



Combined Heat and Mass Transfer

Lec 1 :liquid-liquid Extraction-Part 1

Content

Introduction, Liquid-Liquid Extraction, Distribution Coefficients, Liquid-Liquid Equilibrium, Operating Modes of Extraction and calculations

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- Introduction
- Liquid-Liquid Extraction
- Distribution Coefficients
- Liquid-Liquid Equilibrium
- Operating Modes of Extraction and calculations



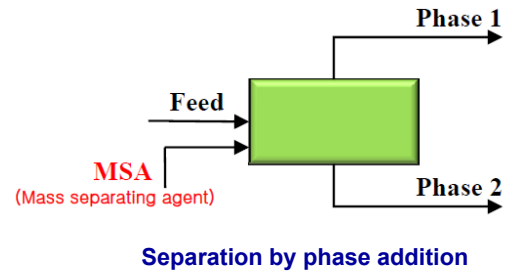
Principal references: Chapter 12 in C.J. Geankoplis book and Chapter 8 in Henley, Seader & Roper book



Overview and definitions



- Liquid-Liquid extraction (solvent extraction) is a mass transfer operation in which a liquid solution (the **feed or aqueous phase: Carrier + Solute**) is **contacted with an immiscible** or nearly immiscible liquid (**solvent or organic phase**) that exhibits **preferential affinity** or selectivity **towards one or more of the components in the feed (solute)**.



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Overview and definitions



- Pioneered during 1940's (uranium purification)
- Alternative to distillation, absorption/stripping
 - Energy savings
 - Sometimes easier separation
 - Lower temperatures
- Usually two distinct phases formed

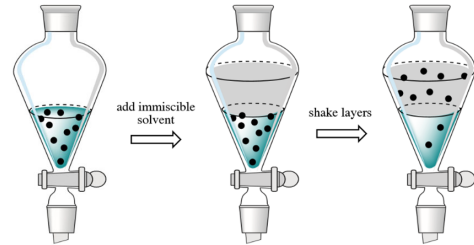
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Three major steps required in LLE:

1. Mixing/contacting:

- turbulent contact between liquid phases
- small droplet dispersion in a continuous phase
- which phase is dispersed?
- mass-transfer between phases
- limited by solute loading in solvent



2. Phase separation:

- reverse of above mixing step
- drops come together and coalesce
- relies on density difference

3. Collection of phases leaving the unit

- split the raffinate from the extract



Solvent Extraction Principle

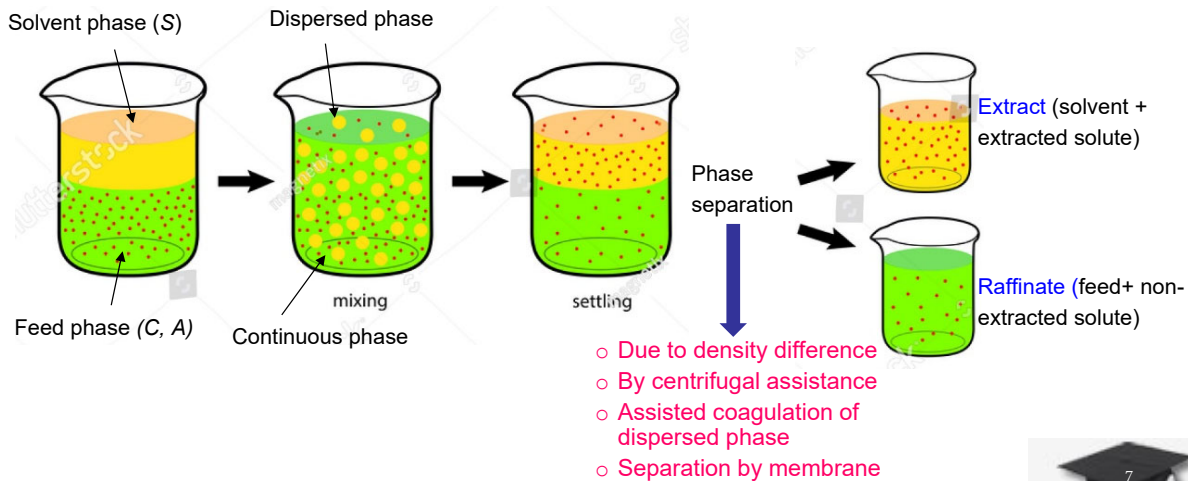
- The simplest liquid-liquid extraction involves only a ternary system.
 - The feed consists of two miscible components, the carrier (C) and the solute (A).
 - Addition of a second phase (solvent phase, S)
 - Components (C,S) are immiscible (**do not dissolve in one another**) or at most only partially soluble in each other.
 - Immiscible liquids form two distinct phases when mixed.
 - Solute (A) is soluble in (C) and completely or partially soluble in S.
 - During the extraction process, mass transfer of (A) from the feed to the solvent occurs, i.e. solute molecules are distributed between phases, with less transfer of (C) to the solvent, or (S) to the feed.
- After extraction; the **feed** and **solvent** phases are called the **Raffinate (R)** and **Extract (E)** phases respectively.



Solvent Extraction Principle



- **Extract** – the layer of solvent + extracted solute
- **Raffinate** – the layer from which solute has been removed (feed phase)



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Liquid-Liquid Extraction



- Normally one of the two phases is an organic phase while the other is an aqueous phase.
- Under equilibrium conditions the distribution of solute A over the two phases is determined by the distribution law.

$$K_D = \frac{(y_A)_E}{(x_A)_R}$$

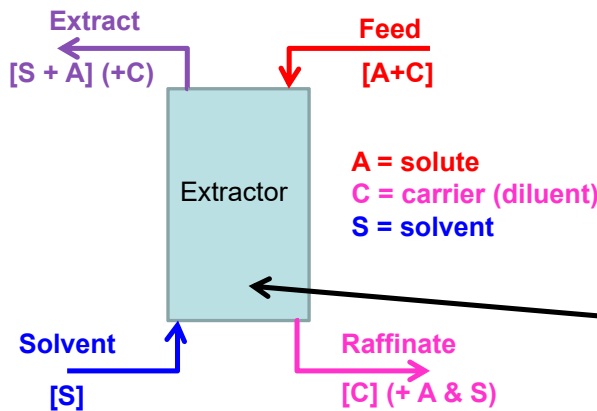
- After the extraction the two phases can be separated because of their immiscibility.
- Component A is then separated from the extract phase by a technique such as distillation and the solvent is regenerated.
- Further extractions may be carried out to remove more component A .



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Liquid-Liquid Extraction



- Separation accomplished by chemical differences
- Usually in two phase
 - - light phase
 - - heavy phase
- Usually coupled with another separation technique

Separator could be:
column w/ stages or packing
column with moving internals
single stage mixer/settler
equilibrium stage(s)

let: $x \equiv$ mass fraction solute in raffinate phase
 $y \equiv$ mass fraction solute in extract phase
 $y^* =$ in equilibrium with associated x

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Why Solvent Extraction?



Liquid-liquid extraction is used to separate components in situations where:

1. Relative volatilities are quite close to unity ($\alpha < 1.1$), or azeotropic making distillation very costly. (Distillation requires tall towers due to the existence of many trays, and high energy consumption because of high reflux ratios.)

e.g. A mixture of benzene and cyclohexane. The normal boiling points of these organics are 80.1°C and 80.7°C, respectively, making their separation by distillation impractical

2. Thermally sensitive components will not permit high enough temperatures to produce a vapor-liquid system at reasonable pressures (pressures greater than 10-50 mm Hg).
3. When Solute concentration is low

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Applications



- Usual purpose, to remove products and pollutants from dilute aqueous streams (purify the Raffinate, or Solute)
- Wash polar compounds or acids/bases from organic streams
- Example:
 - recovery of penicillin from fermentation broth
solvent: butyl acetate
 - recovery of acetic acid (b.p 118⁰c) from dilute aqueous (b.p 100⁰c) solutions
solvent: ethyl-acetate



Example Industrial Processes



Table 8.1 Representative Industrial Liquid-Liquid Extraction Processes

Solute [A]	Carrier [C]	Solvent [S]	Solute	Carrier	Solvent
Acetic acid	Water	Ethyl acetate	Glycerol	Water	High alcohols
Acetic acid	Water	Isopropyl acetate	Hydrogen peroxide	Anthrahydroquinone	Water
Aconitic acid	Molasses	Methyl ethyl ketone	Methyl ethyl ketone	Water	Trichloroethane
Ammonia	Butenes	Water	Methyl borate	Methanol	Hydrocarbons
Aromatics	Paraffins	Diethylene glycol	Naphthenes	Distillate oil	Nitrobenzene
Aromatics	Paraffins	Furfural	Naphthenes/ aromatics	Distillate oil	Phenol
Aromatics	Kerosene	Sulfur dioxide	Phenol	Water	Benzene
Aromatics	Paraffins	Sulfur dioxide	Phenol	Water	Chlorobenzene
Asphaltenes	Hydrocarbon oil	Furfural	Penicillin	Broth	Butyl acetate
Benzoic acid	Water	Benzene	Sodium chloride	aq. Sodium hydroxide	Ammonia
Butadiene	1-Butene	aq. Cuprammonium acetate	Vanilla	Oxidized liquors	Toluene
Ethylene cyanohydrin	Methyl ethyl ketone	Brine liquor	Vitamin A	Fish-liver oil	Propane
Fatty acids	Oil	Propane	Vitamin E	Vegetable oil	Propane
Formaldehyde	Water	Isopropyl ether	Water	Methyl ethyl ketone	aq. Calcium chloride
Formic acid	Water	Tetrahydrofuran			



SOLVENT MISCIBILITY TABLE

Solvent	Polarity Index	Refractive Index @20°C	UV(nm) Cutoff @1AU	Boiling Point(°C)	Viscosity (cPoise)	Solubility in water (%w/w)
Acetic Acid	6.2	1.372	230	118	1.26	100
Acetone	5.1	1.359	330	56	0.32	100
Acetonitrile	5.8	1.344	190	82	0.37	100
Benzene	2.7	1.501	280	80	0.65	0.18
n-Butanol	4.0	1.394	254	125	0.73	0.43
Butyl Acetate	3.9	1.399	215	118	2.98	7.81
Carbon Tetrachloride	1.6	1.466	263	77	0.97	0.08
Chloroform	4.1	1.446	245	61	0.57	0.815
Cyclohexane	0.2	1.426	200	81	1.00	0.01
1,2-Dichloroethane ¹	3.5	1.444	225	84	0.79	0.81
Dichloromethane ²	3.1	1.424	235	41	0.44	1.6
Dimethylformamide	6.4	1.431	268	155	0.92	100
Dimethyl Sulfoxide ³	7.2	1.478	268	189	2.00	100
Dioxane	4.8	1.422	215	101	1.54	100
Ethanol	5.2	1.360	210	78	1.20	100
Ethyl Acetate	4.4	1.372	260	77	0.45	8.7
Di-Ethyl Ether	2.8	1.353	220	35	0.32	6.89
Heptane	0.0	1.387	200	98	0.39	0.003
Hexane	0.0	1.375	200	69	0.33	0.001
Methanol	5.1	1.329	205	65	0.60	100
Methyl-t-Butyl Ether ⁴	2.5	1.369	210	55	0.27	4.8
Methyl Ethyl Ketone ⁵	4.7	1.379	329	80	0.45	24
Pentane	0.0	1.358	200	36	0.23	0.004
n-Propanol	4.0	1.384	210	97	2.27	100
Iso-Propanol ⁶	3.9	1.377	210	82	2.30	100
Di-Iso-Propyl Ether	2.2	1.368	220	68	0.37	
Tetrahydrofuran	4.0	1.407	215	65	0.55	100
Toluene	2.4	1.496	285	111	0.59	0.051
Tichloroethylene	1.0	1.477	273	87	0.57	0.11
Water	9.0	1.333	200	100	1.00	100
Xylene	2.5	1.500	290	139	0.61	0.018

<p>■ Immiscible</p> <p>□ Miscible</p> <p>Immiscible means that in some proportions two phases will be produced</p>	<p>Synonym Table</p> <p>¹ Ethylene Chloride</p> <p>² Methylene Chloride</p> <p>³ Methyl Sulfoxide</p> <p>⁴ tert-Butyl Methyl Ether</p> <p>⁵ 2-Butanone</p> <p>⁶ 2-Propanol</p>
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Extraction is Used in a Wide Variety of Industries

Chemical	<ul style="list-style-type: none"> • Washing of acids/bases, polar compounds from organics
Pharmaceuticals	<ul style="list-style-type: none"> • Recovery of active materials from fermentation broths • Purification of vitamin products
Effluent Treatment	<ul style="list-style-type: none"> • Recovery of phenol, DMF, DMAC • Recovery of acetic acid from dilute solutions
Polymer Processing	<ul style="list-style-type: none"> • Recovery of caprolactam for nylon manufacture • Separation of catalyst from reaction products
Petroleum	<ul style="list-style-type: none"> • Lube oil quality improvement • Separation of aromatics/aliphatics (BTX)
Petrochemicals	<ul style="list-style-type: none"> • Separation of olefins/parafins • Separation of structural isomers
Food Industry	<ul style="list-style-type: none"> • Decaffeination of coffee and tea • Separation of essential oils (flavors and fragrances)
Metals Industry	<ul style="list-style-type: none"> • Copper production • Recovery of rare earth elements
Inorganic Chemicals	<ul style="list-style-type: none"> • Purification of phosphoric acid
Nuclear Industry	<ul style="list-style-type: none"> • Purification of uranium



Analogy Between Extraction and Distillation



Distillation	Extraction
Addition of heat	Addition of solvent
Reboiler	Solvent mixer
Removal of heat	Removal of solvent
Condenser	Solvent separator
Vapor at the boiling point	Solvent-rich solution saturated with solvent
Superheated vapor	Solvent-rich solution containing more solvent than that required to saturate it
Liquid below the boiling point	Solvent-lean solution, containing less solvent than that required to saturate it
Liquid at the boiling point	Solvent-lean solution saturated with solvent
Mixture of liquid and vapor	Two-phase liquid mixture
Relative volatility	Relative selectivity
Change of pressure	Change of temperature
D = distillate	D = extract product (solute on a solvent-free basis)
B = bottoms	B = raffinate (solvent-free basis)
L = saturated liquid	L = saturated raffinate (solvent-free)
V = saturated vapor	V = saturated extract (solvent-free)
A = more volatile component	A = solute to be recovered
C = less volatile component	C = carrier from which A is extracted
F = feed	F = feed
x = mole fraction A in liquid	X = mole or weight ratio of A (solvent-free), $A/(A + C)$
y = mole fraction A in vapor	$Y = S/(A + C)$

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Properties of Extraction Solvents



- The chosen solvent has to meet certain requirements for an efficient extraction
 - Selectivity
 - The solvent should be immiscible or only slightly be miscible with “Feed aqueous phase” to be extracted.
 - The target compound should dissolve very well in the solvent at room temperature (“**like dissolves like**” rule applies) → a large difference in solubility leads to a large value for the *partition coefficient* (also called *distribution coefficient*), which is important for an efficient extraction
 - Relatively low boiling point (Low vapour pressure) for easy removal at a later stage of the product isolation
 - Chemical reactivity: Stable and inert (non-reactive).

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Properties of Extraction Solvents



- Recoverability of solute from solvent
 - No azeotrope formed between solvent and solute
 - Mixtures should have a high relative volatility
 - Solvent should have a small latent heat of vapourisation
- A density difference is required between the two phases (densities determine top or bottom)
- Interfacial tension:

The larger the interfacial tension between the two phases the more readily coalescence of emulsions will occur to give two distinct liquid phases but the more difficult will be the dispersion of one liquid in the other to give efficient solute extraction.
- Non-toxic, Low viscosity, Non-flammable (high flash point), cheap, available

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Solvent miscibility



- Completely miscible
 - Unsuitable for extraction
- Immiscible
 - Ideally suited for extraction
- Partially miscible
 - Composition dependent
 - Various possibilities
 - Could be used for extraction
- Solubility of organic compounds is a function of the polarities of both the solvent and the solute:
 - “Like Dissolves Like”
 - Polar solvents dissolve polar solutes
 - Nonpolar solvents dissolve nonpolar solutes

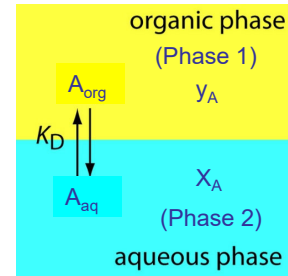
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Distribution Coefficients



- The partitioning of solute s between two chemical phases (1 and 2) is described by the equilibrium constant K_D (the partition or distribution coefficient)



- It is defined as the ratio of concentrations of a solute that is distributed between two immiscible solvents at equilibrium.

$$K_D = \frac{\text{solute concentration in extract phase}}{\text{solute concentration in raffinate phase}}$$

$$K_D = \frac{(y_A)_E}{(x_A)_R}$$



Distribution Coefficients



- Higher value of K_D indicates higher extraction efficiency.

Ideal condition, when $K_D \gg \gg 1$ or $\ll \ll \ll 1$

e.g. water – chloroform

Consider a feed of water/acetone(solute).

$$K = \frac{\text{mass fraction acetone in chloroform phase}}{\text{mass fraction acetone in water phase}}$$

$$K = \frac{\text{kg acetone/kg chloroform}}{\text{kg acetone/kg water}} = y/x$$

👉 $K = 1.72$

i.e. acetone is preferentially soluble in the chloroform phase



Distribution Coefficients for Immiscible Extraction



$$K_d = \frac{y_A}{x_A}$$

K_d = distribution coeff.

y_A = Solute frac. in Extract

x_A = Solute frac. in Raffinate

Solute (A)	Solvent	Diluent	T, °C	$K_d = y_A/x_A$
<i>Equilibrium in Weight Fraction Units (Perry and Green, 1984)</i>				
Acetic acid	Benzene	Water	25	0.0328
Acetic acid	Benzene	Water	30	0.0984
Acetic acid	Benzene	Water	40	0.1022
Acetic acid	Benzene	Water	50	0.0588
Acetic acid	Benzene	Water	60	0.0637
Acetic acid	1-Butanol	Water	26.7	1.613
Furfural	Methylisobutyl ketone	Water	25	7.10
Ethyl benzene	β, β' -Thiodipropionitrile	n-Hexane	25	0.100
m-Xylene	β, β' -Thiodipropionitrile	n-Hexane	25	0.050
o-Xylene	β, β' -Thiodipropionitrile	n-Hexane	25	0.150
p-Xylene	β, β' -Thiodipropionitrile	n-Hexane	25	0.080
<i>Equilibrium in Mass Ratio Units (Brian, 1972)</i>				
Linoleic acid ($C_{17}H_{31}COOH$)	Heptane	Methylcellosolve + 10 vol % water		2.17
Abietic acid ($C_{19}H_{29}COOH$)	Heptane	Methylcellosolve + 10 vol % water		1.57
Oleic acid	Heptane	Methylcellosolve + 10 vol % water		4.14

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Distribution Coefficients for Immiscible Extraction



Solute	Organic solvent	K_D (mol/L) at 25°C
Amino acids		
Glycine	n-butanol	0.01
Alanine	n-butanol	0.02
2-aminobutyric acid	n-butanol	0.02
Lysine	n-butanol	0.20
Glutamic acid	n-butanol	0.07
Antibiotics		
Erythromycin	Amyl acetate	120
Novobiocin	Butyl acetate	100 at pH 7.0 0.01 at pH 10.5
Penicillin F	Amyl acetate	32 at pH 4.0 0.06 at pH 6.0
Penicillin K	Amyl acetate	12 at pH 4.0 0.1 at pH 6.0

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Example



An organic molecule with distribution coefficient (or partition coefficient) of 10 between ether and water, and 150 mL of ether will be used to extract 5.0 g of such organic molecule from 100 mL of water.

Assuming m_1 gram of molecule is left in aqueous phase after the equilibrium is reached, then $5 - m_1$ gram of organic will enter the ether layer. Thus, we have:

$$K_D = 10 = \frac{\frac{5.0 - m_1}{150} \frac{g}{mL_{ether}}}{\frac{m_1}{100} \frac{g}{mL_{water}}} \longrightarrow 10 = \frac{(5.0 - m_1)(100)}{150m_1}$$

$$1500m_1 = 500 - 100m_1 \longrightarrow 1600m_1 = 500$$

$$m_1 = 0.31g$$

$$5.0 - m_1 = 5.0 - 0.31 = 4.69g$$

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Example Cont.d



After single extraction, only 0.31 g of organic left in water, and 4.69 g is separated from water.

The extraction efficiency = $4.69/5.0 = 93.8\%$.

Example 2

Same amount of ether (150 mL) is used to extract the same organic from 100 mL water but in three portions, each with 50 mL of ether.

Similar calculation can be applied for each cycle of extraction, as follows:

- First cycle of extraction

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Example Cont.d



$$K_D = 10 = \frac{\frac{5.0 - m_1}{g}}{\frac{m_1}{g}} \cdot \frac{50 \text{ mL}_{ether}}{100 \text{ mL}_{water}}; \quad 10 = \frac{(5.0 - m_1)(100)}{50m_1}$$

$$500m_1 = 500 - 100m_1$$

$$600m_1 = 500$$

$$m_1 = 0.83$$

Hence, 0.83 g of organic remains in aqueous phase, 4.17 g of organic stays in ether

Extraction efficiency = $4.17/5.0 = 83.4\%$



Example Cont.d



2nd Cycle of Extraction

$$K_D = 10 = \frac{\frac{0.83 - m_1}{g}}{\frac{m_1}{g}} \cdot \frac{50 \text{ mL}_{ether}}{100 \text{ mL}_{water}}; \quad 10 = \frac{(0.83 - m_1)(100)}{50m_1}$$

$$500m_1 = 83 - 100m_1$$

$$600m_1 = 83$$

$$m_1 = 0.14$$

- After 2nd cycle of extraction, only 0.14 g of organic is left in aqueous phase, 0.69 g of organic stay in 50 mL of ether.
- 4.86 g of organic is extracted into 100 mL of ether, the extraction efficiency is $4.86/5.0 = 97.2\%$, already greater than the single extraction (93.8%).



Example Cont.d



3rd Cycle of Extraction

$$K_D = 10 = \frac{\frac{0.14 - m_1}{50} \frac{g}{mL_{ether}}}{\frac{m_1}{100} \frac{g}{mL_{water}}}; \quad 10 = \frac{(0.14 - m_1)(100)}{50m_1}$$

$$500m_1 = 14 - 100m_1$$

$$600m_1 = 14$$

$$m_1 = 0.02$$

- After 3rd cycle of extraction, only 0.02 g of organic remains in aqueous phase, and 4.98 g of organic in total is extracted into 150 mL of ether.
- The extraction efficiency is $4.98/5 = 99.6\%$.
- If same amount of organic solvent is used for extraction of one molecule, the extraction in several portions is much more efficient than single extraction using the whole amount of solvent.

