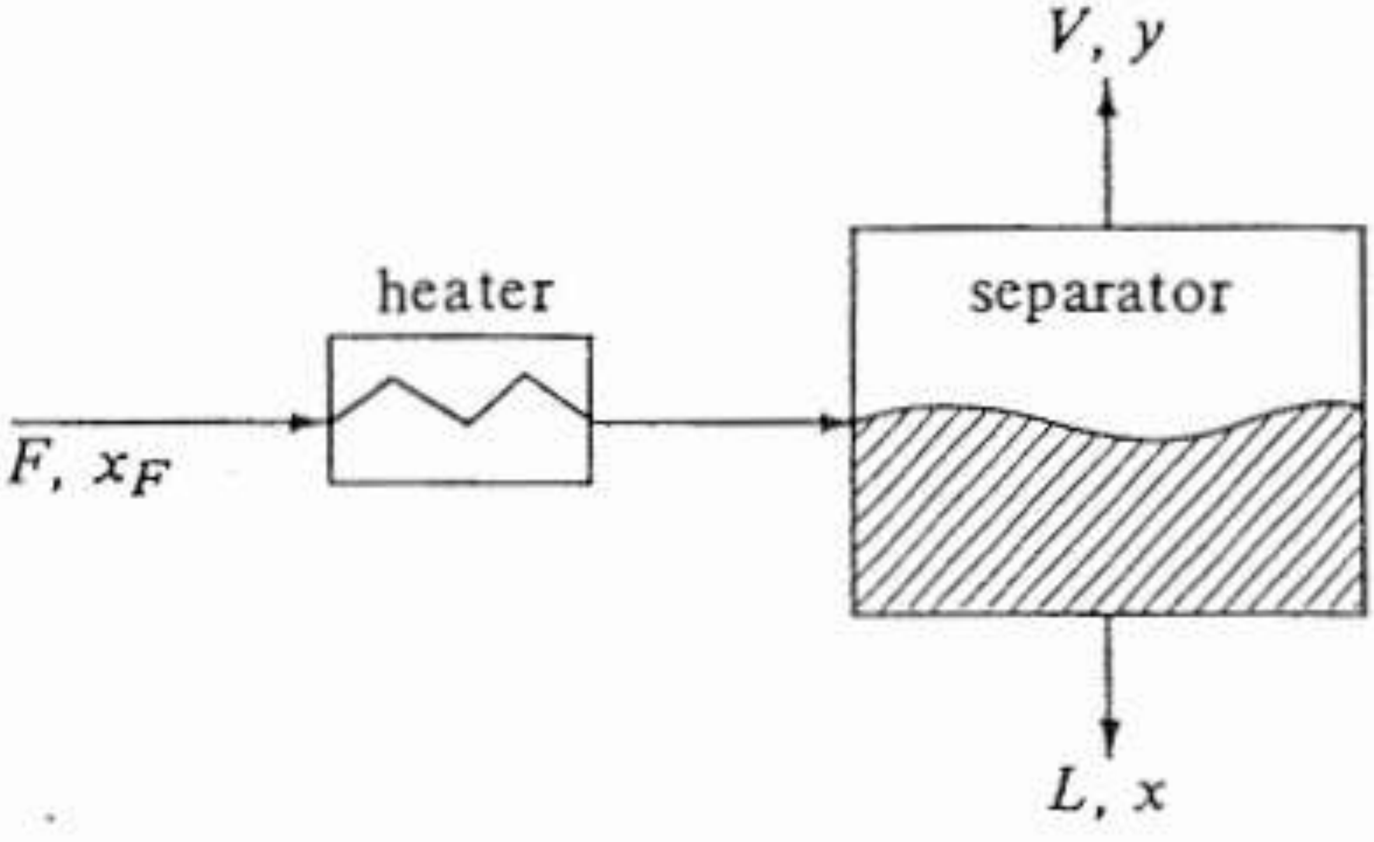
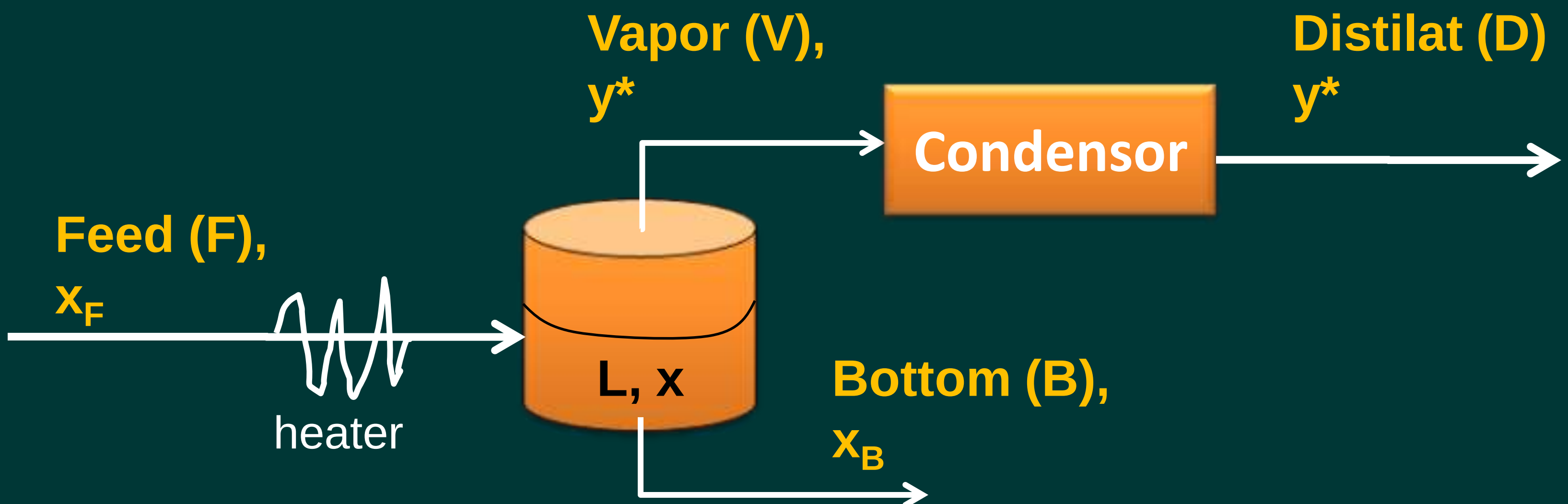


