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Vapour-Liquid Equilibria

Objectives

- Reminder of vapour-liquid equilibria
- Reminder of relative volatility

Vapour-Liquid Equilibria

- For each component in a mixture, the vapour and liquid are in thermodynamic equilibrium when,

$$f_i^V = f_i^L$$

f is the fugacity

- Which are defined as,

$$f_i^V = \phi_i^V y_i P$$

$$f_i^L = \phi_i^L x_i P$$

ϕ is the fugacity coefficient

- Or more typically,

$$f_i^L = \gamma_i x_i P_i^0$$

γ is the activity coefficient

Reminder – Vapour-Liquid Equilibria

- This means that,

$$\phi_i^V y_i P = \gamma_i x_i P_i^o$$

- Which can be rearranged to give,

$$K_i = \frac{y_i}{x_i} = \frac{\gamma_i P_i^o}{\phi_i^V P}$$

- At moderate pressures, $\phi \rightarrow 1$

$$K_i = \frac{\gamma_i P_i^o}{P}$$

- And if ideal system,

$$K_i = \frac{P_i^o}{P}$$

Reminder – Relative Volatility

- The relative volatility is the ratio of the *K* values for two components,

$$\alpha_{ij} = \frac{K_i}{K_j} = \frac{\frac{y_i}{x_i}}{\frac{y_j}{x_j}}$$

$$K_i = \frac{P_i^o}{P}$$

- The geometric mean of the top and bottom product values is most commonly used as a column average,

$$(\alpha_{ij})_{mean} = \sqrt{(\alpha_{ij})_{top}(\alpha_{ij})_{bottom}}$$

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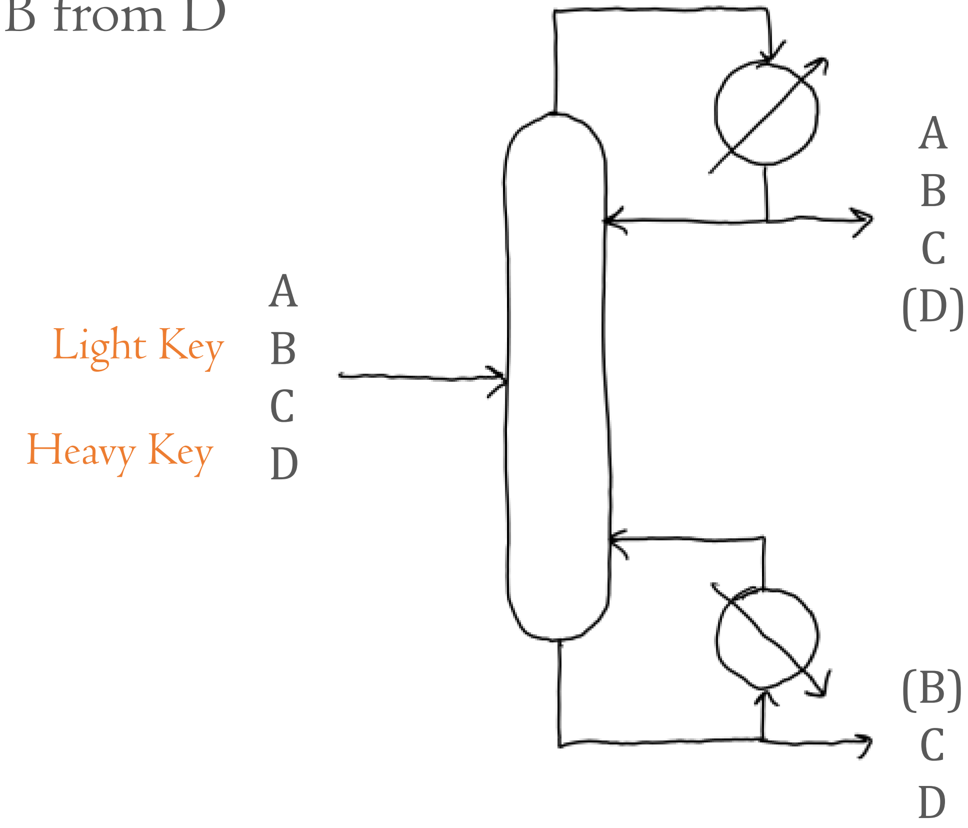
Multi-Component Distillation

Objectives

- Define multicomponent key components
- Short-cut process for multicomponent distillation
- FUG equations

Multicomponent Distillation

- Separate B from D



A
Light Key B
C
Heavy Key D

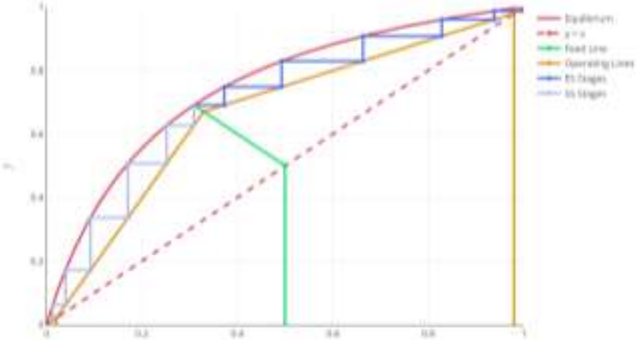
A
B
C
(D)

(B)
C
D

C is an intermediate boiling component

Binary Distillation Reminder

- For binary distillation there are several short-cut methods to calculate the number of stages needed, e.g.:
  - McCabe-Thiele



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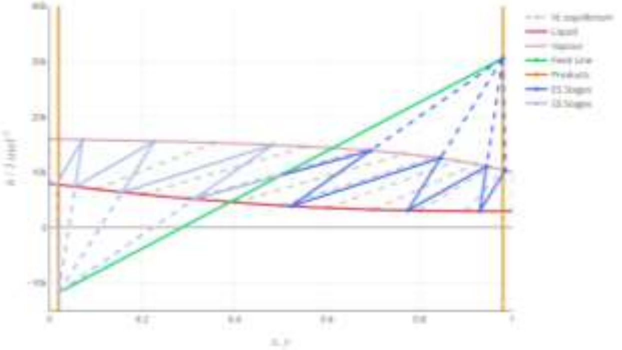
  - Ponchon-Savarit



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Multi-component Distillation

- As many components are present in the feed, fixing the recovery or mole fraction of a single component does not specify the product composition or temperature.
- The selection of the key components is a process engineering decision, usually based on the objectives of the separation

Short-Cut Method

- There are a number of short-cut methods for simple columns.

- These are based on,
  - Theoretical analysis of a simplified column, or
  - Empirical correlations

Specify the Column Separation

1

Specify top product phase, i.e. total or partial condenser

2

Select light and heavy key components

3

Specify mole fraction of light key in the bottoms and heavy key in the distillate

4

Specify pressure of condenser and reboiler

Calculate N_{min}

1

Calculate relative volatilities – top, bottom and feed point

2

Calculate N_{min} – Fenske equation

3

Estimate the split of the non-key components – Hengstebeck-Geddes

4

Repeat steps 1 to 3 until N_{min} doesn't change

Calculate R and Actual Stages

1

Calculate R_{min} – Underwood equations

2

Pick a $R = aR_{min}$ value

3

Calculate the actual number of stages, N – Gilliland correlation

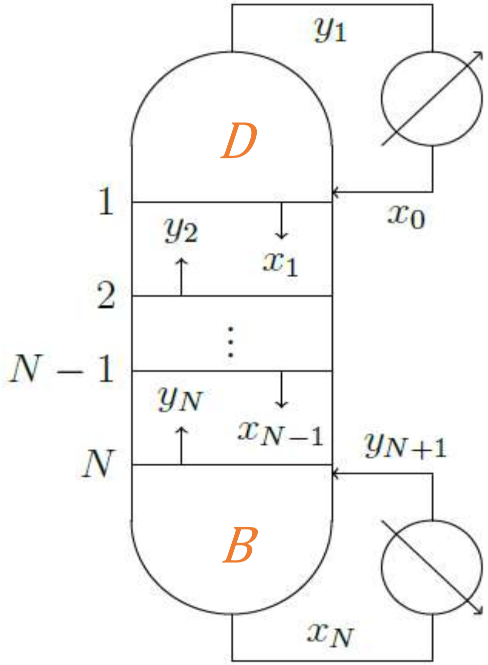
4

Calculate the ratio of rectifying to stripping stages – Kirkbride correlation

The Fenske Equation

- Estimation of the minimum number of the theoretical stages

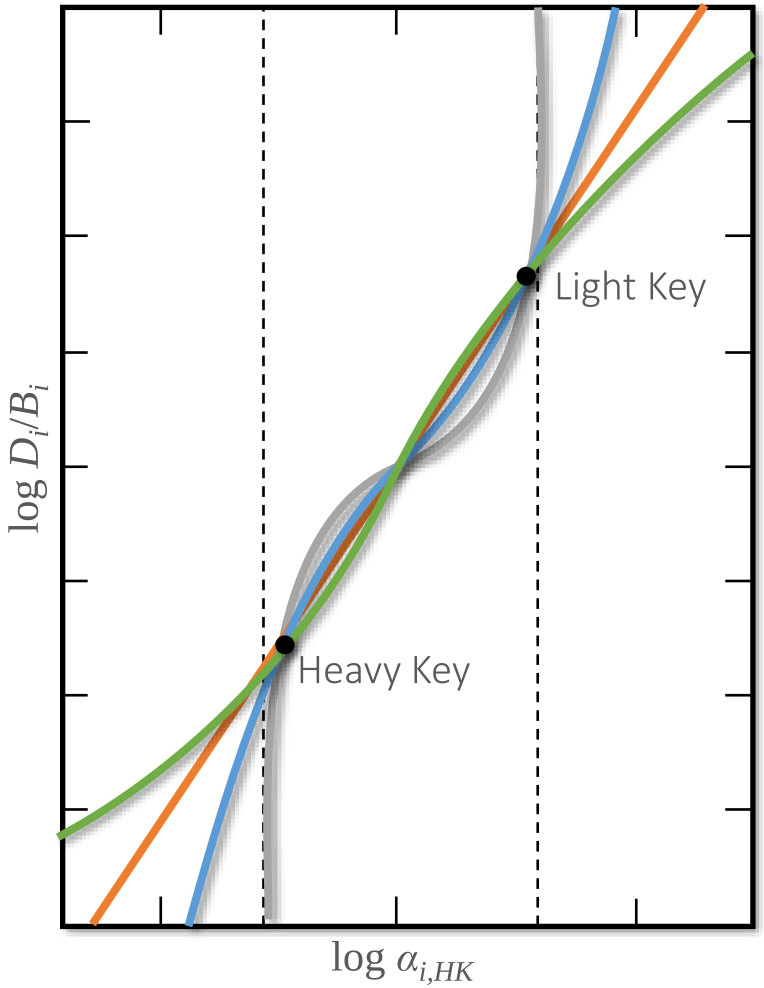
$$N_{min} = \frac{\log\left[\left(\frac{x_L}{x_H}\right)_D \left(\frac{x_H}{x_L}\right)_B\right]}{\log \bar{\alpha}_{LH}} = \frac{\log\left[\frac{D_L}{D_H}\,\frac{B_H}{B_L}\right]}{\log \bar{\alpha}_{LH}}$$



The Hengstebeck-Geddes Method

- Total reflux (Fenske equation)
- Minimum reflux (little distribution of the non-key components)
- High L/D (~$5R_{min}$)
- Low L/D (~$1.1R_{min}$)

$$\log \frac{D_i}{B_i} = A + C \log \alpha_{i,r}$$



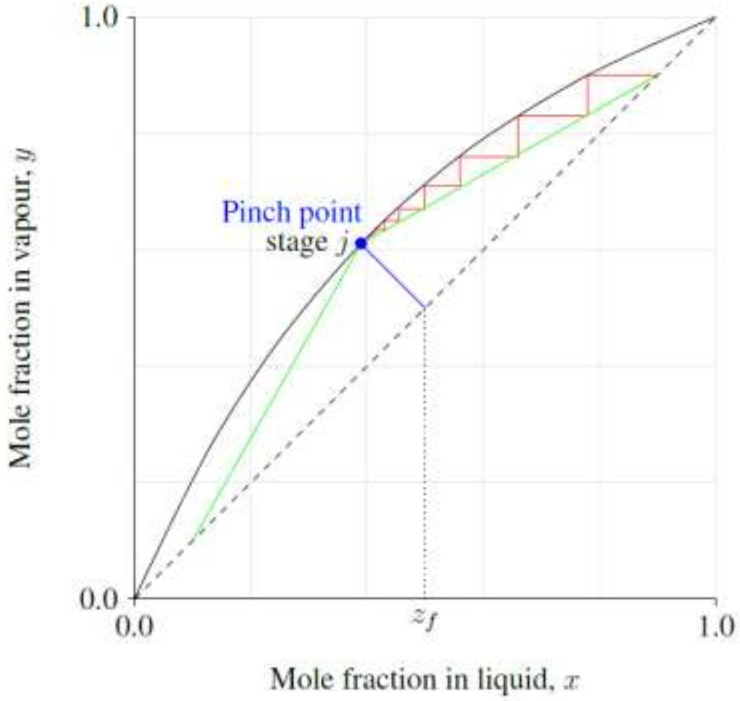
The Underwood Equation

- Estimation of the minimum reflux ratio

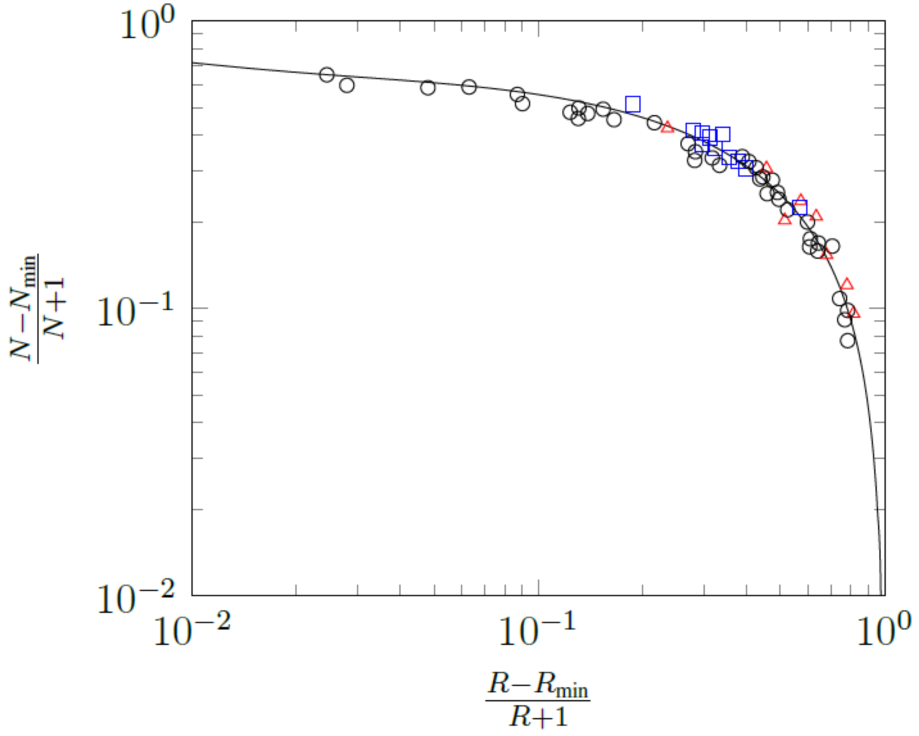
$$1 - q = \sum_i \frac{\alpha_{i,r} z_{F,i}}{(\alpha_{i,r} - \phi)}$$

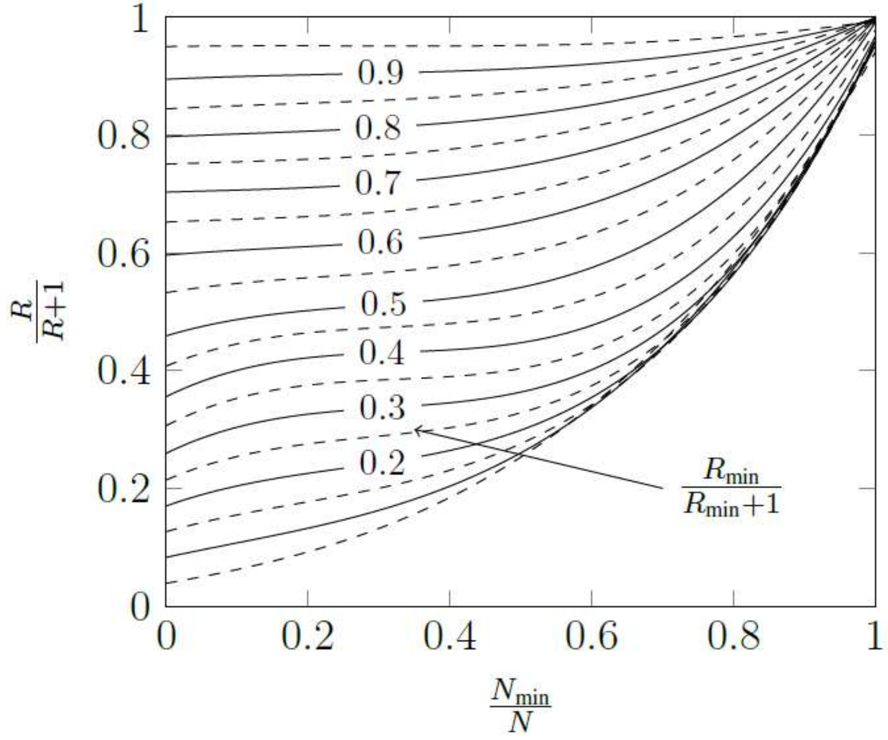
Solve for ϕ in 1 then use in 2

$$R_{min} + 1 = \sum_i \frac{\alpha_{i,r} x_{D,i}}{(\alpha_{i,r} - \phi)}$$



Actual Number of Stages


Gilliland Correlation


Erbar-Maddox Correlation

The Kirkbride Correlation

- An approximation of the optimal feed-stage location can be made with the Kirkbride empirical equation

$$\log\left(\frac{N_r}{N_s}\right) = 0.206 \log\left[\left(\frac{B}{D}\right)\left(\frac{x_{HK,f}}{x_{LK,f}}\right)\left(\frac{x_{LK,b}}{x_{HK,d}}\right)^2\right]$$

- N_r is the number of stages in the rectifying section (above feed stage)
- N_s is the number of stages in the stripping section (below feed stage)

Distillation Operation

Objectives

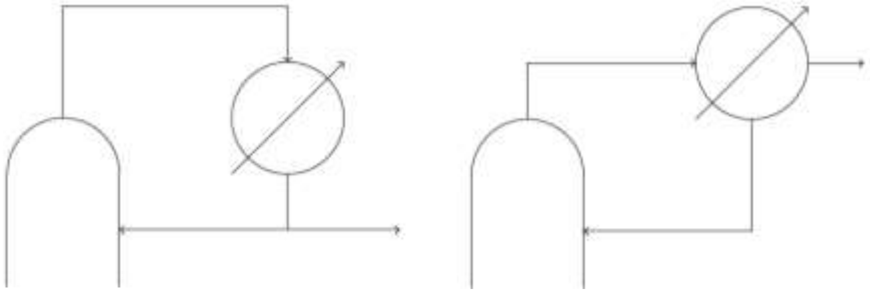
- Considerations for distillation design
- Performance indicators

Distillation Operating Parameters

- Usually the feed composition and flow rate are considered fixed.
- The product specifications are usually fixed.
- There are parameters that can be changed.

Operating Pressure

- The condenser temperature sets the operating pressure:
  - If cooling water is to be used, the condensing temperature of the overhead product should be typically 10°C above the summer cooling water temperature.
  - If air cooling is to be used, the condensing temperature of the overhead product should be typically 20°C above the summer air temperature.

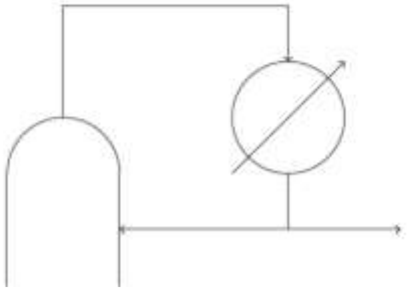


Total Condenser
Bubble point

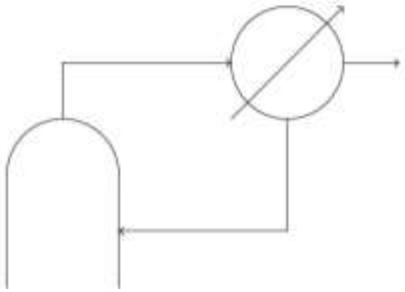
Partial Condenser
Dew point

Type of Condenser

- Total condenser
  - Liquid product for intermediate or final product storage.
  - The top product is to be fed to another distillation at a higher pressure.



- Partial condenser
  - Reduce the condenser duty
  - Avoid expensive refrigeration

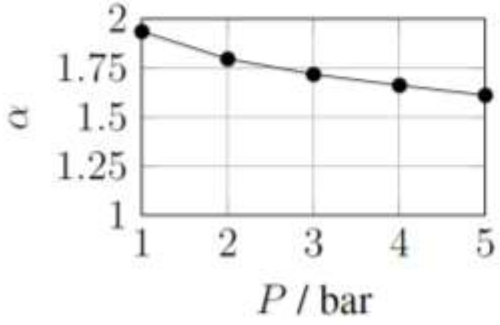


Operating Pressure - Exceptions

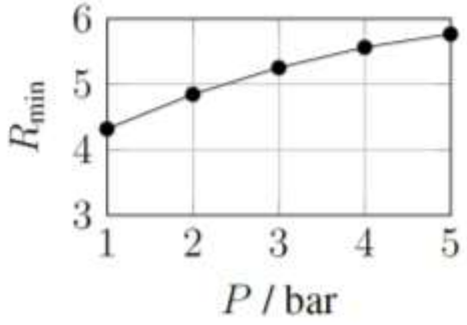
- In the separation of gases and light hydrocarbons
  - Pressure maybe too high to allow cooling water
- For distilling high molecular weight materials
  - Max temperature maybe set by product decomposition
- In a sequence of columns
  - Heat integration

Operating Pressure

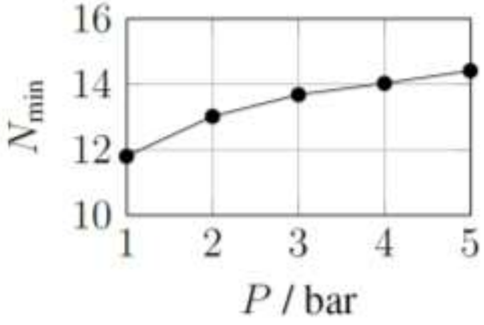
- Benzene-Toluene(LK)-Ethylbenzene(HK)-Styrene – Constant separation