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Distribution Coefficients



- In general, using this partition coefficient, one could determine how much of the compound is extracted in each extraction or after n extractions:

$$\frac{x}{W_o} = \frac{(Final\ mass\ of\ solute)_{remaining}}{(Initial\ mass\ of\ solute)_{feed}} = \left(\frac{V_{feed}}{V_{feed} + K_D V_{Solvent}} \right)^n$$

- K_D = Partition coefficient or distribution coefficient
- $V_{solvent}$ = Volume of the organic solvent in each extraction
- V_{feed} = Original volume of feed
- n = number of extractions
- W_o = Initial mass of solute in the feed
- x = Solute remains in aqueous phase

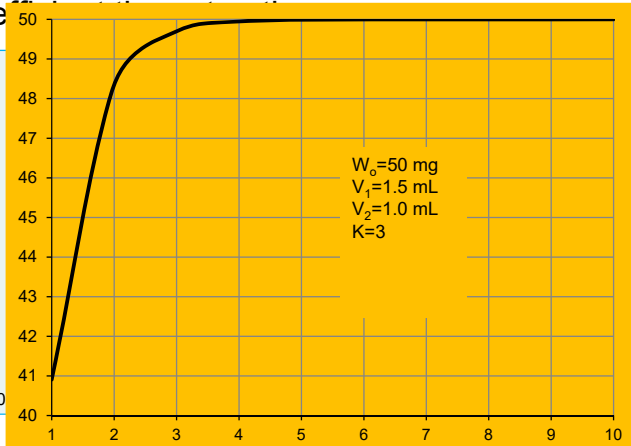
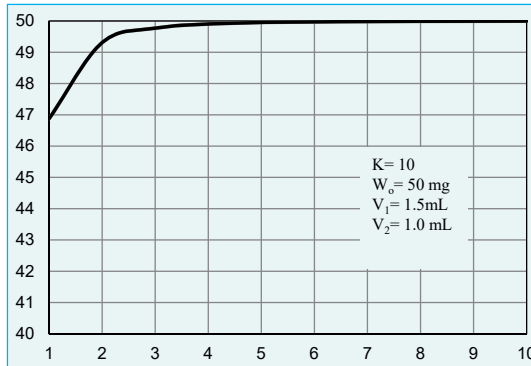
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Distribution Coefficients



- The larger the K-value, the more efficient the extraction process



- For $K=10$, two extractions are sufficient to extract about 99.9%.
- For $K=3$, four extractions are required to accomplish the same degree of the extraction.



Task



The distribution ratio for iodine between water and carbon disulfide is 650. Calculate the concentration of I_2 remaining in the aqueous phase after 50.0 mL of 0.10M I_2 in water is shaken with 10.0 mL of CS_2 .

SOLUTION

The number of moles of solute is $(50 \text{ mL}) \times (0.10 \text{ mmol mL}^{-1}) = 5.00 \text{ mmol}$,

Assuming m_1 gram of molecule is left in aqueous phase after the equilibrium is reached, then $5 - m_1$ gram of organic will enter the ether layer. Thus, we have:

The equilibrium constant is

$$K_D = 650 = \frac{\frac{5.0 - m_1 \text{ mmol}}{10 \text{ mL}_{ether}}}{\frac{m_1 \text{ mmol}}{50 \text{ mL}_{water}}} \longrightarrow 650 = \frac{(5.0 - m_1)(50)}{10m_1}$$



Task



$$6500m_1 = 250 - 50m_1 \longrightarrow 6550m_1 = 250$$

$$m_1 = 0.0382 \text{ mmol}$$

The concentration of solute in the water layer is $(0.0382 \text{ mmol}) / (50 \text{ mL}) = \mathbf{0.000763 \text{ M}}$, showing that almost all of the iodine has moved into the CS_2 layer.



Excursion



Liquid-liquid Equilibrium

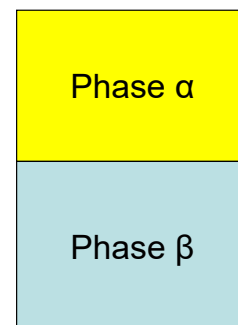
- The fugacities of species a in each liquid phase are equal:

$$\hat{f}_a^\alpha = \hat{f}_a^\beta$$
$$x_a^\alpha \gamma_a^\alpha f_a^\alpha = x_a^\beta \gamma_a^\beta f_a^\beta$$

- For the same reference state gives:

$$\cancel{x_a^\alpha \gamma_a^\alpha f_a^\alpha} = \cancel{x_a^\beta \gamma_a^\beta f_a^\beta}$$

➔ $x_a^\alpha \gamma_a^\alpha = x_a^\beta \gamma_a^\beta$



γ_a^α : Activity coefficient (obtained from Activity coefficient model for g^E)



- **Two phases:**
 - Extract
 - Raffinate phases
- **Usually three components:**
 - Solute (A)
 - Carrier (C)
 - Solvent (S)**(ternary system)**



At certain Temperature and pressure

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- The immiscible liquid phases put in contact (the feed and the solvent) form a closed system evolving towards the thermodynamic equilibrium. According to the Gibbs law:

$$\begin{aligned} F &= C - P + 2 \\ &= 3 - 2 + 2 = 3 \end{aligned}$$

- The system can be defined by three parameters ($f = 3$), the number of components being $c = 3$, (solvent, solute and carrier), and the phases number $P = 2$.
- Usually, the parameters taken into account are the temperature (T), the concentration of the solute in the raffinate (x) and the concentration in the extract (y).
- So, the equilibrium general equation in this case is:

$$y = f(x)_{t=const}$$

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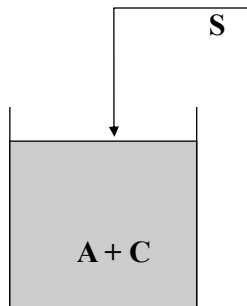


Liquid-Liquid Systems



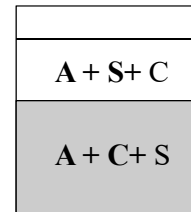
- The solvent S is added into the mixture of the diluent C and the solute A, when the system reaches the equilibrium, it

could be *homogeneous* (i.e. all species are mixed together into a single phase)



could be divided into 2 phases

Type 0, Type I, Type II



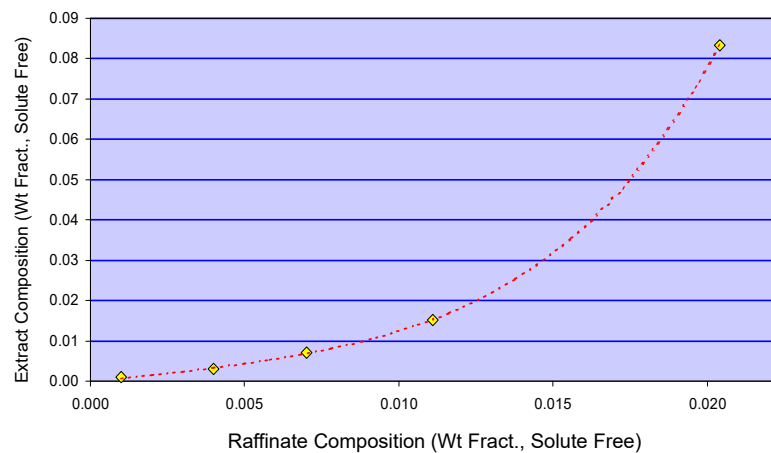
Liquid-Liquid Equilibrium



Typical LLE Equilibrium Curve

$$Y_{AE} = \frac{y_{AE}}{y_{SE} + y_{CE}}$$

$$= \frac{y_{AE}}{1 - y_{AE}}$$



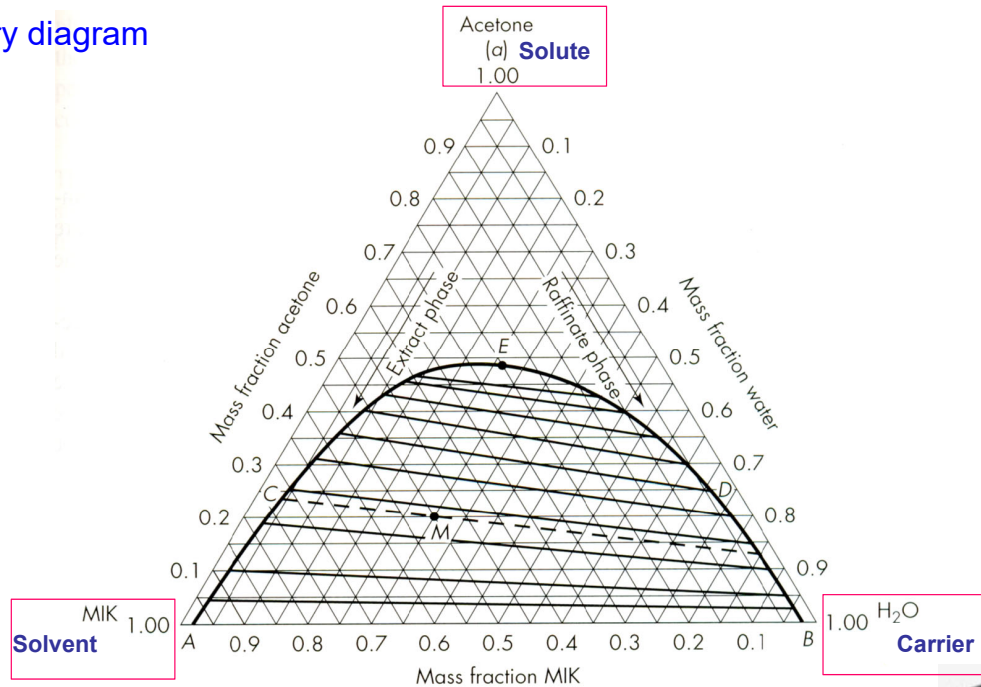
$$X_{AR} = \frac{x_{AR}}{x_{SR} + x_{CR}} = \frac{x_{AR}}{1 - x_{AR}}$$



Liquid-Liquid Equilibrium



Ternary diagram



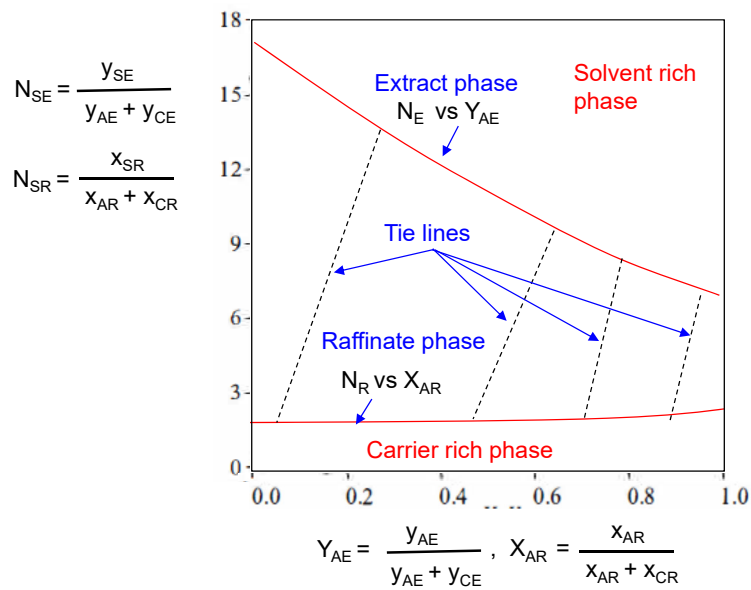
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Liquid-Liquid Equilibrium



Janecke plot of liquid-liquid equilibrium data. (A few tie lines are also shown.)



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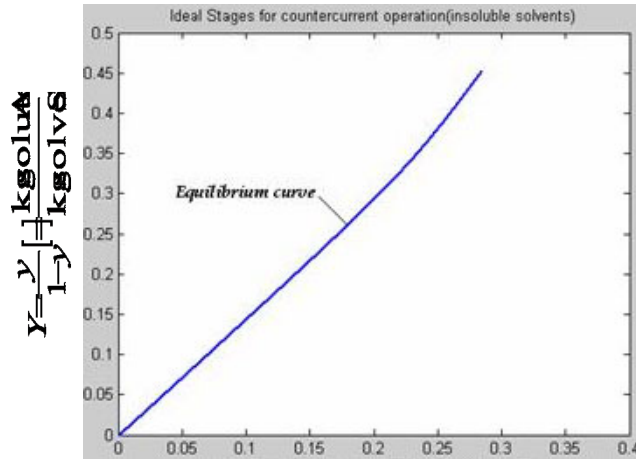
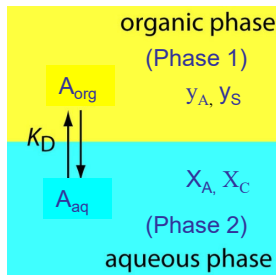


Liquid-Liquid Systems



Type 0

- If the **solute A** is **soluble in Carrier (C) and Solvent (S)**, while **C and S are completely insoluble**, then the extract and raffinate phases will be binary mixtures.
- Thus, solute is the only component distributed in extract and raffinate.



$$X = \frac{x}{1-x} \left[= \right] \frac{\text{kg solute A}}{\text{kg carrier C}}$$

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Liquid-Liquid Systems



- Equilibrium relationships are more complicated – 3 or more components present in each phase
 - **Type I:** A dissolves completely in S and C, while C and S are only **partially miscible**
 - **Type II:** the pairs A and S, S and C, are partially miscible pairs, while A and C are totally soluble

Triangular Diagrams

Ternary systems are represented on two types of triangular diagrams:

1. Equilateral triangles
2. Right Triangles

Assumptions

- 1) The system is isothermal
- 2) The system is isobaric
- 3) The heat of mixing is negligible
- 4) No chemical reactions

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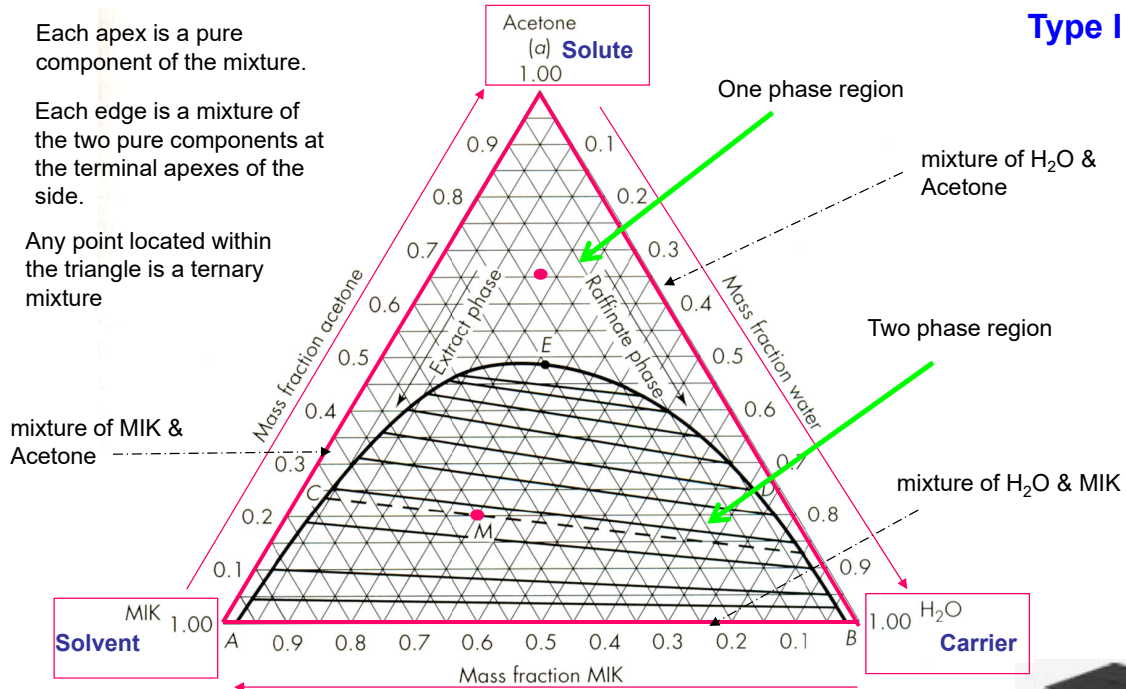


Liquid-Liquid Systems



- Each apex is a pure component of the mixture.
- Each edge is a mixture of the two pure components at the terminal apexes of the side.
- Any point located within the triangle is a ternary mixture

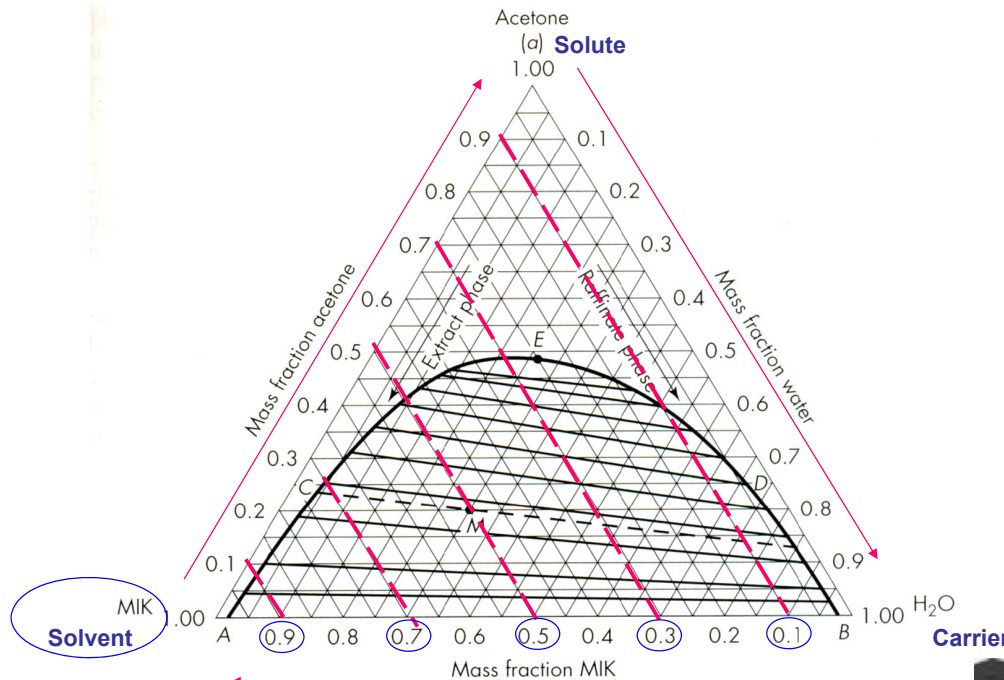
Type I



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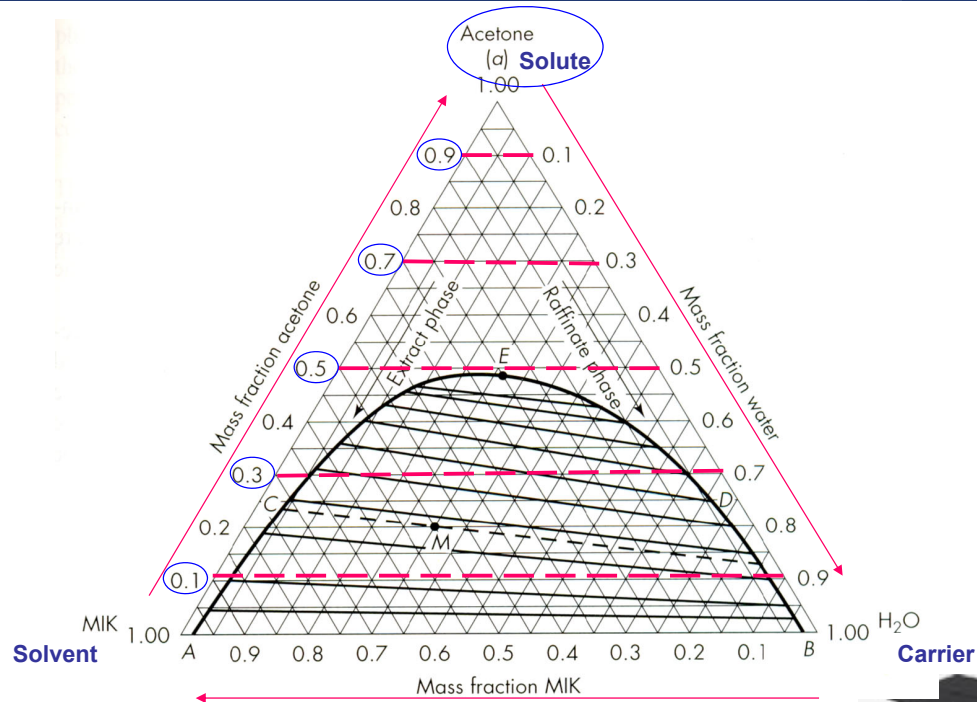
Liquid-Liquid Systems