FIGURE 11.3-1. *Equilibrium or flash distillation.*

Continuous flash distillation

- Merupakan metode distilasi paling sederhana
- Feed masuk dalam bentuk liquid, kemudian dilakkukan *pre-heating* sebelum masuk chamber distilasi
- Setelah dipanaskan, volatile compound (vapor) akan naik ke atas untuk dikondensasi (distilat) sedangkan non-volatile akan turun ke bawah sebagai bottom



Kesetimbangan massa continuous flash distillation

Overall mass balance:

$$F = B + V$$

Enthalpy balance:

$$F.h_F = B.h_B + V.h_V$$

Volatile compound mass balance:

$$F.x_F = B.x_B + V.y^*$$

$$y^* = -(B/V).(x_B - x_F) + x_F$$

Contoh Soal

Suatu larutan dengan massa 100 kg mengandung komponen flavor dengan konsentrasi 0.5% b/b. Komponen flavor tersebut kemudian dipisahkan dengan flash distillation. Larutan dipanaskan pada suhu T dan dimasukkan dalam tangki distilasi dengan tekanan 20 kPa. Jika fungsi liquid-vapor equilibrium dari larutan adalah y* = 63x (x dan y* adalah konsentrasi flavor dalam cairan dan uap, b/b).

Tentukan suhu minimal supaya didapatkan 70% komponen vapor dalam distilat.

asumsi: karakteristik termal larutan sama seperti air

Penyelesaian

Overall mass balance:

$$F = B + V = 100 \rightarrow B = 100 - V$$

Flavor compound mass balance:

flavor comp. pada feed = 50% x 100 = 50 kg

flavor comp. pada vapor = 70% x 50 = 35 kg → $y^* = 35/V$

flavor comp. pada botom = 30% x 50 = 15 kg → $x = 15/(100-V)$

Fungsi kesetimbangan:

$$y^* = 63\,x$$

$$35/V = 63 \times 15/(100-V)$$

$$V = 3.57 \text{ kg} \rightarrow B = 96.43 \text{ kg}$$

Penyelesaian

Enthalpy balance:

$$F.h_F = B.h_B + V.h_V$$

Pada tekanan 20 kPa: (steam table)

h_B =251 kJ/kg, h_V= 2610 kJ/kg

$$100.h_F = 96.43 \times 251 + 3.57 \times 2610$$

$$h_F = 335.2 \text{ kJ/kg}$$

(pada steam table) ↓

Suhu pada entalpi 335.2 kJ/kg adalah **80°C**

Simple batch distillation

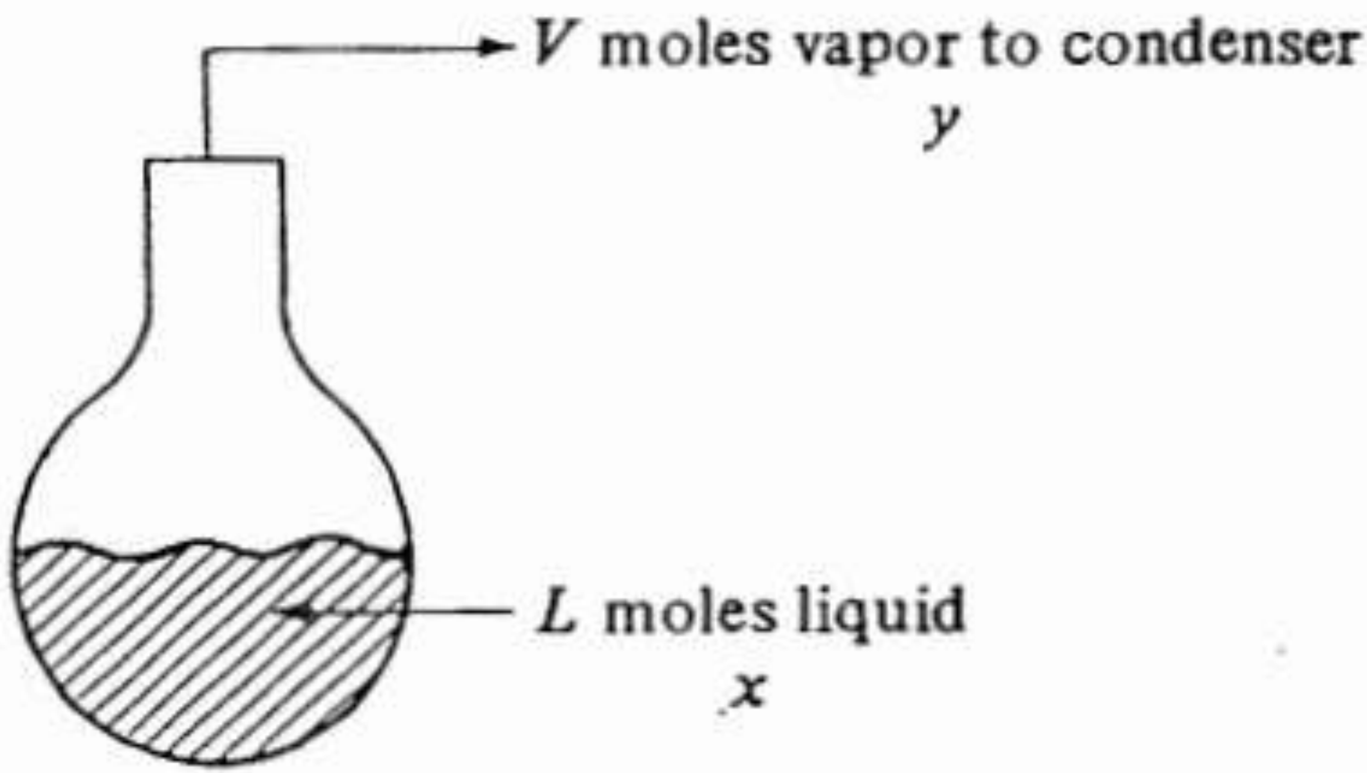
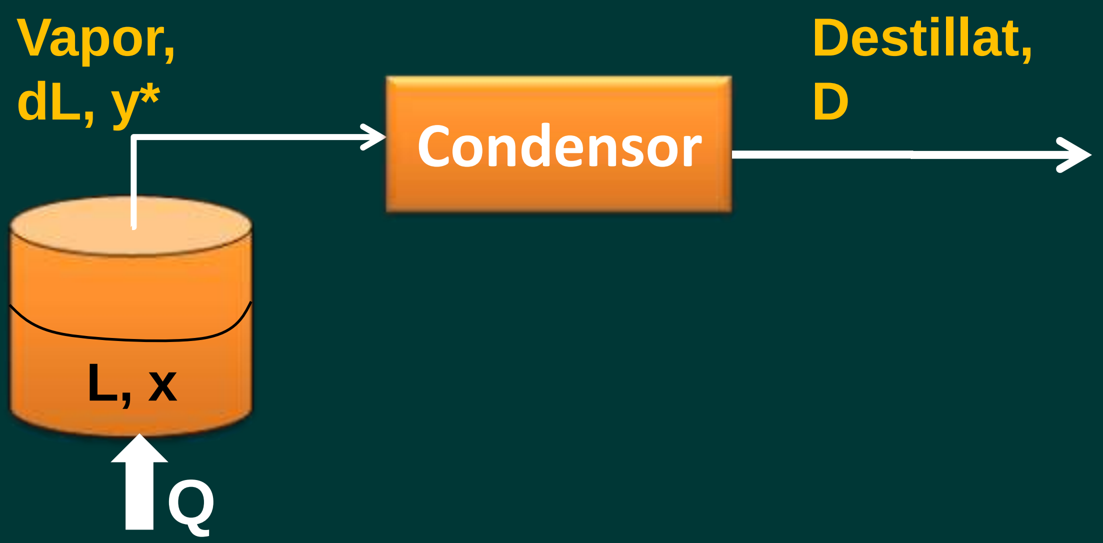