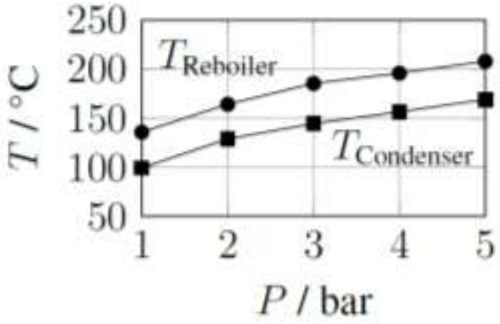
α decreases



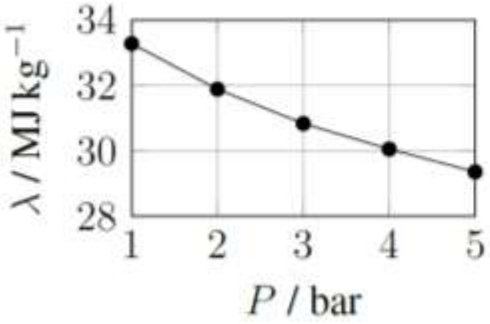
R_{min} increases



N_{min} increases

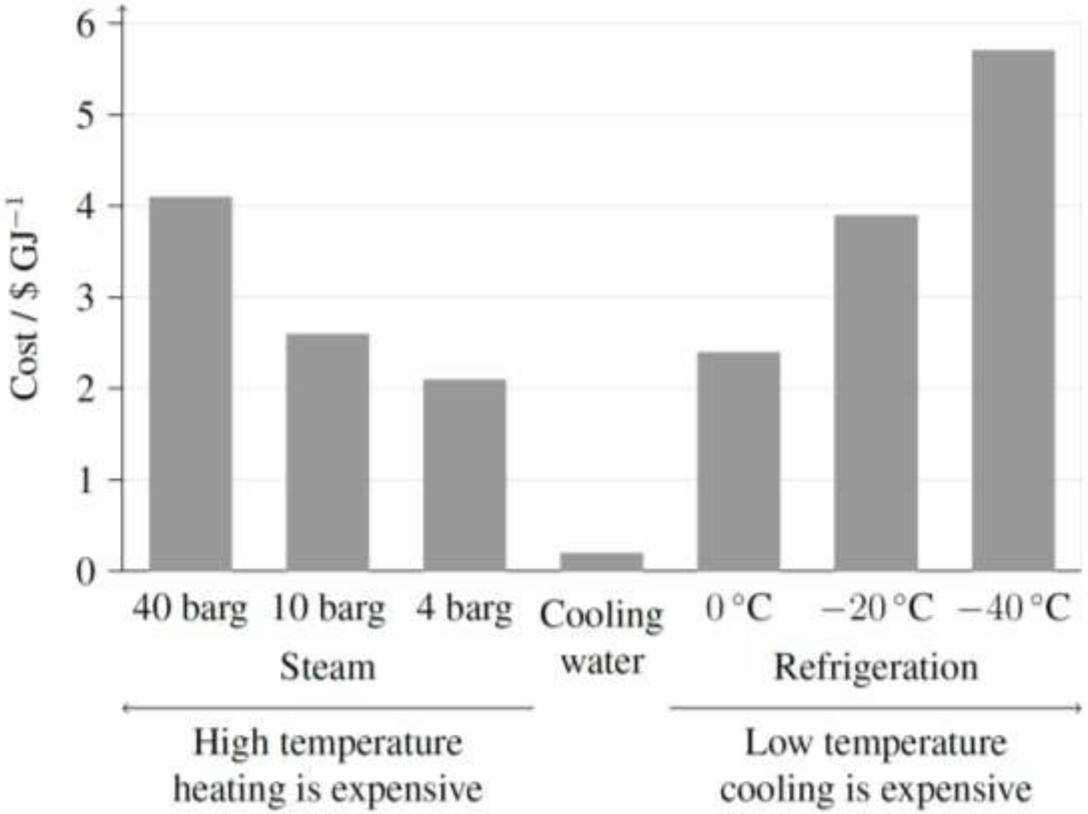


T increases



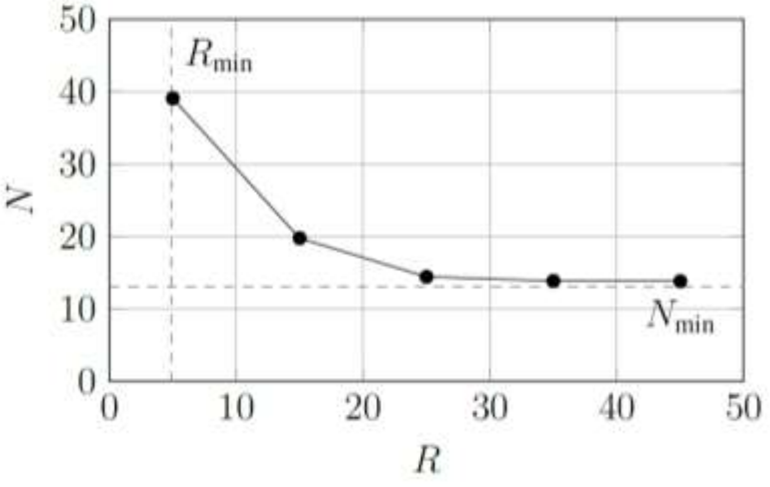
λ decreases

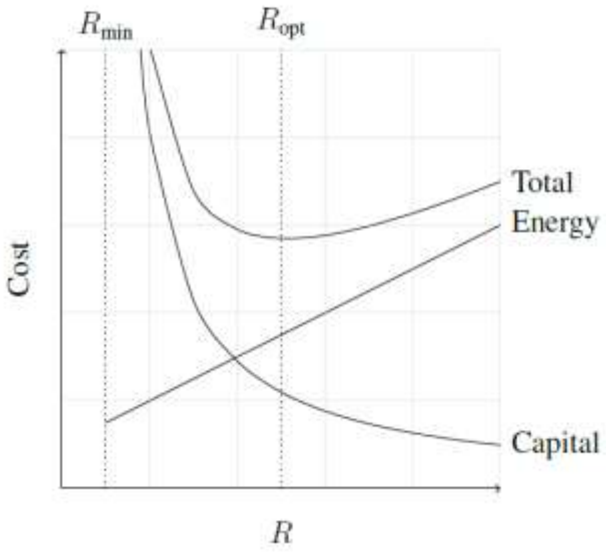
Condenser/Reboiler Temperature



Choice of Reflux Ratio

- Rule of thumb -
    - Use a reflux ratio of 1.1 to 1.3 times the minimum reflux ratio.





Choice of Feed Condition

$$q = \frac{\text{Heat required to vapourise 1 mole of feed}}{\text{Molar latent heat of vapourisation of feed}}$$

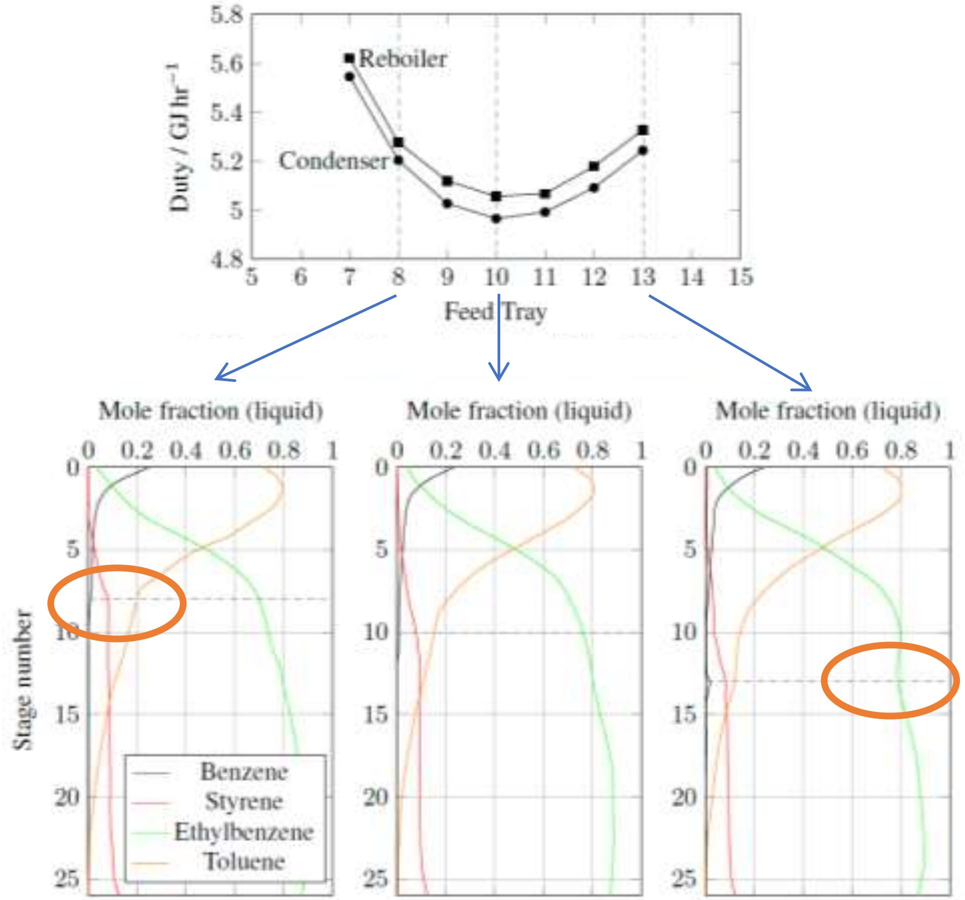
- $q = 1$, (saturated liquid feed) is preferred
    - This makes the column pressure easy to control by pumping the liquid to the correct pressure.
- In columns with multiple feeds, it is not unusual to use feeds with $q < 0$ or $q > 1$, which would be determined by the practical needs.

Choice of Feed Condition

- Heating or cooling the feed can reduce overall energy costs.
- The best feed condition can only be determined by considering heat recovery opportunities with the overall process.

Choice of Feed Stage Location

- Increased duty caused by disruptions in profile



Performance Indicators

- Some quantitative performance indicators are proposed for screening distillation sequences.
- These are,
  - Total vapour load
  - Total energy demand
  - Operating cost
  - Capital cost
  - Total annualised cost including capital, energy trade-offs, and the consideration of heat integration

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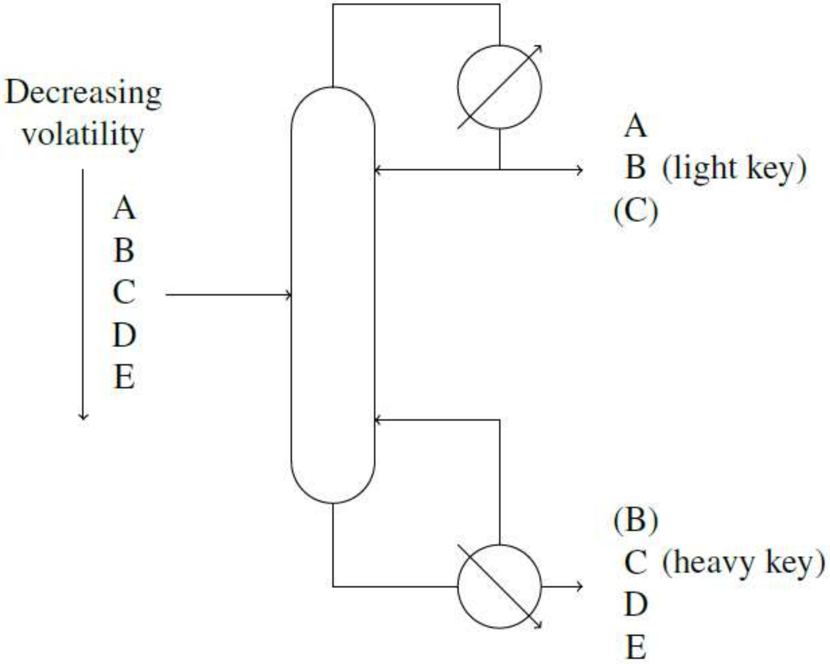
Simple Distillation Column Sequencing

Objectives

- Definition of a simple distillation column
- Introduction to distillation column sequencing
- Design heuristics for simple distillation columns

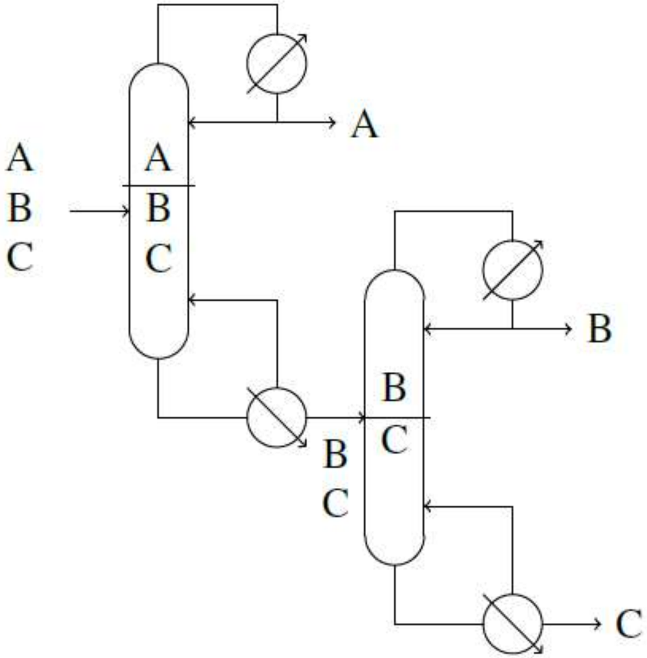
Simple Distillation Columns

- A simple distillation column means that it has
    - A single feed,
    - Two products,
    - The key components are adjacent in volatility, and
    - The column has a reboiler and a condenser.

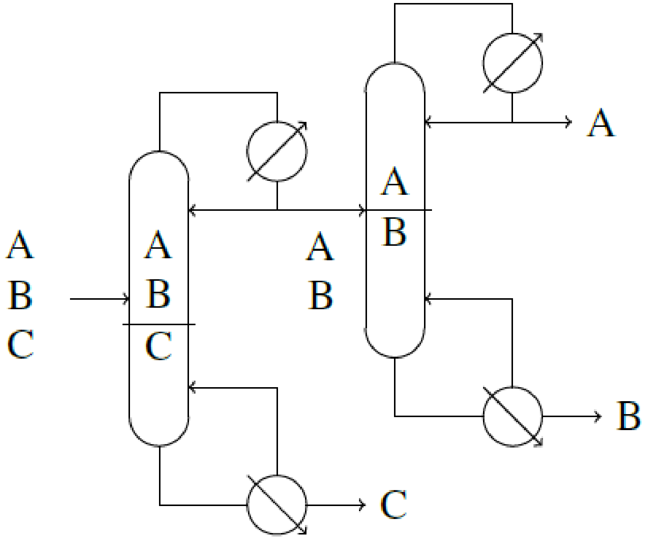


Sequencing Simple Distillation Columns

- Suppose there is a homogeneous mixture of A, B, and C



Direct Sequence
Lightest component separated



Indirect Sequence
Heaviest component separated

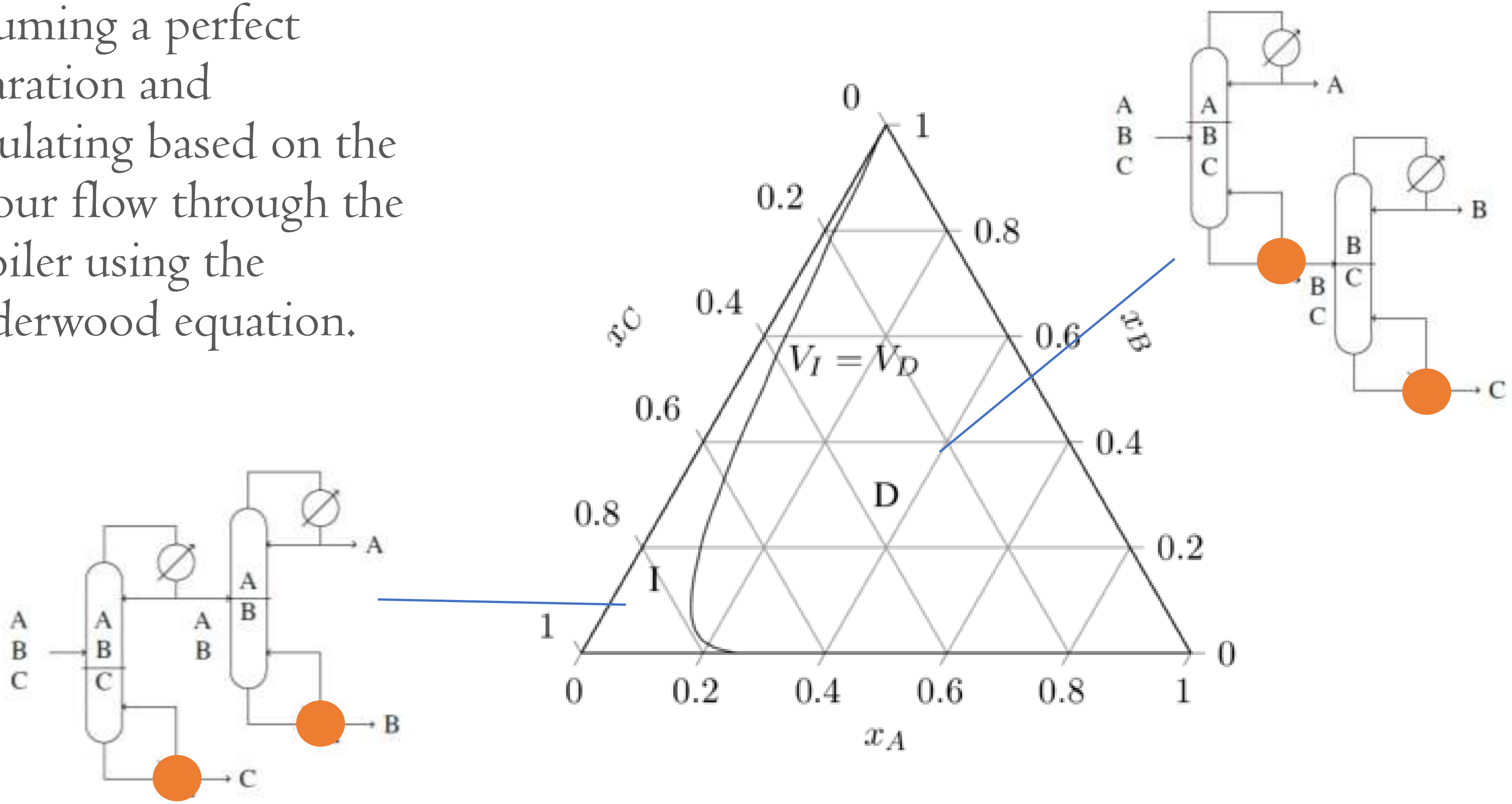
Sequencing Simple Distillation Columns

- The direct sequence often requires less energy than the indirect one.
    - Light component only vapourised once

- The indirect sequence can be quite energy efficient compared to the direct one if the light component in the feed has a low flowrate but the heavy component has a high flowrate.

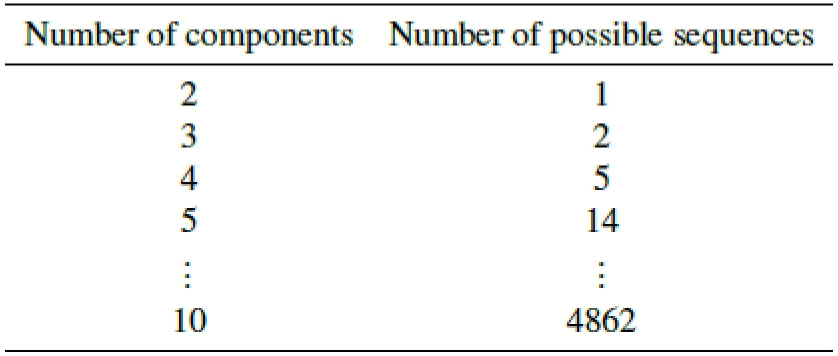
Direct Sequence

- Assuming a perfect separation and calculating based on the vapour flow through the reboiler using the Underwood equation.



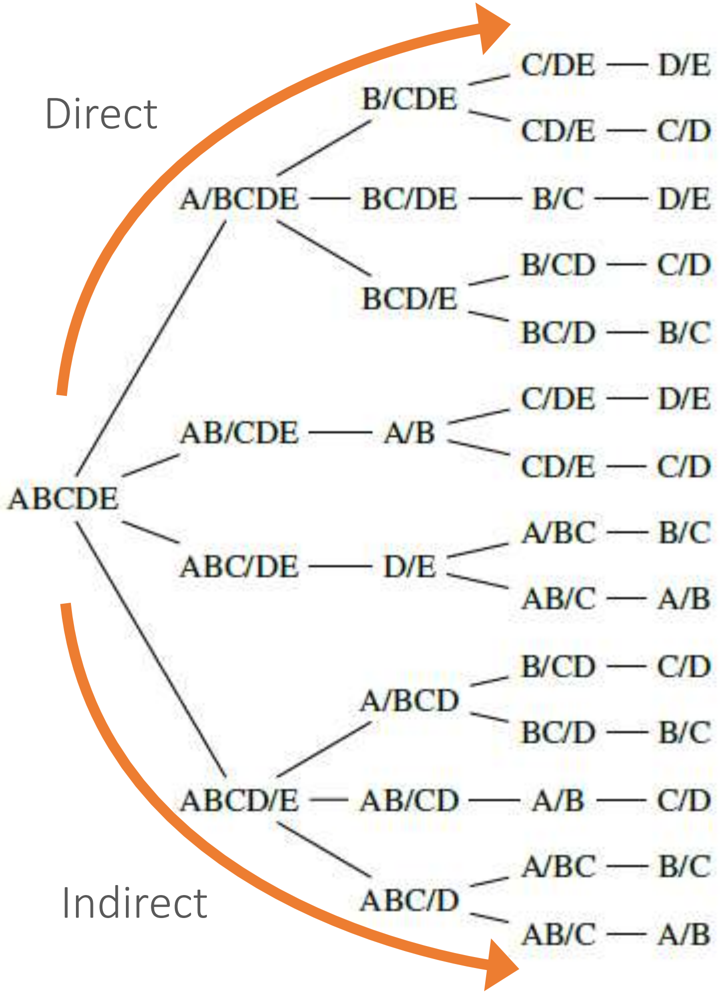
Sequencing Simple Distillation Columns

- When the number of components in a mixture increases, the number of sequences increases dramatically.



Number of components	Number of possible sequences
2	1
3	2
4	5
5	14
⋮	⋮
10	4862

Sequencing Simple Distillation Columns



Distillation Columns Sequencing Rules

- Certainly, not every sequence is to be feasible, there are practical constraints to sequencing columns including,
- Some components need to be remove early:
    - hazardous component,
    - reactive and heat-sensitive components, and
    - corrosive components.

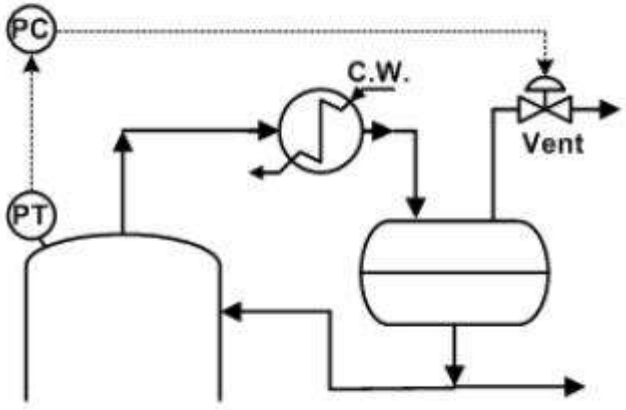
Distillation Columns Sequencing Rules

- Decomposition in the reboilers would contaminate products,
  - Finished products should not be taken from bottoms of columns if possible.

- Compounds which polymerise when distilled would require chemicals added to inhibit polymerisation,
  - These tend to be involatile and end up in the column bottoms, so it would be necessary to take finished products overhead.

Distillation Columns Sequencing Rules

- For those components that are difficult to condense, a total condensation would normally be required under low-temperature refrigeration and/or very high operating pressures.
  - The light components are normally removed from the top of the first column to minimise use of refrigeration and high pressures.



Distillation Columns Sequencing Heuristics

- For systems that do not include these issues, or after these materials have been removed, there are some heuristics developed for sequencing columns,

- They are useful as a guide line.

Heuristic Rules

- Do the most difficult separation last
- Remove large fraction components first
- Favour the direct sequence
- Separate each feed 50/50

Distillation Columns Sequencing Heuristics

- Difficulties can arise when those heuristics are in conflict with each other.
- Heuristics can be contradictory to each other but they do have advantages.