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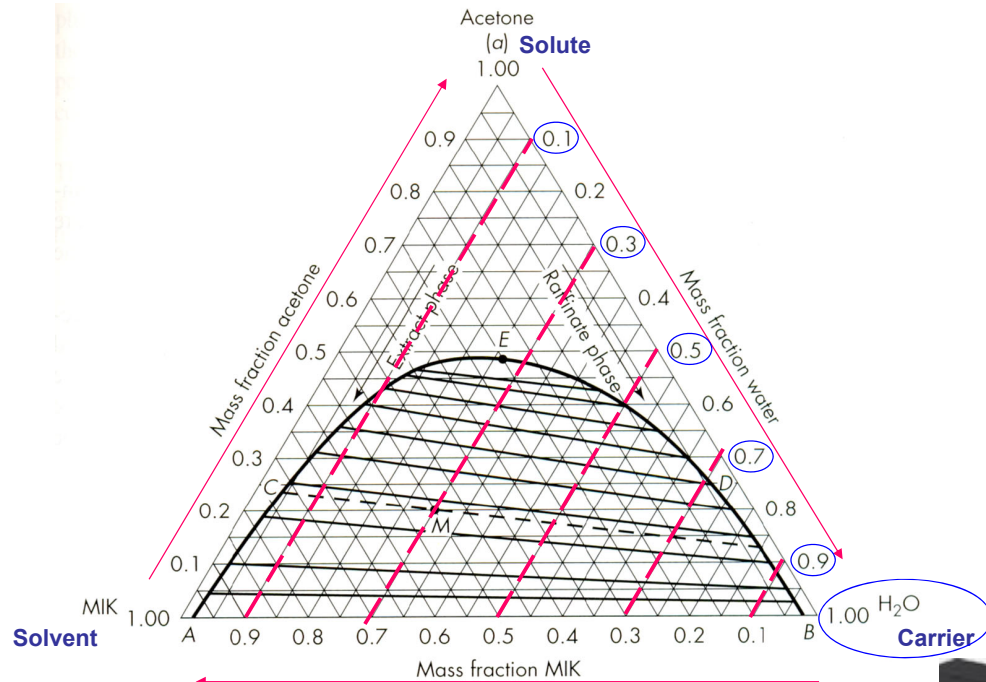
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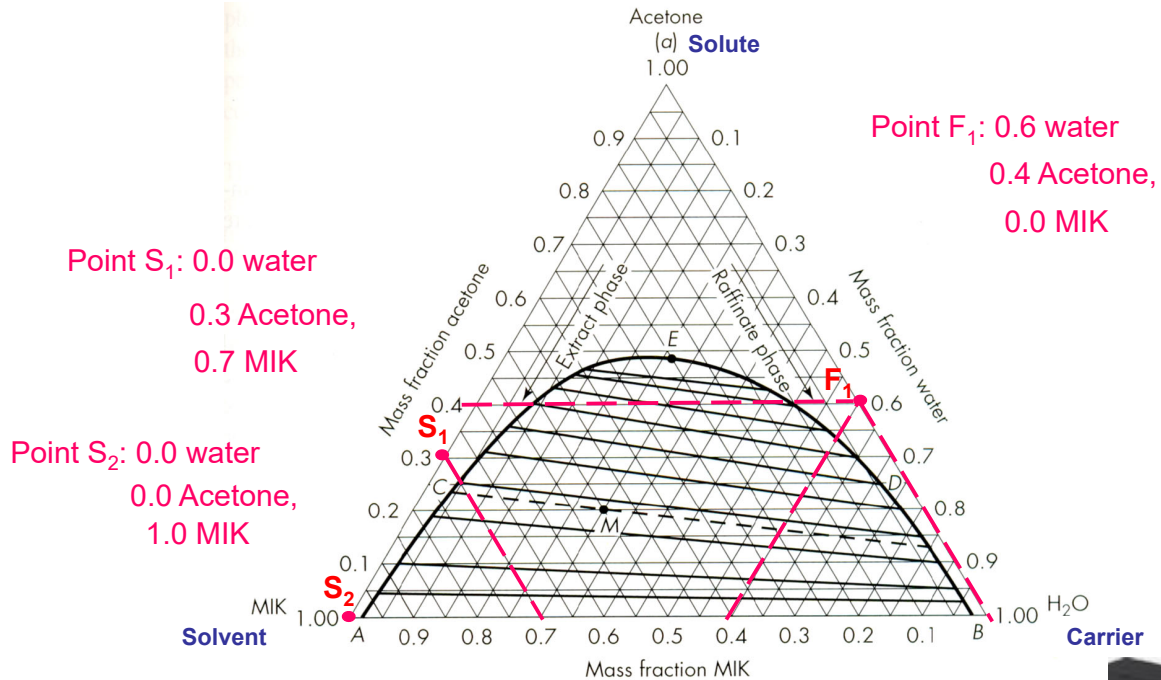
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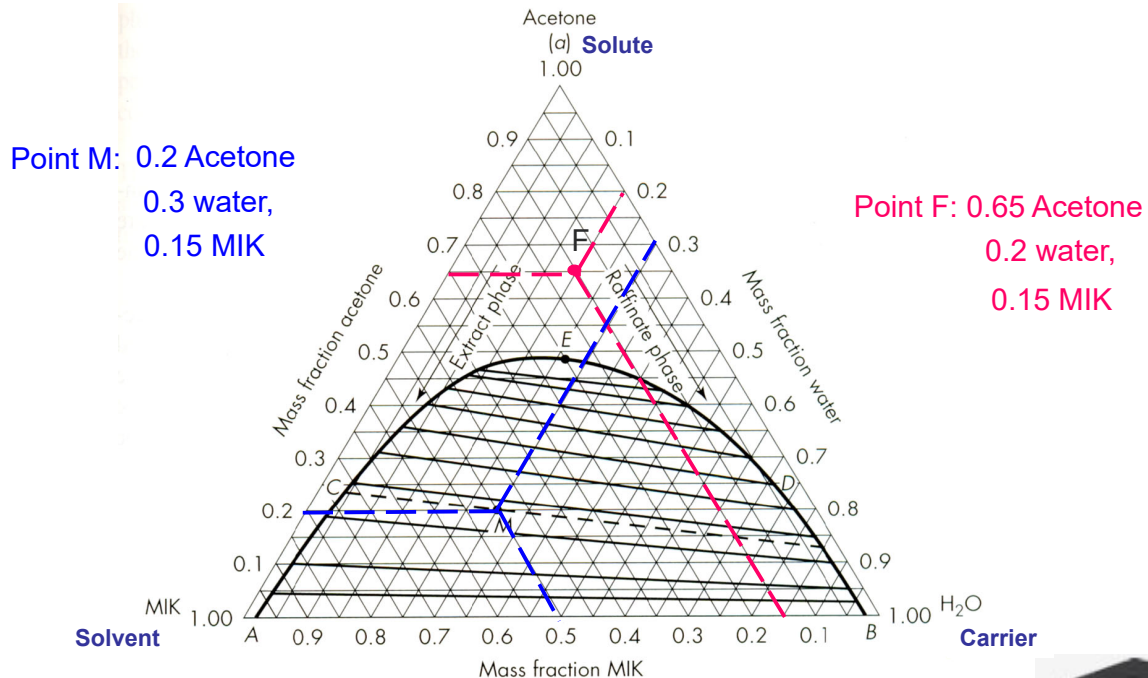
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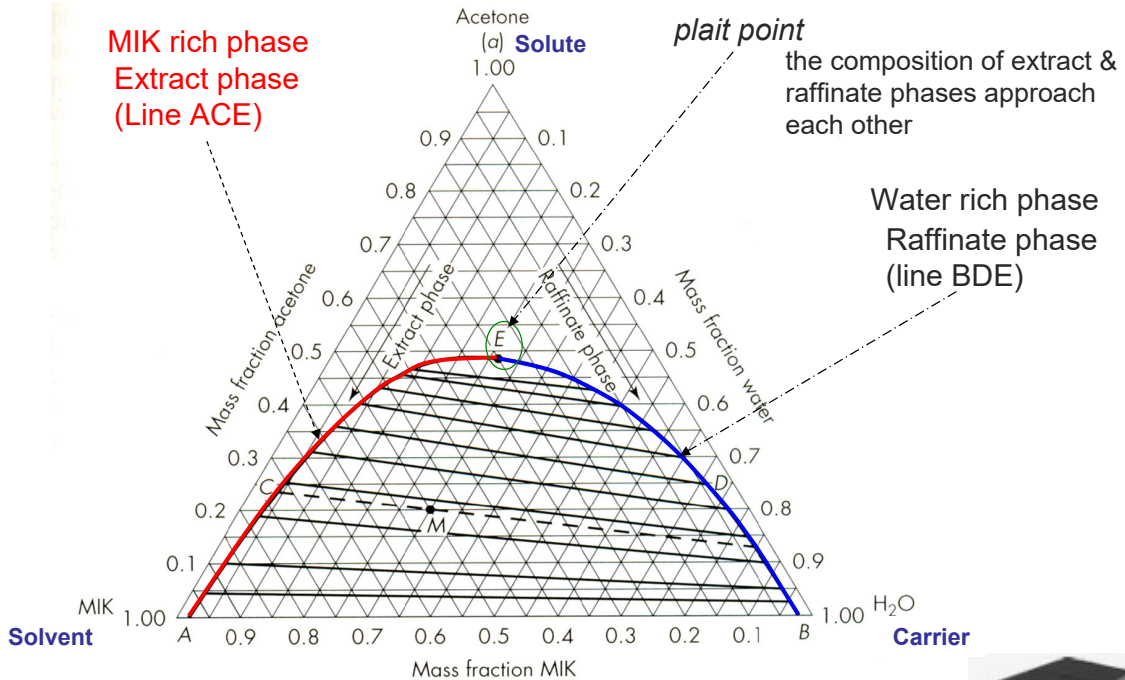
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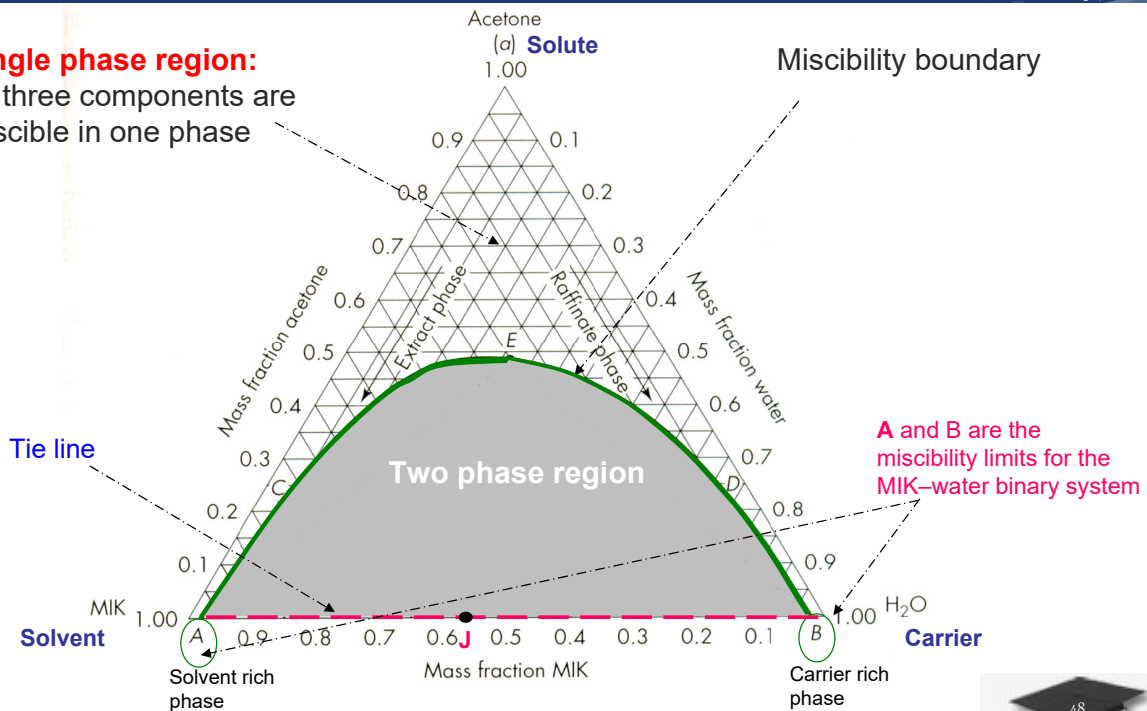


Liquid-Liquid Systems



Single phase region:

All three components are miscible in one phase



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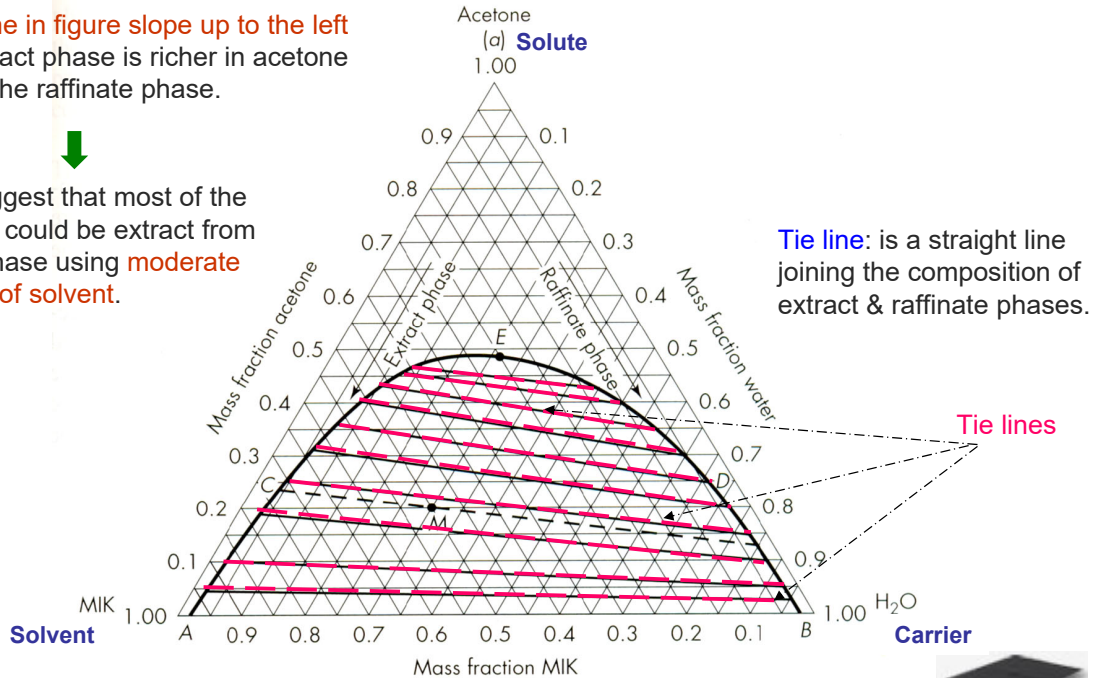


Liquid-Liquid Systems

- Tie line in figure slope up to the left – extract phase is richer in acetone than the raffinate phase.



This suggest that most of the acetone could be extract from water phase using moderate amount of solvent.



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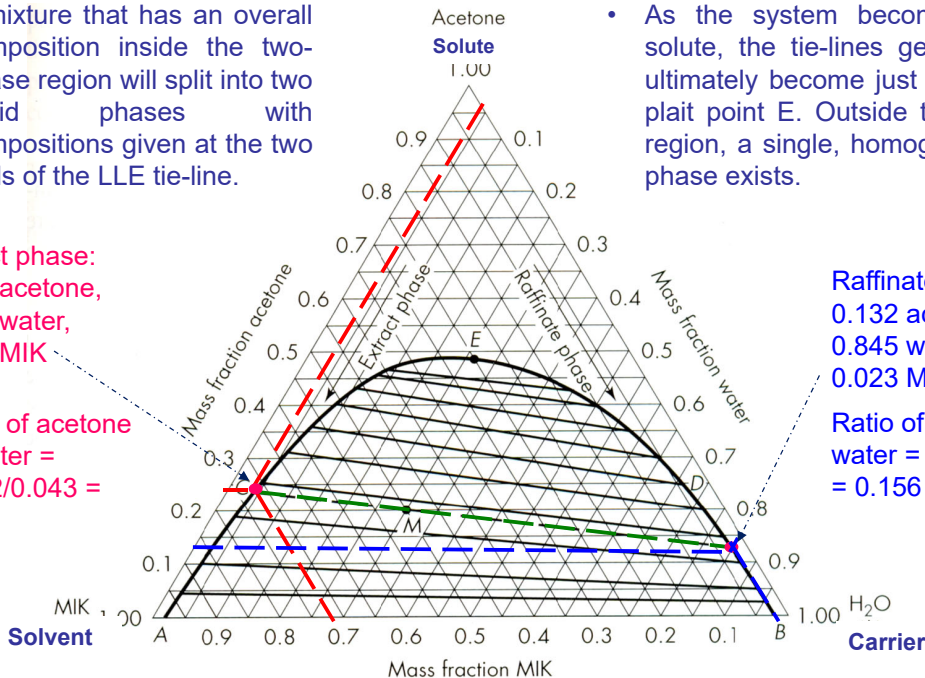
Liquid-Liquid Systems

- A mixture that has an overall composition inside the two-phase region will split into two liquid phases with compositions given at the two ends of the LLE tie-line.

- As the system becomes richer in solute, the tie-lines get shorter and ultimately become just a point at the plait point E. Outside the two-phase region, a single, homogeneous liquid phase exists.

Extract phase:
0.232 acetone,
0.043 water,
0.725 MIK

Ratio of acetone
to water =
 $0.232/0.043 =$
5.4



Raffinate phase:
0.132 acetone,
0.845 water,
0.023 MIK

Ratio of acetone
to water =
 $0.132/0.845 =$
0.156

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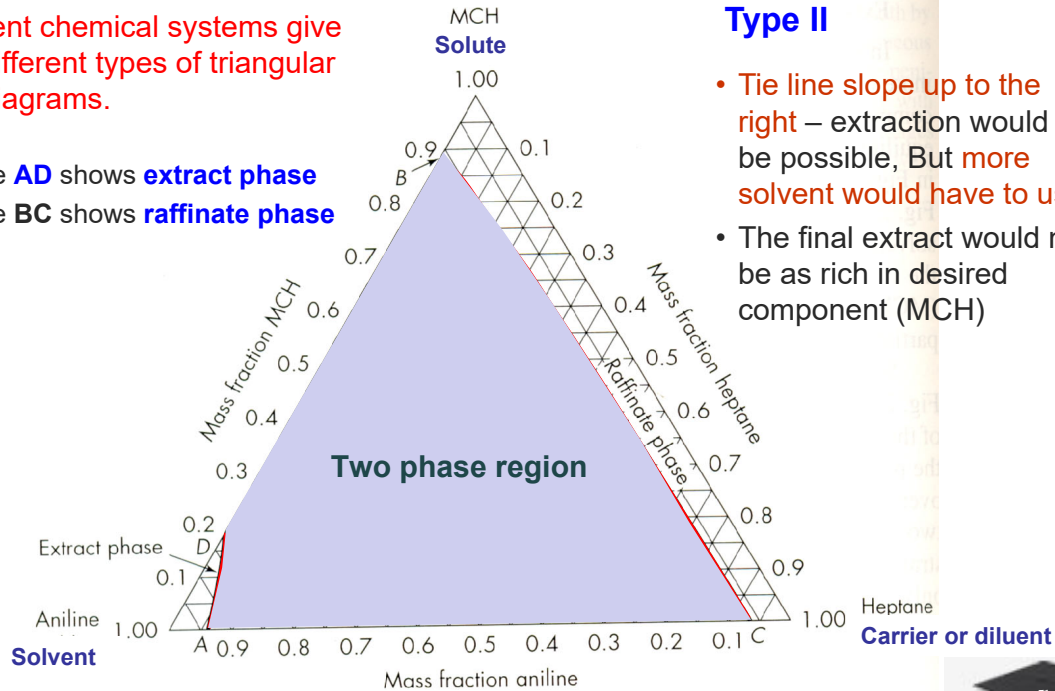


Liquid-Liquid Systems



Different chemical systems give different types of triangular diagrams.

- Line **AD** shows **extract phase**
- Line **BC** shows **raffinate phase**



Type II

- Tie line slope up to the right – extraction would still be possible, But **more solvent would have to use**.
- The final extract would not be as rich in desired component (MCH)

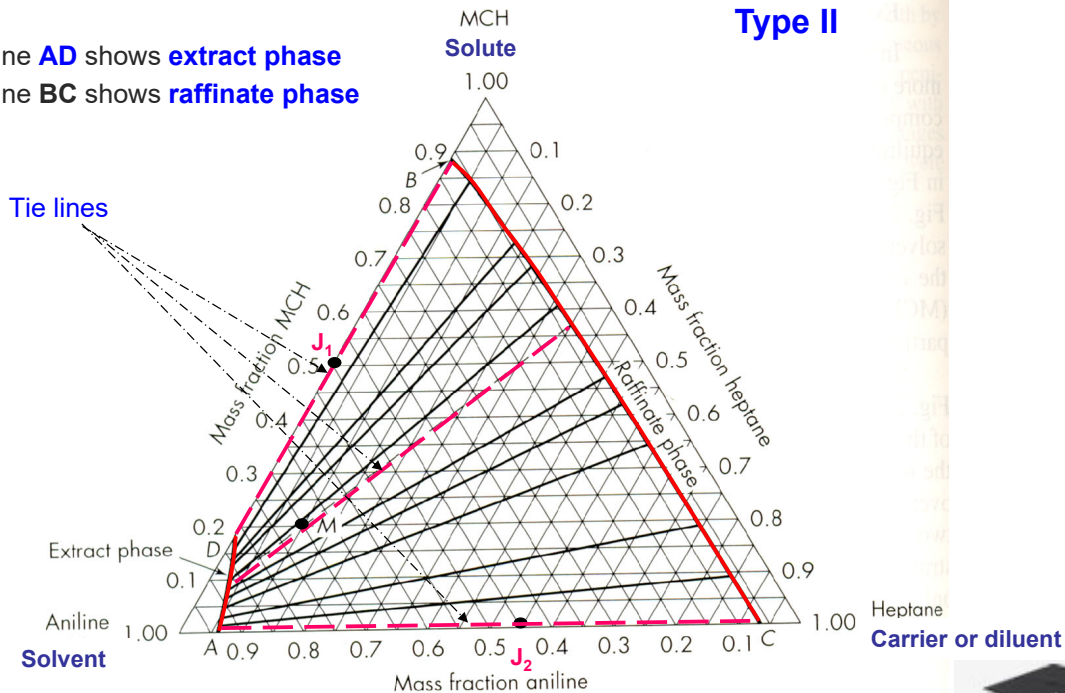
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Liquid-Liquid Systems



- Line **AD** shows **extract phase**
- Line **BC** shows **raffinate phase**

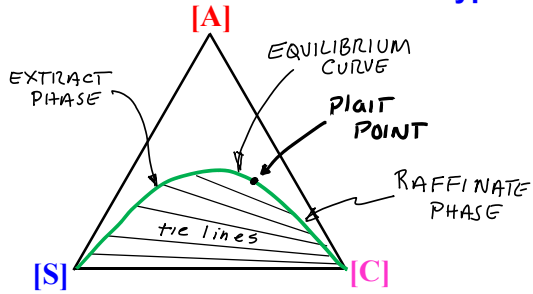


Type II

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Type-I (closed ternary system)

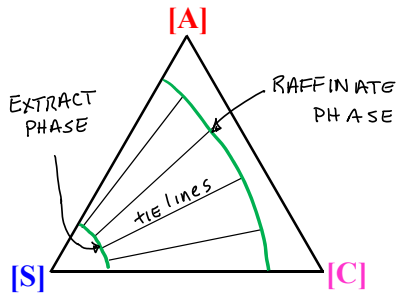


- The solute is miscible (form homogenous single phase) with the carrier and with the solvent in all proportions (A-C and A-S are miscible pairs); but the carrier and the solvent are only partially miscible (the pair C-S is partially miscible, form two separate phases).

Examples:

- water (C), ethylene glycol (a), furfural (s)
- water (C), acetone (a), chloroform (s)

Type-II (open ternary system)



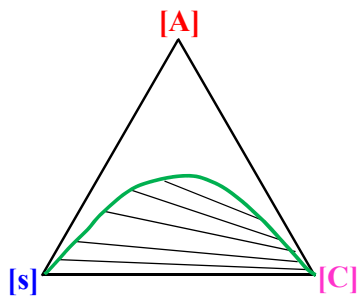
- The solute is completely miscible with the carrier (the binary A-C is miscible in all proportions); but both the solute and the carrier have limited miscibility with the solvent (the pairs C-S and A-S are only partially miscible, i.e. form two separate phases).

Example:

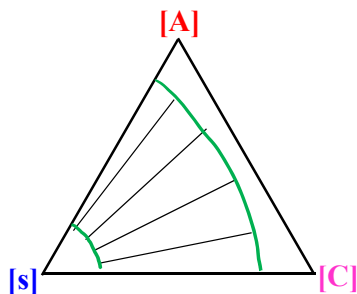
- n-heptane (C), methylcyclohexane (a), aniline (S)



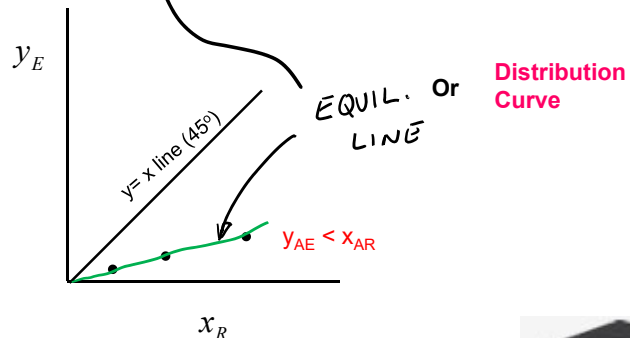
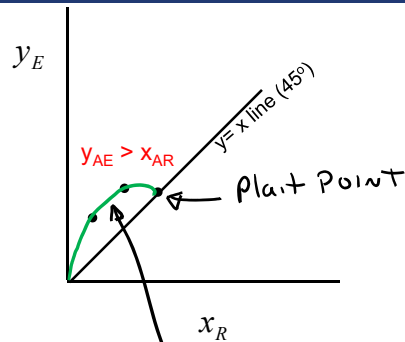
Liquid-Liquid Systems



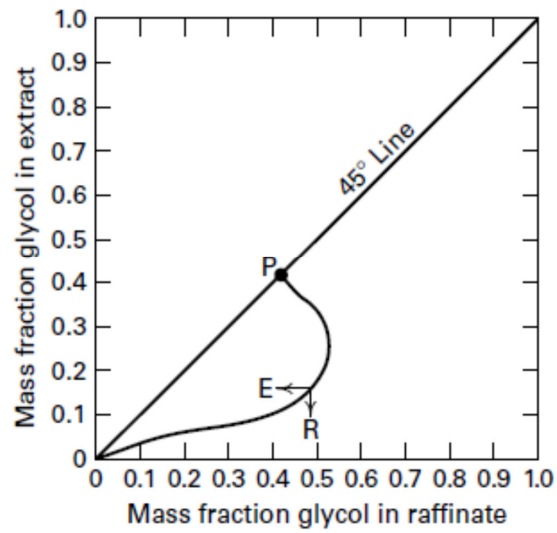
Type I



Type II



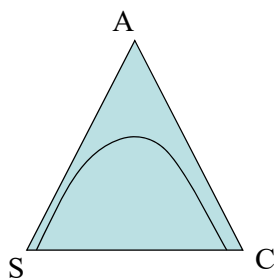
Liquid-Liquid Systems



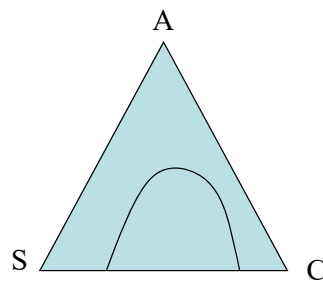
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Liquid-Liquid Systems



Preferred solvent – S and C
have limited solubility



S very soluble in C and C
very soluble in S

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Liquid-Liquid Systems



Example:

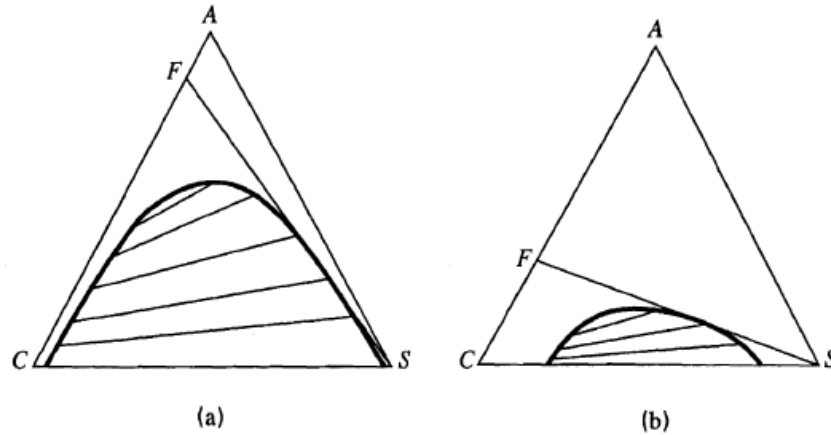


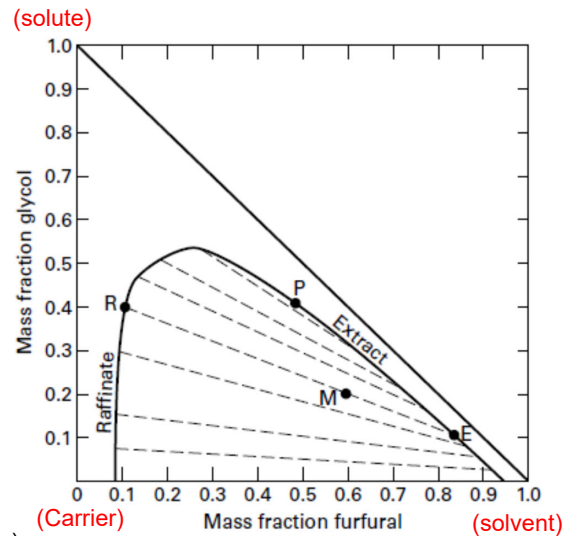
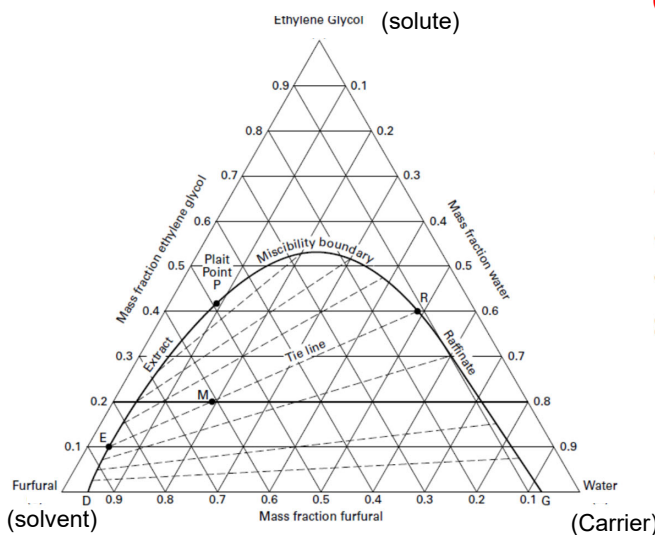
Figure 8.11 Effect of solubility on range of feed composition that can be extracted.