FIGURE 11.3-2 *Simple batch or differential distillation.*

Kesetimbangan massa batch distillation



Massa awal = massa tersisa dalam vessel + massa vapor

$$xL = (x - dx)(L - dL) + y\, dL$$

$$\cancel{xL} = \cancel{xL} - x\, dL - L\, dx + \cancel{dx\, dL} + y\, dL$$

$$y\,dL - x\,dL = L\,dx$$

$$(y - x)\,dL = L\,dx$$

$$1/L\,dL = 1/(y - x)\,dx$$

$$\ln \frac{L_1}{L_2} = \int_{x_2}^{x_1} \frac{dx}{y - x}$$

Rerata komposisi distilat (y_{av})

$$L_1 x_1 = L_2 x_2 + (L_1 - L_2) y_{av}$$

L_1 = Jumlah sampel awal (mol)
L_2 = Jumlah sampel akhir (mol)
x_1 = konsentrasi awal komponen X dalam vessel
x_2 = konsentrasi akhir komponen X dalam vessel
y = konsentrasi komponen X dalam vapor

Simple Batch/differential Distillation

EXAMPLE 11.3-2. Simple Differential Distillation

A mixture of 100 mol containing 50 mol % *n*-pentane and 50 mol % *n*-heptane is distilled under differential conditions at 101.3 kPa until 40 mol is distilled. What is the average composition of the total vapor distilled and the composition of the liquid left? The equilibrium data are as follows, where x and *y* are mole fractions of *n*-pentane.

x	y	x	y	x	y
1.000	1.000	0.398	0.836	0.059	0.271
0.867	0.984	0.254	0.701	0	0
0.594	0.925	0.145	0.521		

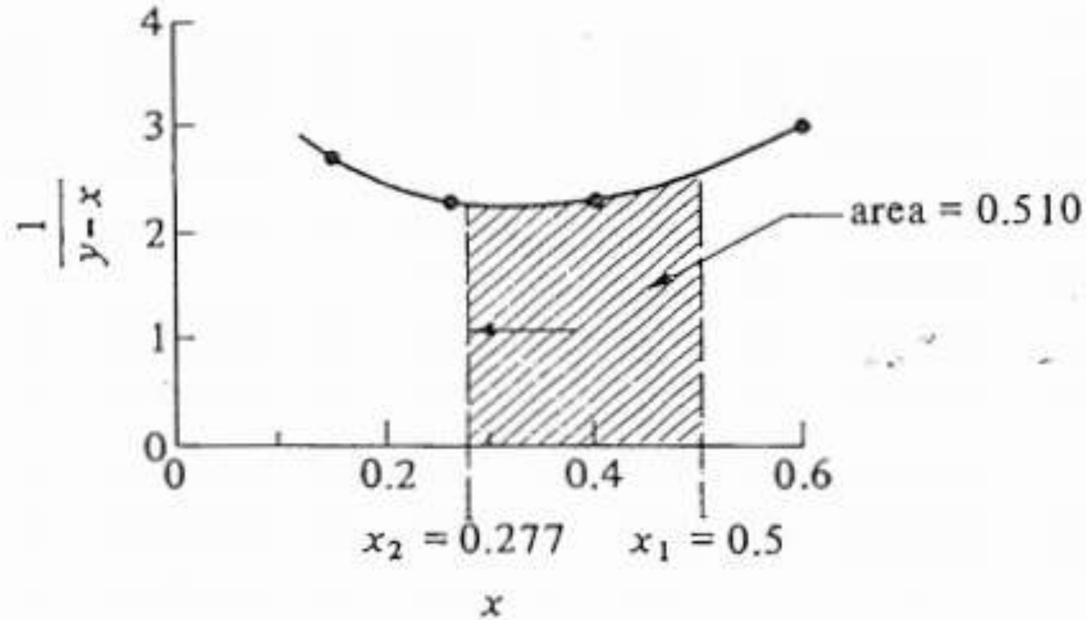
Simple Batch/differential Distillation

Solution: The given values to be used in Eq. (11.3-10) are $L_1 = 100$ mol, $x_1 = 0.50$, $L_2 = 60$ mol, V (moles distilled) $= 40$ mol. Substituting into Eq. (11.3-10),

$$\ln \frac{100}{60} = 0.510 = \int_{x_2}^{x_1=0.5} \frac{dx}{y-x} \qquad \textbf{(11.3-12)}$$

The unknown is x_2, the composition of the liquid L_2 at the end of the differential distillation. To do the graphical integration a plot of $1/(y-x)$ versus x is made in Fig. 11.3-3 as follows. For $x = 0.594$, the equilibrium value of $y = 0.925$. Then $1/(y-x) = 1/(0.925 - 0.594) = 3.02$. The point $1/(y-x) = 3.02$ and $x = 0.594$ is plotted. In a similar manner, other points are plotted.