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Complex Distillation Column Sequencing

Objectives

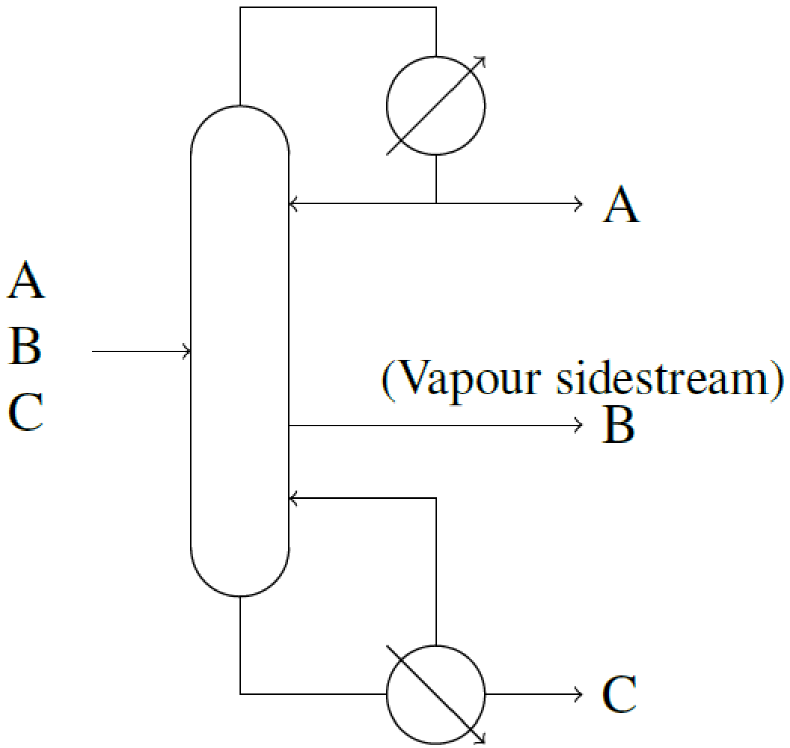
- Introduction to complex distillation columns
- Design heuristics for complex distillation columns
- Thermally coupled utilities

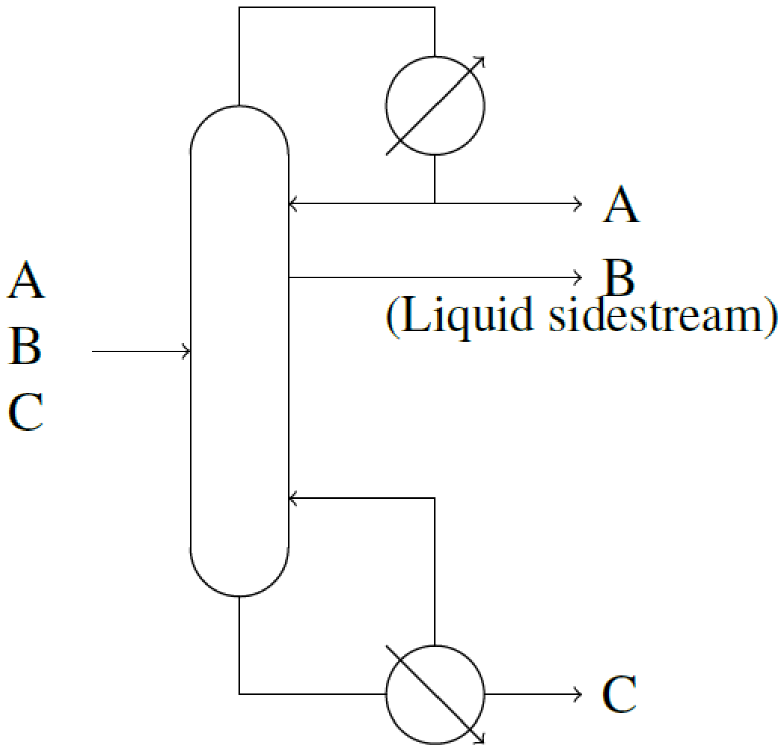
Complex Distillation Columns

- Complex distillation configurations can sometimes be referred to as thermally coupled distillation columns

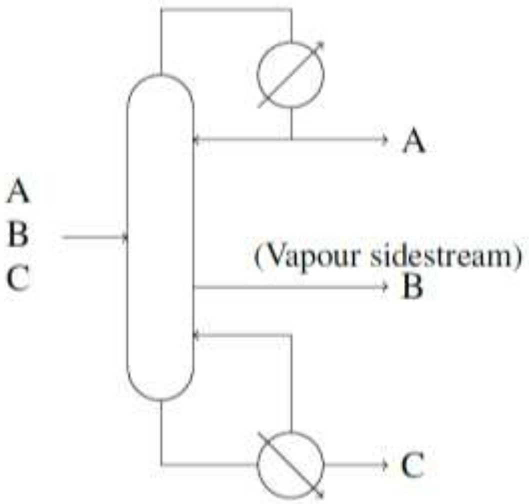
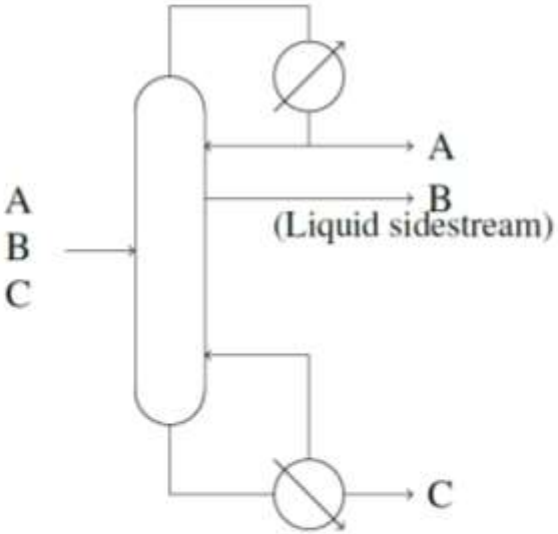
- Columns are linked by two-directional flows and one column has no condenser, and/or no reboiler.

Side-Stream Columns

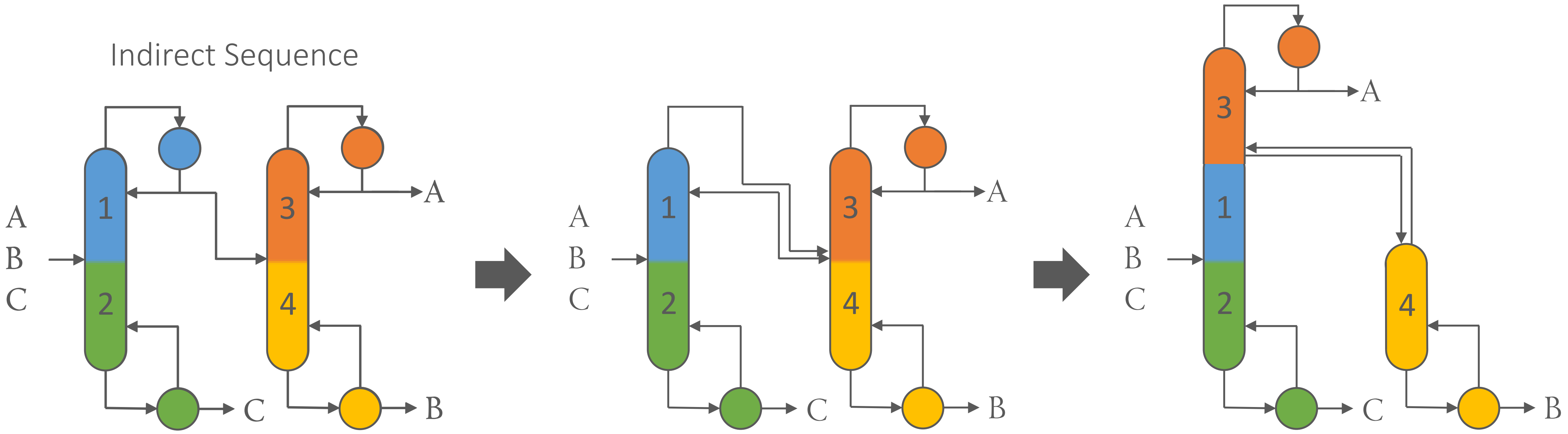




Heuristics for Side-Streams

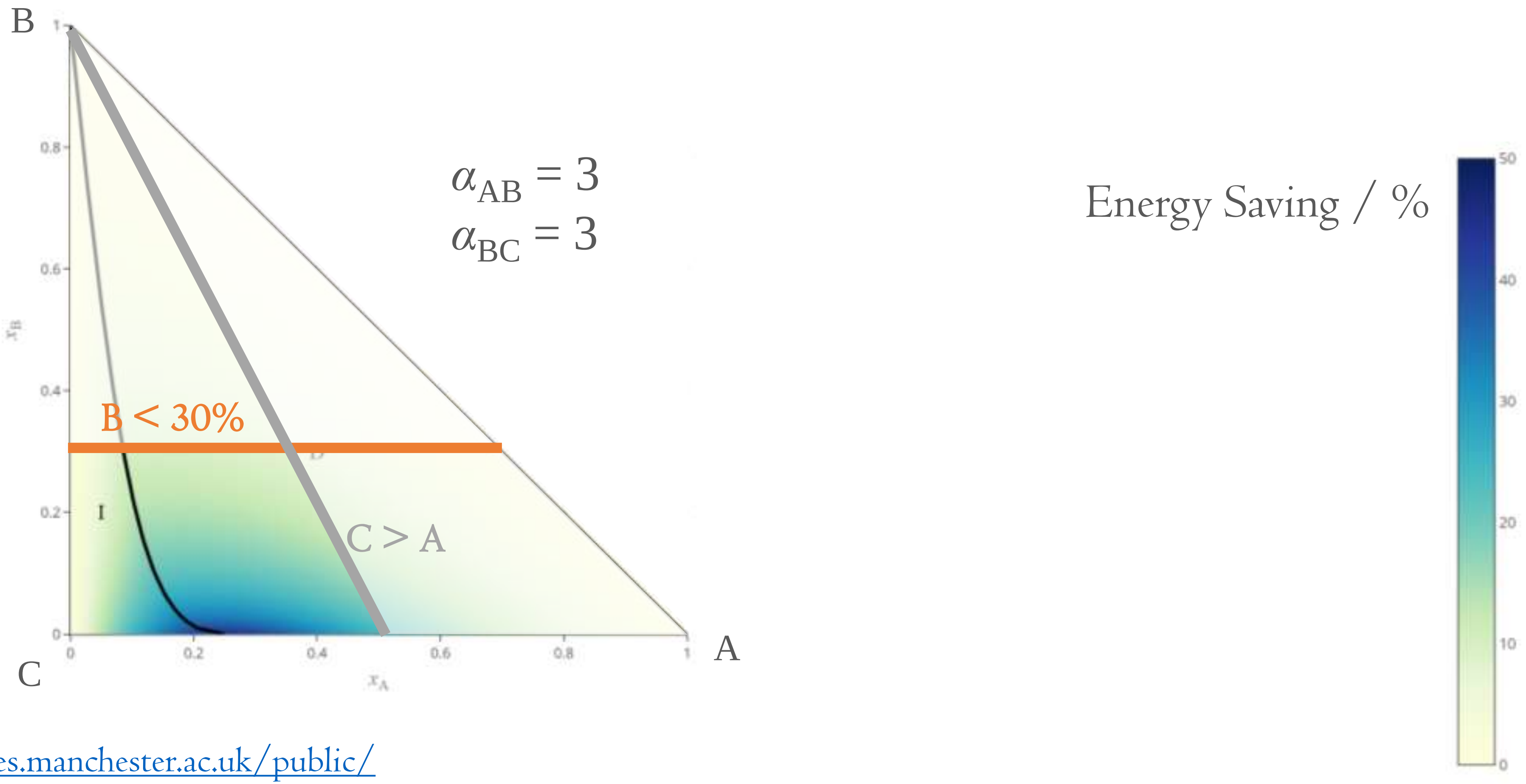
		
Feed Composition	B > 50% C < 5%	B > 50% A < 5%
Relative volatility	$\alpha_{BC} >> \alpha_{AB}$	$\alpha_{AB} >> \alpha_{BC}$

Side-Stripper Arrangement



Indirect Sequence

Side-Stripper Energy Saving



$\alpha_{AB} = 3$
$\alpha_{BC} = 3$

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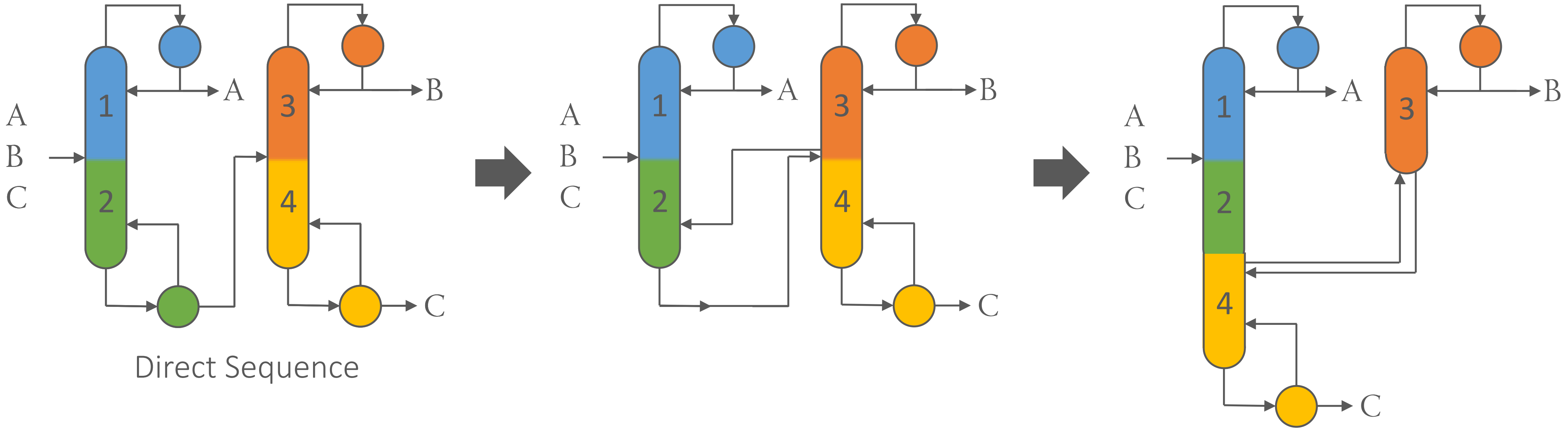
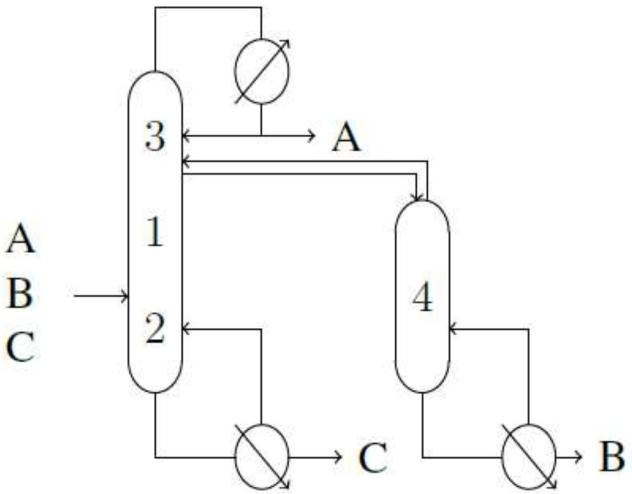
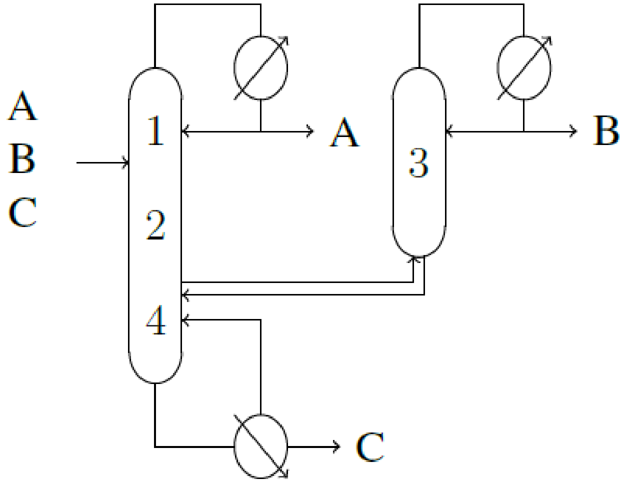
Side-Rectifier Arrangement

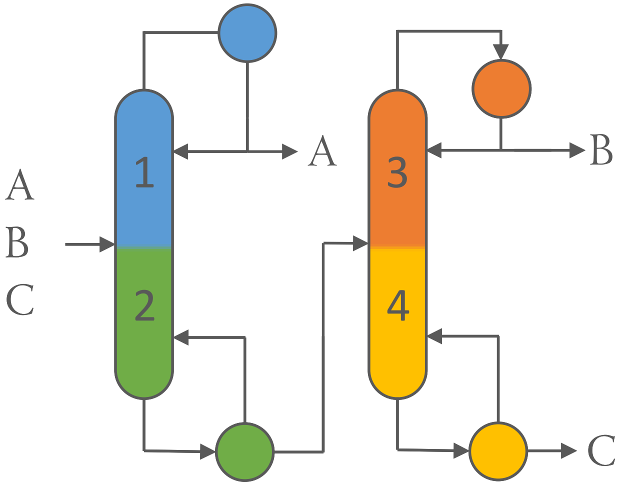


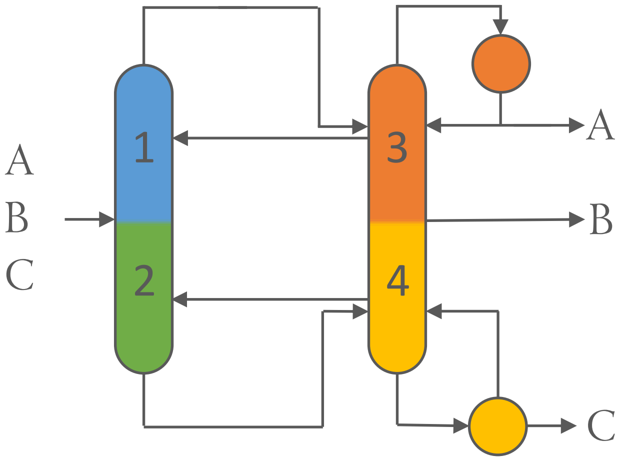
Direct Sequence

Heuristics for Side-Columns

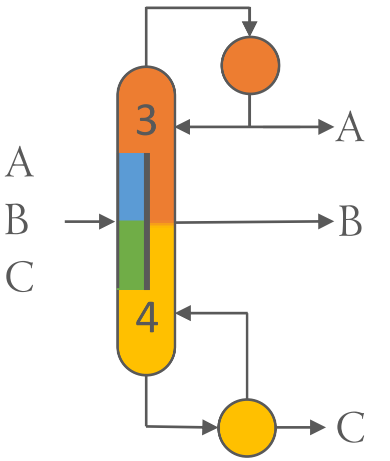
	Side-Stripper	Side-Rectifier
		
Feed Composition	B < 30% C > A	B < 30% A > C

Pre-Fractionation Arrangements



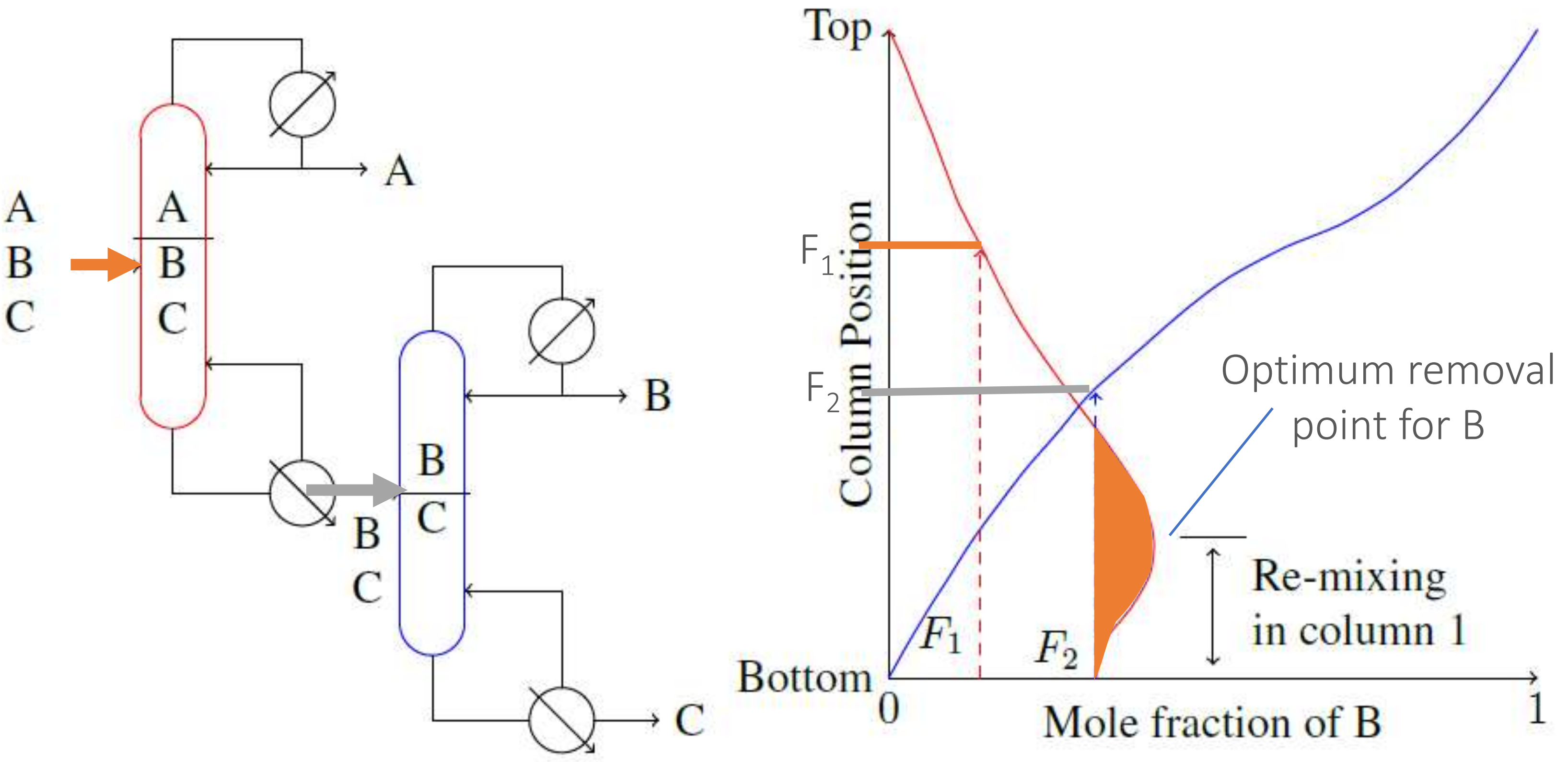


Thermally-coupled pre-fractionator (Petlyuk) column



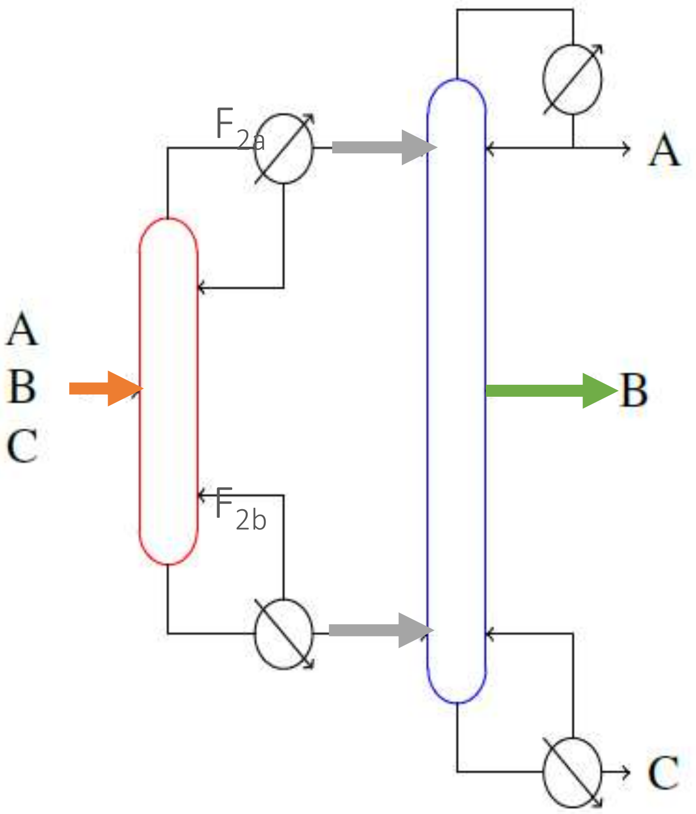
Dividing wall column

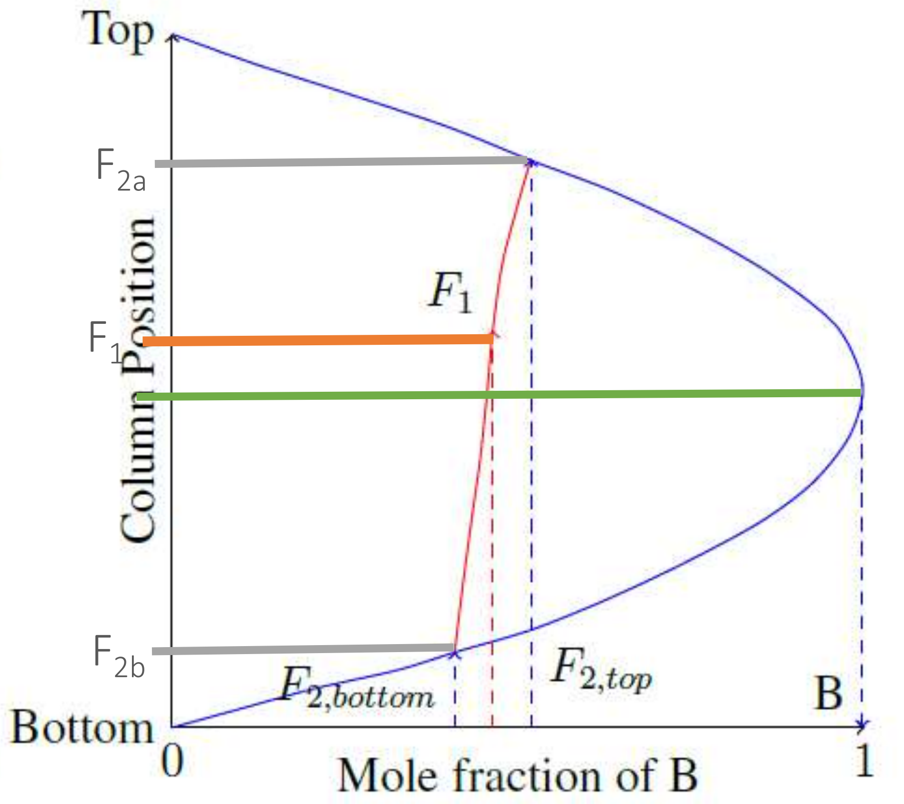
Composition Profiles – Standard Configuration