Phase diagram in (a) has a wider range of feed composition than that of (b)

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Liquid-Liquid Systems



- Furfural can be used as a solvent to remove the solute, ethylene glycol, from water, where the furfural-rich phase is the extract, and the water-rich phase is the raffinate.

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Liquid-Liquid Equilibrium



Raffinate phase

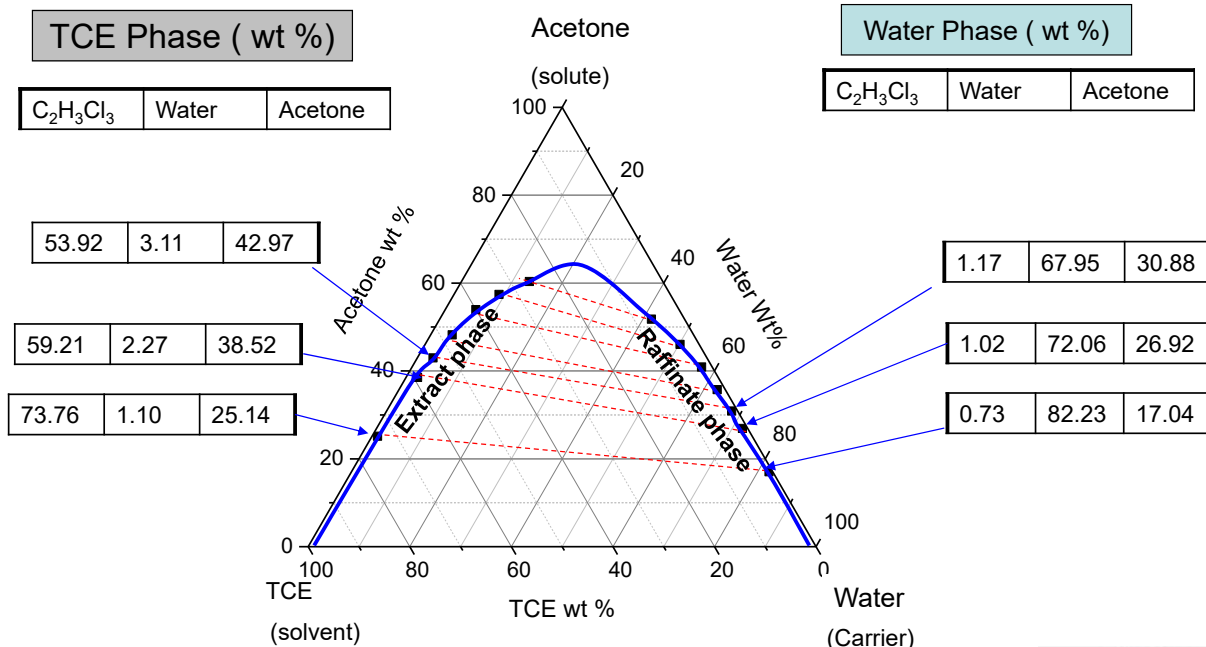
Extract phase

Water Phase (wt %)			TCE Phase (wt %)		
C ₂ H ₃ Cl ₃	Water	Acetone	C ₂ H ₃ Cl ₃	Water	Acetone
0.73	82.23	17.04	73.76	1.10	25.14
1.02	72.06	26.92	59.21	2.27	38.52
1.17	67.95	30.88	53.92	3.11	42.97
1.60	62.67	35.73	47.53	4.26	48.21
2.10	57.00	40.90	40.00	6.05	53.95
3.75	50.20	46.05	33.70	8.90	57.40
6.52	41.70	51.78	26.26	13.40	60.34

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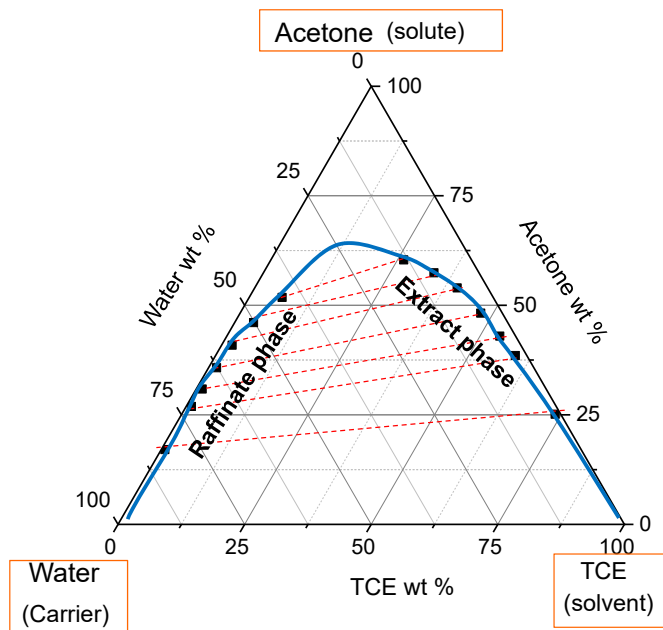
Liquid-Liquid Equilibrium



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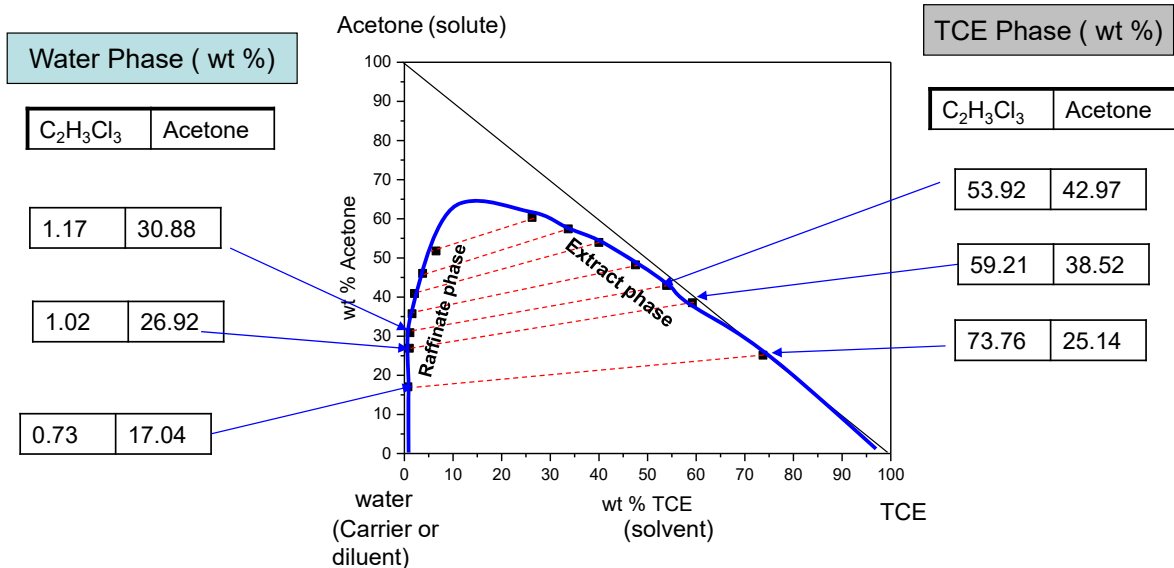
Liquid-Liquid Equilibrium



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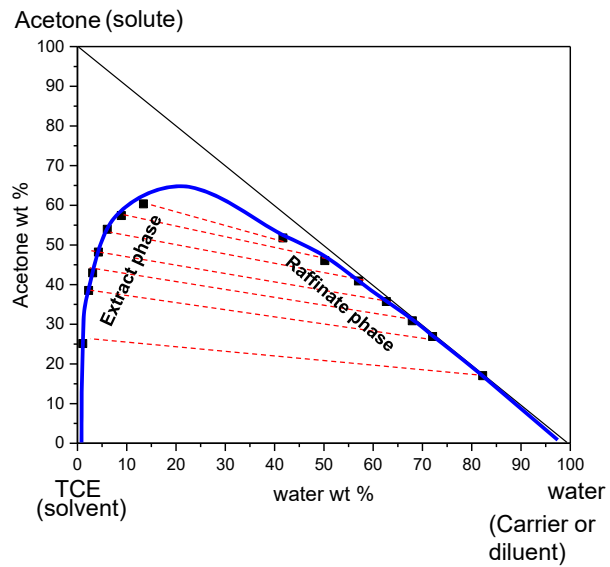
Liquid-Liquid Equilibrium



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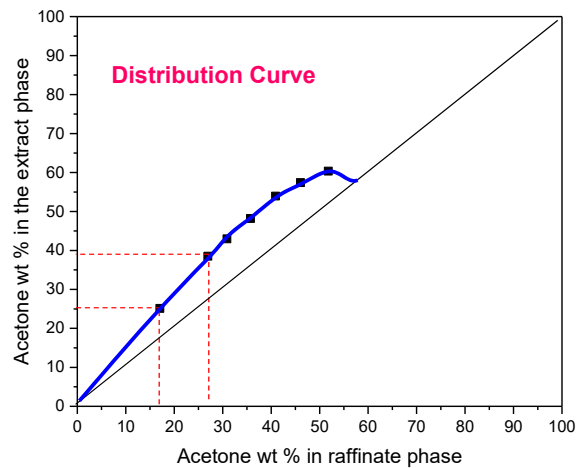
Liquid-Liquid Equilibrium



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Liquid-Liquid Equilibrium



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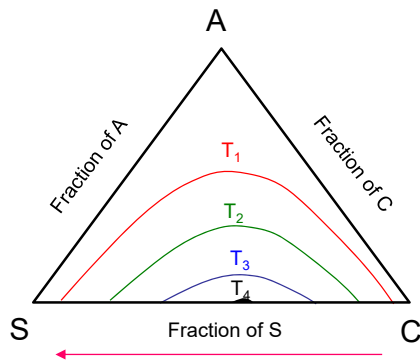


Liquid-Liquid Equilibrium



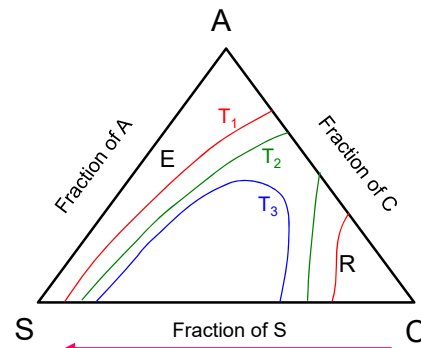
➤ Effect of Temperature on solubility

- Usually, the solubility increases as the temperature increases, for this reason, most liquid-liquid extraction systems operate at low temperatures and some times even require refrigeration.
- Pressure, on the other hand, has little effect on solubility.



Type-I (closed ternary system)

$$T_1 < T_2 < T_3 < T_4$$



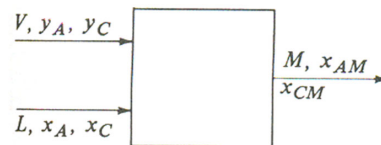
Type-II (open ternary system)



Single-Stage Equilibrium



Derivation of lever-arm rule for graphical addition



An overall mass balance: $V + L = M$ 5.1

A balance on A: $Vy_A + Lx_A = Mx_{AM}$ 5.2

Where x_{AM} is the mass fraction of A in the M stream.

A balance on S: $Vy_C + Lx_C = Mx_{CM}$ 5.3



Single-Stage Equilibrium

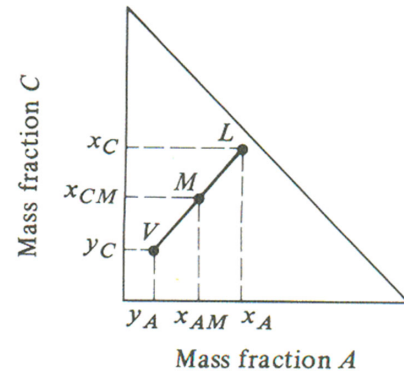


Derivation of lever-arm rule for graphical addition

$$\text{Sub 5.1 into 5.2} \quad \frac{L}{V} = \frac{y_A - x_{AM}}{x_{AM} - x_A} \quad (5.4)$$

$$\text{Sub 5.1 into 5.3} \quad \frac{L}{V} = \frac{y_C - x_{CM}}{x_{CM} - x_C} \quad (5.5)$$

$$\text{Equating 5.4 and 5.5 and rearranging} \quad \frac{x_C - x_{CM}}{x_A - x_{AM}} = \frac{x_{CM} - y_C}{x_{AM} - y_A} \quad (5.6)$$



Eqn. 5.6 shows that points L, M, and V must lie on a straight line. By using the properties of similar right triangles,

$$\text{Lever arm's rule} \quad \frac{L(\text{kg})}{V(\text{kg})} = \frac{\overline{VM}}{\overline{LM}} \quad (5.7)$$

$$\frac{L(\text{kg})}{M(\text{kg})} = \frac{\overline{VM}}{\overline{LV}} \quad (5.8)$$

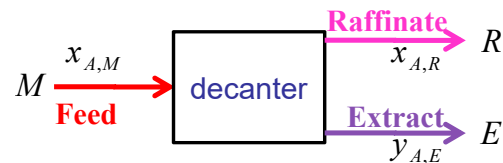
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Single-Stage Equilibrium Extraction



A mixture feed stream M, containing A, S and C is allowed to reach equilibrium



Overall material balance:

$$R + E = M \quad [\text{kg}]$$

R [kg] raffinate mixture

E [kg] extract mixture

M [kg] combined mixture

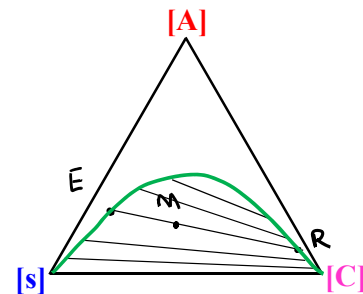
Component material balance (on A):

$$x_{A,R}R + y_{A,E}E = x_{A,M}M$$

$$\text{Rearrange:} \quad \frac{R}{E} = \frac{y_{A,E} - x_{A,M}}{x_{A,M} - x_{A,R}}$$

Or

$$\text{Lever principle:} \quad \frac{R}{E} = \frac{\bar{e}}{\bar{m}} = \frac{\overline{EM}}{\overline{MR}}$$



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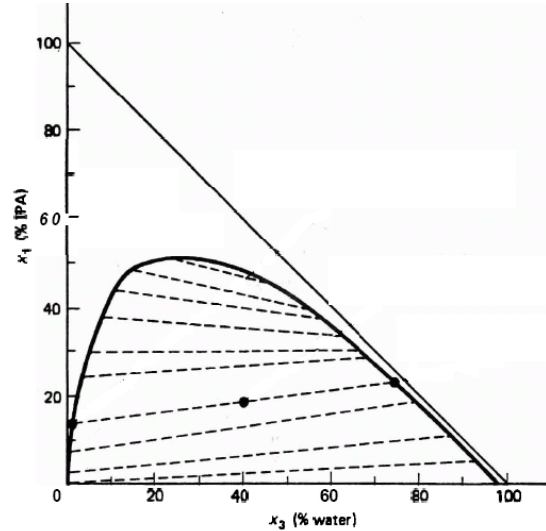
Example



Extraction of isopropyl alcohol (IPA) from toluene to water

Thirty thousand kg/hr of a ternary mixture of: isopropyl alcohol (IPA) $x_1=19$ weight percent, toluene $x_2=41$ weight percent, and water $x_3=40$ weight percent are fed into a decanter operating at 25°C.

the figure gives the LLE data for the system. Determine the compositions and flow rates of the two liquid streams leaving the decanter.



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Example Cont.d

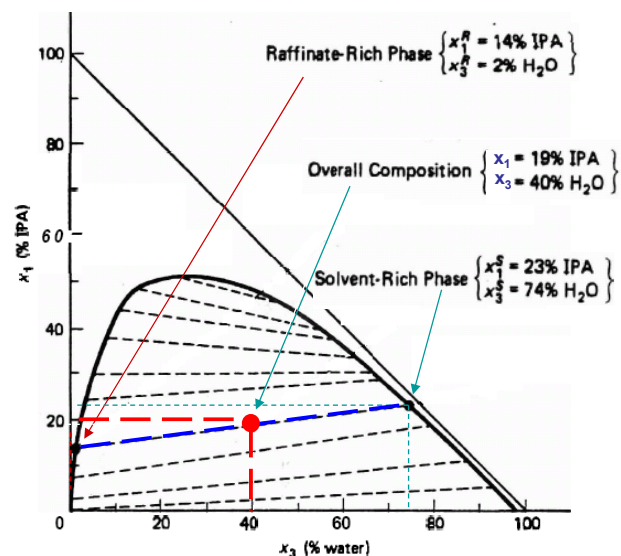


The overall compositions of the feed ($x_1 = 19$ percent and $x_3 = 40$ percent) are located on the diagram.

The compositions of the two liquid phases are read off the diagram at the two ends of the LLE tie-line.

The raffinate-rich phase is 14 percent IPA and 2 percent water (the rest being toluene).

The solvent-rich phase is 23 percent IPA and 74 percent water.



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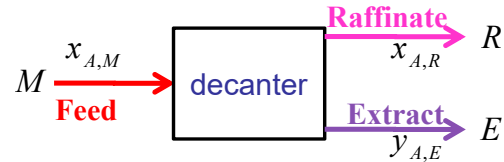


Example Cont.d



Total mass: $M = 30000 = E + R$

Water = $(30000)(0.4) = E(0.74) + R(0.02)$



Solving the last two equations simultaneously gives

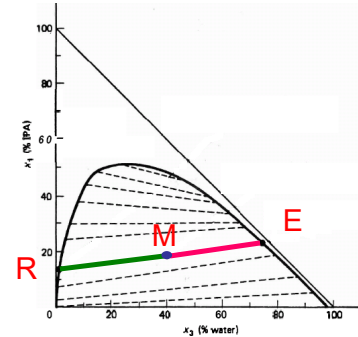
$$E = 15833 \text{ kg/h}$$

$$R = 14176 \text{ kg/h}$$

Or applying Lever principle:

$$\frac{R}{E} = \frac{\bar{e}}{\bar{m}} = \frac{\overline{EM}}{\overline{MR}} = \frac{0.74 - 0.4}{0.4 - 0.02} = 0.894 \quad (1)$$

$$\text{Total mass: } M = 30000 = E + R \quad (2)$$



Solve (1) and (2)

$$E = 15833 \text{ kg/h}$$

$$R = 14176 \text{ kg/h}$$

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Example Cont.d



Check the balance for IPA

$$\text{IPA in the Feed} = (30000)(0.19) = 5700 \text{ kg/h}$$

$$\text{IPA out} = S(0.23) + R(0.14)$$

$$= (15833)(0.23) + (14176)(0.14) = 5625 \text{ kg/h}$$

The difference is due to the accuracy of reading composition from the diagram

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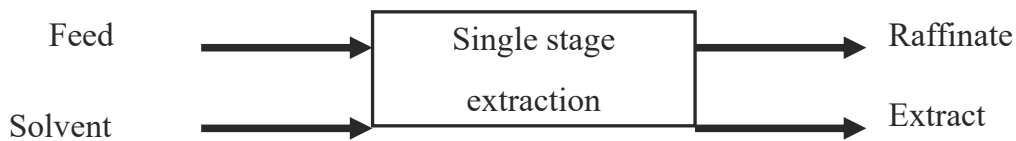
Operating Modes of Extraction



- Batch or continuous extractions
- Batch extraction – single stage or multiple stage
- Continuous extraction – co-current or countercurrent extraction

Batch Extraction

- The aqueous feed is mixed with the organic solvent
- After equilibration, the extract phase containing the desired solute is separated out for further processing.
- A schematic representation of a single batch operation:



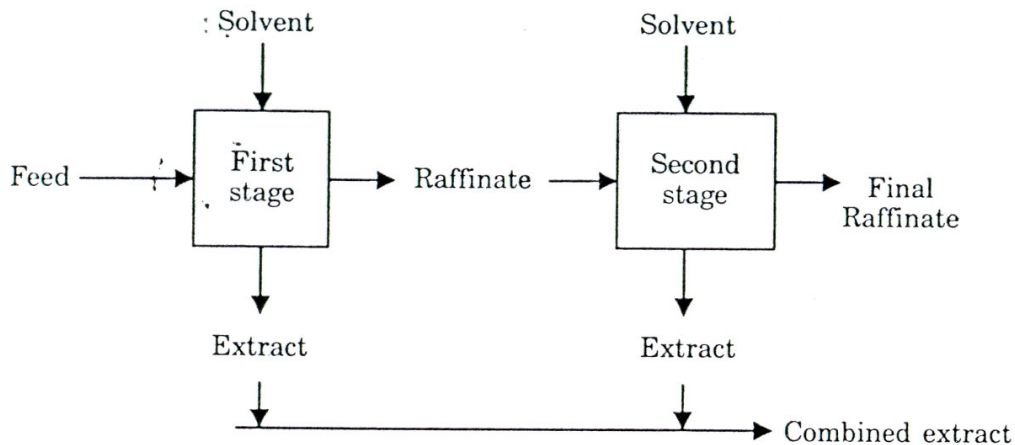
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Operating Modes of Extraction



Batch Extraction in Multiple Stage



Schematic representation of a two-stage batch extraction

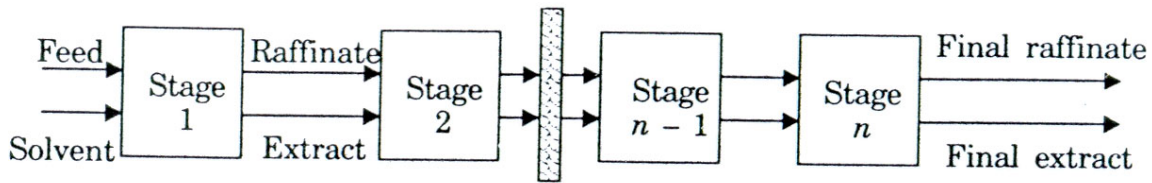
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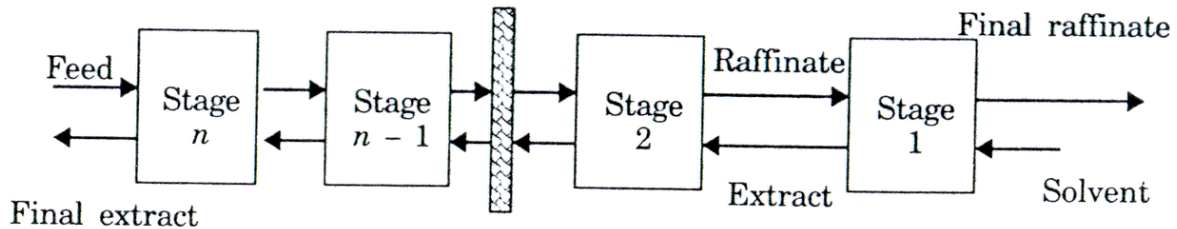
Operating Modes of Extraction



Continuous Extraction



(a) Co-current extraction



(b) Counter-current extraction

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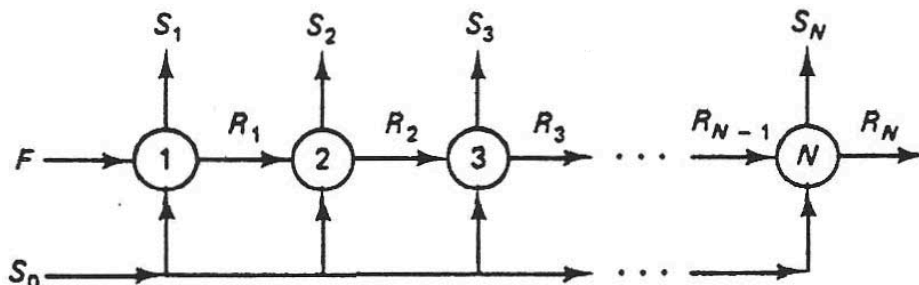


Operating Modes of Extraction



Multiple stages with crossflow of solvent

- If the process liquid stream from the first stages fed into a second extractor and mixed with more fresh solvent, as shown in the Figure, we have what is called cross-flow extraction.
- The process can be described the same as for a single stage .it is simply repeated again for each stage, using the raffinate phase from the upstream stage as the feed to each stage.