Simple Batch/differential Distillation

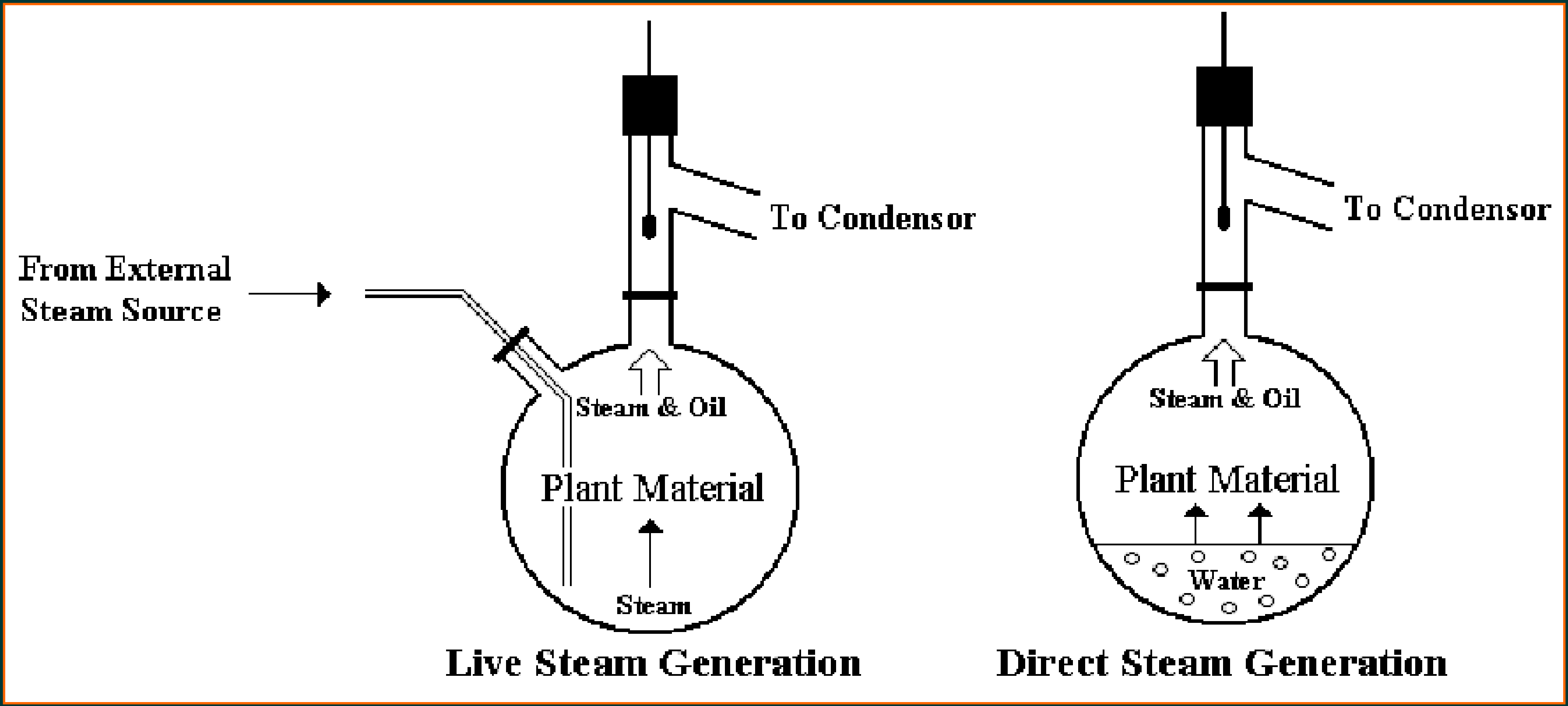


To determine the value of x_2, the area of Eq. (11.3-12) is obtained under the curve from $x_1 = 0.5$ to x_2 such that the area = 0.510. Hence, $x_2 = 0.277$. Substituting into Eq. (11.3-11) and solving for the average composition of the 40 mol distilled,

$$100(0.50) = 60(0.277) + 40(y_{av})$$

$$y_{av} = 0.835$$

Steam distillation



Steam & Vacuum Distillations

- Used for components that decompose at or near its bp
- Steam
  - Limited to those components that are immiscible with water
    - Problem – Emulsion form
      - Usually forms when densities of 2 liquids are similar
      - Breaking emulsions
        - » Glass wool
        - » Centrifuge
        - » Salts
        - » Acids
        - » Phase separation paper (Whatman PS-1)

Steam & Vacuum Distillations

- Vacuum
  - Any distillation below atmospheric pressure
  - Advantage boiling pt **differences** increase at reduced pressures