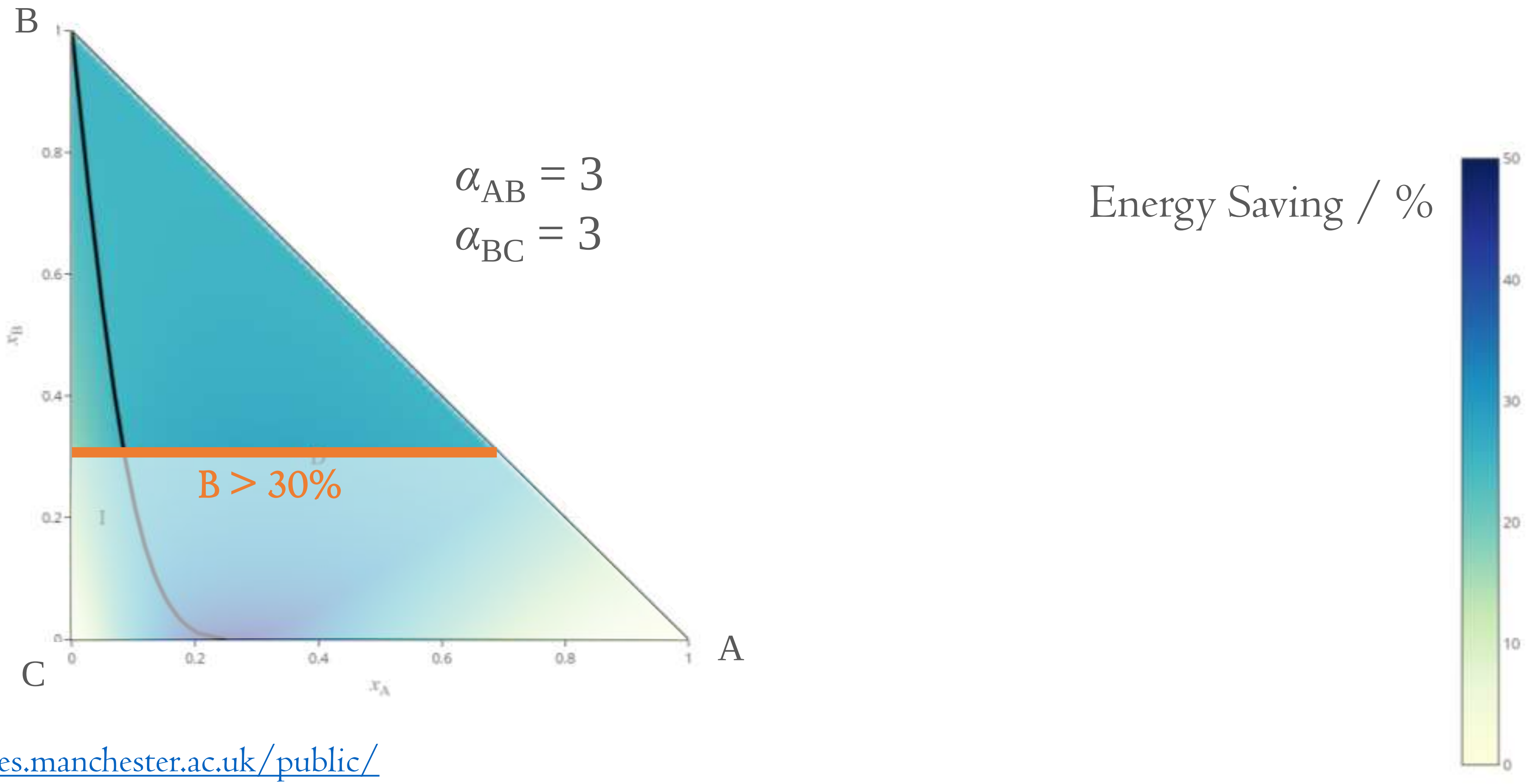
Optimum removal point for B

Composition Profiles – Pre-fractionation





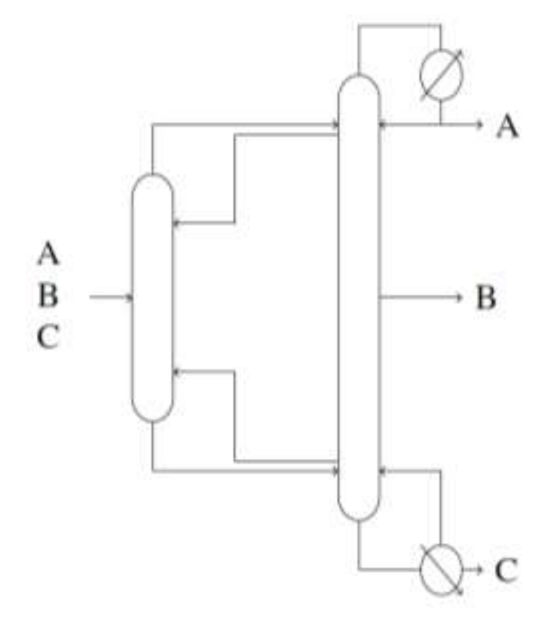
Pre-fractionation Energy Saving



$\alpha_{AB} = 3$
$\alpha_{BC} = 3$

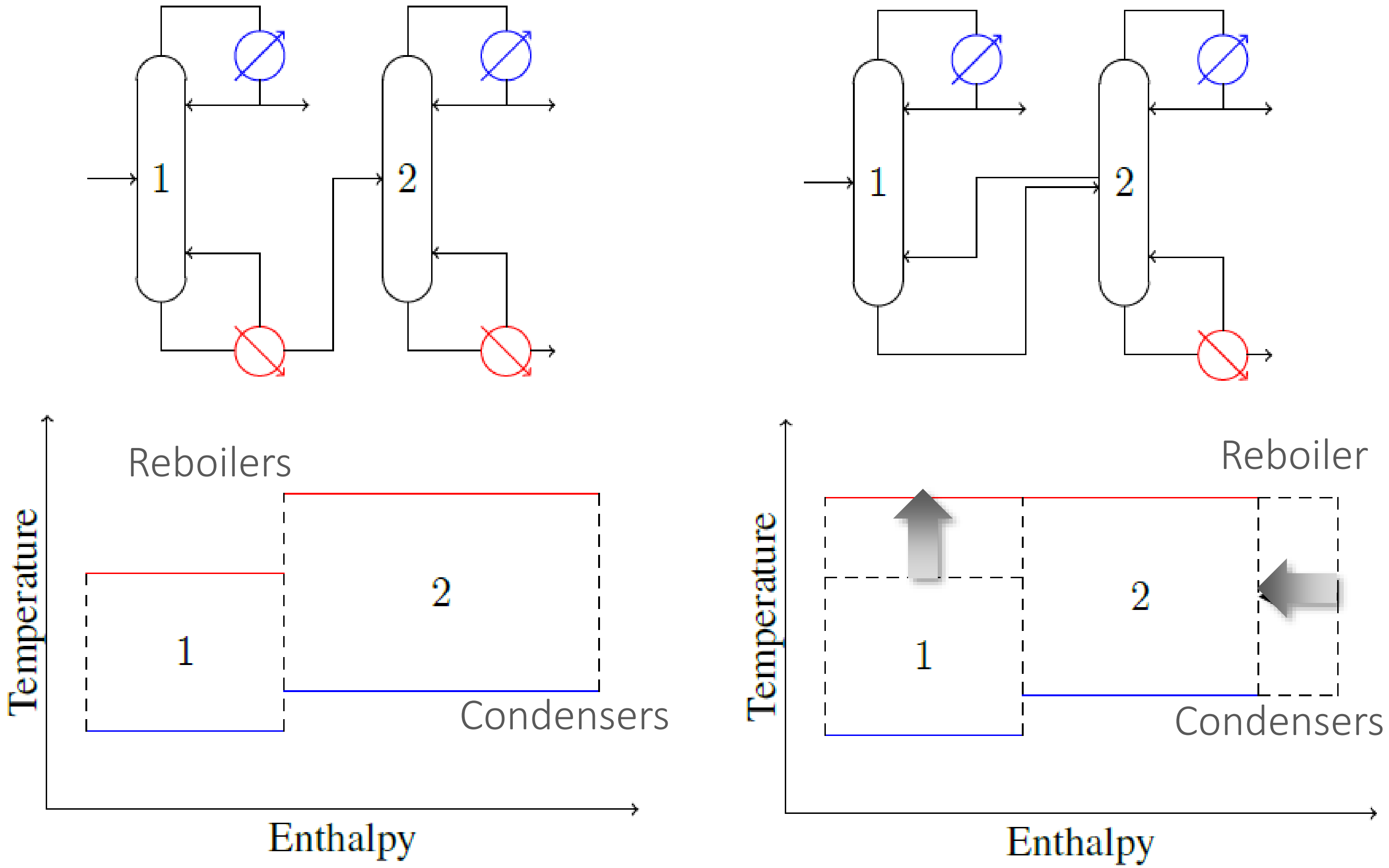
https://www.training.itservices.manchester.ac.uk/public/gced/separations.html?separations/multi_dist/index.html

Heuristics for Pre-fractionators



Feed Composition	B is a large fraction
Relative volatility	$\alpha_{AB} \approx \alpha_{BC}$

Thermally Coupled Utilities



Implementing Thermal Coupling

- Thermal coupling arrangements have great capabilities to reduce energy consumption compared to that from the conventional arrangements.
    - Can be up to 30% reduction in energy
- Thus, it is necessary in the design distillation sequences, to take this into account.

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Short-Cut Design for Complex Columns

Objectives

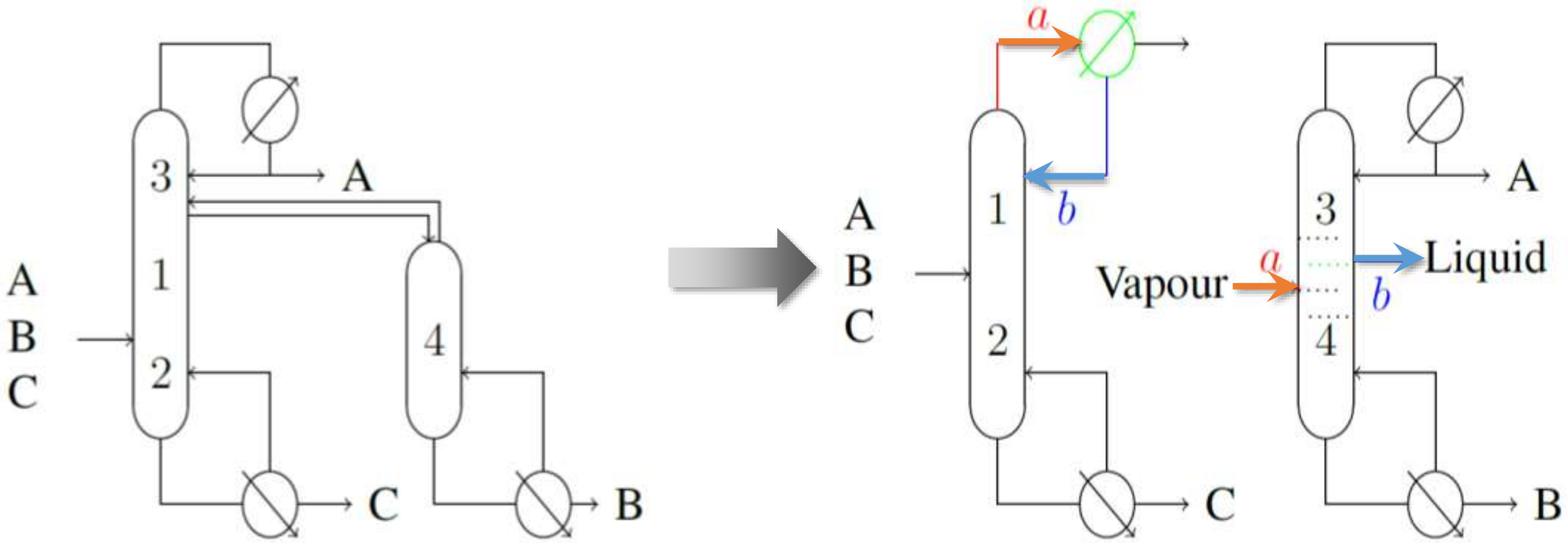
- Decomposition of complex distillation columns
- Modifications to short-cut design for complex columns

Design of Complex Columns

- When it comes to mass and energy balance analysis, it is practically useful to decompose complex columns into simple columns so that the calculations can be carried out relatively easily.

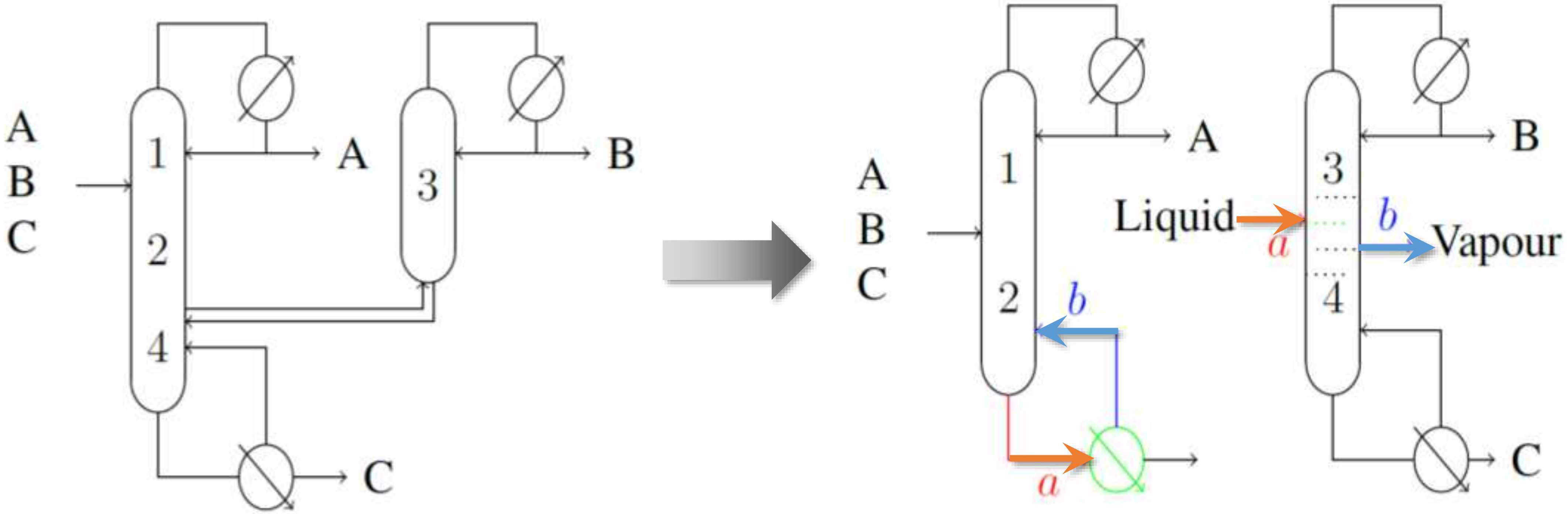
Side-Stripper Decomposition

- The liquid side stream is one stage above feed stage



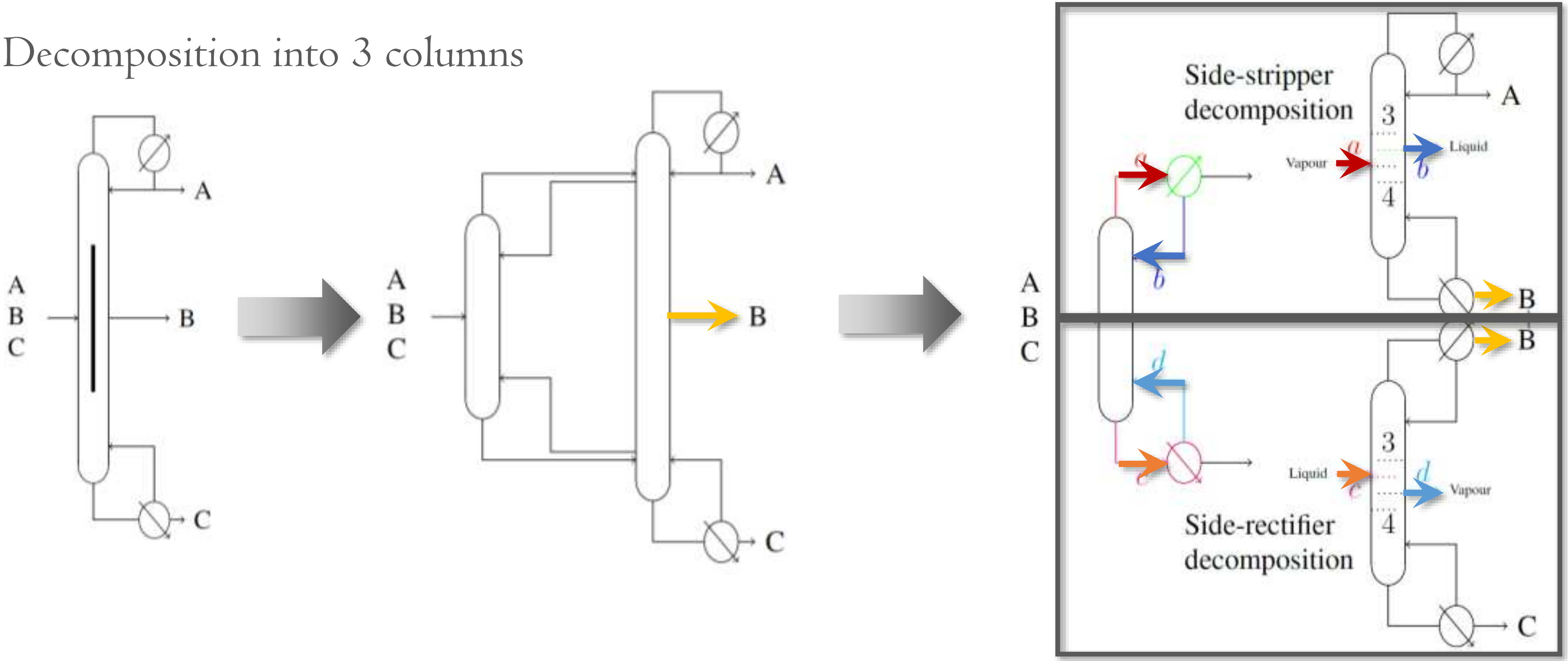
Side-Rectifier Decomposition

- The vapour side stream is one stage below feed stage



Pre-Fractionator Decomposition

- Decomposition into 3 columns

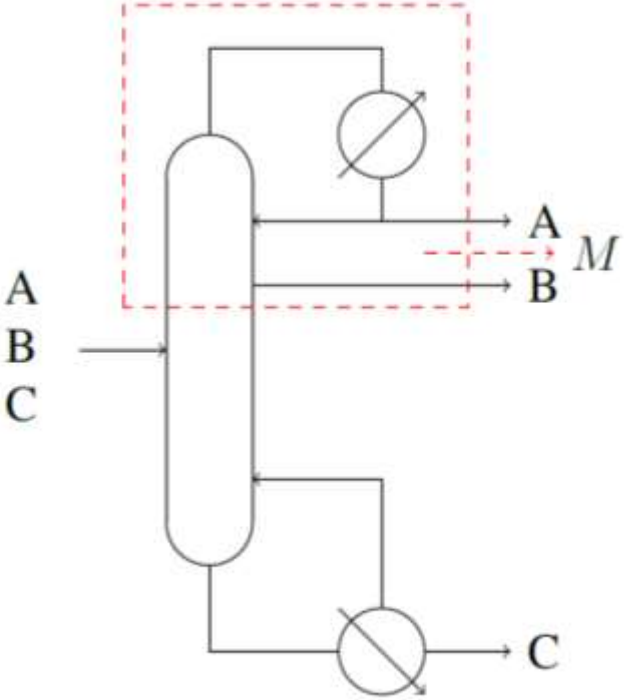


Extension to Underwood Equations

- The Underwood equations only work for simple distillation columns
- So how do we use it for complex distillation columns?
  - We have shown thermally coupled distillation columns can be represented as **simple distillation columns** and **side-stream distillation columns**.
  - Therefore we only need an extension to account for side-stream distillation columns.