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Extraction Equipment



➤ Extraction Equipments:

- Mixer settlers
- Packed extraction towers
- Perforated plate towers
- Baffle towers
- Agitated tower extractors

➤ Auxiliary equipment:

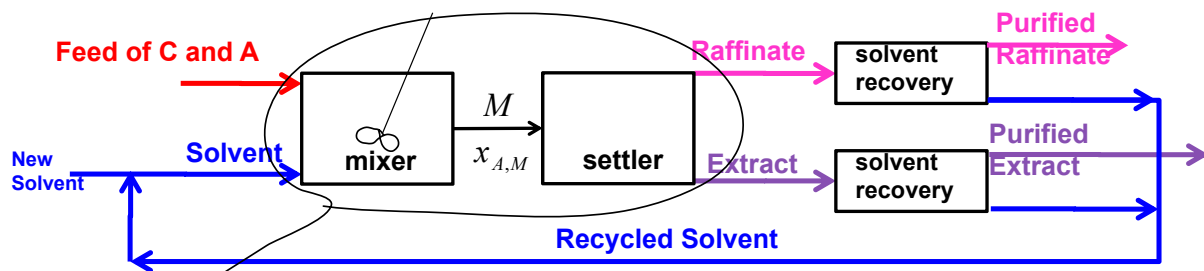
- stills, evaporators, heaters and condenser

➤ Concept of operation: Batchwise or continuous operation

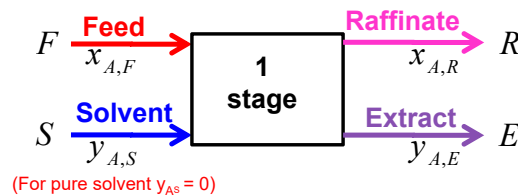
- Feed liquid + solvent (put in agitated vessel) = layers (to be settled and separated)
 - **Extract** – the layer of solvent + extracted solute
 - **Raffinate** – the layer from which solute has been removed
 - Extract may be lighter or heavier than raffinate.
- Continuous flow – more economical for more than one contact process



Mixer – Settler (single stage extraction)



Black Box:

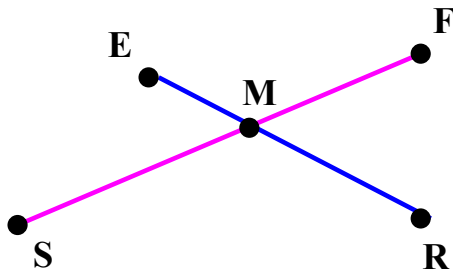


Material balance:

$$F + S = R + E = M$$

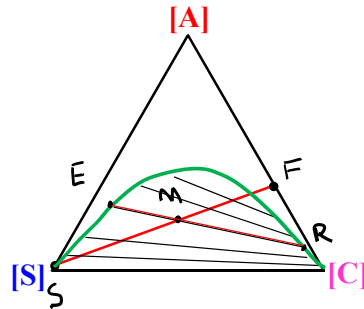


Mixer – Settler (single stage extraction)



→Lever-arm rule:

- F, S, and M must located on the same straight line and mixture point M is between F and S.
- E, R, and M must located on the same straight line and mixture point M is between E and R.

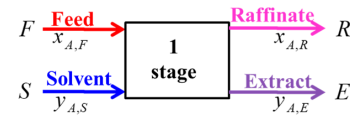


Mixer – Settler (single stage extraction)



given: $x_{A,F}, F, y_{A,S}, S$

find: $x_{A,M}, M, x_{A,R}, y_{A,E}, E, R$



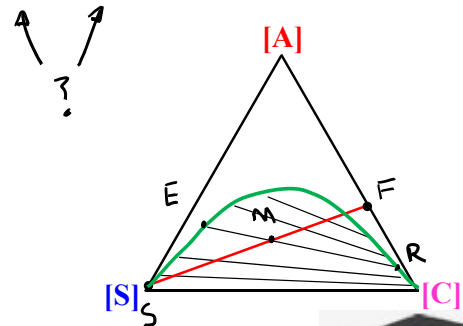
Component material balance (on A in feeds): $x_{A,F}F + y_{A,S}S = x_{A,M}M$

$$x_{A,M} = \frac{x_{A,F}F + y_{A,S}S}{F + S}$$

Component material balance (on A in products): $x_{A,R}R + y_{A,E}E = x_{A,M}M$

$$E = \frac{M(x_{A,M} - x_{A,R})}{y_{A,E} - x_{A,R}}$$

$$R = \frac{M(x_{A,E} - x_{A,M})}{y_{A,E} - x_{A,R}}$$



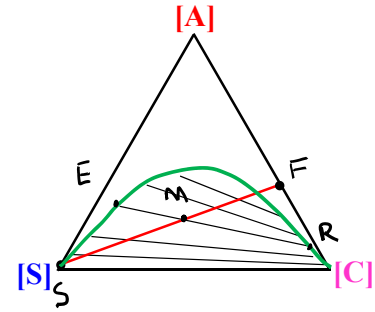
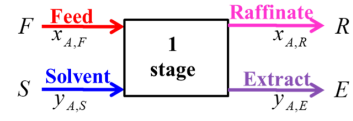
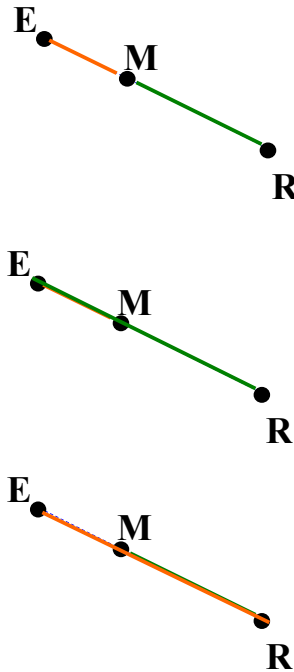
Mixer – Settler (single stage extraction)



$$\frac{R}{E} = \frac{y_A - x_{AM}}{x_{AM} - x_A} = \frac{\overline{EM}}{\overline{RM}}$$

$$\frac{R}{M} = \frac{y_A - x_{AM}}{y_A - x_A} = \frac{\overline{EM}}{\overline{RE}}$$

$$\frac{E}{M} = \frac{x_{AM} - x_A}{y_A - x_A} = \frac{\overline{RM}}{\overline{RE}}$$



Mixer – Settler (single stage extraction)



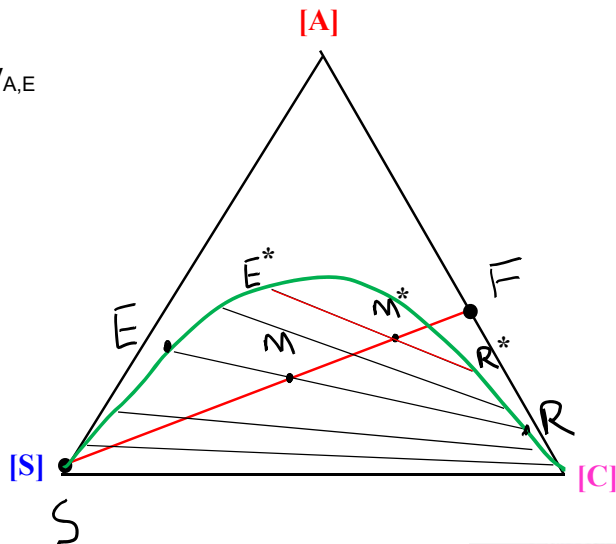
but now lower solvent flow rate (to try save money!). What happens to (a) extract concentration and (b) solute recovery?

Extract concentration increases: $y_{A,E^*} > y_{A,E}$

Solute recovery drops: $x_{A,R^*} > x_{A,R}$

Fraction of solute recovered:

$$f = 1 - \frac{(x_{A,R})(R)}{(x_{A,F})(F)}$$



Mixer – Settler (single stage extraction)



- The minimum solvent required for single stage extraction is the quantity when the M point falls at the intersection of the line FS with the Raffinate side of the solubility curve

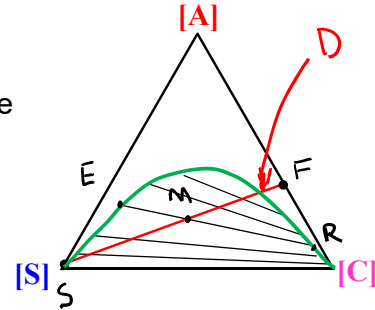
Minimum Solvent (rate):

Material balance:

$$F + S_{\min} = D$$

$$x_{A,F}F + y_{A,S}S_{\min} = x_{A,D}D$$

$$\frac{S_{\min}}{F} = \frac{\overline{FD}}{\overline{DS}} = \frac{x_{A,F} - x_{A,D}}{x_{A,D} - y_{A,S}}$$



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Mixer – Settler (single stage extraction)



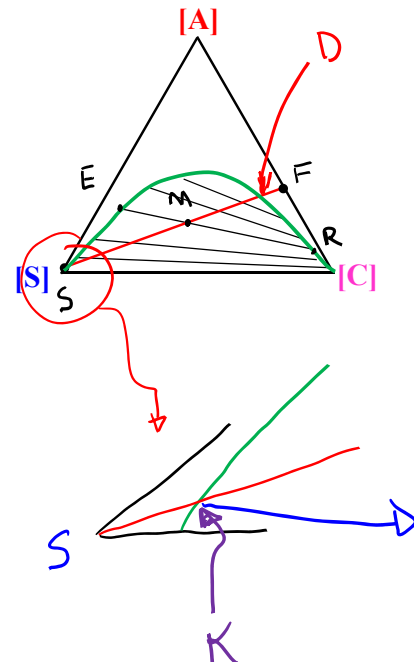
Maximum Solvent (rate):

Material balance:

$$F + S_{\max} = K$$

$$x_{A,F}F + y_{A,S}S_{\max} = x_{A,K}K$$

$$\frac{S_{\max}}{F} = \frac{\overline{FK}}{\overline{KS}} = \frac{x_{A,F} - x_{A,K}}{x_{A,K} - y_{A,S}}$$



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Example



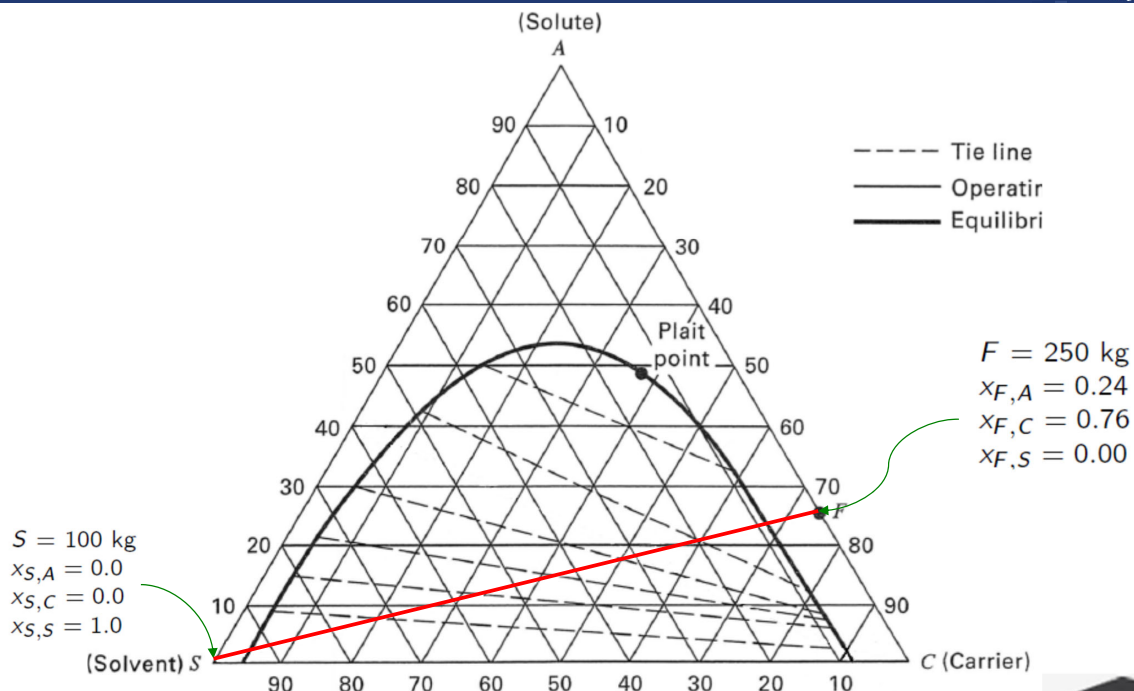
In mixer-settler extraction unit, 250 kg of feed which contains 24 wt% solute(A) 76 wt% carrier(C) is mixed with pure 100 kg solvent (S).

- 1) Find the overall composition of mixture at equilibrium using:
 - a) The phase diagram given below
 - b) Mass balances
- 2) Find the amounts and compositions of raffinate and extract phases.
The phase diagram is given on the next slide.

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Example Cont.d



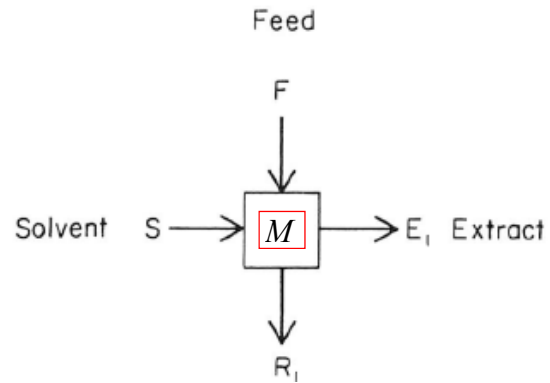
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Example Cont.d



Feed	Solvent
$F = 250 \text{ kg}$	$S = 100 \text{ kg}$
$x_{F,A} = 0.24$	$x_{S,A} = 0.0$
$x_{F,C} = 0.76$	$x_{S,C} = 0.0$
$x_{F,S} = 0.00$	$x_{S,S} = 1.0$



Material balance:

$$M = F + S = 350 \text{ kg}$$

Component balance (on A in feeds):

$$x_{A,F}F + y_{A,S}S = x_{A,M}M$$

$$250(0.24) + 0 = x_{A,M}(350)$$

$$x_{A,M} = 0.17$$

Component balance (on C in feeds):

$$x_{C,F}F + y_{C,S}S = x_{C,M}M$$

$$250(0.76) + 0 = x_{C,M}(350)$$

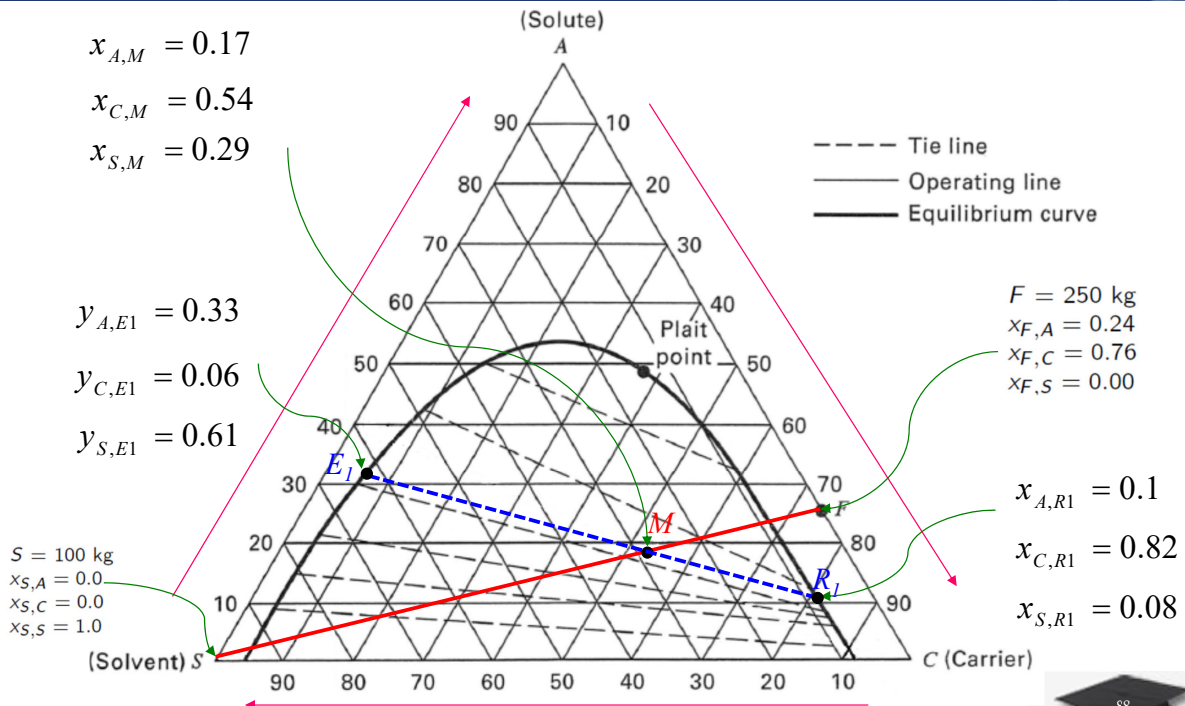
$$x_{C,M} = 0.54$$

$$x_{S,M} = 0.29$$

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Example Cont.d



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Example Cont.d



Material balance: $M = R_1 + E_1 = 350 \text{ kg}$

Component material balance (on A in products):

$$0.1 R_1 + 0.33 E_1 = 350 (0.17)$$

$$R_1 = 222 \text{ kg} \quad E_1 = 128 \text{ kg}$$

$$\text{Solute recovery} = \frac{F x_{F,A} - R x_{R,A}}{F x_{F,A}} = 1 - \frac{R x_{R,A}}{F x_{F,A}}$$

$$\text{Solute recovery} = 1 - \frac{(222)(0.1)}{(250)(0.24)} = 63\%$$

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Example Cont.d



Min. solvent

$$F + S_{\min} = D, \quad x_{A,F} F + y_{A,S} S_{\min} = x_{A,D} D$$

$$x_{C,D} = 0.68$$

$$x_{S,D} = 0.09$$

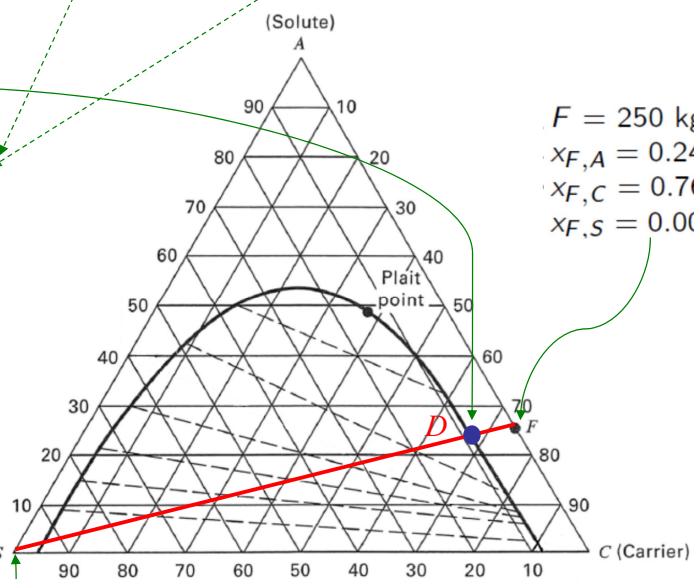
$$x_{A,D} = 0.23$$

$$\frac{S_{\min}}{F} = \frac{\overline{FD}}{\overline{DS}} = \frac{x_{A,F} - x_{A,D}}{x_{A,D} - y_{A,S}}$$

$$\frac{S_{\min}}{250} = \frac{0.24 - 0.23}{0.23 - 0}$$

$$S_{\min} = 10.87 \text{ kg}$$

$$\begin{aligned} x_{S,A} &= 0.0 \\ x_{S,C} &= 0.0 \\ x_{S,S} &= 1.0 \end{aligned}$$