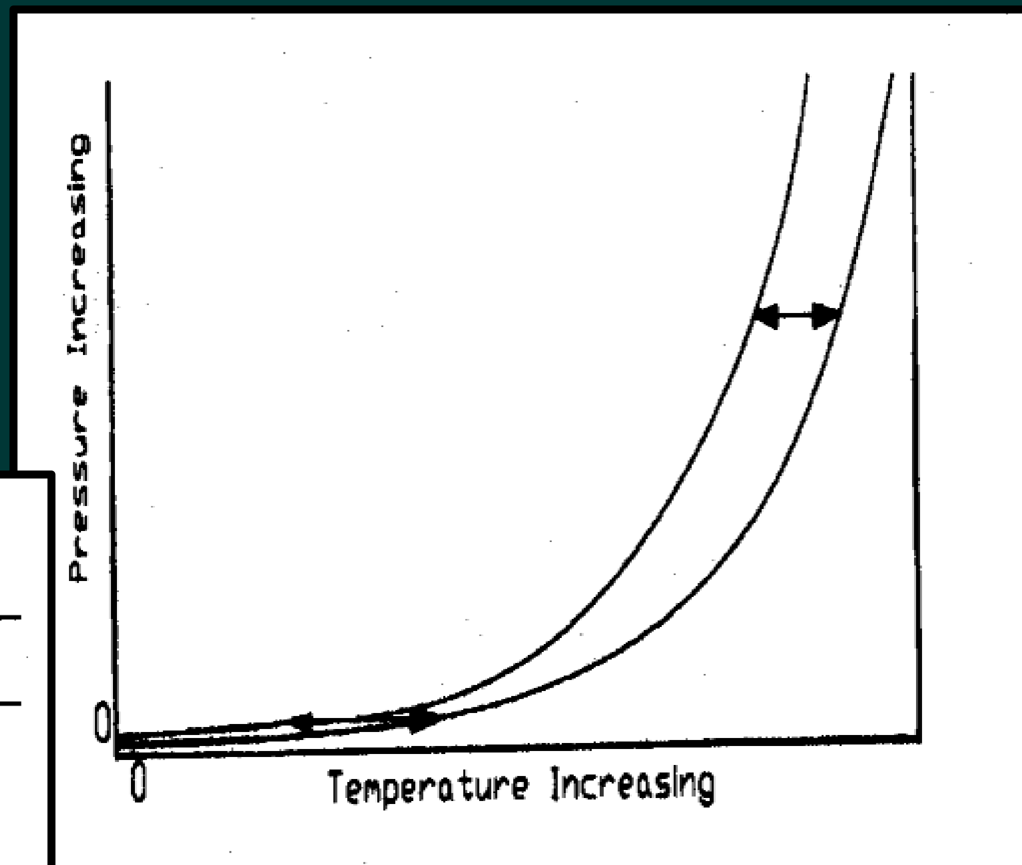
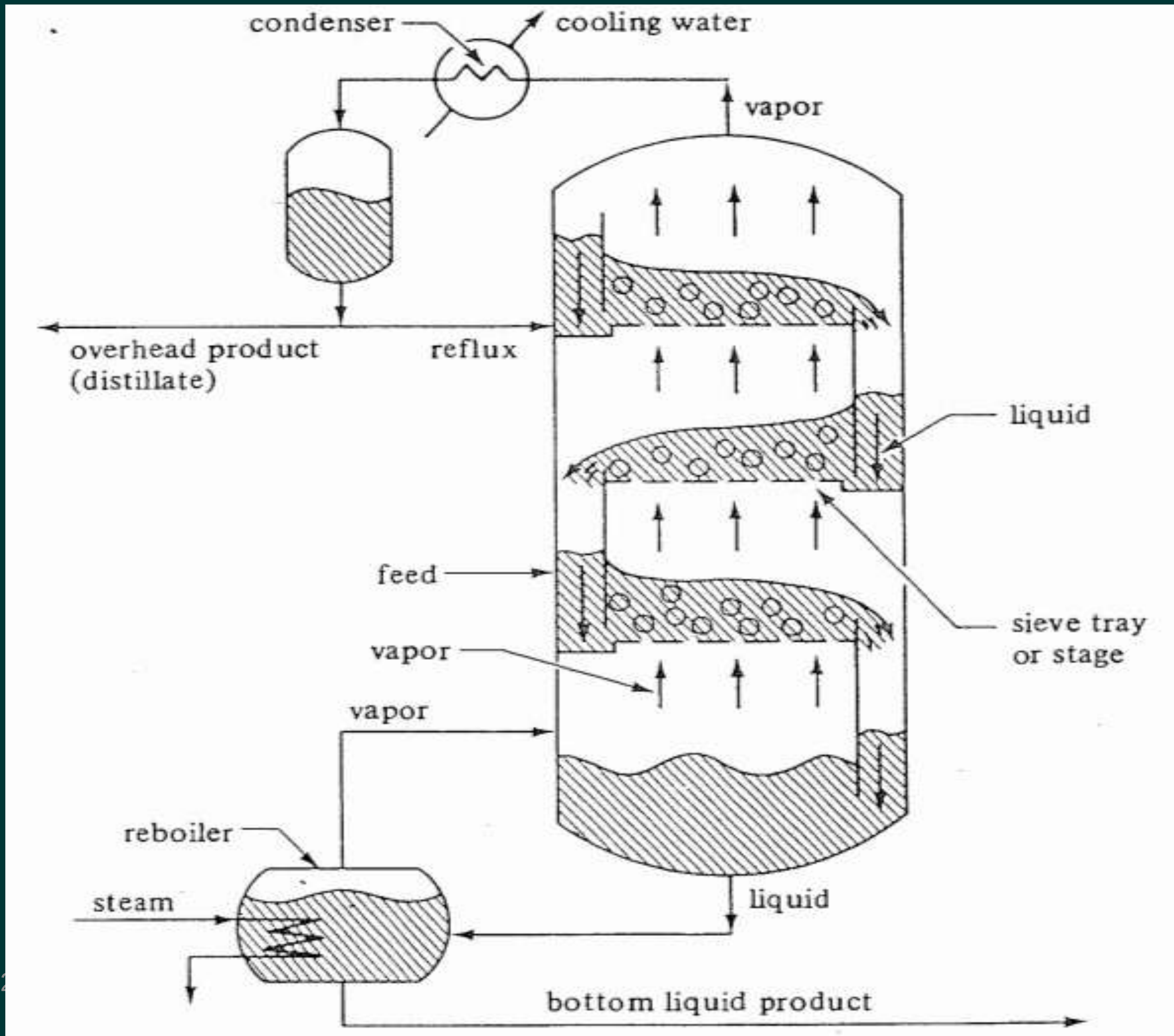