Extension to Underwood Equations

- Liquid Side-Stream



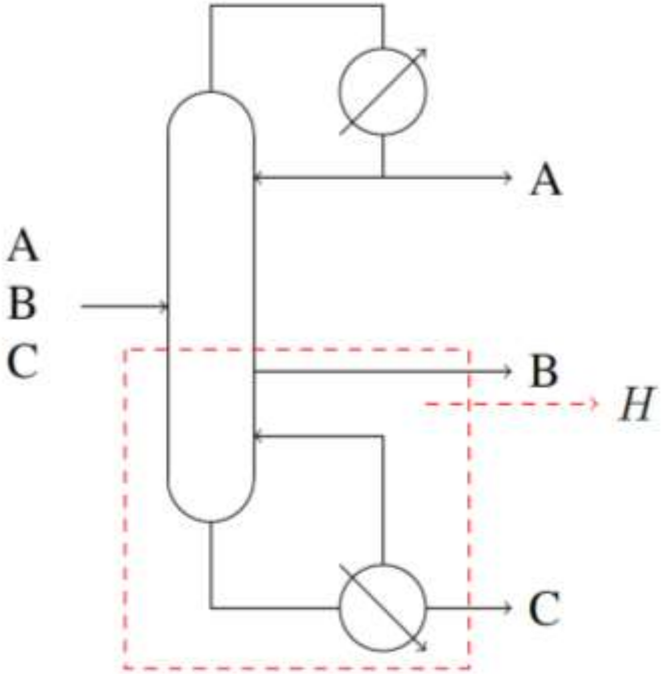
$$1 - q = \sum_{i=1}^{n} \frac{\alpha_{i,r} z_{F,i}}{(\alpha_{i,r} - \phi)}$$

$$V_{min} = M \sum_{i=1}^{n} \frac{\alpha_{i,r} x_{i,M}}{(\alpha_{i,r} - \phi)}$$

$$\alpha_{A,r} > \phi > \alpha_{B,r}$$

Extension to Underwood Equations

- Vapour Side-Stream



$$1 - q = \sum_{i=1}^{n} \frac{\alpha_{i,r} z_{F,i}}{(\alpha_{i,r} - \phi)}$$

$$-V_{min} = H \sum_{i=1}^{n} \frac{\alpha_{i,r} x_{i,H}}{(\alpha_{i,r} - \phi)}$$

$$\alpha_{B,r} > \phi > \alpha_{C,r}$$

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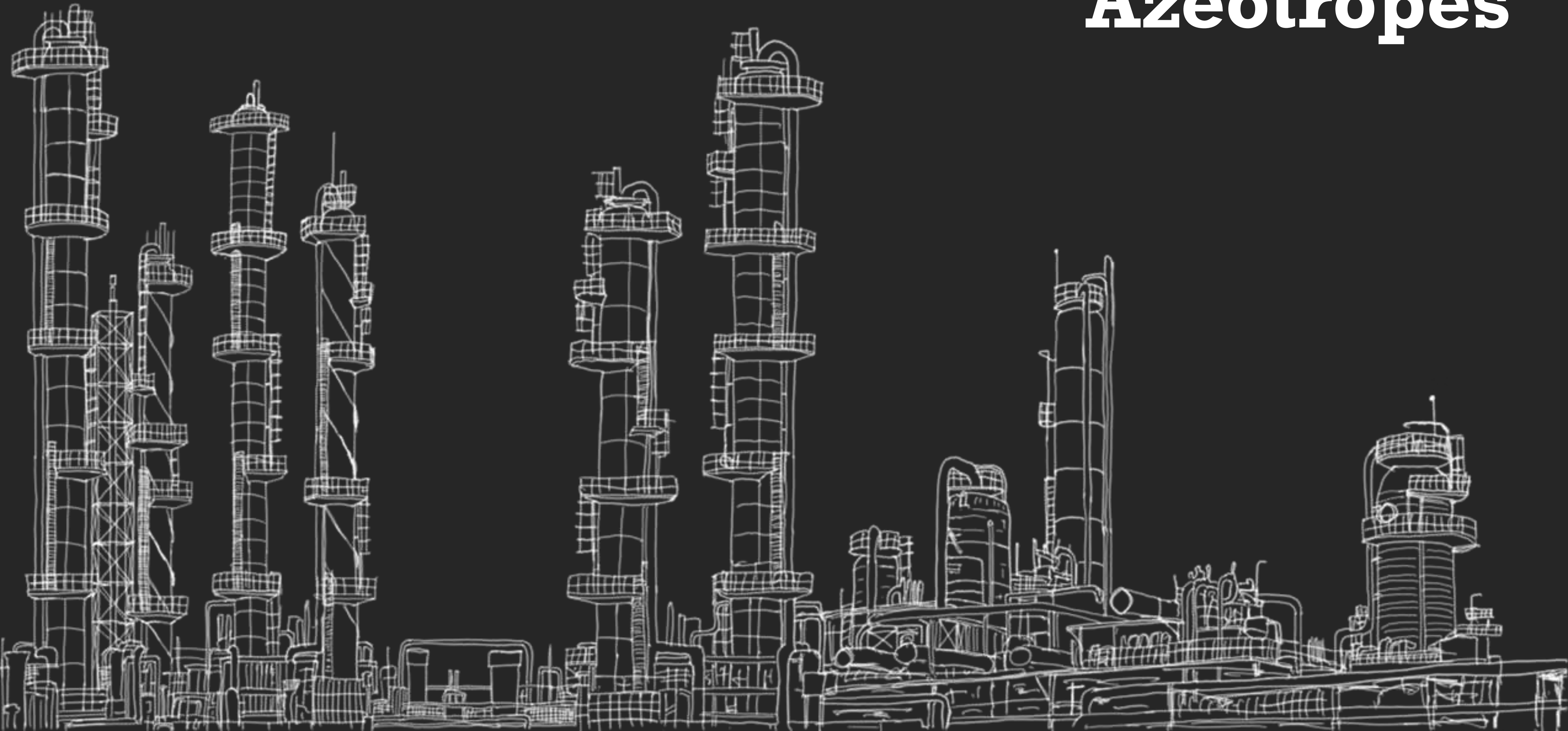
Azeotropes



Objectives

- Reminder of VLE with azeotropes

Reminder – Vapour-Liquid Equilibria

- At equilibrium between vapour and liquid phases,

$$\phi_i^V y_i P = \gamma_i x_i P_i^o$$

- At moderate pressures, $\phi \rightarrow 1$, Raoult's law for non-ideal systems,

$$y_i P = \gamma_i x_i P_i^o$$

- These non-ideal systems commonly manifest themselves in the formation of azeotropes.
- Many close boiling, non-ideal mixtures form azeotropes, particularly those of different chemical types

Azeotropes

- At an azeotrope the vapour and liquid compositions are identical
    - Thus, all K values are 1
    - Dewpoint and bubble point temperatures are equal
- $\alpha_{AB} = 1$ – no separation can take place (via distillation)

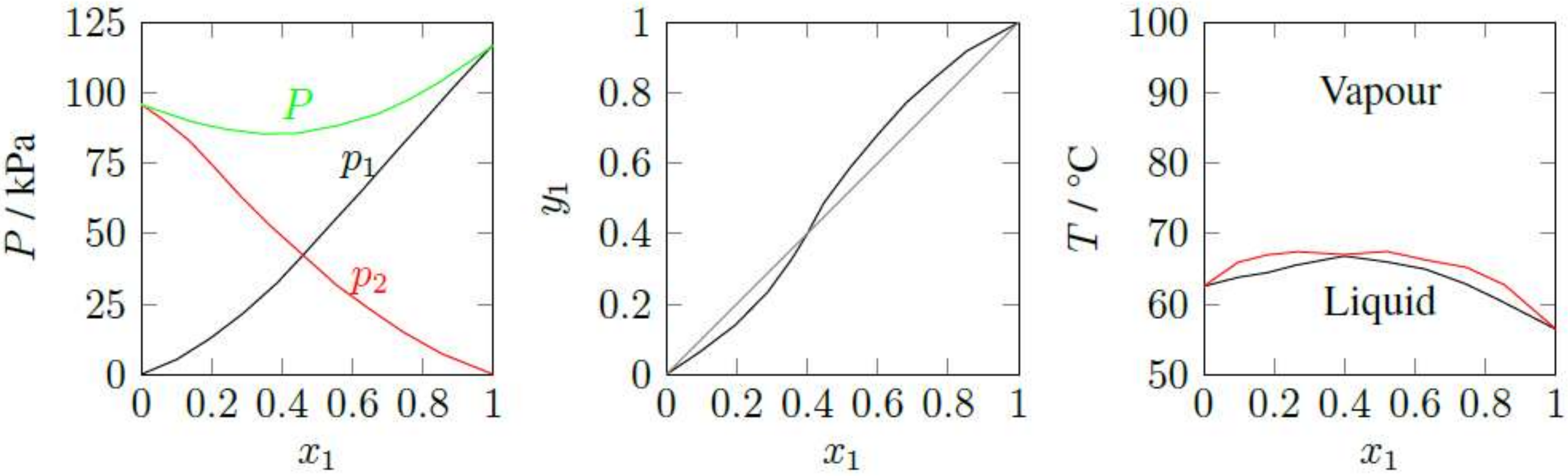
$$y_i P = \gamma_i x_i P_i^\circ$$

- with $x_i = y_i$ at non-trivial solutions of $x_i = 0$ and $x_i = 1$.

Types of Azeotropes

- Maximum boiling ($\gamma < 1$)
  - e.g. acetone-chloroform

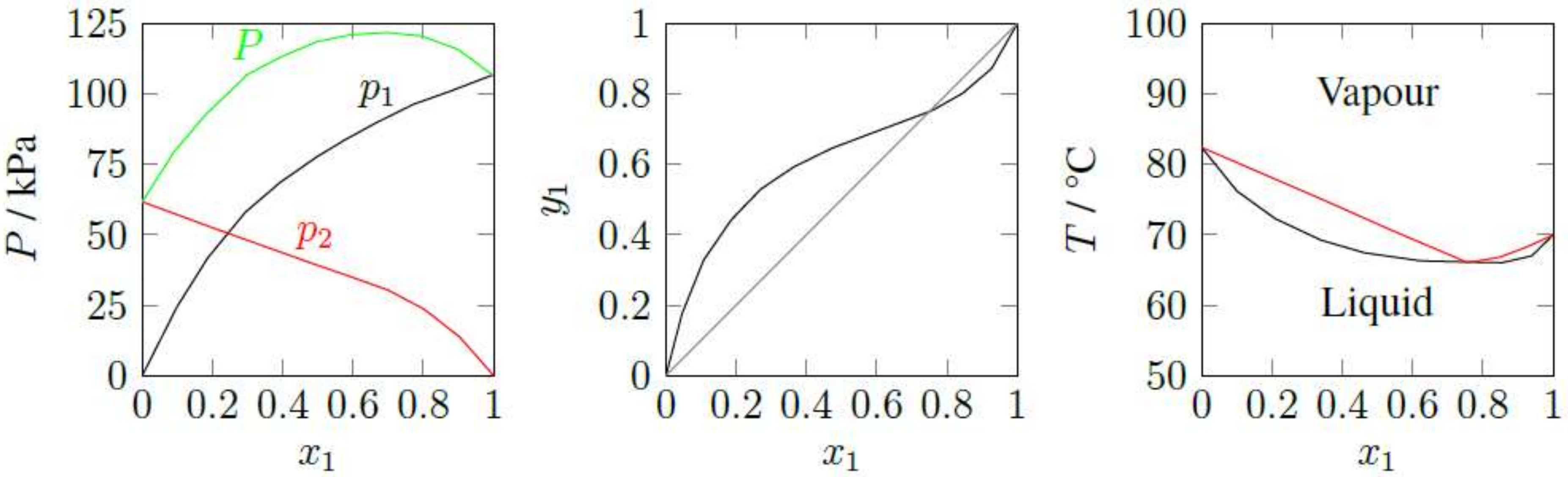
$$y_1 P = \gamma_1 x_1 P_1^0$$
$$y_2 P = \gamma_2 x_2 P_2^0$$
$$\overline{}$$
$$P = \gamma_1 x_1 P_1^0 + \gamma_2 (1 - x_1) P_2^0$$



Types of Azeotropes

- Minimum boiling ($\gamma > 1$)
    - e.g. isopropyl ether-isopropyl alcohol

$$y_1 P = \gamma_1 x_1 P_1^0$$
$$y_2 P = \gamma_2 x_2 P_2^0$$
$$P = \gamma_1 x_1 P_1^0 + \gamma_2 (1 - x_1) P_2^0$$

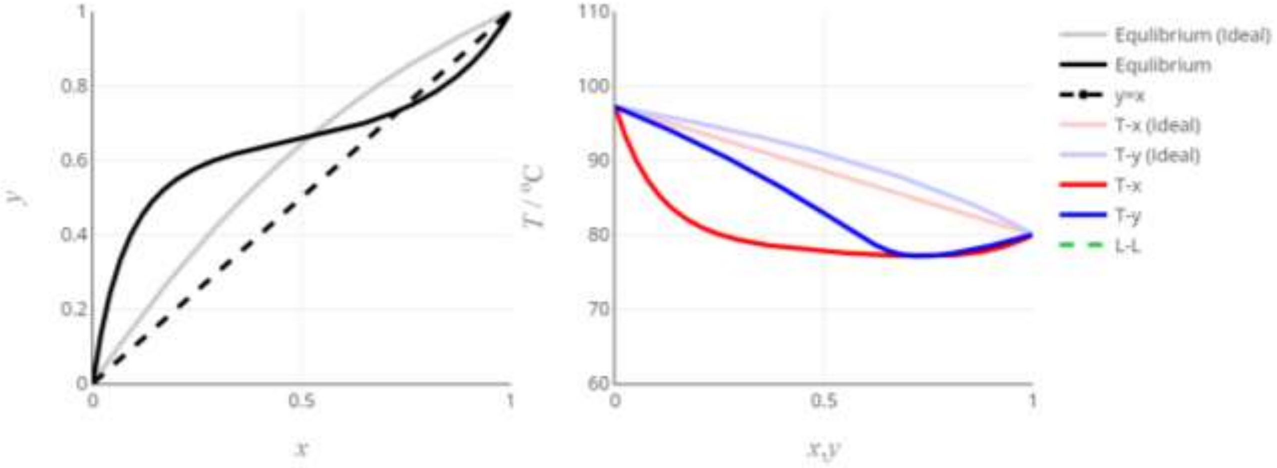


Types of Azeotropes

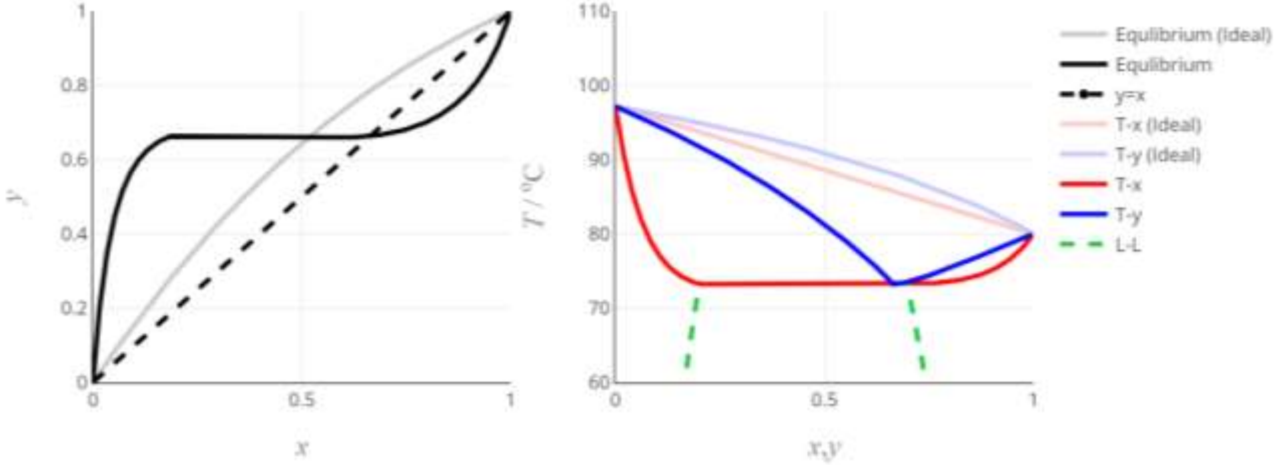
- These are **homogeneous** azeotropes
  - 1 vapour phase and 1 liquid phase in equilibrium
- For a minimum-boiling azeotrope with large deviation from Raoult's law ($\gamma >> 1$), phase splitting may occur and a minimum-boiling **heterogeneous** azeotrope forms
  - 1 vapour phase and 2 liquid phases in equilibrium

Types of Azeotropes

Homogeneous azeotrope



Heterogeneous azeotrope

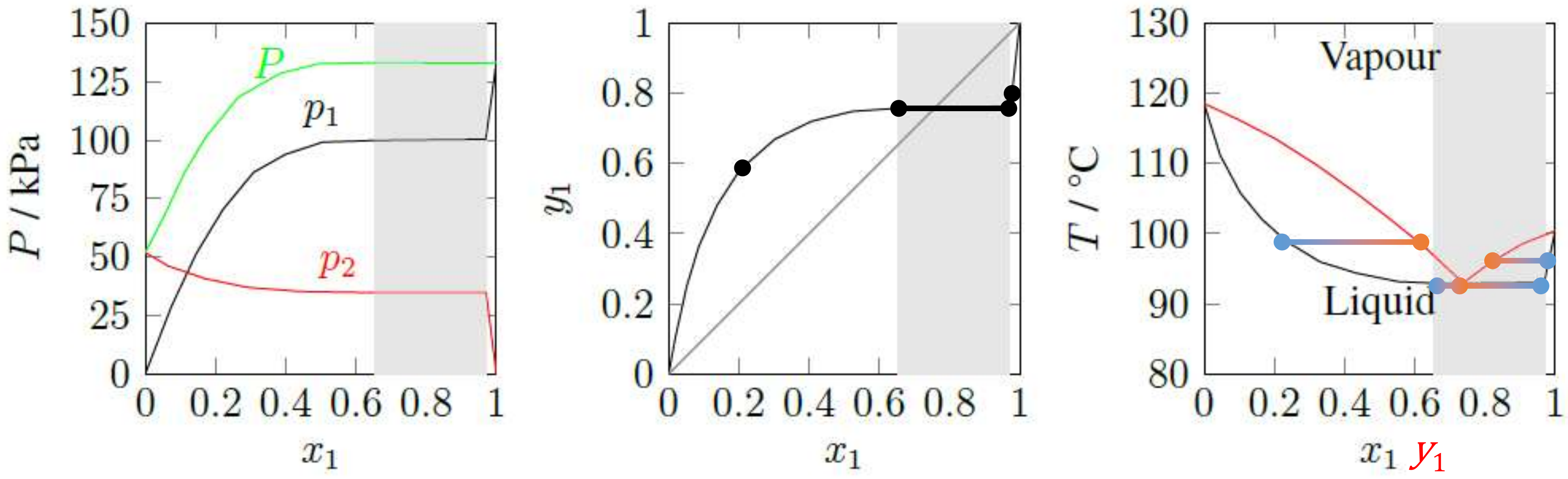


https://www.training.itservices.manchester.ac.uk/public/gced/separations.html?separations/VLE/index.html

Types of Azeotropes

- Heterogeneous azeotropes must be minimum-boiling
  - $\gamma >> 1$ to get 2 liquid phases
  - e.g. water-*n*-butanol

$$y_1 P = \gamma_1 x_1 P_1^0$$
$$y_2 P = \gamma_2 x_2 P_2^0$$
$$\overline{\qquad\qquad\qquad\qquad\qquad\qquad}$$
$$P = \gamma_1 x_1 P_1^0 + \gamma_2 (1 - x_1) P_2^0$$



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Feasible Distillation Regions

Objectives

- Introduction to ternary diagrams for distillation
- Introduction to residue curves
- Using residue curves for distillation design

Ternary Mixtures

- In the distillation of a ternary mixture, possible equilibrium compositions do not lie uniquely on a single, isobaric equilibrium curve because the Gibbs phase rule gives an additional degree of freedom.

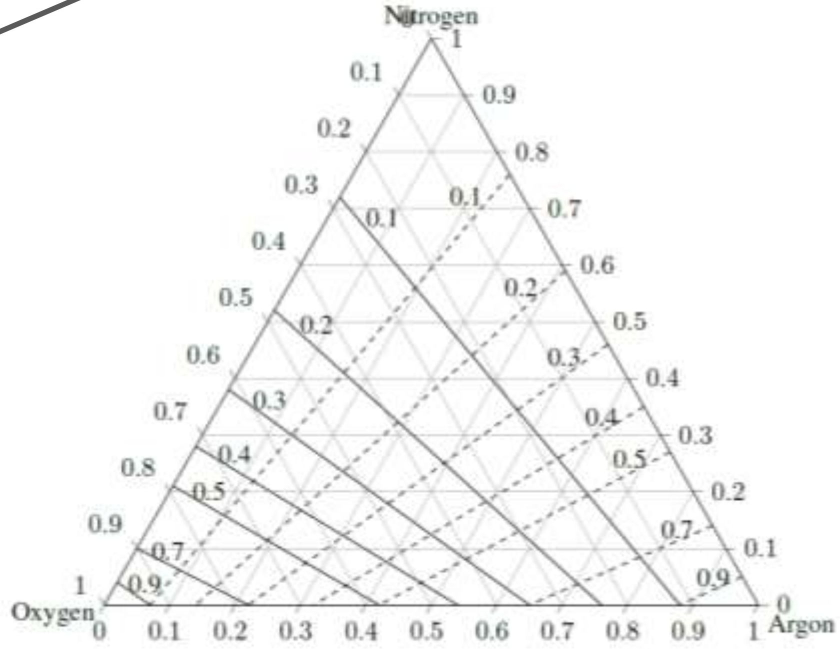
$$N_{freedom} = N_{components} - N_{phases} + 2$$

- The other compositions are determined only if the temperature, pressure, and composition of one component in one phase are fixed.

Ternary Phase Equilibrium

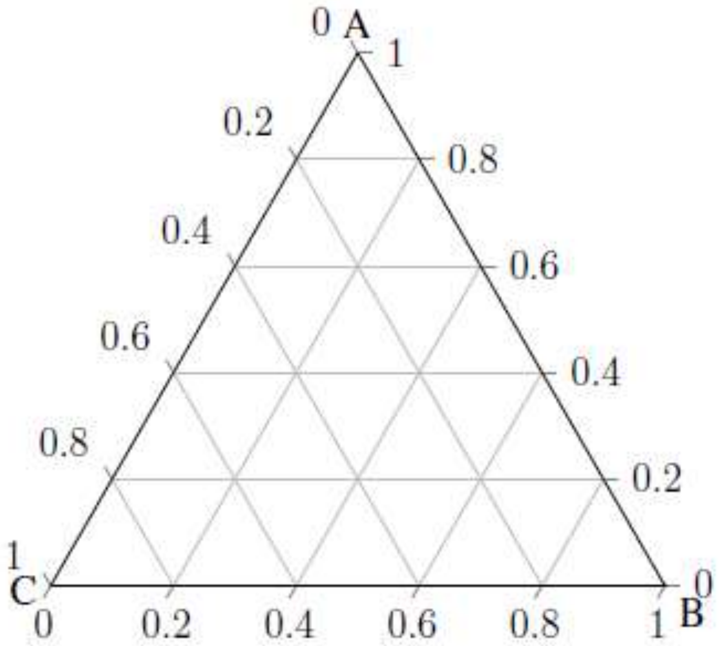
- The composition of a ternary mixture can be represented o
  triangular diagram
- Vapour-liquid phase equilibria at a fix
  - triangular grid represent the li
  - lines of constant e … our composition

Rarely Used

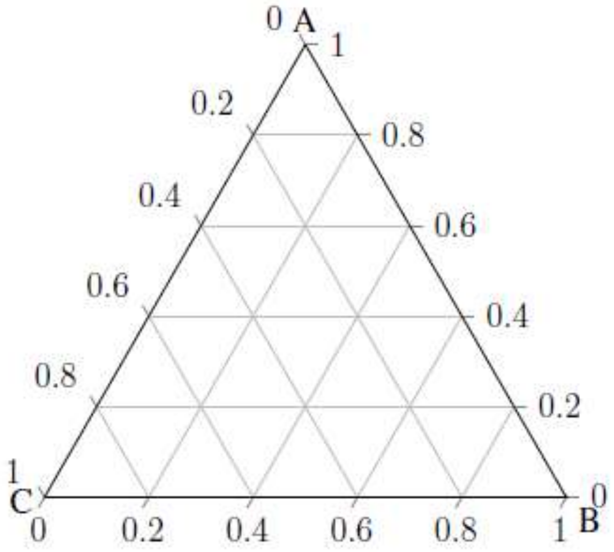


Ternary Phase Diagrams

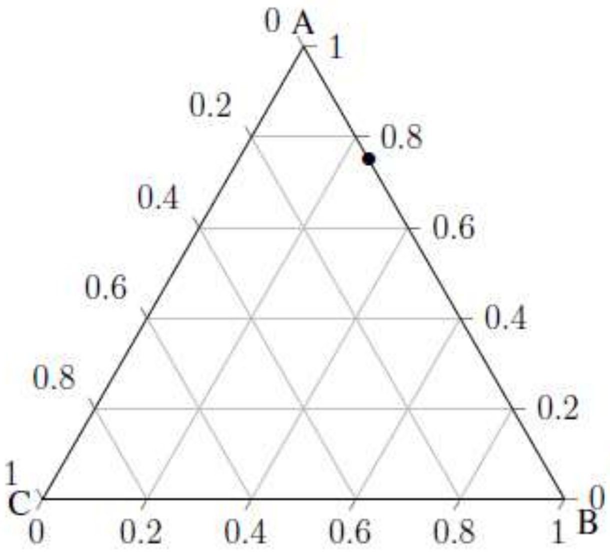
- It is more useful, when developing a feasible-separation process for a ternary mixture, to plot only equilibrium-liquid-phase compositions on the triangular diagram.