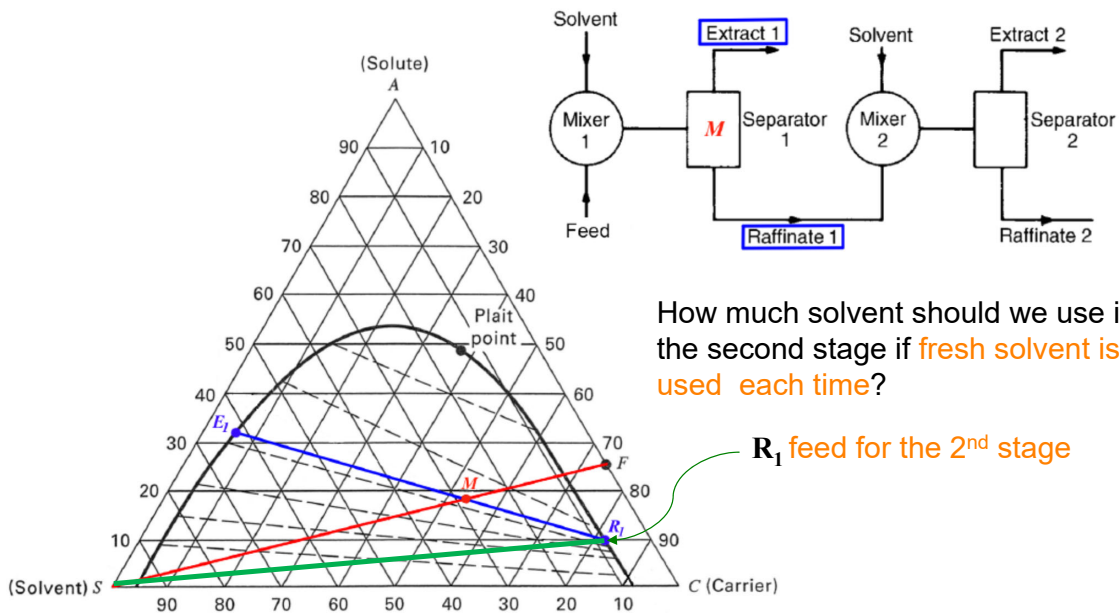


$$\begin{aligned} F &= 250 \text{ kg} \\ x_{F,A} &= 0.24 \\ x_{F,C} &= 0.76 \\ x_{F,S} &= 0.00 \end{aligned}$$

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Example



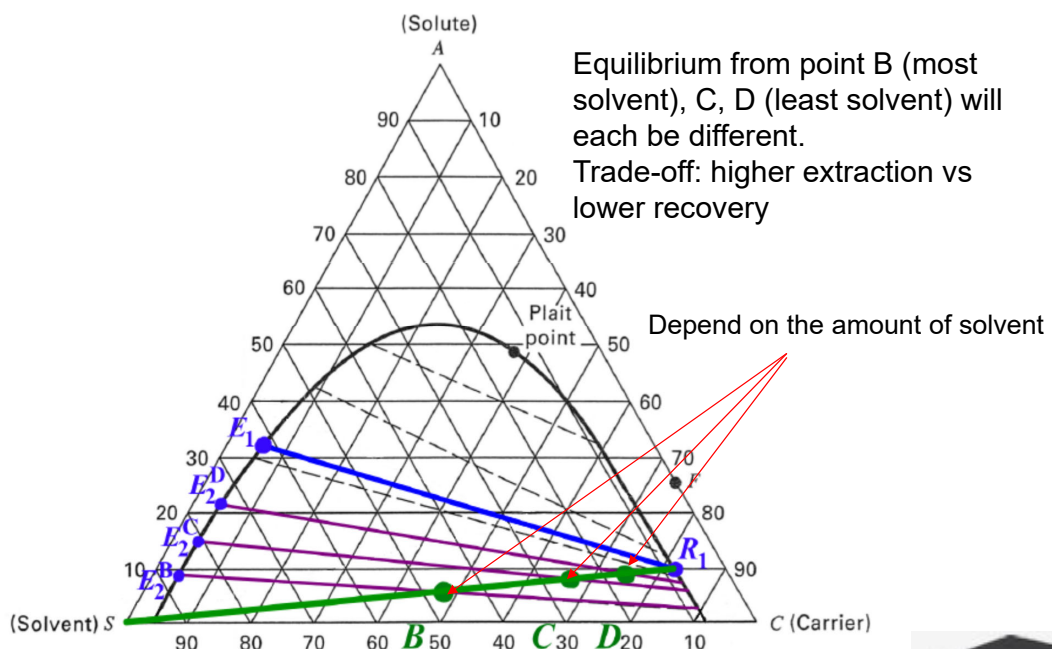
How much solvent should we use in the second stage if fresh solvent is used each time?

R_1 feed for the 2nd stage

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Example Cont.d



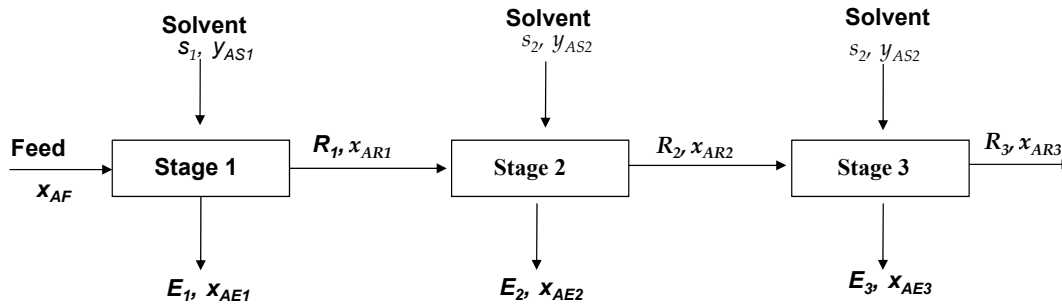
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Multistage Cross-current Extraction



Process flow



Material balance on Stage 1:

$$F + S_1 = R_1 + E_1$$

given: $x_{A,F}, F, y_{A,S}, S$

find: $x_{A,M1}, M_1, x_{A,R1}, y_{A,E1}, E, R_1$

Component material balance (on A in feeds):

$$x_{A,F}F + y_{A,S1}S_1 = x_{A,M1}M_1$$

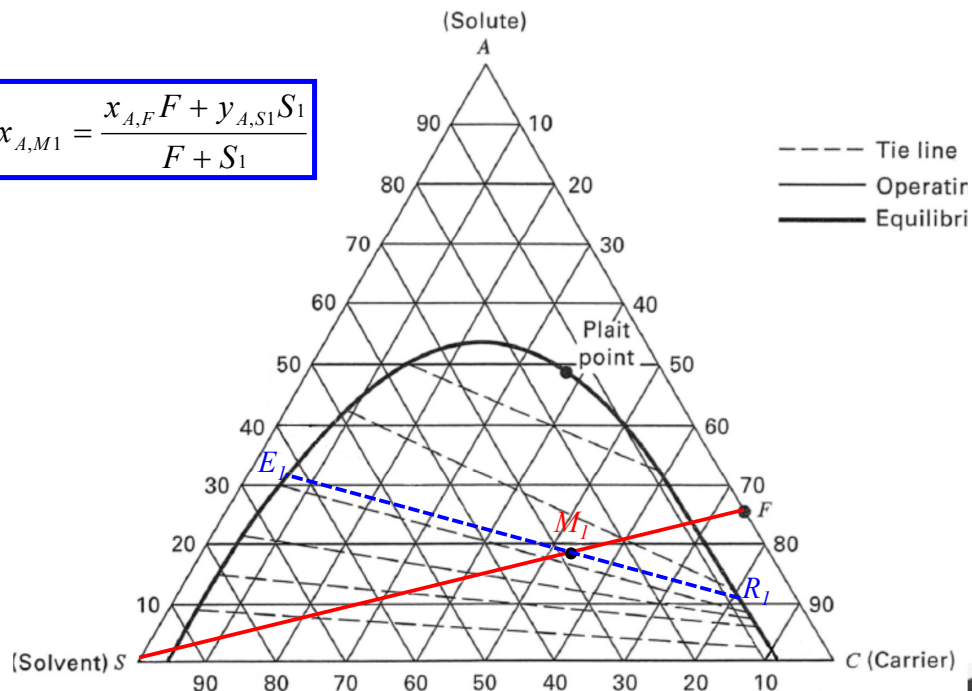
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Multistage Cross-current Extraction



$$x_{A,M1} = \frac{x_{A,F}F + y_{A,S1}S_1}{F + S_1}$$



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Multistage Cross-current Extraction



Component material balance (on A in products): $x_{A,R1}R_1 + y_{A,E1}E_1 = x_{A,M1}M_1$

$$E_1 = \frac{M_1(x_{A,M1} - x_{A,R1})}{y_{A,E1} - x_{A,R1}}$$

$$R_1 = \frac{M(x_{A,E1} - x_{A,M1})}{y_{A,E1} - x_{A,R1}}$$

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Multistage Cross-current Extraction



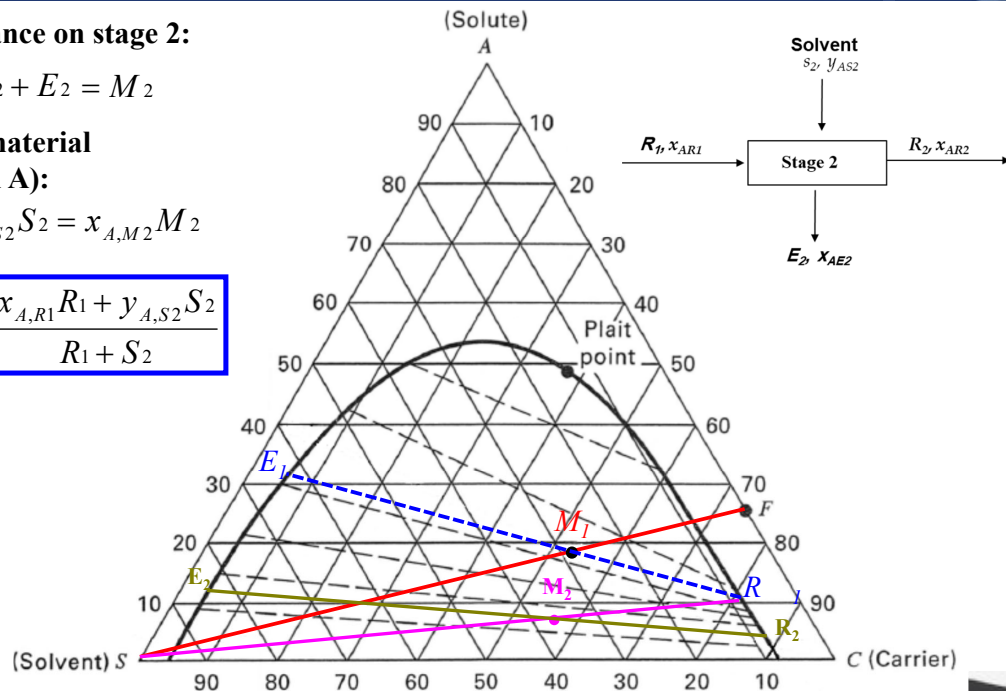
Material balance on stage 2:

$$R_1 + S_2 = R_2 + E_2 = M_2$$

Component material balance (on A):

$$x_{A,R1}R_1 + y_{A,S2}S_2 = x_{A,M2}M_2$$

$$\rightarrow x_{A,M2} = \frac{x_{A,R1}R_1 + y_{A,S2}S_2}{R_1 + S_2}$$



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Multistage Cross-current Extraction



Component material balance (on A in products):

$$x_{A,R2}R_2 + y_{A,E2}E_2 = x_{A,M2}M_2$$

$$E_2 = \frac{M_2(x_{A,M2} - x_{A,R2})}{y_{A,E2} - x_{A,R2}}$$

$$R_2 = \frac{M(x_{A,E2} - x_{A,M2})}{y_{A,E2} - x_{A,R2}}$$

In similar Manner you can obtain the relations for Stage 3

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Multistage Cross-current Extraction



- **Recovery** \equiv fraction of solute recovered

$$\text{Solute recovery} = 1 - \frac{R_N x_{R_N,A}}{F x_{F,A}}$$

N: number of stages

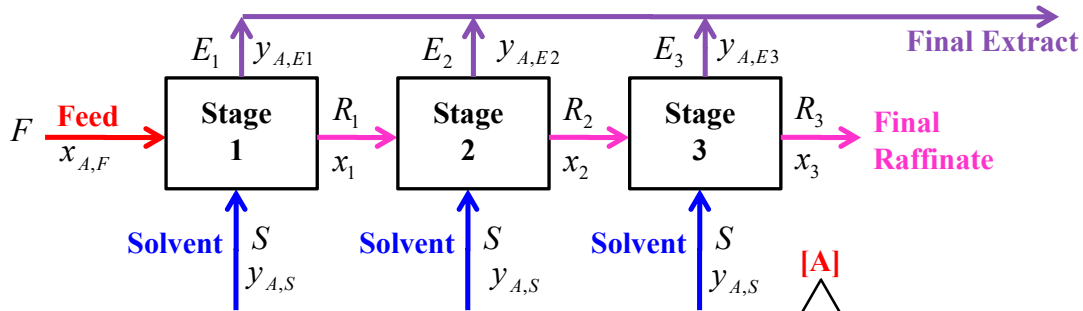
- Overall **solute concentration** in the extract

$$\bar{y}_{E,A} = \frac{\sum_{i=1}^N E_i y_{E_i,A}}{\sum_{i=1}^N E_i}$$

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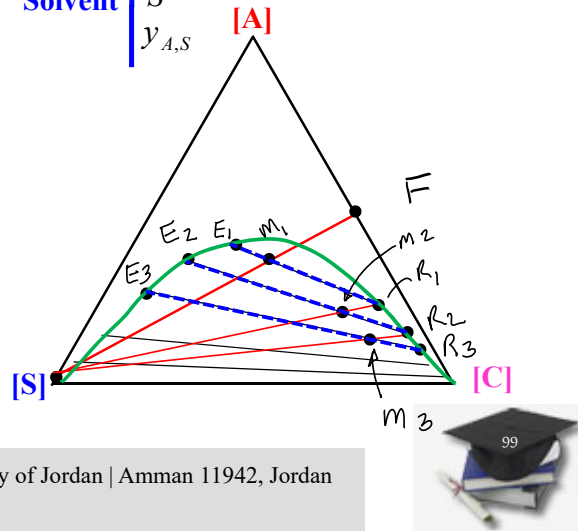


Cross-Current (multi-stage extraction)



Final Extract: $E_1 + E_2 + E_3$

$$y_{A,E} = \frac{y_{A,E1}E_1 + y_{A,E2}E_2 + y_{A,E3}E_3}{E_1 + E_2 + E_3}$$



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Example



Cross-current mixer-settler extraction units are used for extraction process of 250 kg of feed which contains 24 wt% solute (A) and 76 wt% carrier(C). Each stage is supplied with pure 100 kg solvent (S). Find the minimum number of stages required to achieve at least 85% solute recovery. Find the corresponding overall solute concentration in the extract.

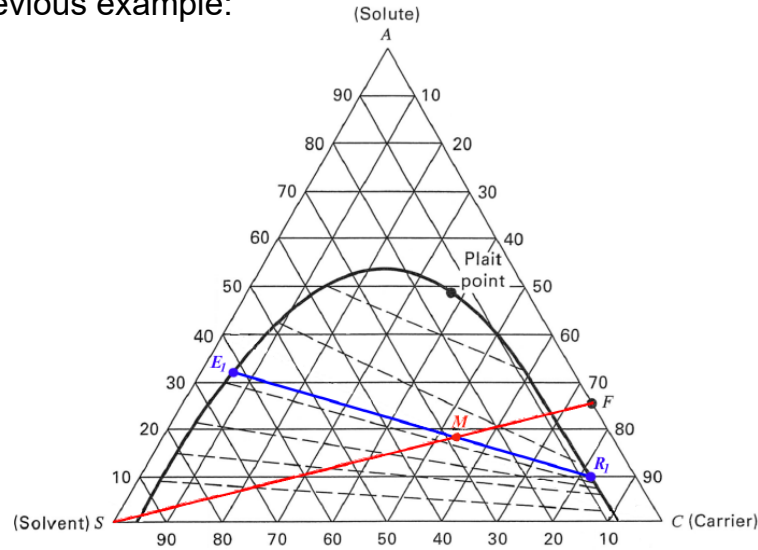
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Example Cont.d



First stage from previous example:



$$R_1 = 222 \text{ kg}; \quad x_{R_1,A} = 0.10; \quad x_{R_1,C} = 0.82; \quad x_{R_1,S} = 0.08$$

$$E_1 = 128 \text{ kg}; \quad y_{E_1,A} = 0.33; \quad y_{E_1,C} = 0.06; \quad y_{E_1,S} = 0.61$$

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Example Cont.d



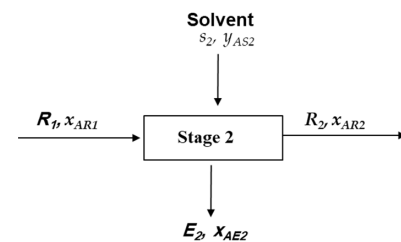
Material balance on stage 2:

$$R_1 + S_2 = 222 + 100 = 322 \text{ kg} = M_2$$

Component material balance (on A):

$$x_{A,R_1} R_1 + y_{A,S_2} S_2 = x_{A,M_2} M_2$$

$$\rightarrow x_{A,M_2} = \frac{x_{A,R_1} R_1 + y_{A,S_2} S_2}{R_1 + S_2} \rightarrow x_{A,M_2} = \frac{0.1(222) + 0}{322} = 0.069$$

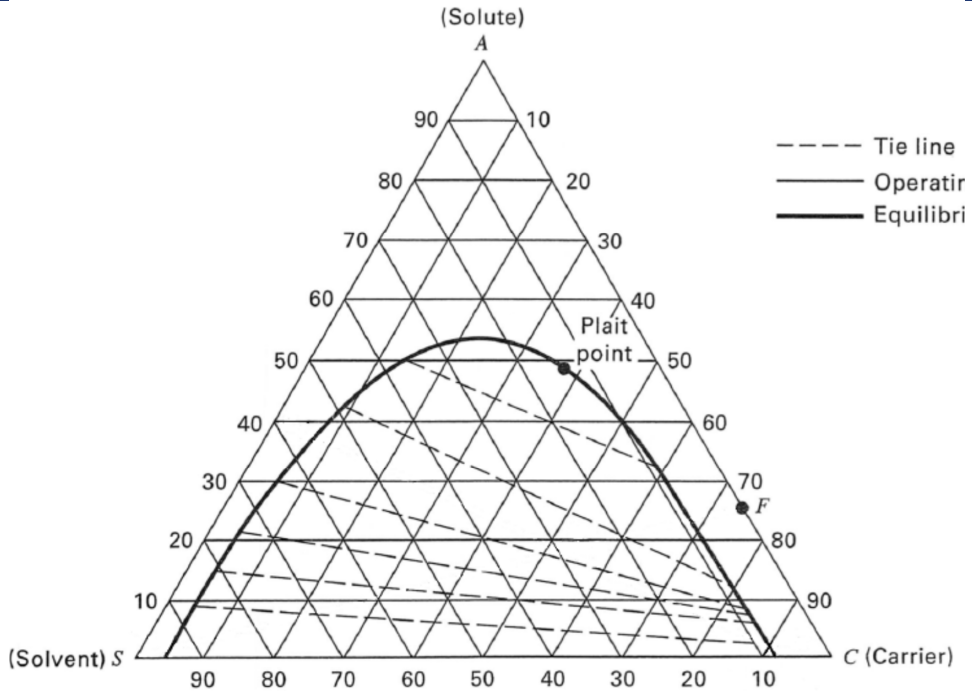


Component material balance (on C):

$$x_{C,R_1} R_1 + y_{C,S_2} S_2 = x_{C,M_2} M_2 \rightarrow x_{C,M_2} = \frac{0.82(222) + 0}{322} = 0.57$$

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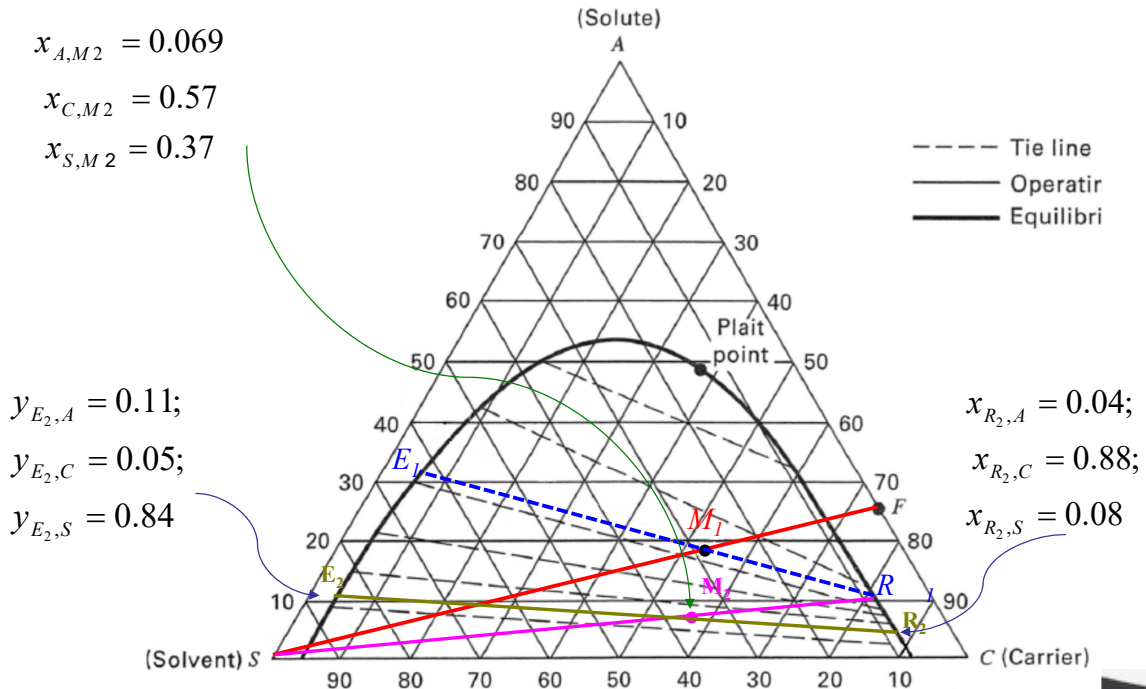




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Example Cont.d



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Example Cont.d



Component material balance (on A in products): $x_{A,R2}R_2 + y_{A,E2}E_2 = x_{A,M2}M_2$

$$E_2 = \frac{M_2(x_{A,M2} - x_{A,R2})}{y_{A,E2} - x_{A,R2}} \rightarrow E_2 = \frac{322(0.069 - 0.04)}{0.11 - 0.04} = 133.4 \text{ kg}$$

$$M_2 = R_2 + E_2 \rightarrow R_2 = M_2 - E_2 = 322 - 133.4 = 188.6 \text{ kg}$$

$$\text{Solute recovery} = 1 - \frac{R_2 x_{R2,A}}{F x_{F,A}} = 1 - \frac{(188.6)(0.04)}{(250)(0.24)} = 87.4\% > 0.85 \text{ (required)}$$

Thus, **two stages** is sufficient to achieve the required solute recovery.