Table 6-1. Boiling point changes with pressure for m-nitrotoluene

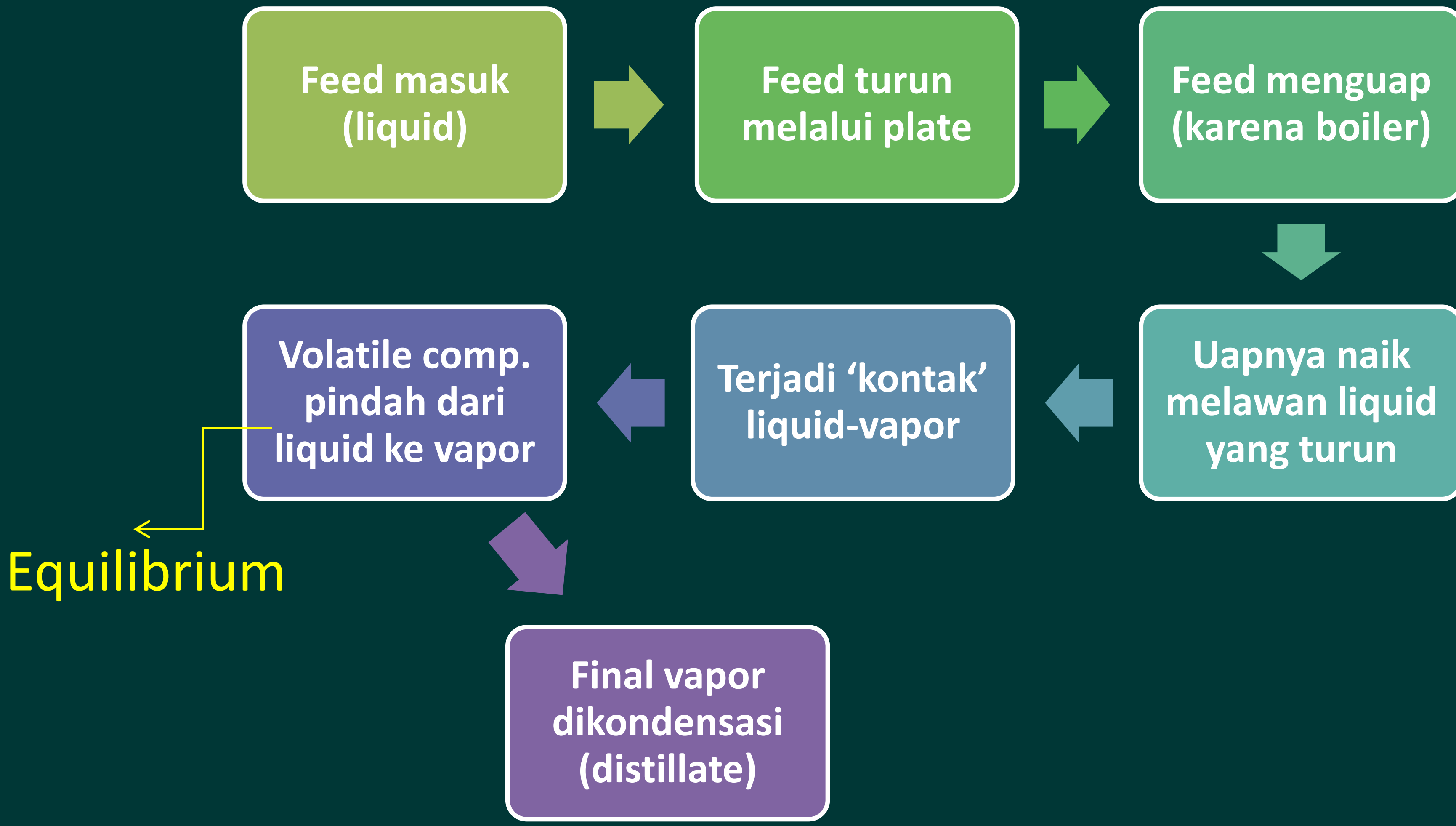
Pressure, torr	(kPa)	B.P. °C
760	101.3	231.9
100	13.3	156.9
40	5.3	130.7
20	2.66	112.6
10	1.33	96.0
5	0.67	81.0
1	0.133	50.2

Multistage distillation

Fractional Distillation



Mekanisme *Fractional Distillation*



Fractional Distillation

- Plates
  - Theoretical plates
    - Represent each equilibrium step in the refluxing system
  - HETP (Height Equivalent to a Theoretical Plate)
    - Takes into account the distance from surface of liquid to the top of the column
    - Measures the efficiency of distillation

Table 3-2. Relationship between the number of theoretical plates and the difference in boiling point for a good separation (Courtesy- K. Wiberg- *Laboratory Technique in Organic Chemistry*, McGraw-Hill, NY, 1960)

Number of plates	Difference in b.p. °C
0	215
1	108
2	72
3	54
4	43
5	36
10	20
15	13
20	10
30	7
50	4
100	2

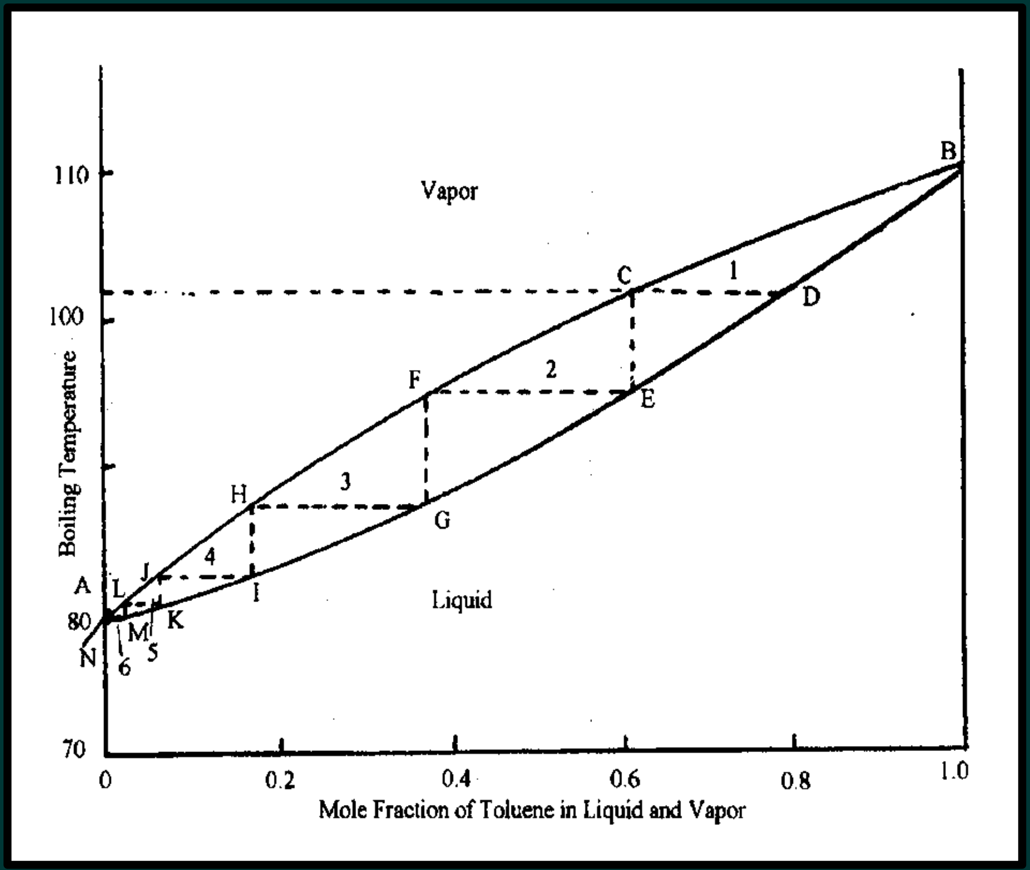
Fractional Distillation

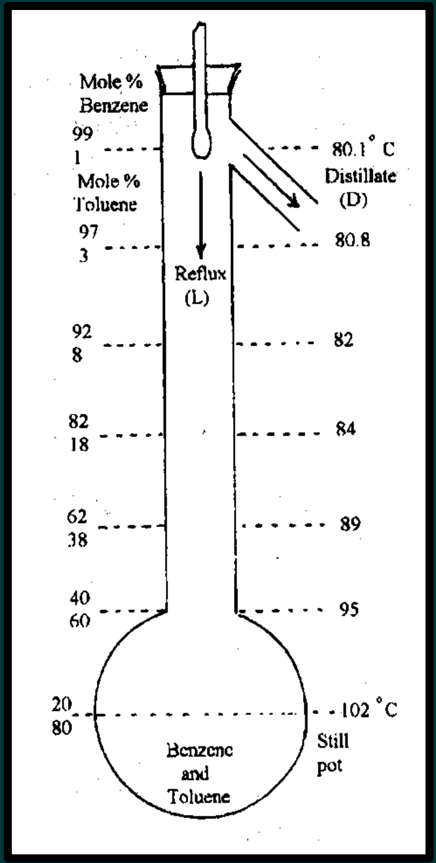
- Continuous Refluxing
  - Total
  - Partial