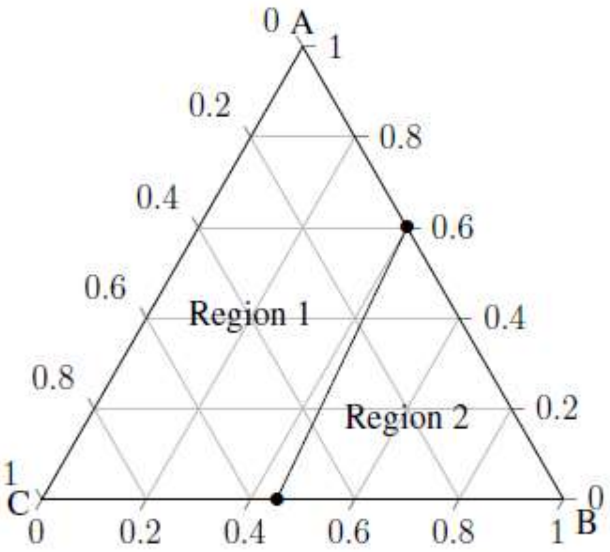
Ternary Systems



Zeotropic system



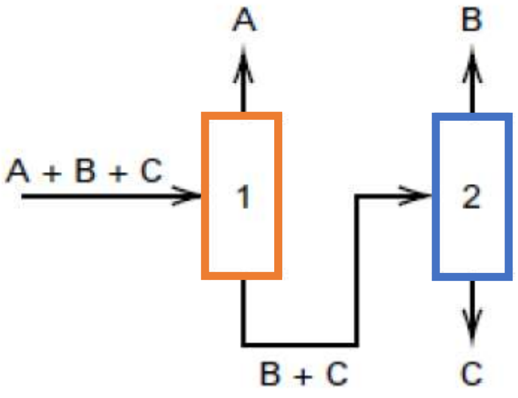
Single
azeotropic system

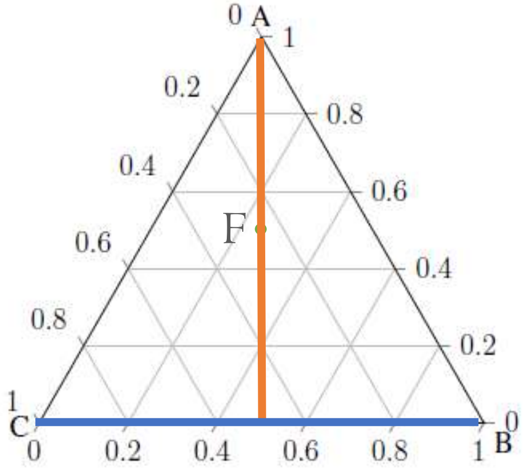


Two azeotropic system
with distillation boundary

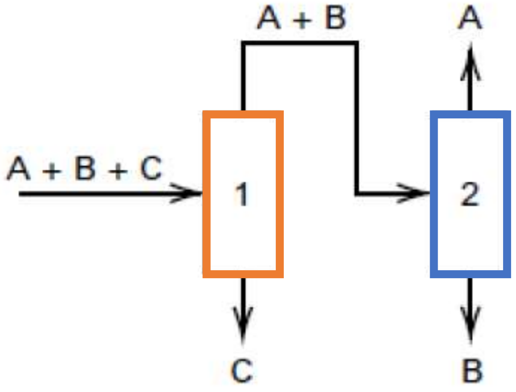
Distillation Options

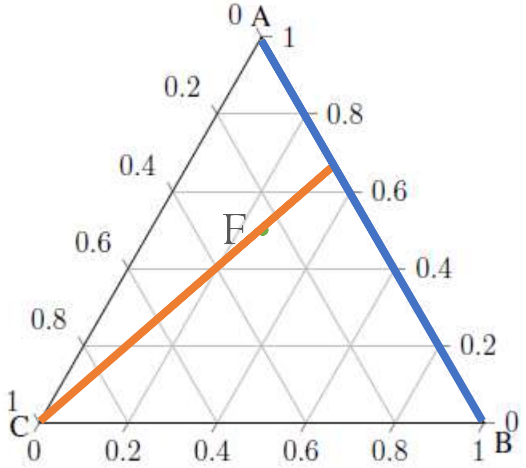
Direct Sequence





Indirect Sequence

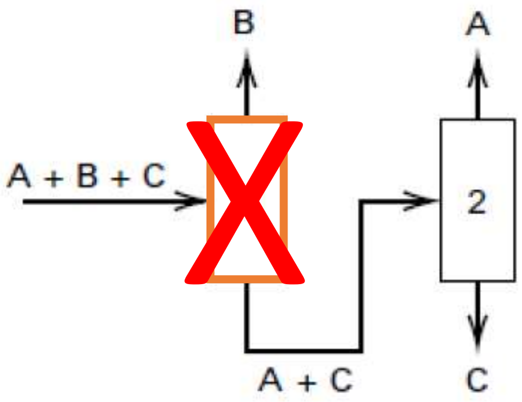


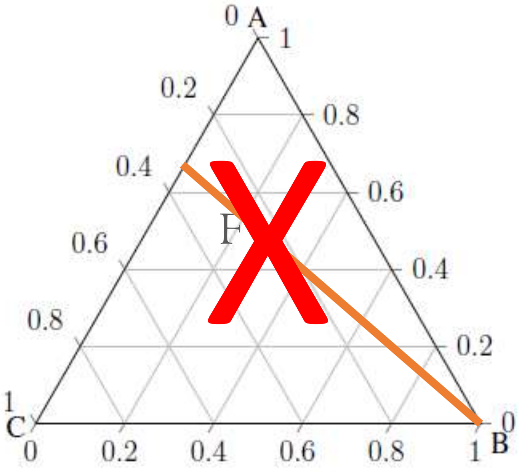


A low boiler
B intermediate boiler
C high boiler

Distillation Options

- Not possible to get pure B in the first column as it is the intermediate boiler



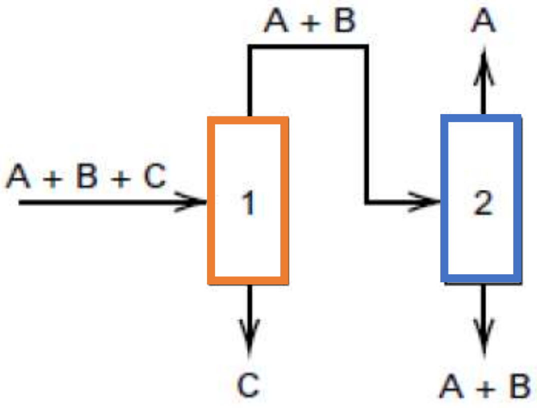


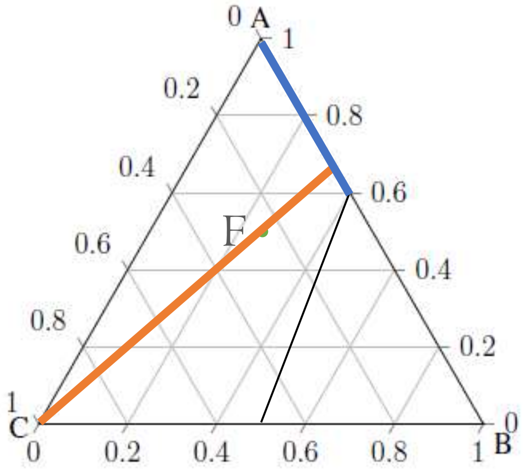
A low boiler
B intermediate boiler
C high boiler

- So how can we know this?

Distillation Options

- Can't cross the distillation boundary
- Can't get all three pure components with a simple distillation sequence





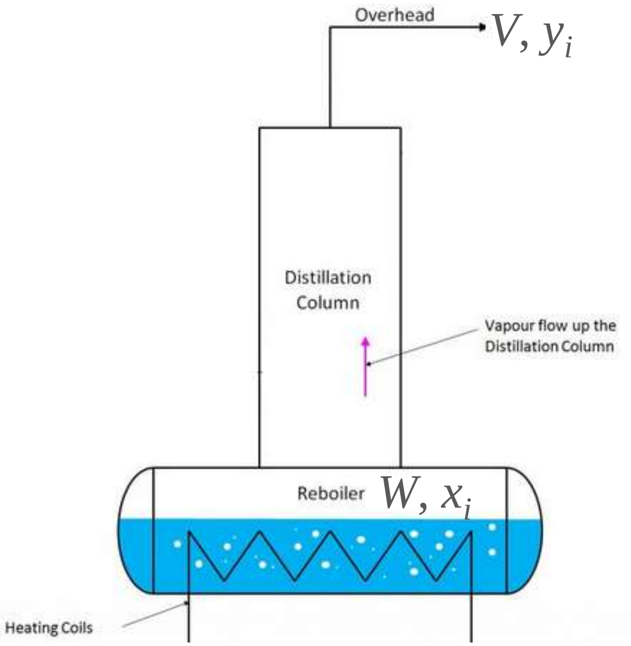
A low boiler
B intermediate boiler
C high boiler

- So how can we know this?

Residue-Curve Maps

- A residue curve describes the change of the composition of the liquid phase of a chemical mixture during continuous evaporation at the condition of vapour-liquid equilibrium.

- Consider the simple batch distillation (no trays, packing, or reflux).



Residue-Curve Maps

- Component mass balance:

$$\frac{d(Wx_i)}{dt} = -Vy_i$$

- Expanding,

$$x_i\frac{dW}{dt} + W\frac{dx_i}{dt} = -Vy_i$$

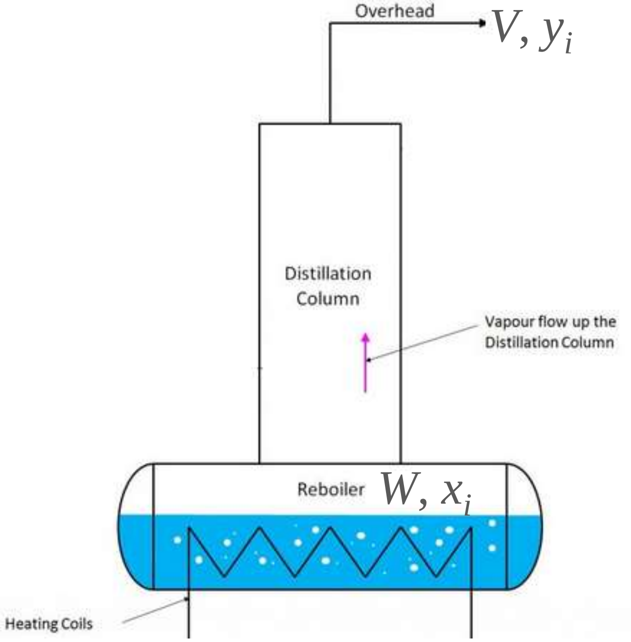
$$W\frac{dx_i}{dt} = -x_i\frac{dW}{dt} - Vy_i$$

- Total mass balance:

$$\frac{dW}{dt} = -V$$

- Thus,

$$\frac{dx_i}{dt} = (y_i - x_i)\frac{1}{W}\frac{dW}{dt}$$



Residue-Curve Maps

$$\frac{dx_i}{dt} = (y_i - x_i)\frac{1}{W}\frac{dW}{dt}$$

- We can combine *W* and *t* into as single variable that can be considered as a dimensionless time, $\xi(t)$

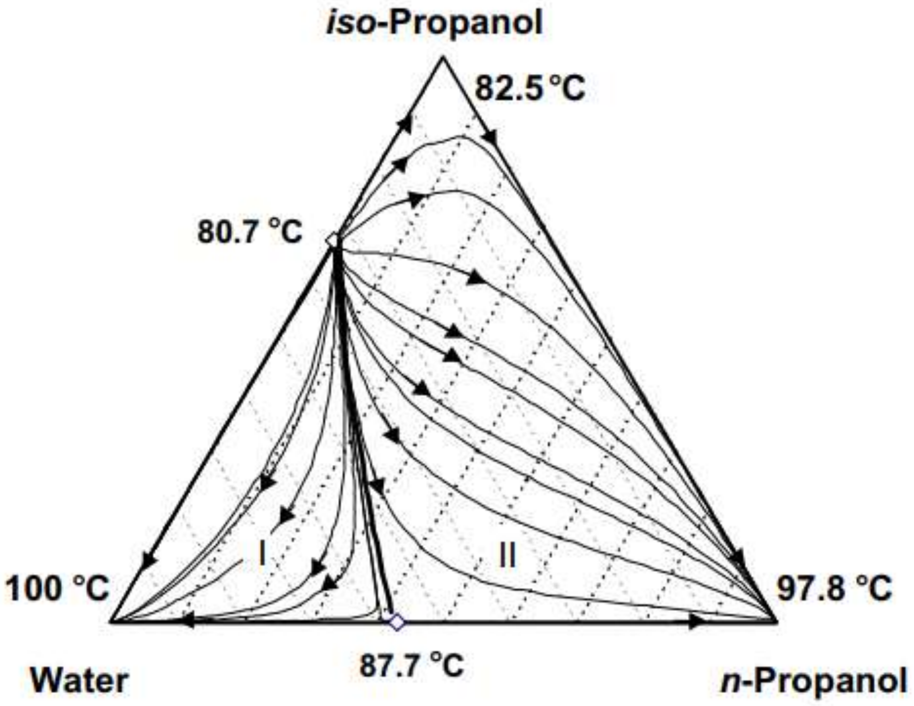
$$\frac{dx_i}{d\xi} = (x_i - y_i), \qquad i = 1, 2 \qquad\qquad \sum_{i=1}^{3} x_i = 1$$

$$y_i = K_i x_i, \qquad i = 1, 2, 3 \qquad\qquad \sum_{i=1}^{3} K_i x_i = 1$$

- Each different feed point will produce a different curve

Residue-Curve Maps

- If we repeat for many feed positions, we can build up the map,

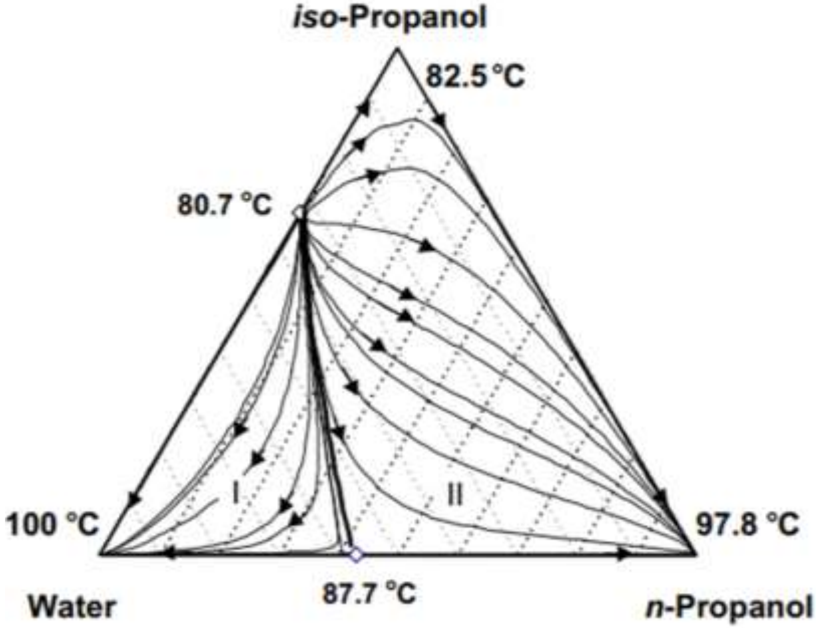


- Arrows point in direction of increasing ξ (increasing T)

- This will take a long time …
- But programs like ASPEN can make these for you.

Feasible Product-Compositions

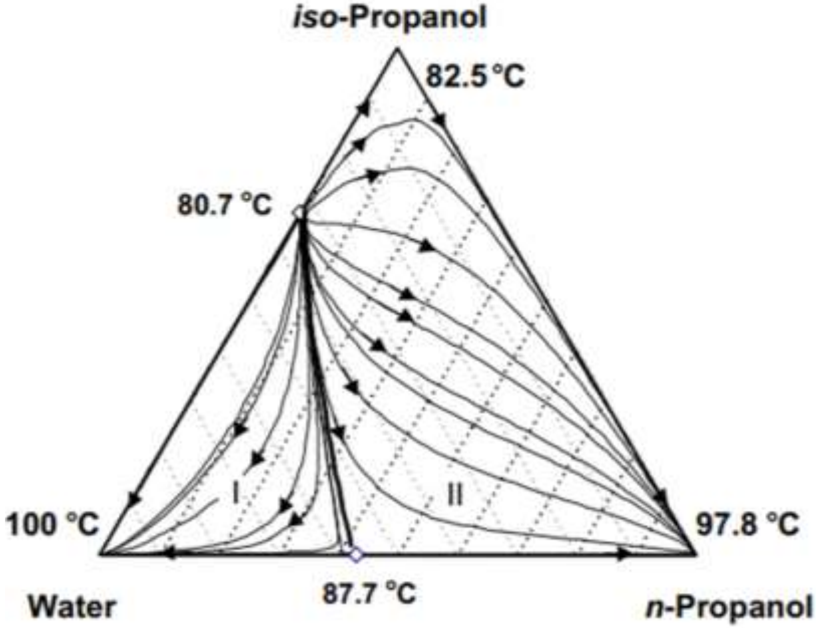
- The feasible-distillation regions for azeotrope-forming ternary mixtures are not obvious
- Residue curve maps can be used to make preliminary estimates of regions of feasible-product compositions
- These regions are determined by using a column material-balance line



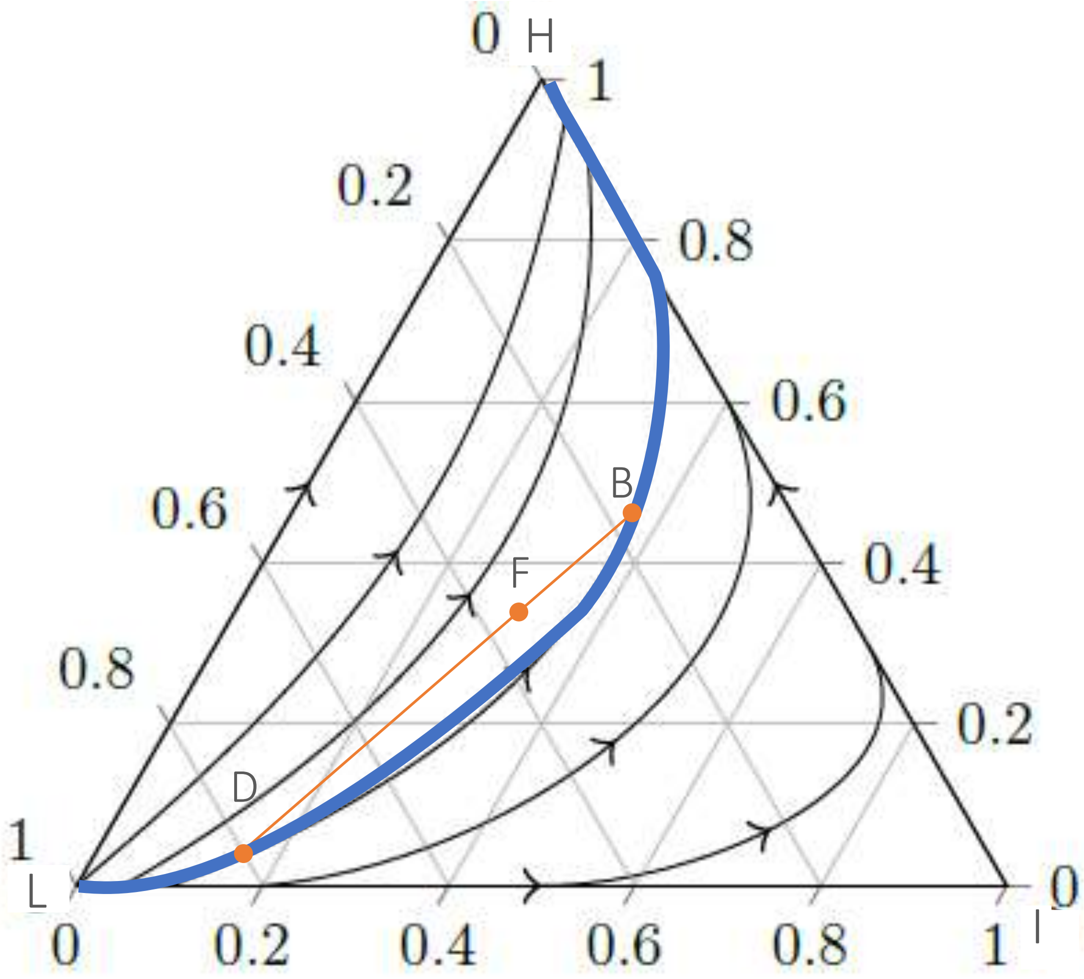
Feasible Product-Compositions

- If a straight line is drawn that connects the distillate and bottoms compositions, that line must pass through the feed composition at some intermediate point to satisfy a **material balance**

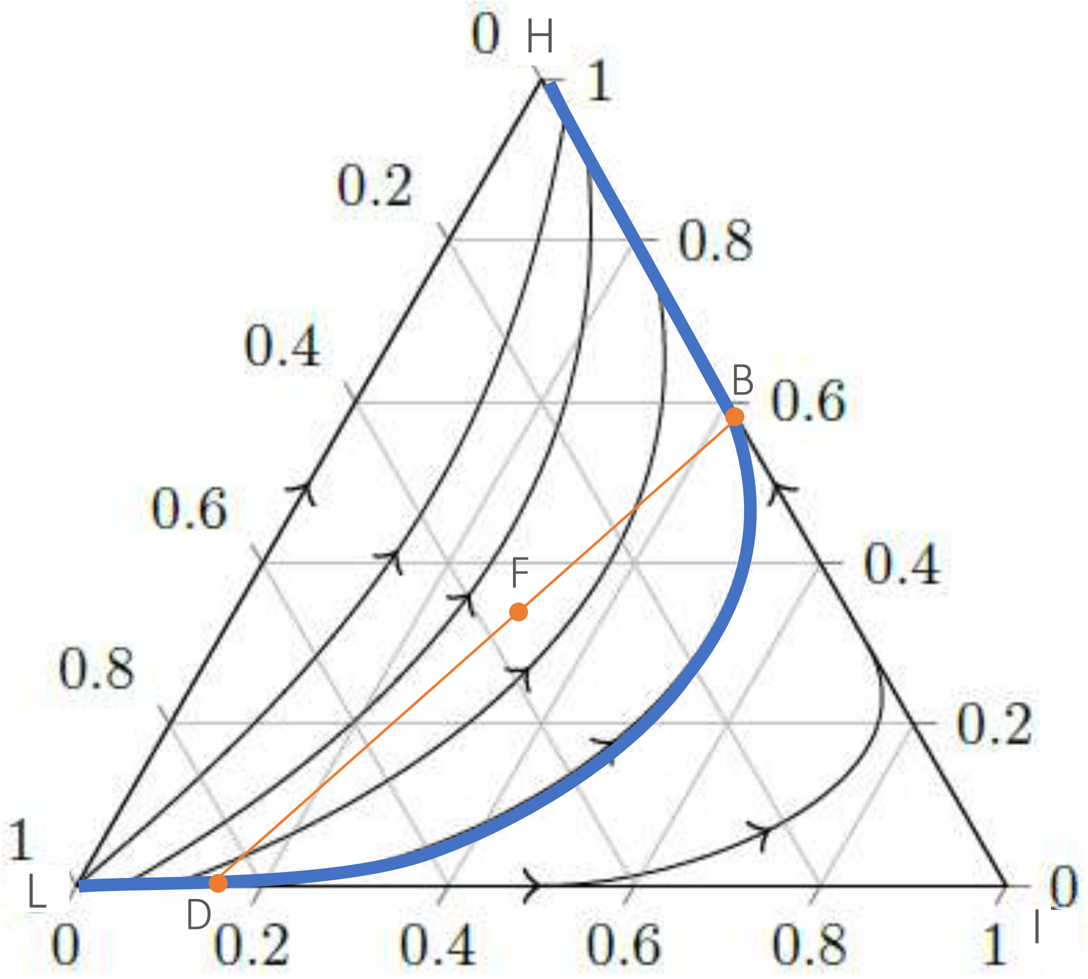
- The distillate and bottoms points (the end of the line) must also lie on the same residue curve to satisfy the **column stage equilibriums**