- Overall solute concentration:

$$\bar{y}_{E,A} = \frac{\sum_{i=1}^2 E_i y_{E_i,A}}{\sum_{i=1}^2 E_i} = \frac{(E_1 y_{E_1,A} + E_2 y_{E_2,A})}{(E_1 + E_2)} = 0.22$$

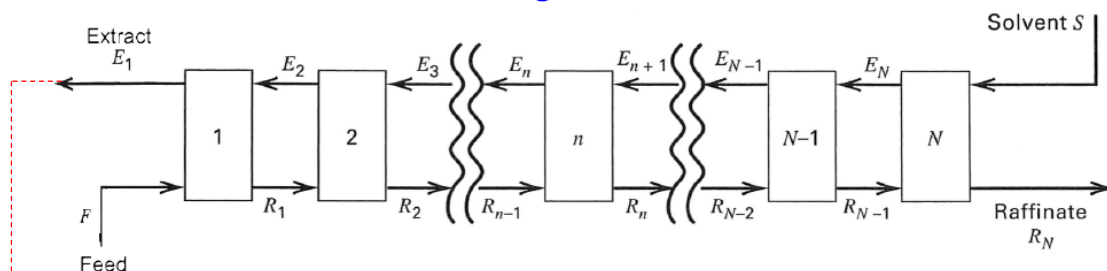
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Continuous Multistage Countercurrent Extraction



N units in counter-current arrangement:



N: index for stage number

- “Re-use” of solvent
- E_n and R_n for $n=1, \dots, N$ leaving each stage are in equilibrium [they are determined via the **tie line**]

- Solute Recovery:**

$$\text{Solute recovery} = 1 - \frac{R_N x_{R_N,A}}{F x_{F,A}}$$

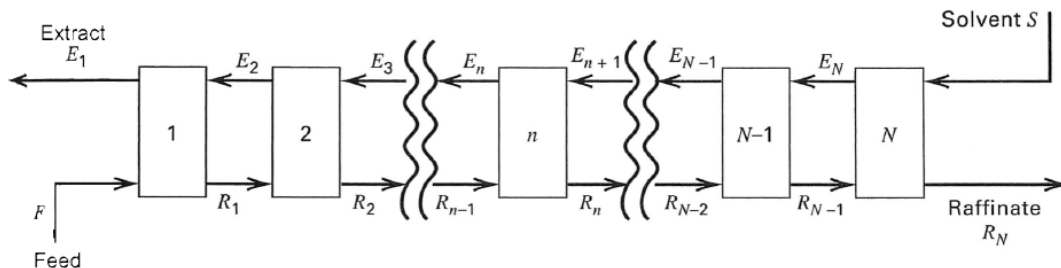
- Overall solute concentration:**

$$\bar{y}_{E,A} = y_{E_1,A}$$

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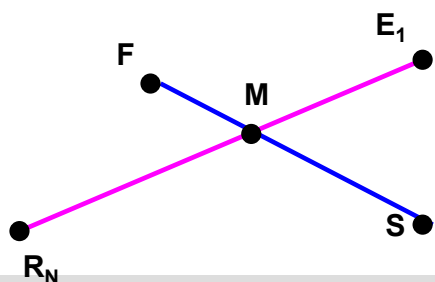


Continuous Multistage Countercurrent Extraction



Apply st. st. total mass balance on the overall system:

$$F + S = E_1 + R_N = M$$



→Lever rule:

- F, S, and M must located on the same straight line and mixture point M is between F and S.
- E_1 , R_N , and M must located on the same straight line and mixture point M is between E_1 and R_N .

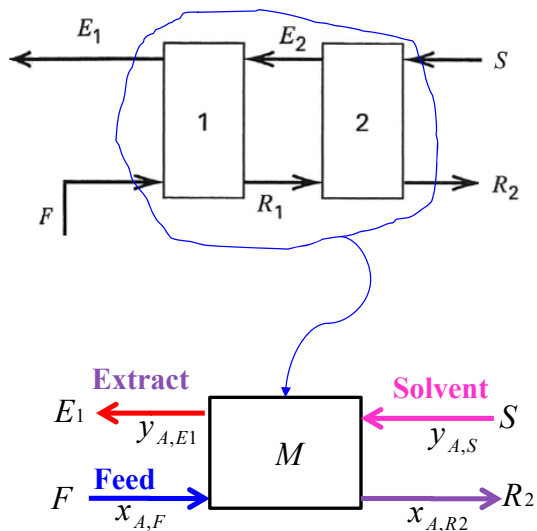
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Continuous Multistage Countercurrent Extraction



Two counter-current units



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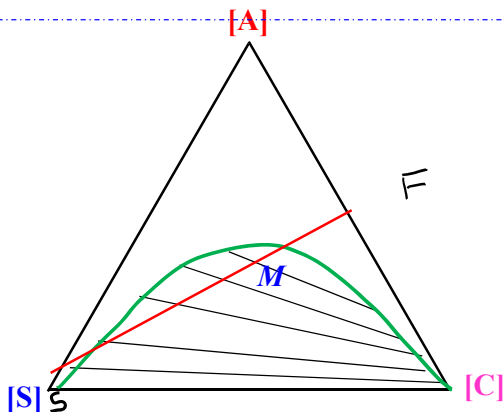


Continuous Multistage Countercurrent Extraction

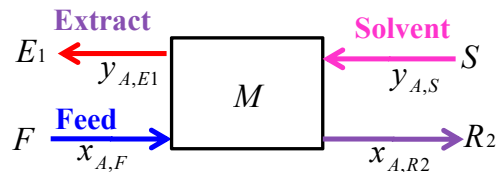


Two counter-current units

- If the fresh solvent flow rate S and composition are given along with the feed flow rate F and composition, we can locate the point M on the straight line connecting the points F and S .



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Overall balance

$$M = F + S = R_2 + E_1$$

Component balance (on A in feeds):

$$x_{A,F}F + y_{A,S}S = x_{A,M}M$$

Component balance (on C in feeds):

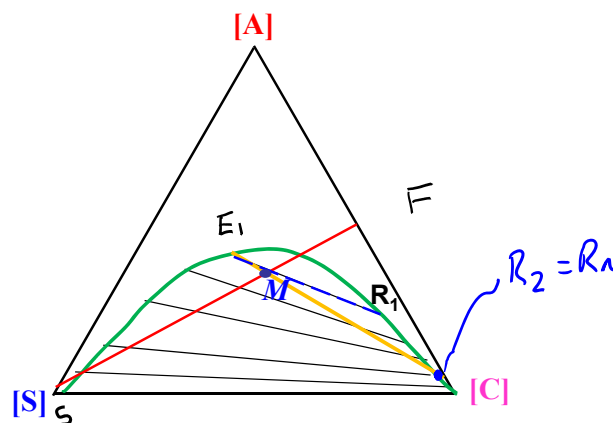
$$x_{C,F}F + y_{C,S}S = x_{C,M}M$$

Continuous Multistage Countercurrent Extraction



➤ Furthermore,

- From the overall balance, it is clear that M must also lie on the straight line connecting E_1 and $(R_2) R_N$, as shown in the figure.
- Since E_1 and R_1 are in phase equilibrium, we can use an LLE tie-line to determine the point R_1 on the solubility curve.



➤ In the typical design problem, you will be given:

The point R_N will be given, i.e., the concentration of the raffinate phase leaving the final stage will be specified so as to recover the desired amount of the solute from the feed.

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Continuous Multistage Countercurrent Extraction

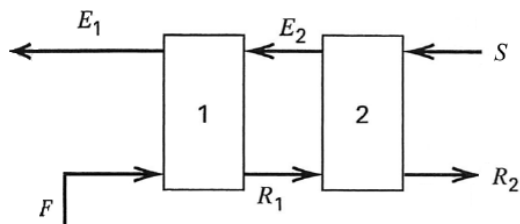


Material balance on stage 1:

$$F + E_2 = R_1 + E_1$$

Material balance on stage 2:

$$R_1 + S = R_2 + E_2$$

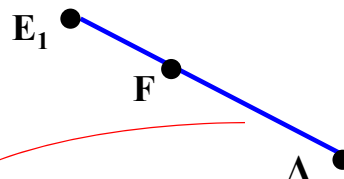


Rearrange

$$F - E_1 = R_1 - E_2$$

$$R_1 - E_2 = R_2 - S$$

$$\longrightarrow F - E_1 = R_1 - E_2 = R_2 - S = \Delta$$



Rearrange again

$$F = E_1 + \Delta$$

$$R_1 = E_2 + \Delta$$

$$R_2 = S + \Delta$$

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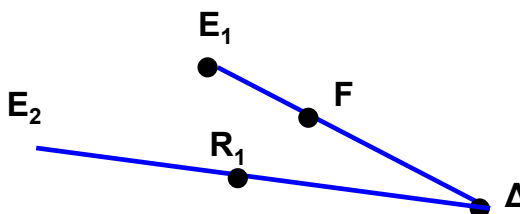


Continuous Multistage Countercurrent Extraction



$$R_1 = E_2 + \Delta$$

→ Lever-arm rule, the three points R_1 , E_2 , and P must be located on the same straight line and R_1 is between E_2 and Δ

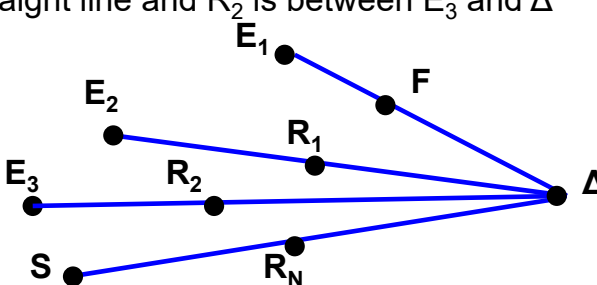


$$R_2 = E_3 + \Delta$$

→ Lever-arm rule, the three points R_2 , E_3 , and P must be located on the same straight line and R_2 is between E_3 and Δ

....and so on to:

$$R_N = S + \Delta$$



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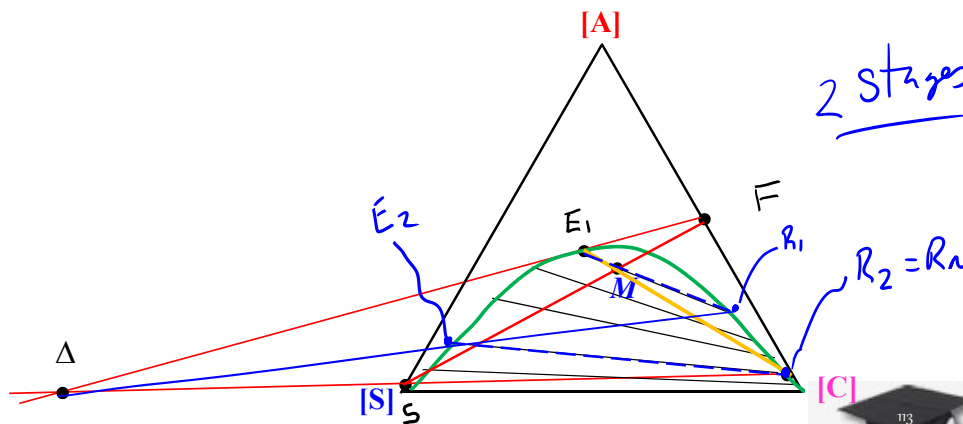


Continuous Multistage Countercurrent Extraction



➤ Interpretation: Δ is a fictitious operating point on the ternary diagram (from lever rule)

- ➔ F is on the line that connects E_1 and Δ
- R_1 is on the line that connects E_2 and Δ
- R_2 is on the line that connects S and Δ



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Continuous Multistage Countercurrent Extraction



Counter-current graphical solution: 2 units

Feed	Solvent
$F = 250 \text{ kg}$	$S = 100 \text{ kg}$
$x_{F,A} = 0.24$	$x_{S,A} = 0.0$
$x_{F,C} = 0.76$	$x_{S,C} = 0.0$
$x_{F,S} = 0.00$	$x_{S,S} = 1.0$

What is $y_{A,E1}$ when the $x_{A,R2} = 0.05$

Overall balance:

$$M = F + S = R_2 + E_1 \rightarrow M = 350 \text{ kg}$$

Component balance (on A in feeds):

$$x_{A,F}F + y_{A,S}S = x_{A,M}M$$

$$250(0.24) + 0 = x_{A,M}(350)$$

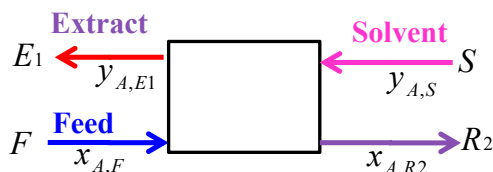
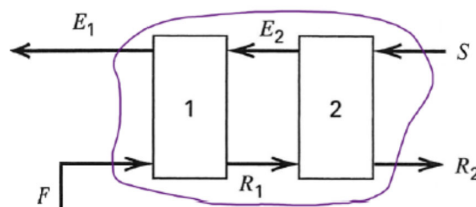
$$x_{A,M} = 0.17$$

Component balance (on C in feeds):

$$x_{C,F}F + y_{C,S}S = x_{C,M}M$$

$$250(0.76) + 0 = x_{C,M}(350)$$

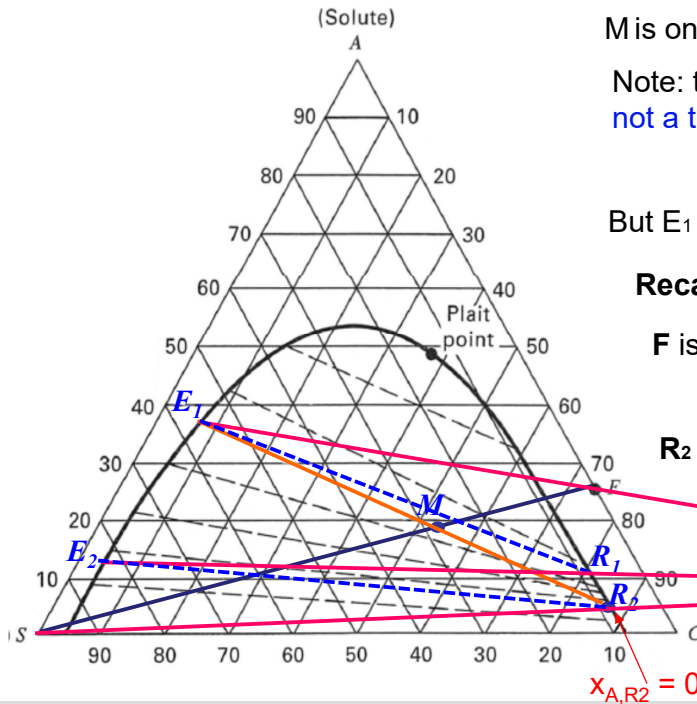
$$x_{C,M} = 0.54 \quad x_{S,M} = 0.29$$



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Continuous Multistage Countercurrent Extraction



M is on the line that connects R_2 and E_1

Note: the line connecting E_1 to R_2 is not a tie line (from lever rule).

$$y_{A,E1} = 0.38$$

But E_1 is in equilibrium with R_1

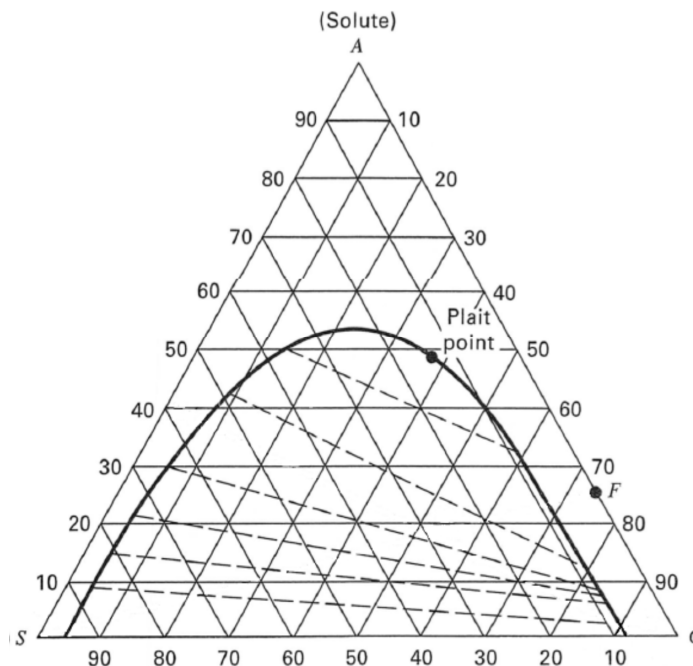
Recall $F = E_1 + \Delta$

F is on the line that connects E_1 and Δ

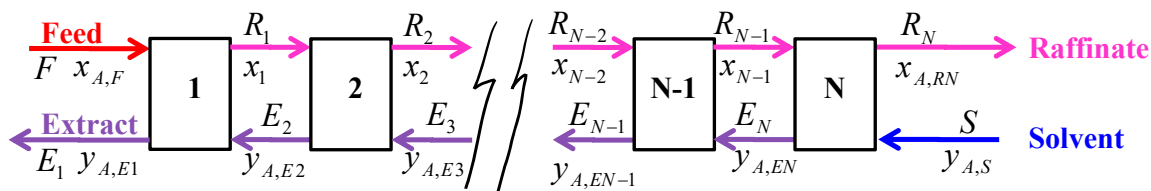
$$R_2 = S + \Delta$$

R_2 is on the line that connects S and Δ

$$x_{A,R2} = 0.05$$



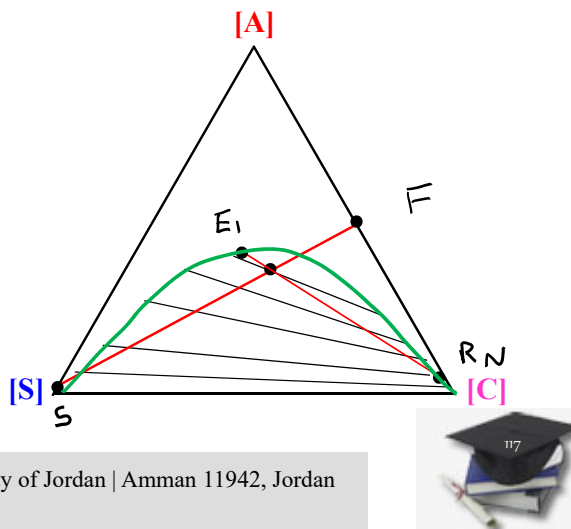
Continuous Multistage Countercurrent Extraction



Total MB: $F + S = E_1 + R_N = M$

Total MB on A: $x_{A,M} = \frac{x_{A,F}F + y_{A,S}S}{F + S}$

If $y_{A,E1}$ & $x_{A,RN}$ known (specified), then Flow rates E_1 & R_N can be found.



Continuous Multistage Countercurrent Extraction



Material balance:

$$F + E_2 = R_1 + E_1 \quad R_2 + E_2 = R_1 + E_3 \quad R_n + E_n = E_{n+1} + R_{n-1}$$

Rearrange

$$F - E_1 = R_1 - E_2 \quad R_1 - E_2 = R_2 - E_3 \quad R_{n-1} - E_n = R_n - E_{n+1}$$

$$F - E_1 = R_1 - E_2 = \dots = R_{n-1} - E_n = \dots = R_n - S = \Delta$$

Notes:

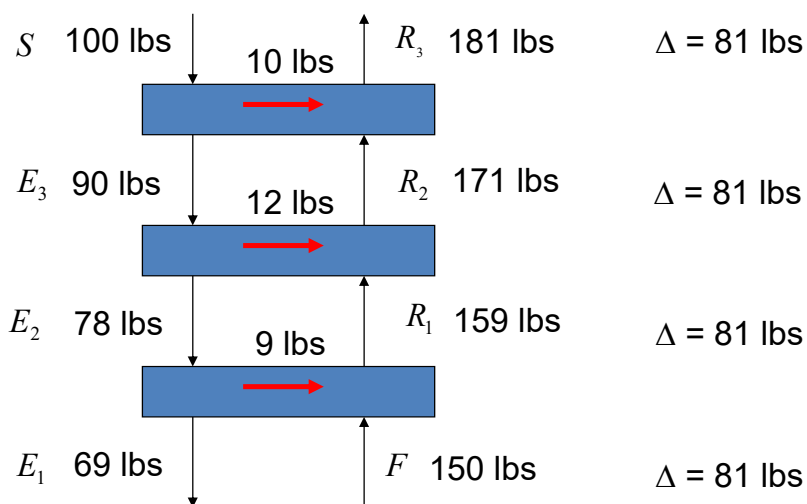
1. each difference is equal to Δ (the difference between flows)
2. E_n and R_n are in equilibrium, leaving each stage [via tie line]



Continuous Multistage Countercurrent Extraction



Δ point



$$F - E_1 = R_1 - E_2 = \dots = R_{n-1} - E_n = \dots = R_N - S = \Delta$$

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Continuous Multistage Countercurrent Extraction



1. We know F and S; Connect S and F points with a line.
2. Locate the mixture point **M** using overall mass balance and lever rule.
3. Either specify E_1 or R_N (we will always know one of them)
4. Connect a straight line through M passing through the one specified
5. Solve for unspecified one [via tie line]
6. Connect S through R_N and extrapolate
7. Connect E_1 through F and extrapolate; cross lines at Δ
8. Locate Δ by intersection of 2 lines
9. In general: connect E_n and R_n via equilibrium tie lines.
10. Repeat until reaching R_N

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Example

Example. Consider a system for which you have been given the ternary diagram. A = solute, S = solvent (100% pure), C = carrier. In a counter-current extraction process, the feed, F enters at 112 kg/hr with composition of 25 wt% solute and 75 wt% carrier. The solvent flow rate is 28 kg/hr.

- Find the number of theoretical stages required to achieve solute concentration in raffinate of 2.5 wt% (at most).
- Calculate the overall recovery and solute concentration of the extract stream.
- Plot solute concentrations in the extract and raffinate streams versus stages number.

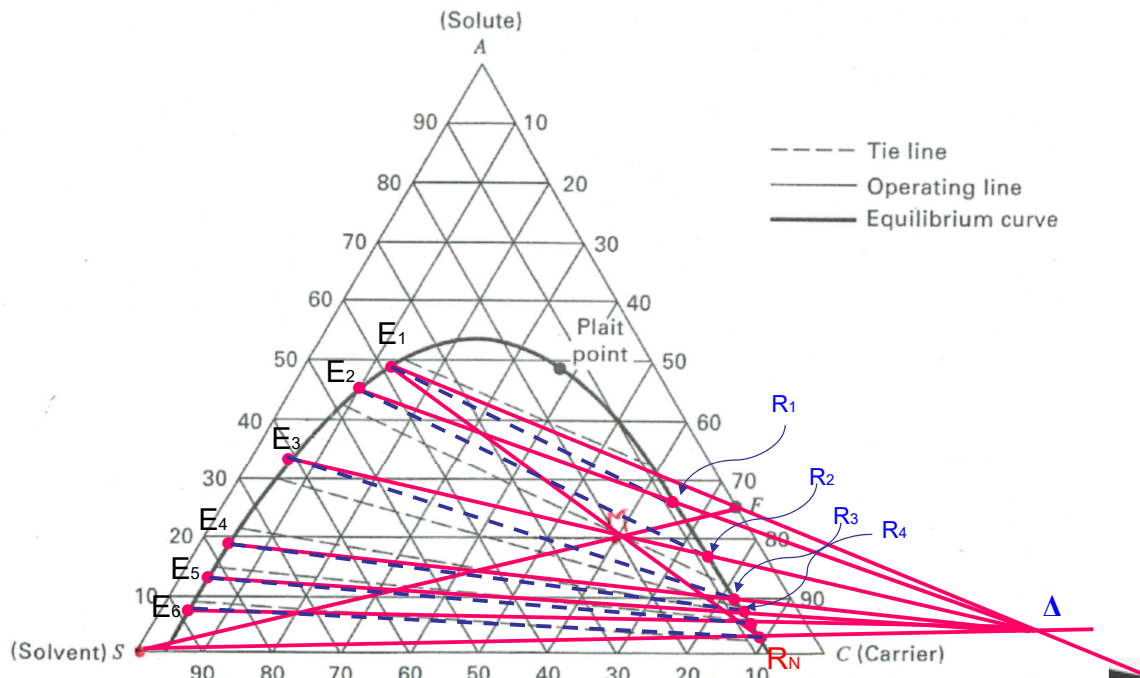
$$S = 28 \text{ kg/h} ; F = 112 \text{ kg/h} ; x_{R_N,A} = 0.025 ; x_{F,A} = 0.25 ; x_{F,C} = 0.75$$

The objective now is to have a counter-current system so the raffinate leaving in the N^{th} stage, R_N has $y_{A,R_N} = 0.025$