Table 3-3. The number of plates required to get a 99 % mole fraction separation depending upon the boiling point difference between two liquids

Boiling Point Difference °C	Number of Plates
15-20	10
8-12	25
5-10	45
3-5	80
2	150

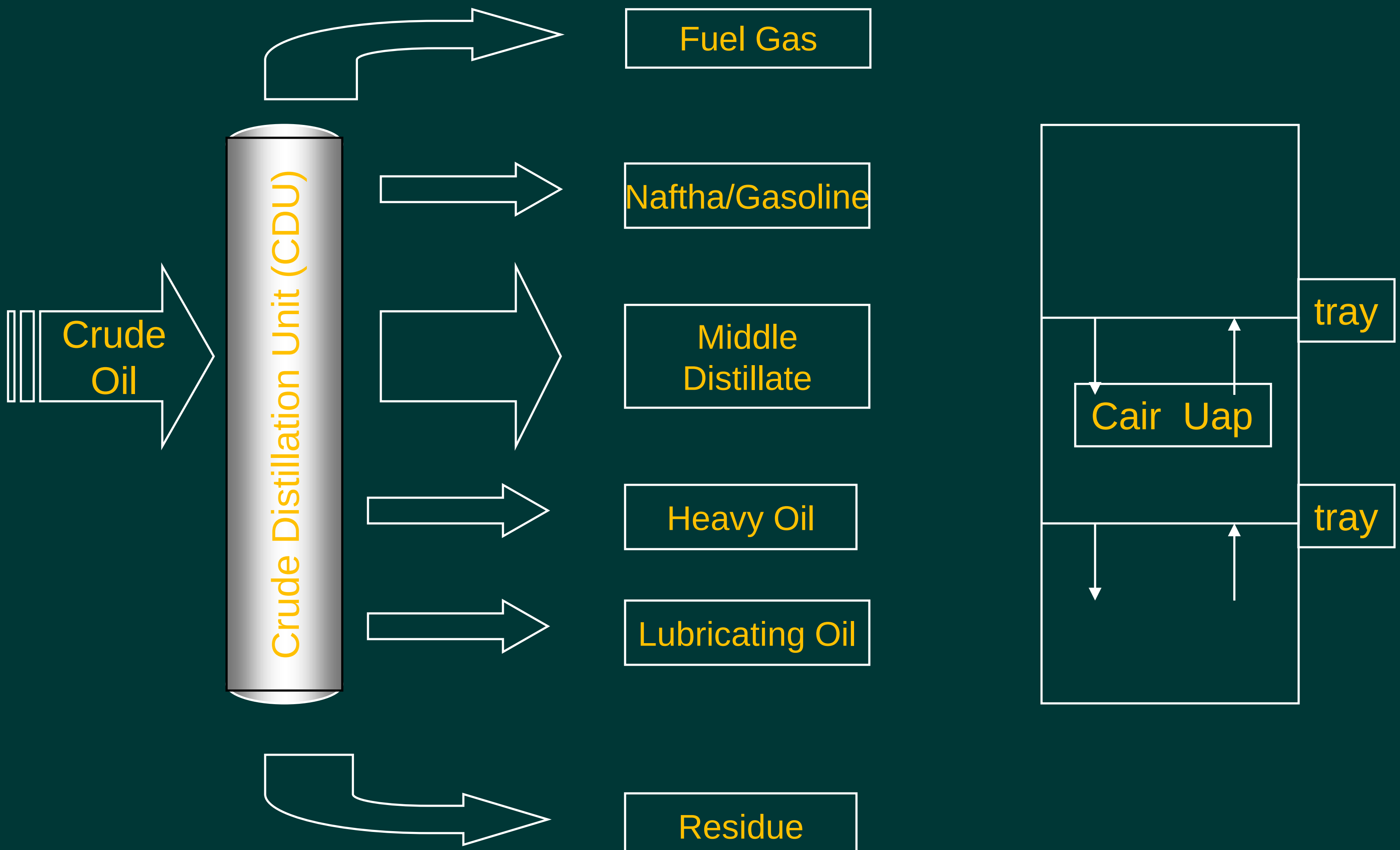
Fractional Distillation





Toluene + Benzene

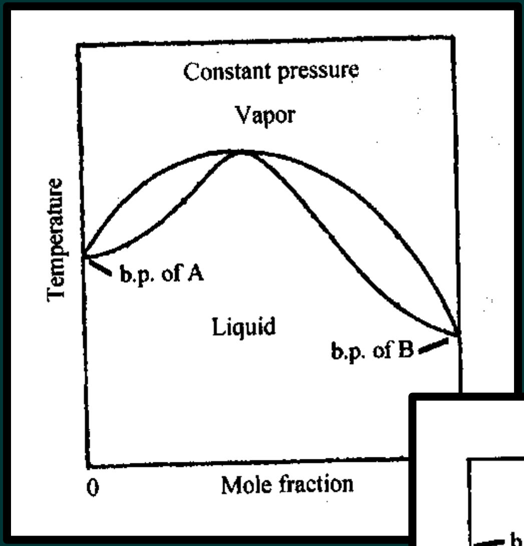
Aplikasi Continuous Distillation/Rectification

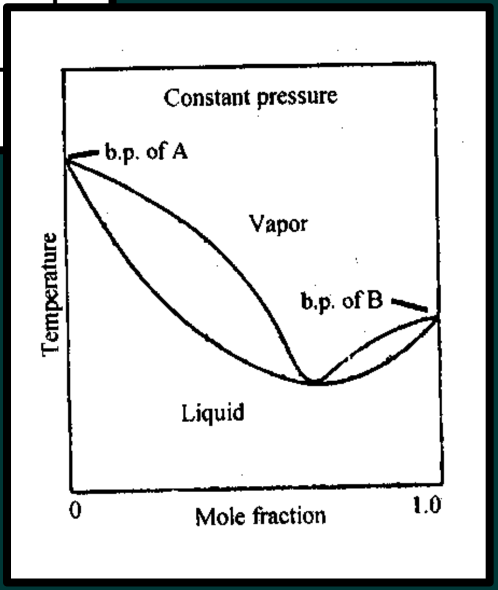


Gambar 1C. Multi Stage Distillation/Crude Distillation Unit

Azeotropic & Extractive Distillations

- Azeotrope
  - Liquid mixture characterized by a maximum or minimum boiling pt. (bp) which is lower or higher than bp for any of the components and that distills without change in composition
  - Distillation – form an azeotrope