Feasible Product-Compositions

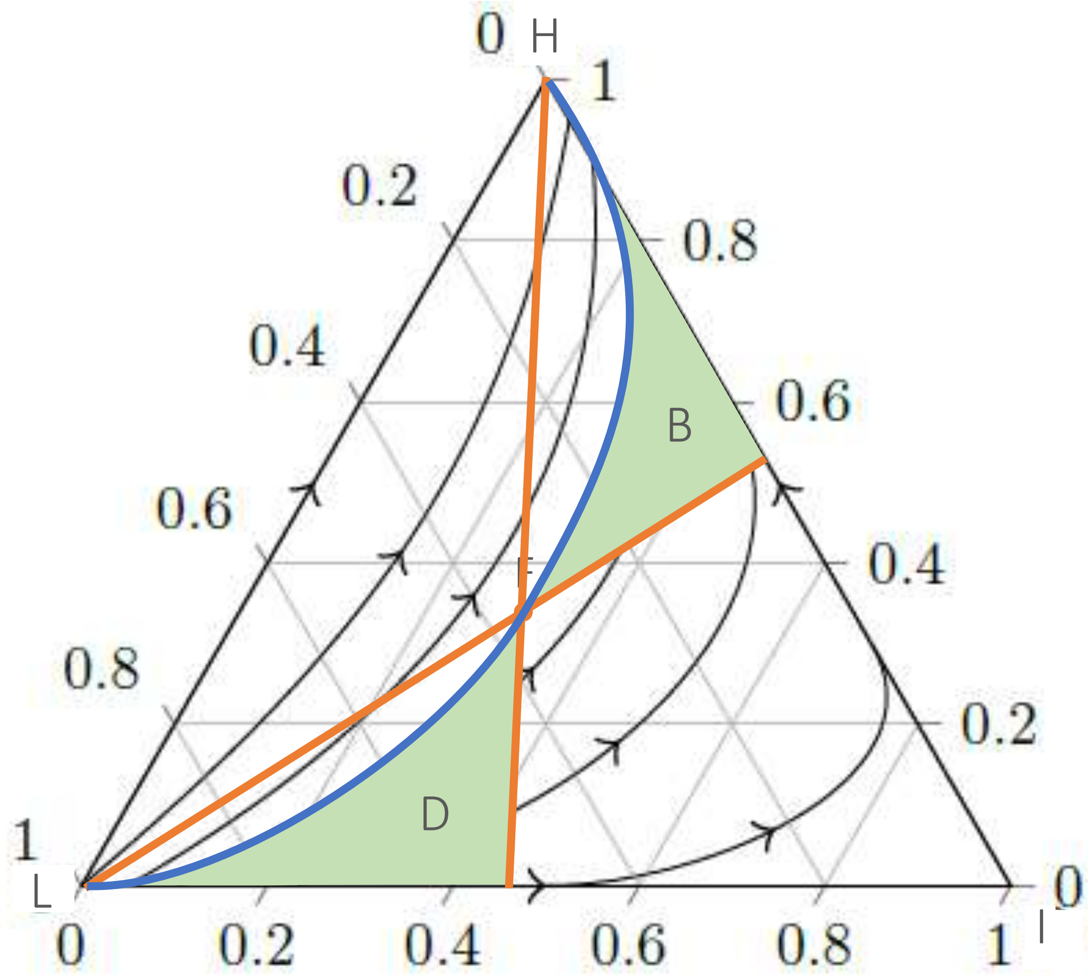


Feasible Product-Compositions



Feasible Product-Compositions

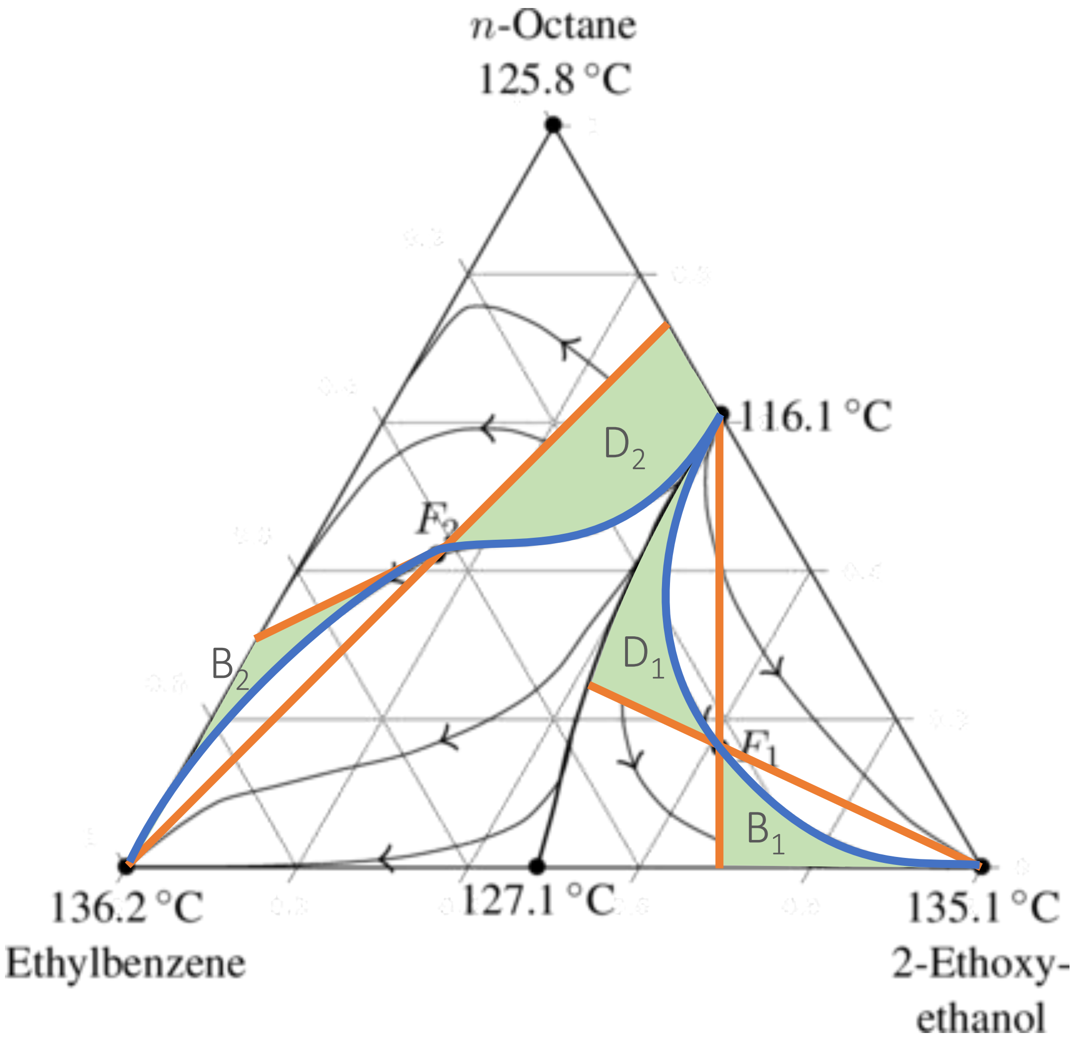
- We can repeat this for all possible combinations to find the range of feasible separations for a given feed



Feasible Product-Compositions

- The green areas are the only feasible product regions for that feed.
- Often called “bow-tie” regions

Feasible Product-Compositions



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Short-Cut Design for Azeotropes

Objectives

- Modifications to short-cut design for azeotropes

Short-cut Equations for Azeotropic Systems

- Short-cut calculations can be performed for non-ideal systems.

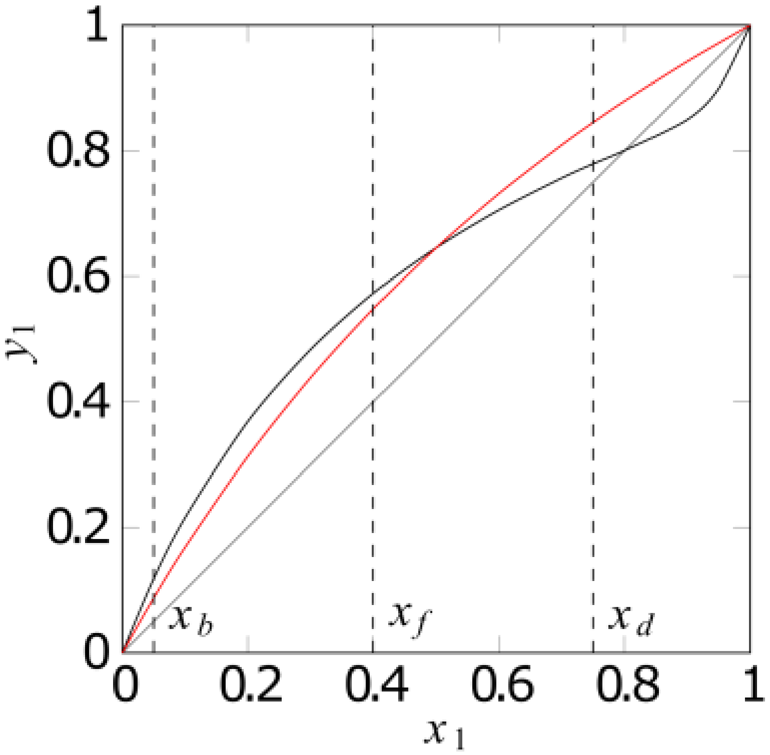
- However, key assumptions for short-cut is an approximately constant relative volatility

- When a system has an azeotrope it is clear that the relative volatility is not constant

Short-cut Equations for Azeotropic Systems

- At $x_1 = 0.05$ $\alpha = 2.55$
- At $x_1 = 0.95$ $\alpha = 0.47$

- Taking an average relative volatility from the feed, distillate, and bottom product composition gives a value of $\alpha = 1.82$



Short-cut Equations for Azeotropic Systems

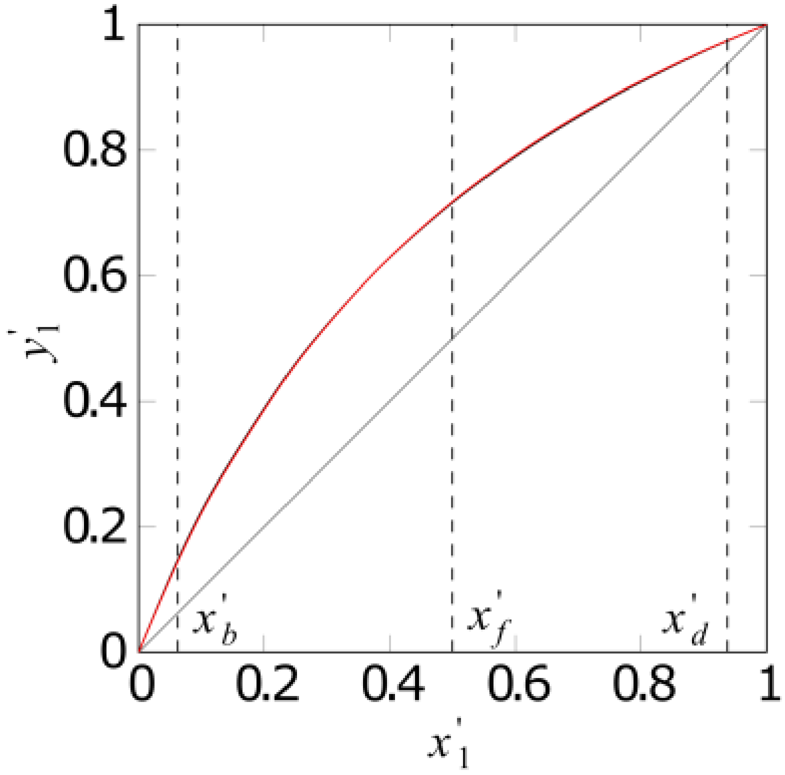
- The short-cut method can still be used for systems of this type by transforming the region of interest,

$$x'_i = \frac{x_i}{a}, \qquad y'_i = \frac{y_i}{a}$$

- For this system $a = 0.8$

$$\alpha' = \frac{y'_i\,(1 - x'_i)}{x'_i\,(1 - y'_i)}$$

- The average relatively volatility of the transformed system is then $\alpha' = 2.54$

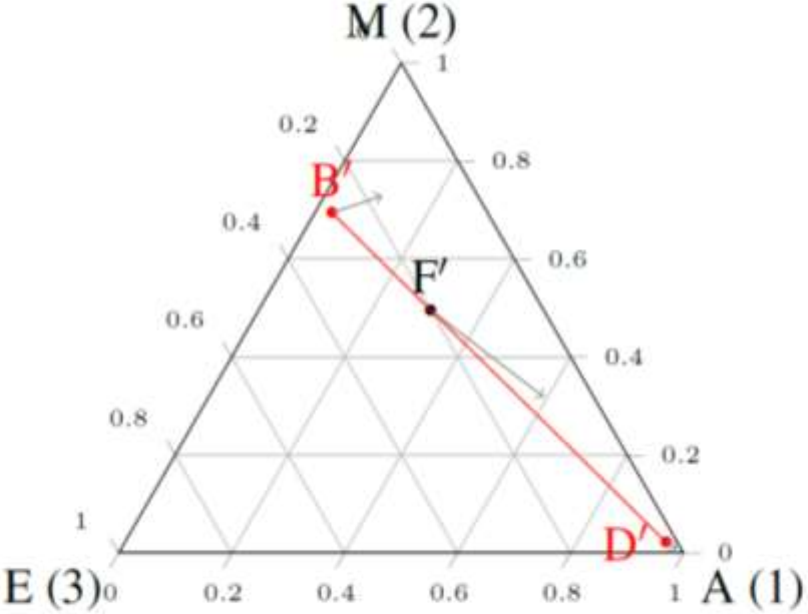


Short-cut Equations for Azeotropic Systems

- This method has been extended to more components, by using a matrix transformation

$$\bar{X} = \bar{M}\bar{X}'$$

- M is the singular point matrix of the 3 corners of the region X
- However, the more non-ideal components the more inaccurate the method will become as the relative volatilities will vary across the column



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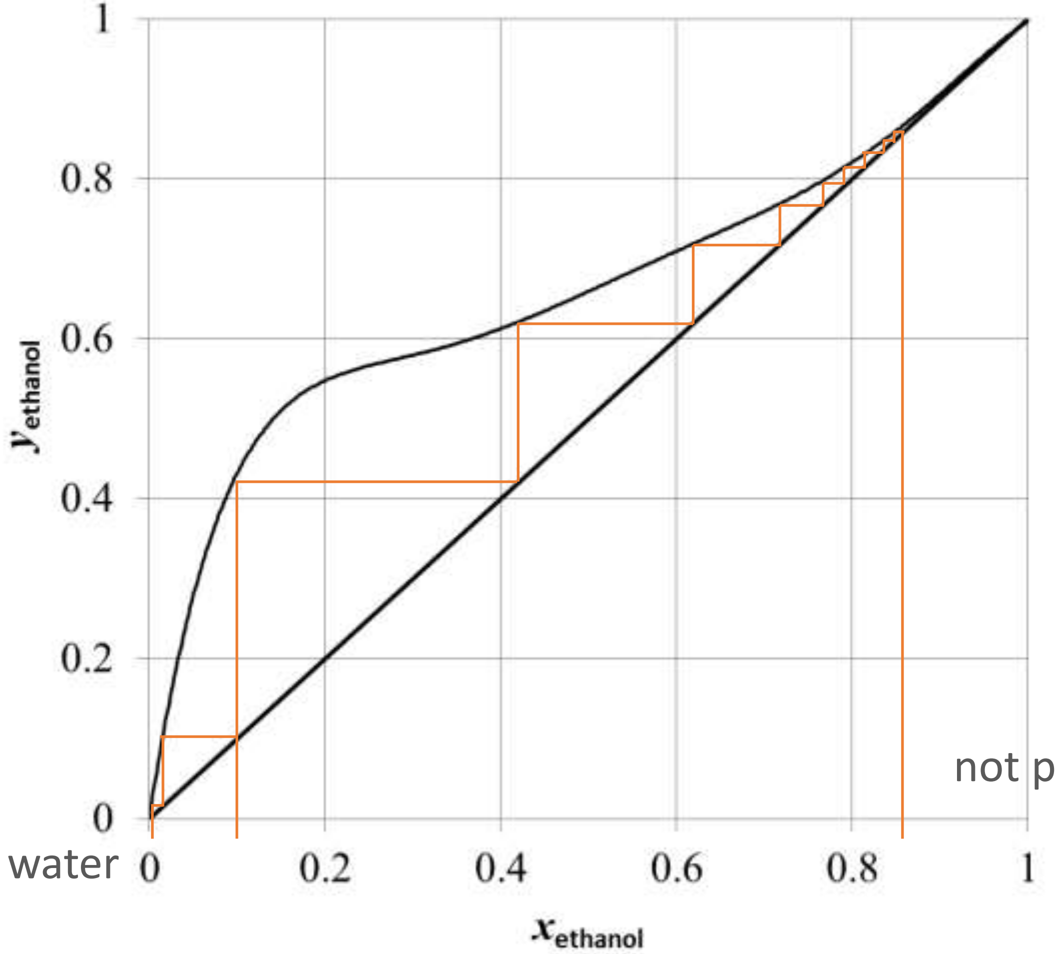
Distillation Sequences for Azeotropes

Objectives

- Distillation sequences for separating azeotropic systems
  - Pressure-swing distillation
  - Extractive distillation
  - Homogeneous Azeotropic Distillation
  - Heterogeneous Azeotropic Distillation
  - Reactive Distillation

Distillation with Azeotropes



“pure” water

not pure ethanol

Azeotropes

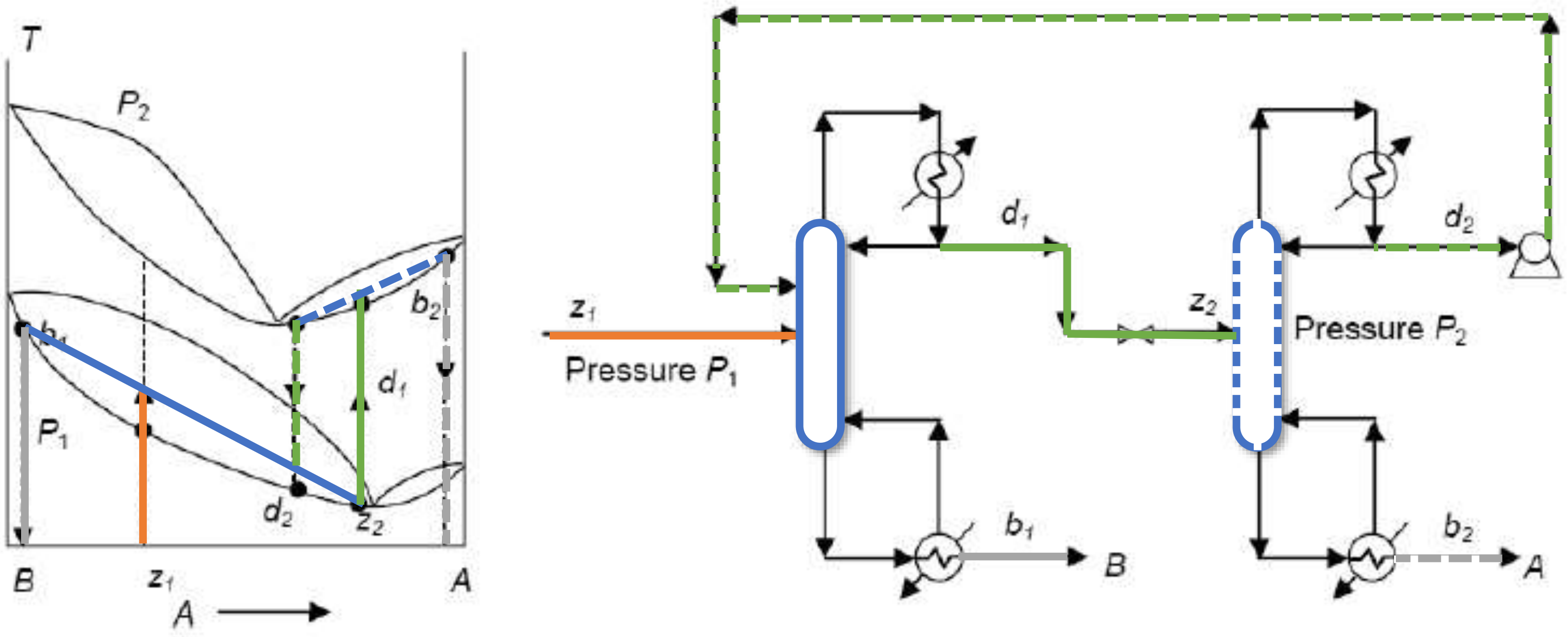
- It is sometimes possible to change the pressure sufficiently to "break" the azeotrope, e.g.
  - Ethyl alcohol and water form a homogeneous minimum-boiling azeotrope of 95.6 wt% alcohol at 78.15 °C and 101.3 kPa.
  - At vacuums of less than 9.3 kPa, no azeotrope is formed.
- Or at least "shift" the azeotrope

Pressure Swing Distillation

- A binary azeotrope that changes composition by > 5 mol% over a moderate range of pressure
- Two ordinary distillation columns operating in series at different pressures.
- A pump/compressor is needed to increase the pressure
- No additional component is needed

Pressure Swing Distillation

- e.g., Minimum-boiling azeotrope



Azeotropes

- Though this is sometimes not possible
- So we can add a third component

Extractive Distillation

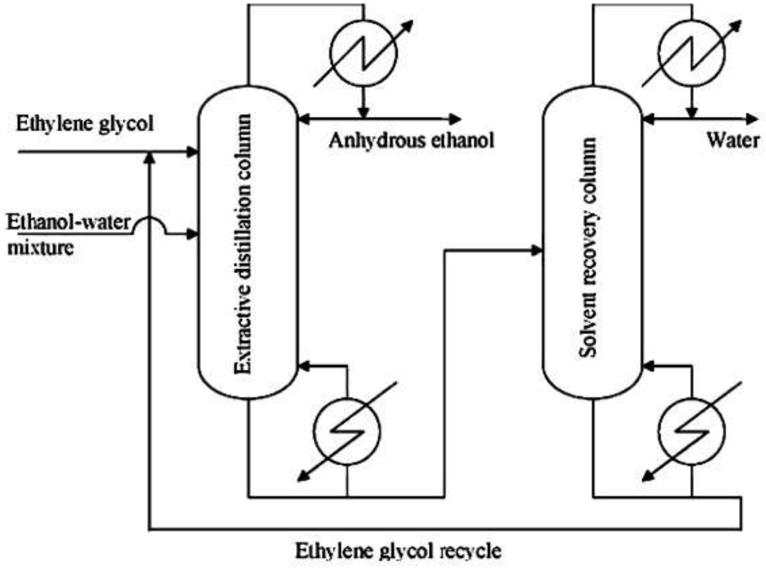
- The components in the feed must have different solvent affinities so that the solvent causes an increase in α of the key components.
- The solvent should not form an azeotrope with any components in the feed.
- Usually, a molar ratio of solvent to feed on the order of 1 is required.
- The bottoms are processed to recover the solvent for recycle and complete the feed separation.

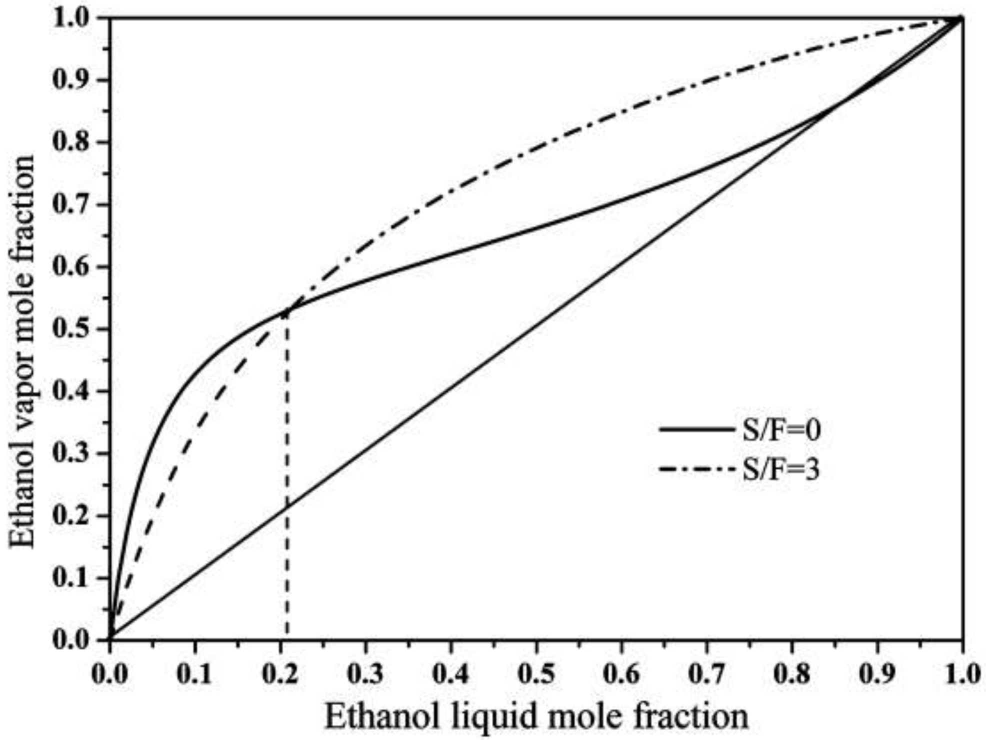
Extractive Distillation

- Minimum-boiling azeotrope system, a solvent with a lower volatility than the key components of the feed, is added just below the top of the column
    - the solvent is present in the down-flowing liquid, and
    - little solvent is stripped and lost to the overhead vapour.
- Maximum boiling azeotrope system, the solvent enters the column with the feed.