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Example cont.d



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Example cont.d



$$M = S + F = 28 + 112 = 140 \text{ kg/h}$$

$$x_{R_N,A} = 0.025 \quad y_{E_1,A} = 48\%$$

$$\rightarrow R_N = 87 \text{ kg/h}$$

$$E_1 = M - R_N = 140 - 87 = 53 \text{ kg/h}$$

$$\text{Recovery} = 1 - \frac{R_N x_{R_N,A}}{F x_{F,A}}$$

$$= 1 - \frac{(87)(0.025)}{(112)(0.25)} = 92\%$$

$$x_{R_1,A} = 27.3\%$$

$$\rightarrow E_2 = 74.3 \text{ kg/h}$$

$$R_1 = E_2 + \Delta = 133.3 \text{ kg/h}$$

$$y_{E_2,A} = 45\%$$

$$x_{R_2,A} = 17.3\%$$

$$y_{E_3,A} = 34\%$$

$$x_{R_3,A} = 10\%$$

$$y_{E_4,A} = 20\%$$

$$x_{R_4,A} = 6.0\%$$

$$y_{E_5,A} = 12.5\%$$

$$x_{R_5,A} = 4.0\%$$

$$y_{E_6,A} = 7.0\%$$

$$x_{R_6,A} = 1.5\%$$

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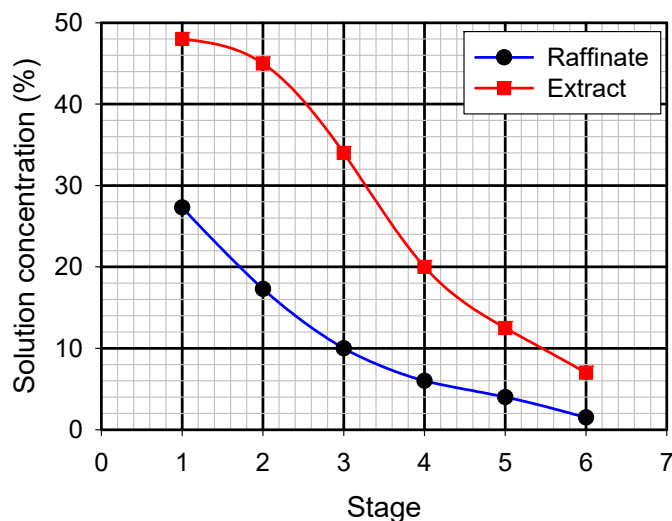


Example cont.d



$$x_{R_6,A} = 1.5\% < 2.5\%$$

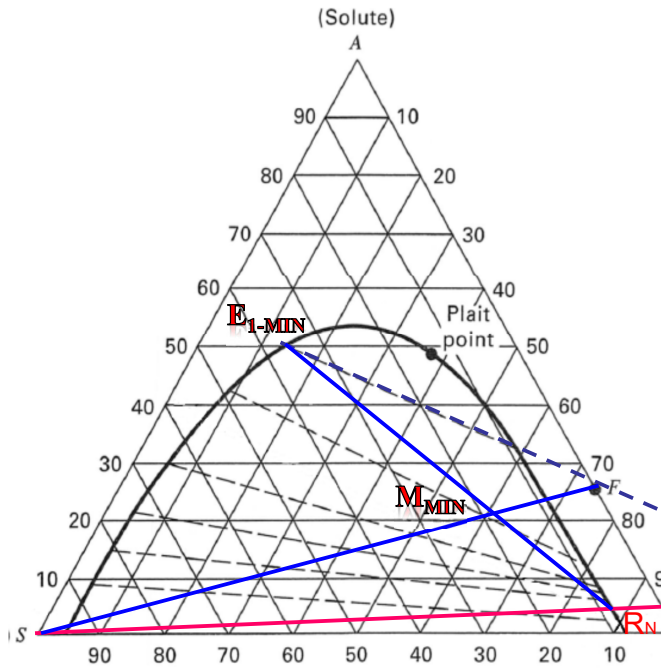
N=6 theoretical stages is enough to have solute concentration less than 2.5 wt% in raffinate stream



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Determining Minimum Solvent



Procedure:

- Draw the tie line passing through the **feed**.
- End of this tie line represents E_1 minimum ($E_{1-\min}$).
- Connect F and S with straight line on ternary phase diagram.
- Connect S and R_N with straight line on ternary phase diagram.
- The intersection of the two lines: $F-S_{\min}$ and $R_N-E_{1-\min}$ represents the minimum mixing point (M_{\min}).
- E_1F line must intersect with SR_N at the operating point denoted as Δ_{\min}

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Determining Minimum Solvent



- Using these fractions, do mass balance to calculate S_{\min}

The material balances can be established as follows

$$F + S_{\min} = M_{\min} = R_N + E_1$$

$$x_{A,F}F + x_{A,S}S_{\min} = x_{A,M}M_{\min}$$

$$x_{A,F}F + x_{A,S}S_{\min} = x_{A,M}(F + S_{\min})$$

$$\frac{S_{\min}}{F} = \frac{x_{A,F} - x_{A,M}}{x_{A,M} - y_{A,S}}$$

- The optimal operating S/F is approximately $1.5 S_{\min}/F$

In the previous figure, calculate S_{\min}/F

Remark. With S_{\min}/F , the required number of theoretical stages:

$N \rightarrow \infty$

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Determining Maximum Solvent

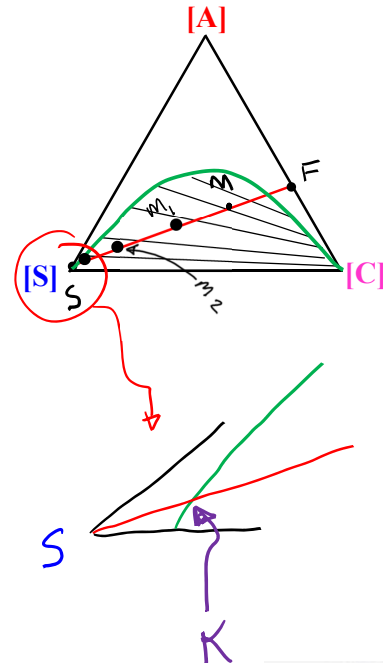


- if the amount of solvent (S) increases, the distance FM becomes longer, which means that Point M moves toward Point S
- The maximum S/F ratio (S_{\max}/F) occurs when Point M reaches the *extract-phase* curve,

$$\frac{S_{\max}}{F} = \frac{x_{A,F} - (x_{A,M})_{\max}}{(x_{A,M})_{\max} - y_{A,S}}$$

- When Point M is on the *extract-phase* curve, no raffinate phase is formed, and **only a single stage is needed**, which is **impractical**

Remark. With S_{\max}/F , the required number of theoretical stages: $N \rightarrow 1$



Determining Maximum Solvent



Remark.

- For extraction operation: $\left(\frac{S_{\min}}{F}\right) < \left(\frac{S}{F}\right) < \left(\frac{S_{\max}}{F}\right)$ or $S_{\min} < S < S_{\max}$
- A reasonable value : $S \approx 1.5S_{\min}$



Counter-current stage extraction with immiscible liquids



- The carries (C) of solute (A) is immiscible in solvent (S).
- In other words, solute is the only component distributed in extract and raffinate.
- This means that the raffinate has a binary mixture of A and C and the extract has binary mixture of A and S.
- In such ternary coordinates is not helpful.
- As in distillation and absorption, the equilibrium xy diagram is used instead.
- Let

x : solute concentration in raffinate

y : solute concentration in extract

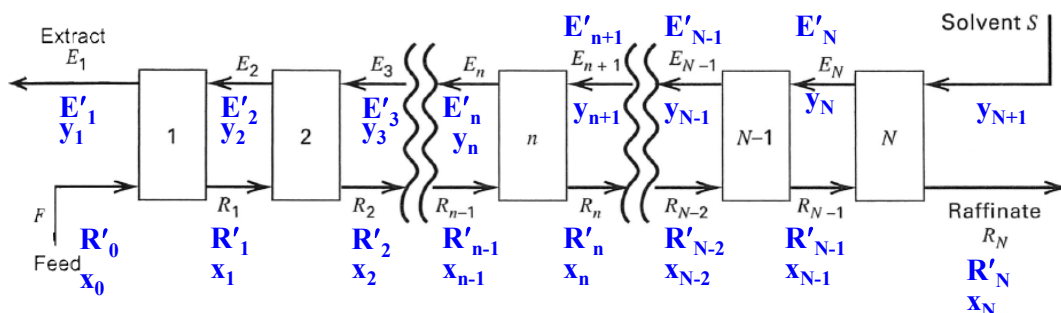
E' : flow rate of solvent in the extract streams

R' : flow rate of carrier B in the raffinate streams

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Counter-current stage extraction with immiscible liquids



- **Applying mass balance for carries B on each stage gives:**

$$R'_0 = (1 - x_0)F = R'_1 = R'_2 = \dots = R'_N = \text{constant} = R'$$

- **Applying mass balance for solvent S on each stage gives:**

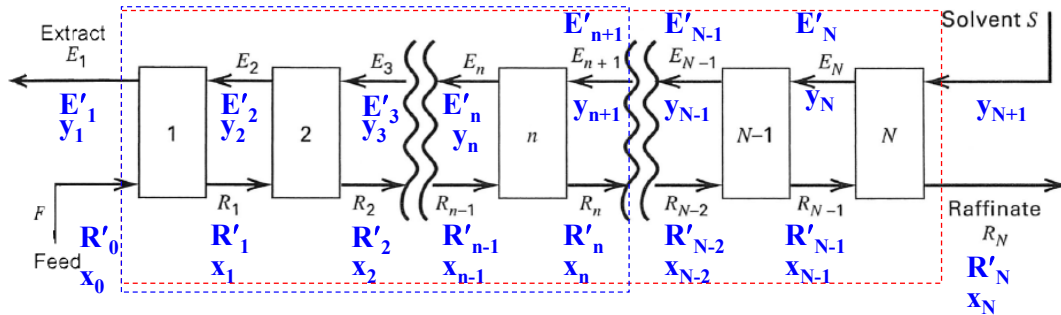
$$E'_1 = E'_2 = E'_3 = \dots = E'_N = (1 - y_{N+1})S = \text{constant} = E'$$

If pure solvent is used: $y_{N+1} = 0 \Rightarrow E' = S$

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Counter-current stage extraction with immiscible liquids



- Let us now define:
 - The **solute-to-carrier mass ratio** in the raffinate:

$$X = \frac{x}{1-x} [=] \frac{\text{kg solute A}}{\text{kg carrier C}}$$

- and **solute-to-solvent mass ratio** in the extract:

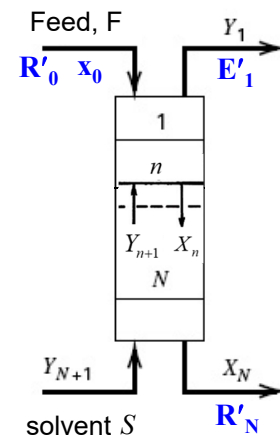
$$Y = \frac{y}{1-y} [=] \frac{\text{kg solute A}}{\text{kg solvent S}}$$



- Applying mass balance for solute A over stages: 1→N :**

$$R' \left(\frac{x_0}{1-x_0} \right) + E' \left(\frac{y_{N+1}}{1-y_{N+1}} \right) = R' \left(\frac{x_N}{1-x_N} \right) + E' \left(\frac{y_1}{1-y_1} \right)$$

$$E'Y_{N+1} = R'X_N + E'Y_1 - R'X_0$$



- Applying mass balance for solute A over stages: 1→n :**

$$R' \left(\frac{x_0}{1-x_0} \right) + E' \left(\frac{y_{n+1}}{1-y_{n+1}} \right) = R' \left(\frac{x_n}{1-x_n} \right) + E' \left(\frac{y_1}{1-y_1} \right)$$

$$E'Y_{n+1} = R'X_n + E'Y_1 - R'X_0$$





➤ **Dividing by E' gives:**
$$Y_{n+1} = \frac{R'}{E'} X_n + Y_1 - \frac{R'}{E'} X_0$$
 Operating line equation

▪ **For the above operating line equation:**

- Plot of Y_{n+1} versus X_n on X-Y diagram gives a straight line with:

$$\text{Slope} = \frac{R'}{E'}$$

$$\text{Intercept} = Y_1 - \frac{R'}{E'} X_0$$

- For certain feed flow rate, the minimum solvent flow rate, S_{\min} must correspond to the maximum allowable slope:

$$E'_{\min} = \frac{R'}{\text{Slope}_{\max}}$$

$$\text{But } (1 - y_{N+1})S = E' \Rightarrow S_{\min} = \frac{E'_{\min}}{(1 - y_{N+1})} = \frac{R'}{(1 - y_{N+1})(\text{Slope}_{\max})}$$



Example



An inlet water solution of 100 kg/h containing 0.01 wt fraction nicotine (A) in water is stripped with kerosene stream of 200 kg/h containing 0.0005 wt fraction nicotine in a countercurrent stage tower. The water and kerosene are essentially immiscible in each other. It is desired to reduce the concentration of exit water to 0.0010 wt fraction nicotine. Determine.

- Number of the theoretical stages needed.
- The minimum solvent rate.

The equilibrium data are as follows:

x	y
0.001010	0.000806
0.002460	0.001959
0.005000	0.004540
0.007460	0.006820
0.009880	0.009040
0.020200	0.018500

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Solution



- Solute (A) \equiv Nicotine; Carrier (B) \equiv Water
Solvent (S) \equiv Kerosene

$$F = 100 \text{ kg/h}; S = 200 \text{ kg/h}$$

$$x_0 = 0.01; x_N = 0.001; y_{N+1} = 0.0005$$

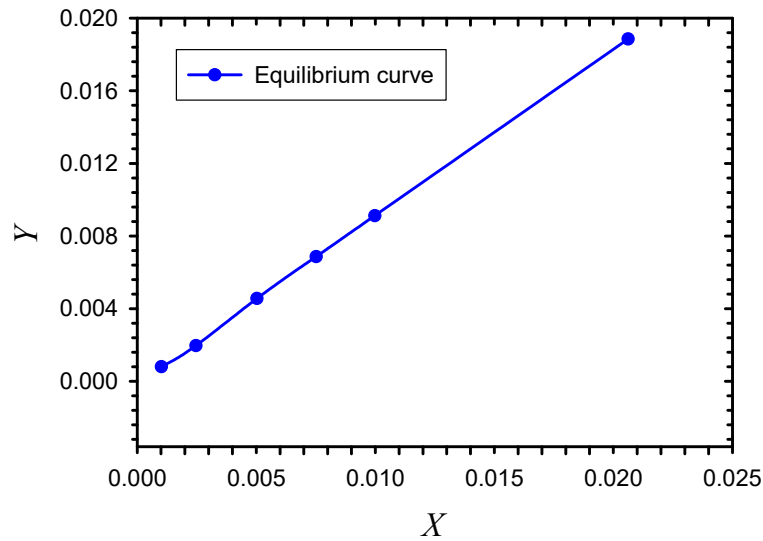
- Re-describe the equilibrium data in terms of solute-to-carrier mass ratio (X) and solute-to-solvent mass ratio in the raffinate

$X=x/(1-x)$	$Y=y/(1-y)$
0.001011	0.000807
0.002466	0.001963
0.005025	0.004561
0.007516	0.006867
0.009979	0.009122
0.020616	0.018849

- Draw XY equilibrium curve

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$$F = 100 \text{ kg/h}; S = 200 \text{ kg/h}$$

$$x_0 = 0.01; x_N = 0.001; y_{N+1} = 0.0005$$

▪ Draw the operating line:

1. Calculate flow rate of water: $R' = (1 - x_0)F = 99.0 \text{ kg/h}$

2. Calculate flow rate of solvent (Kerosene):

$$E' = (1 - y_{N+1})S = 200(1 - 0.0005) = 199.9 \text{ kg/h}$$

3. Find the slope of operating line equation:

$$\text{Slope} = R'/E' = 0.50$$

4. Calculate:

$$X_0 = x_0 / (1 - x_0) = 0.01$$

$$X_N = x_N / (1 - x_N) = 0.001$$

$$Y_{N+1} = y_{N+1} / (1 - y_{N+1}) = 0.0005$$



Solution cont.d



5. Determine Y_1 . The point $(X_N, Y_{N+1})=(0.001, 0.0005)$ must lie on the operating line, thus

$$Y_{N+1} = \frac{R'}{E'} X_N + Y_1 - \frac{R'}{E'} X_0$$

$$0.0005 = (0.50)(0.001) + Y_1 - (0.50)(0.01) \rightarrow Y_1 = 0.005$$

The point $(X_0, Y_1)=(0.01, 0.005)$ must also lie on the operating line.

6. With the two point: $(X_N, Y_{N+1})=(0.001, 0.0005)$ and $(X_0, Y_1)=(0.01, 0.005)$ we can now draw the operating line.

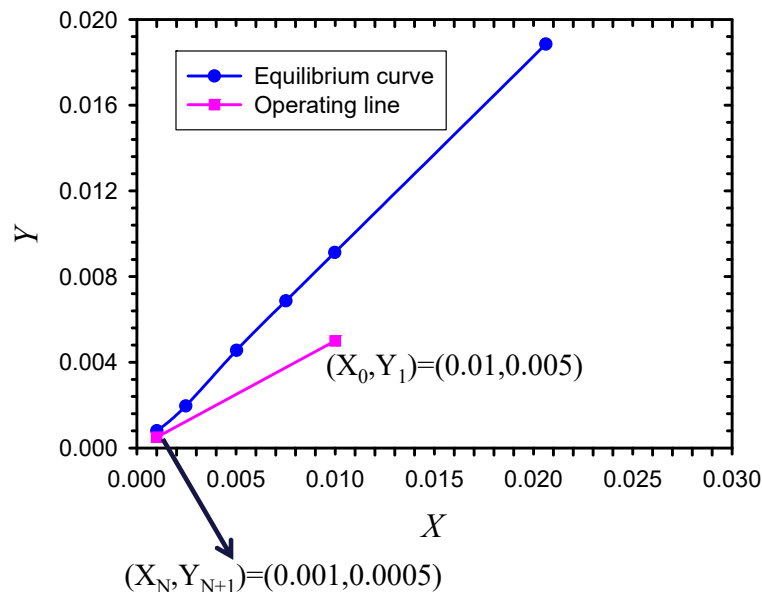
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Solution cont.d



- If x and y are quite small (dilute solutions): $X \approx x ; Y \approx y$



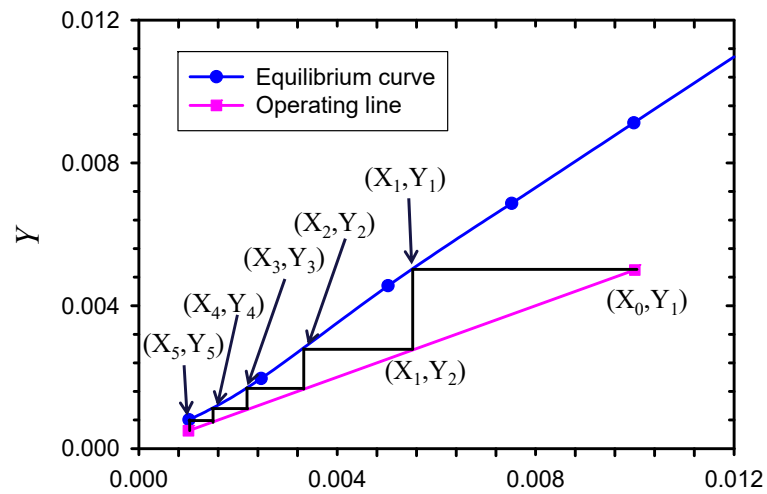
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Solution cont.d



- The number of theoretical stages are stepped off, as in distillation and absorption, from as (X_0, Y_1) to (X_N, Y_{N+1}) :



a) The number of theoretical stages; $N=5$ X

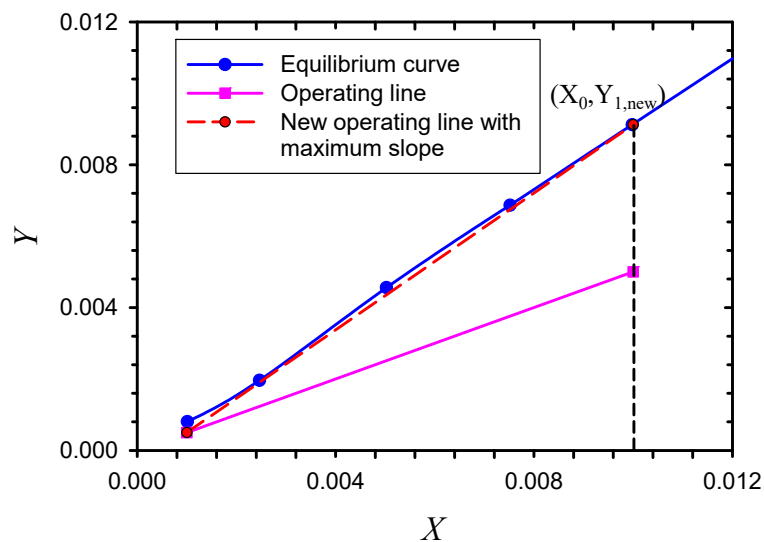
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Solution cont.d



b) Minimum solvent flow \rightarrow Maximum allowable slope



$$\text{Slope}_{\max} = \frac{Y_{1,\text{new}} - Y_{N+1}}{X_0 - X_N} = \frac{0.009122 - 0.0005}{0.01 - 0.001} = 0.96$$

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b) Minimum solvent flow → Maximum allowable slope

$$\text{Slope}_{\max} = 0.96 = \frac{R'}{E'_{\min}} = \frac{99.0}{E'_{\min}} \rightarrow E'_{\min} = 103.1 \text{ kg/h}$$

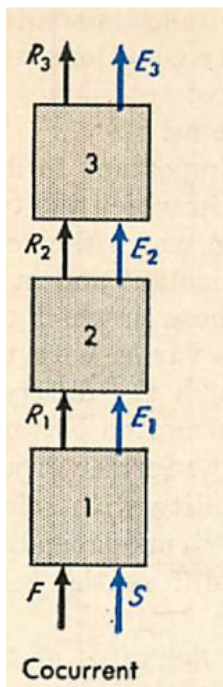
$$E'_{\min} = 103.1 = S_{\min} (1 - y_{N-1}) = S_{\min} (1 - 0.0005)$$

$$\rightarrow S_{\min} = 103.1 \text{ kg/h}$$

- If reasonable solvent flow rate; $S = 1.5S_{\min}$, for such process we should use around 155 kg/h. Try to find number of stages with this solvent flow rate.

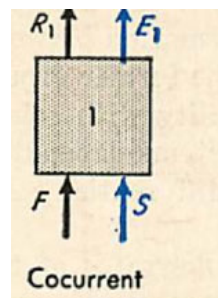


Cocurrent one stage extraction



$$M = S + F = R_1 + E_1 = R_2 + E_2 = \dots\dots\dots = R_N + E_N$$

Obviously, always there will be only one theoretical stage operating in cocurrent mode.



Converting theoretical stages to actual equipment size



Assume the counter-current stage extraction needs 6 theoretical stages to achieve some required recovery:

- This does not mean we require 6 mixer-settlers (though we could do that, but costly).
- It means we need an extraction column which has equivalent operation of 6 counter-current mixer-settlers that fully reach equilibrium.
- At this point, we resort to correlations and vendor assistance.
- Vendors: provide **HETS** = Height Equivalent to a Theoretical Stage.
- Use that to size the extraction column:

$$H = \frac{\text{HETS} \times N}{\text{Stage efficiency}}$$

Where H is the height of extraction tower

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Converting theoretical stages to actual equipment size



TABLE 12.6-1. Typical Performance for Several Types of Commercial Extraction Towers

Type	Capacity of Combined Streams, $(V_D + V_C)$, $m^3/m^2 \cdot h$	Approximate Flooding, $(V_D + V_C)$, $m^3/m^2 \cdot h$	Spacing between Stages, T , cm	Overall Height of Transfer Unit, H_{OU} , m	Plate Efficiency, E_o , %	Height of Equilibrium Stage, HETS, m	Ref.
Spray Tower	15-75			3-6		3-6	M4, S5
Packed Tower	12-30			0.9-1.7		0.4-1.5	S4, S5, W1
Structured Packing Tower	65-90					0.5-1.6	H4
Sieve-Tray Tower	27-60		10-25		8-30	0.8-1.2	M4, P4, S4
Pulsed Packed Tower	17-23	40				0.15-0.3	P4, S4, W1
Pulsed Sieve-Tray Tower	25-35	60	5.1			0.15-0.3	S4, W1
Scheibel Tower	10-14*	40	2.5-20			0.1-0.3	P4, S2, S3, W1
Karr Tower	30-40	80-100	5-15			0.2-0.6	S2, S4

*Throughput for diameter $D_t = 76$ cm. For larger towers of D_t diameter see Eq. (12.6-3).

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