Azeotropic & Extractive Distillations

Table 4-2. Some binary system azeotropes (weight %; b.p. °C)

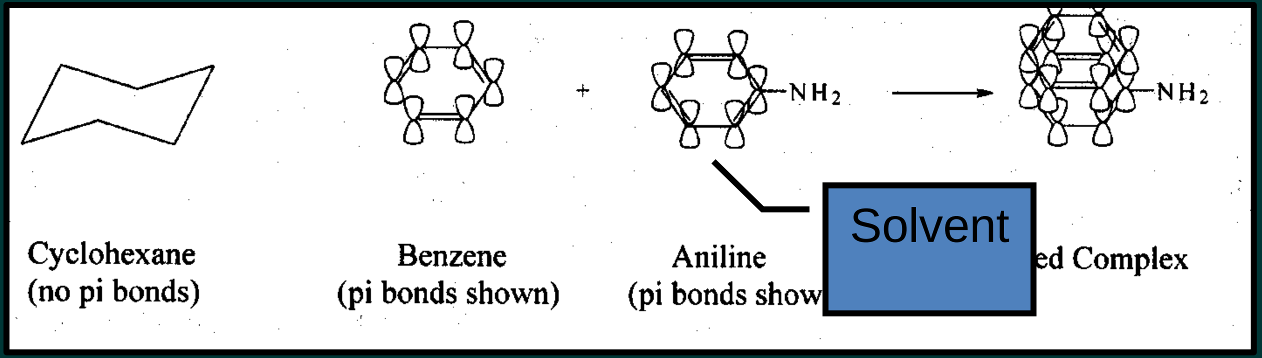
A Component	Weight %	B.P.	B Component	Weight %	B.P.	B.P. Azeotrope
Ethanol	53.	78.3	1,3-Butadiene	47.	-4.5	74.5
Nitromethane	56.5	101.1	Dioxane	43.5	101.3	100.5
Methanol	22.3	64.7	2-Methylfuran	77.7	63.1	51.5
Acetic acid	30.4	118.5	3-Picoline	69.6	144.	152.5
Acetamide	23.	221.2	Camphor	77.	209.1	199.8
Acetone	61.	56.3	2-Propanol	39.	69.0	54.2
2-Butanone	30.	79.0	Butyl nitrite	70.	78.2	76.7
Ethyl acetate	71.	77.2	Butyl nitrite	29.	78.2	76.2
Butyl amine	60.	77.8	Cyclohexane	40.	80.7	76.5
Pyridine	45.	115.5	3-Pentanol	55.	116.0	117.4
Cyclohexanone	65.	156.7	Cumene	35.	152.8	152.

Table 4-3. Some ternary azeotropic systems

A	B	C	Weight % A	Weight % B	Weight % C	B.P.°C
HBr	Water	Chlorobenzene	10.4	11.0	78.6	105.
HF	Water	Ethanol	30.	10.	60.	103.
Water	CCl_4	t-Butanol	3.1	85.	11.9	64.7
Water	$CHCl_3$	Acetone	40.	57.6	38.4	60.4
Water	Formic acid	m-Xylene	10.6	40.4	49.	97.5
Water	Trichloroethylene	Acetonitrile	6.4	73.1	20.5	67.
Water	Acetonitrile	Benzene	8.2	23.3	68.5	66.
Water	Ethanol	Ethyl chloroacetate	17.5	61.7	20.8	81.3
Water	Ethanol	Acetal	11.4	27.6	61.0	77.8
Water	Ethanol	Triethylamine	9.	13.	78.	74.7
Water	Propanol	Ethoxypropoxymethane	17.6	22.9	59.6	83.8
Water	Butanol	Butyl chloroacetate	41.8	50.3	7.9	93.1
Water	2-Butanol	Isobutyl chloroacetate	33.6	53.1	13.3	90.2
Water	Isoamyl alcohol	Isoamyl chloroacetate	46.2	47.3	6.5	95.4
$CHCl_3$	Methanol	Acetone	47.	23.	30.	57.5

Azeotropic & Extractive Distillations

- Extractive
  - A third component is added to extract one of the major components



  - Other interactions
    - Hydrogen, dipole-dipole, ion-dipole, pi bonds

Latihan soal

Diberikan data tekanan uap hexane-octane sebagai berikut:

		Vapor Pressure			
		n-Hexane		n-Octane	
T(°F)	T(°C)	kPa	mm Hg	kPa	mm Hg
155.7	68.7	101.3	760	16.1	121
175	79.4	136.7	1025	23.1	173
200	93.3	197.3	1480	37.1	278
225	107.2	284.0	2130	57.9	434
258.2	125.7	456.0	3420	101.3	760

(a) Menggunakan Hukum Raoult, hitung komposisi liqud-vapor dari hexane(A) dan octane(B) pada tekanan total 101.32 kPa
(b) Blot data komposisi tadi dengantitik didihnya

Latihan soal

Tentukan tekanan total campuran water-benzene pada masing-masing suhu jika komposisi liquid benzene adalah 0.4 fraksi mol?

Temperature		P_{water}	$P_{benzene}$
K	°C	(mm Hg)	(mm Hg)
308.5	35.3	43	150
325.9	52.7	106	300
345.8	72.6	261	600
353.3	80.1	356	760

Latihan soal

Sebuah 100 mol campuran mengandung 60% mol n-pentane dan 40% mol n-heptane diuapkan pada tekanan 101.32 kPa didapatka kesetimbangan 40 mol vapor dan 60 mol liquid melalui *single stage distillation*. Hitunglah komposisi liquid dan komposisi vapor jika diketahui data kesetimbangan sebagai berikut:

x	y	x	y	x	y
1.000	1.000	0.398	0.836	0.059	0.271
0.867	0.984	0.254	0.701	0	0
0.594	0.925	0.145	0.521		

THANKS FOR YOUR ATTENTION

The best person is one give something useful always