Extractive Distillation – "Dissolved" Solvent

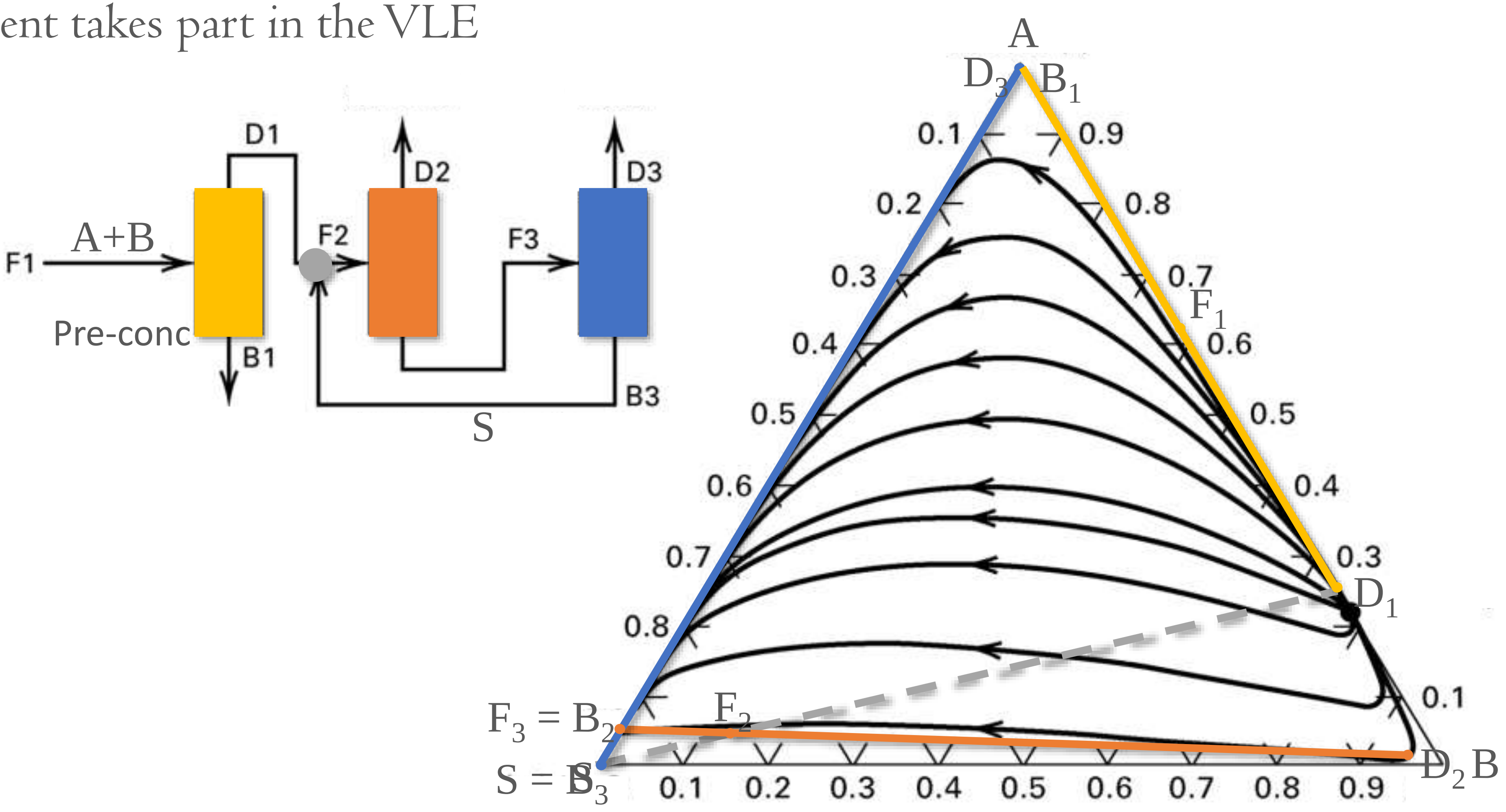
- For example, adding ethylene glycol to ethanol and water
  - The ethylene glycol never vapourises





Extractive Distillation – “Alternate Path”

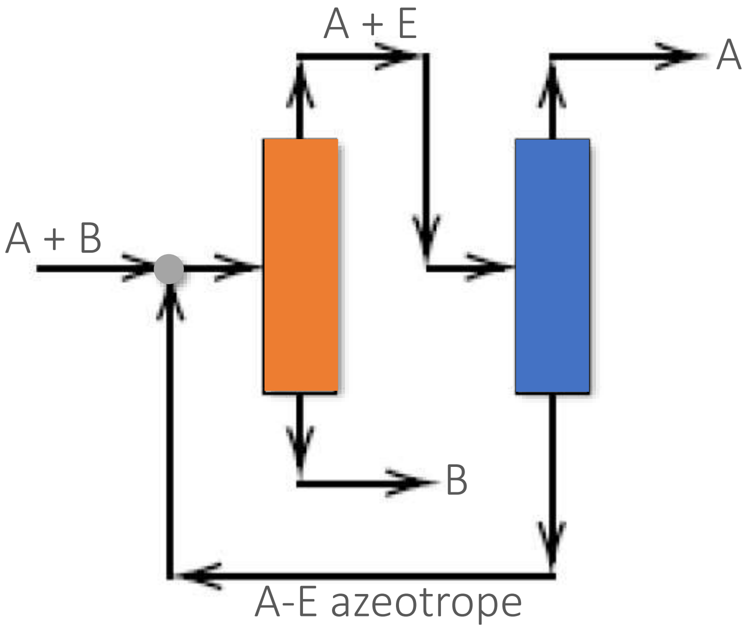
- The solvent takes part in the VLE

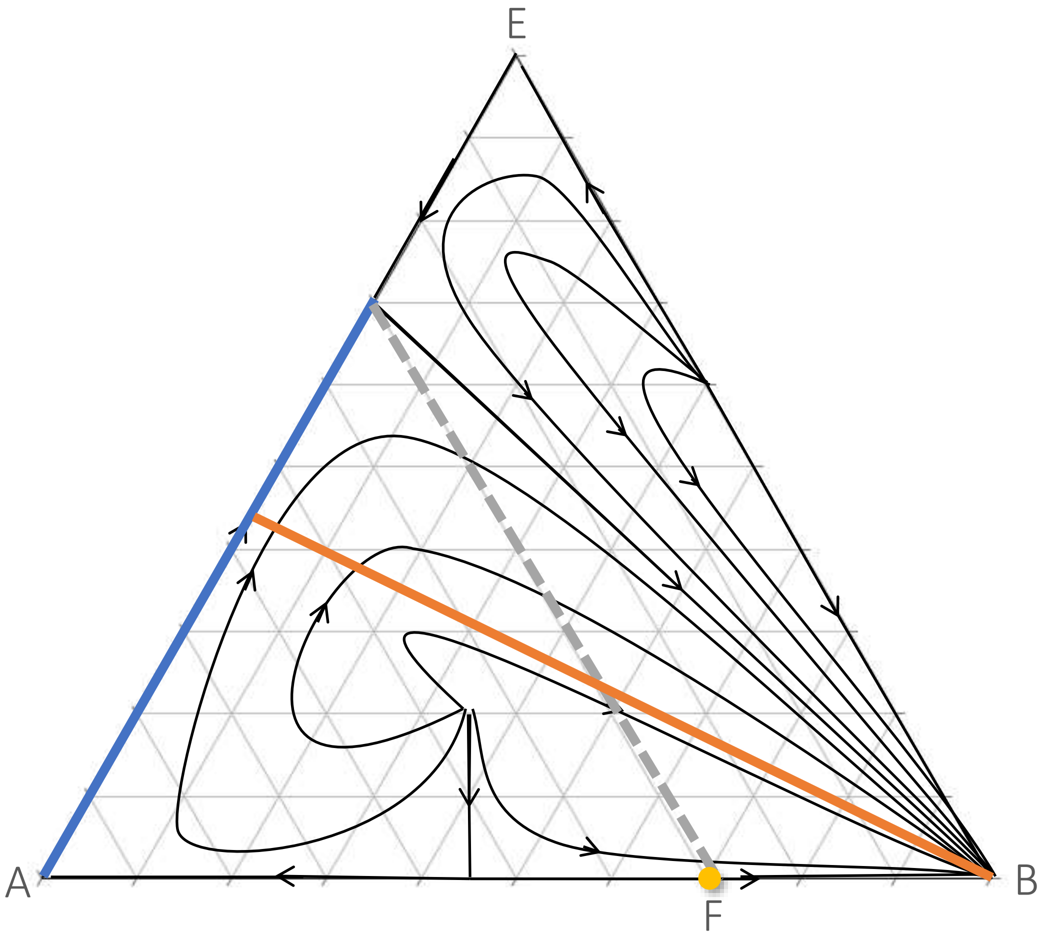


Homogeneous Azeotropic Distillation

- An azeotrope can be separated by extractive distillation, using a solvent that is higher boiling than the feed components and does not form any azeotropes.
- Alternatively, the separation can be made by homogeneous azeotropic distillation, using an entrainer not subject to such restrictions.
- Instead of recycling the pure entrainer, an azeotrope of the entrainer and one of the other components is recycled.

Homogeneous Azeotropic Distillation





Heterogeneous Azeotropic Distillation

- For homogeneous azeotropic distillation the patterns of the distillation boundaries are important for the separation
  - Often at least one maximum-boiling azeotrope is needed
  - However, such azeotropes are far less common than minimum-boiling azeotropes.
- Thus, sequences based on homogeneous azeotropic distillation are rare and a better alternative is needed.

Heterogeneous Azeotropic Distillation

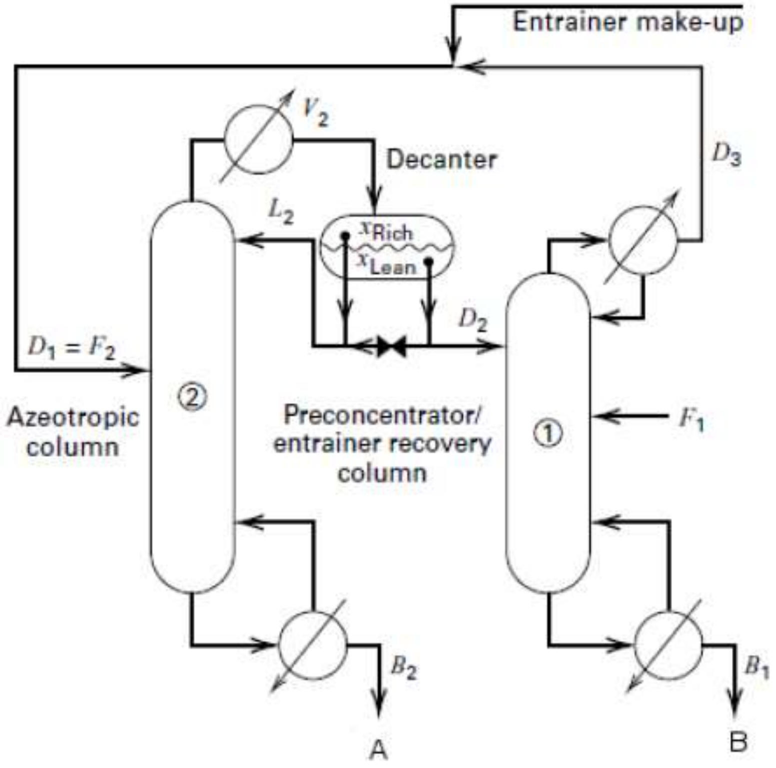
- An entrainer that forms a binary and/or ternary heterogeneous azeotrope is used.
- The overhead vapour from the column is set so it is close to the composition of the heterogeneous azeotrope.
- When condensed, two liquid phases form in a decanter which are then often separated by gravity separation.

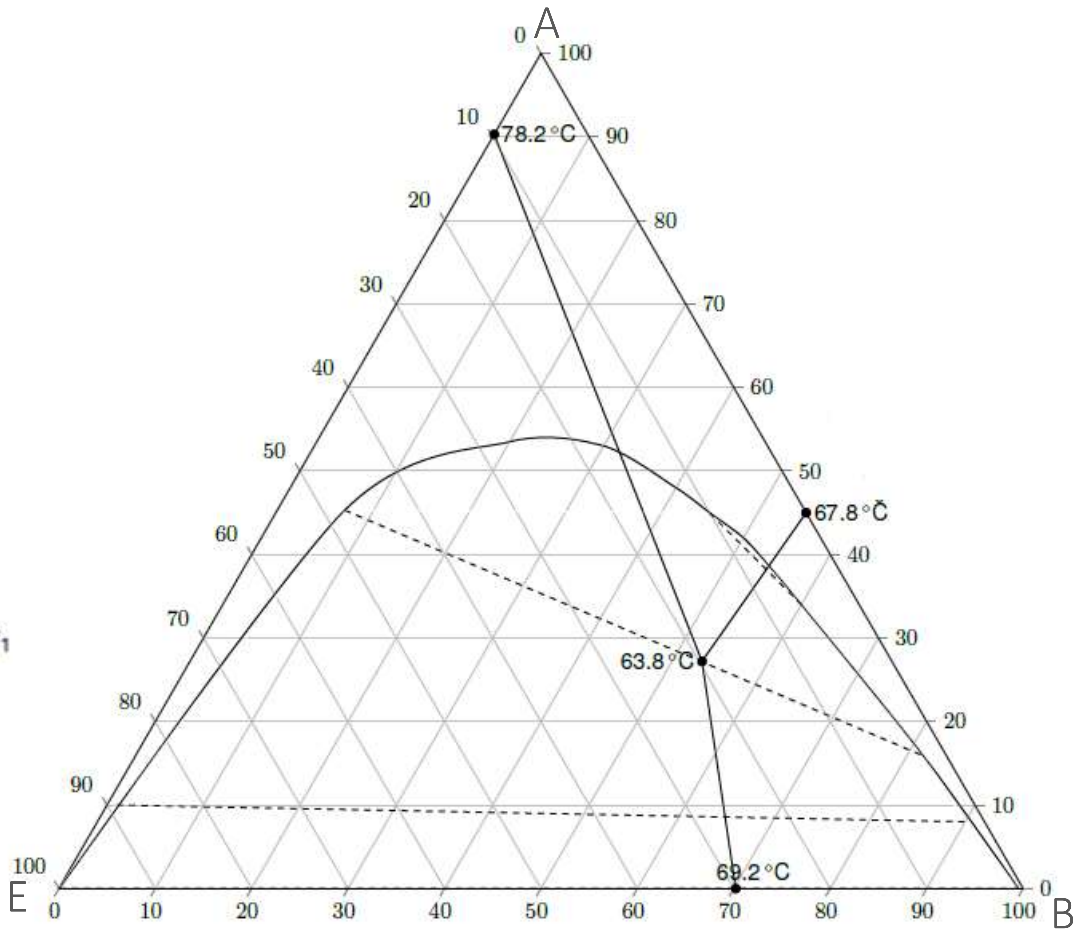
Heterogeneous Azeotropic Distillation

- As these two liquid phases usually lie in different distillation regions of the residue-curve map, the restriction that dooms homogeneous azeotropic distillation is overcome.

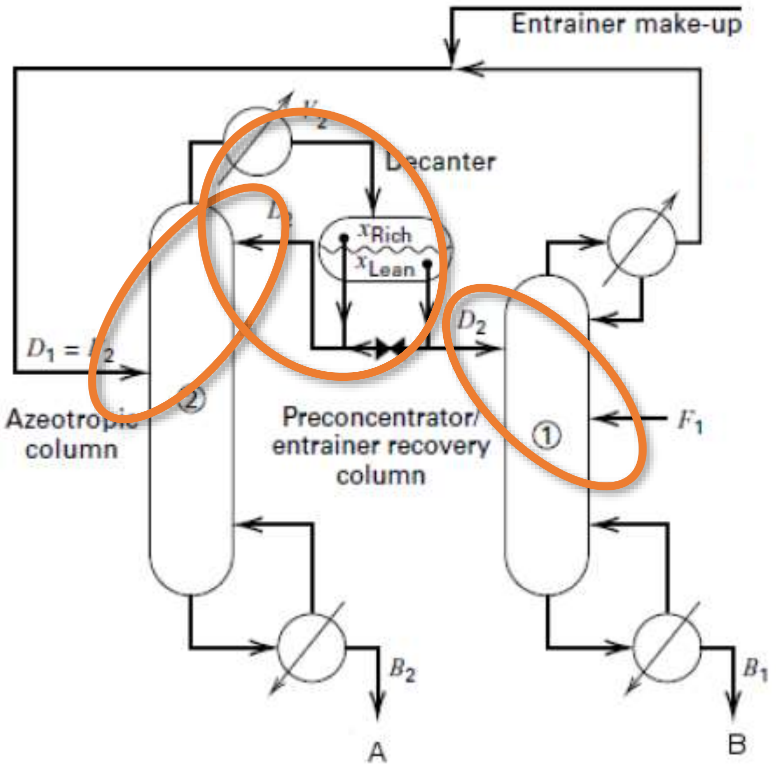
- Thus, in heterogeneous azeotropic distillation, the components to be separated need not lie in the same distillation region.

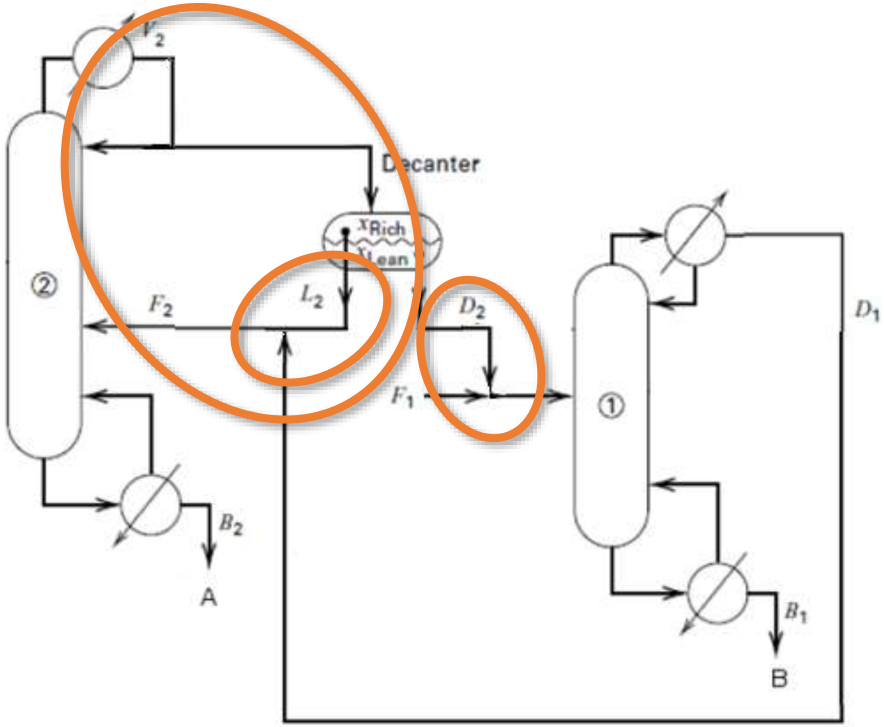
Heterogeneous Azeotropic Distillation





Heterogeneous Azeotropic Distillation





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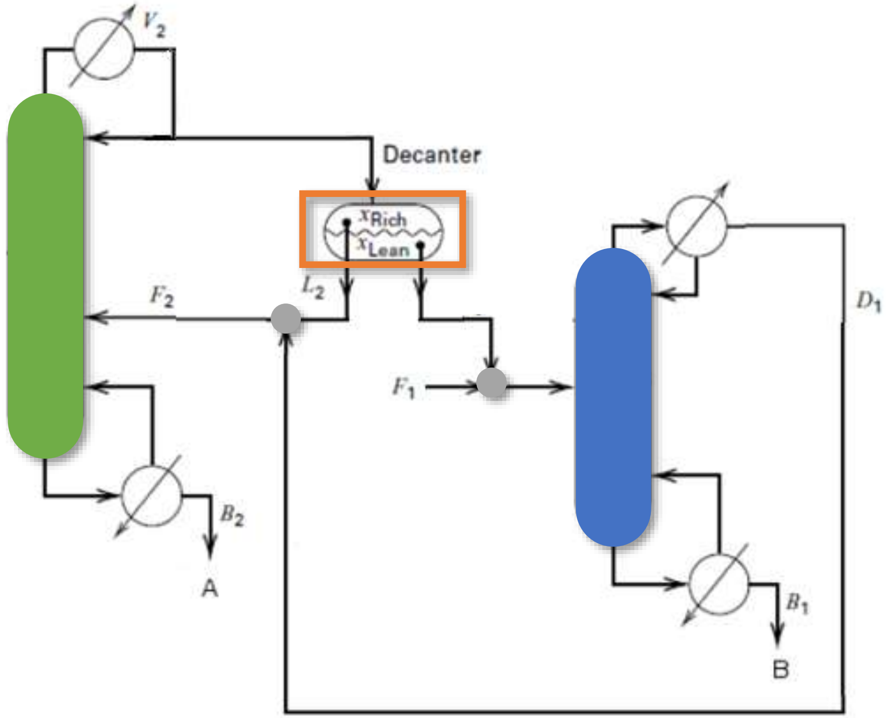
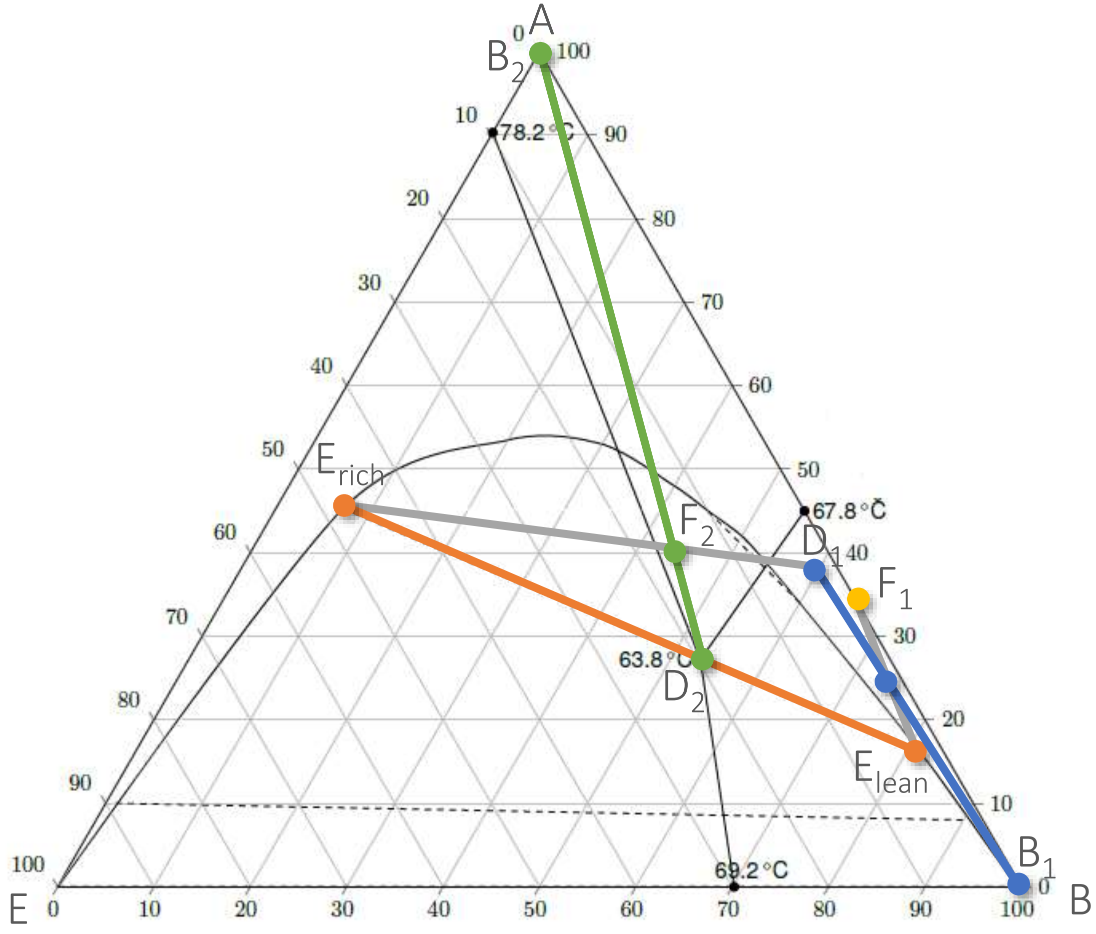
Heterogeneous Azeotropic Distillation





https://www.training.itservices.manchester.ac.uk/public/gced/separations.html?separations/Het_azeo/index.html

Reactive Distillation

- Reactive distillation denotes simultaneous chemical reaction and distillation.
- The reaction usually takes place in the liquid phase or at the surface of a solid catalyst in contact with the liquid.
- The separation of a close-boiling or azeotropic mixture of components A and B can be enhanced if a chemically reacting entrainer E is introduced into the column.

Reactive Distillation

- If A is the lower-boiling component,
  - E be higher boiling than B
  - react reversibly with B to produce reaction product C
- Component A is removed as distillate, components B, C, and E, are removed as bottoms.
- Components B and E are recovered from C in a separate distillation, where the reaction is reversed.

	α
A	1.01
B	1
E	0.8
C	0.5

$$B + E \rightleftharpoons C$$