4. INTRODUCTION TO PROCESS INTEGRATION

- **Process integration** is an approach to process design which emphasizes the unit of the process and considers the interaction between different unit operations from the outset, rather than optimizing them separately.

- It exploits the **interactions between different units in order to employ resources effectively and minimize costs**.

- The main advantage of process integration is to **consider a system as a whole in order to improve this design**.

- In contrast, an analytical approach would attempt **to improve or optimize process units separately** without necessarily taking advantage of potential interactions among them.

4.1. SYNTHESIS OF MASS EXCHANGE NETWORKS

- Most chemical processes are dominated by the need to separate **multi-component chemical mixture**.

- In general **a number of separation steps** must be employed, where each step separates between two components of the feed to that step.

- During process design, separation methods **must be selected and sequenced for these step**s.

- This section discusses some of the techniques for the synthesis of mass exchange network.

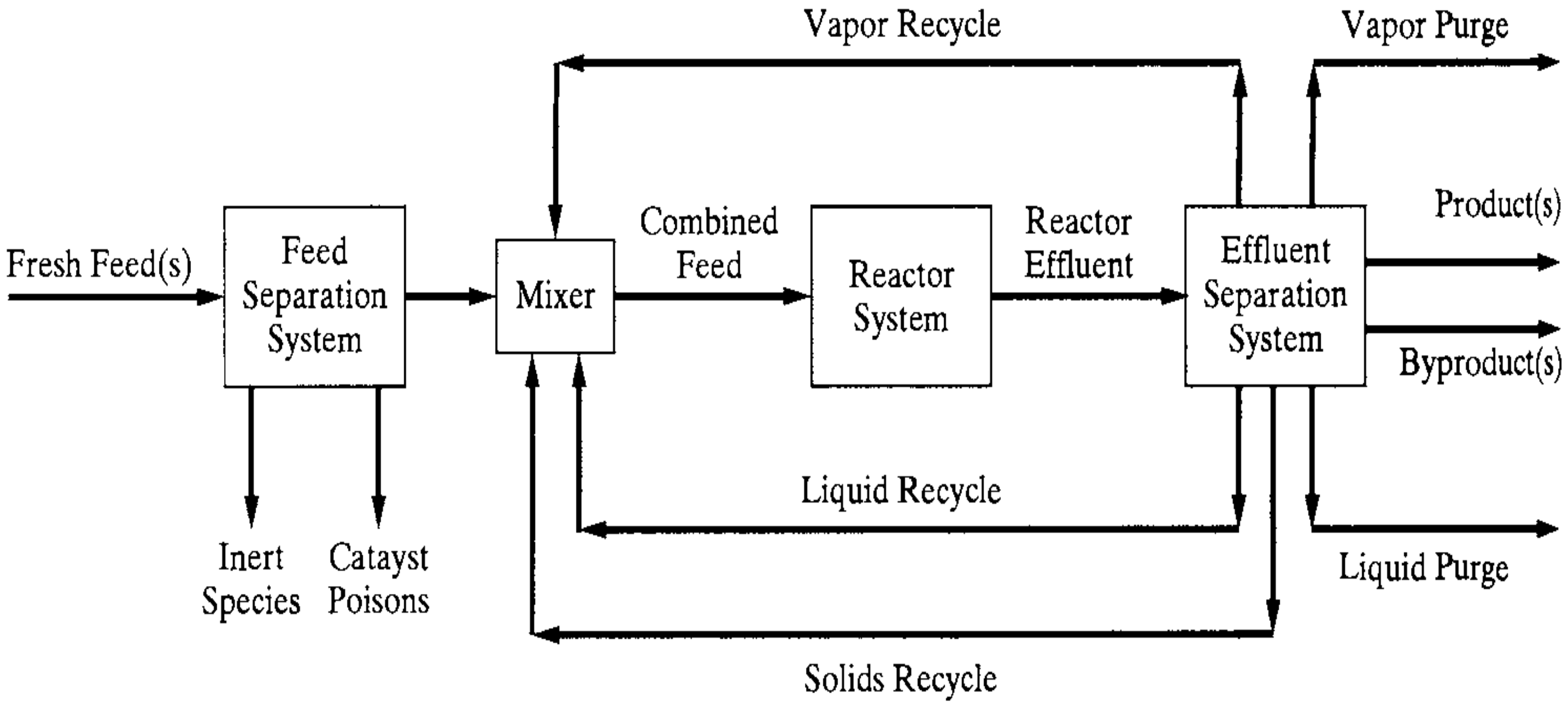
- **A feed separation** system is required to purify the reactor feed by removing catalyst poisons and inert species, especially if present as a significant percentage of the feed.

- **An effluent separation system**, which follows the reactor system and is almost always required, recovers unconverted reactants(in gas, liquid, and /or solid phases) to recycle to the reactor system and separates and purify products and byproducts.

- Where separations are too difficult **purge streams are used to prevent buildup** of certain species in recycle streams.

- Frequently, the major investment and operating costs of a process will be **those costs associated with the separation equipment, rather than with chemical reactor**(s).

- The following figure shows a general flow sheet for a process involving one reactor system where separation systems are shown before and as well as after the reactor section.



- The reactor effluent may be heterogeneous(two or more phases) mixture but most often is a homogenous mixture.

- When the effluent is homogenous, it is often advantageous to change the temperature or pressure **to obtain a partial separation of the components** by forming a heterogeneous mixture of two or more phases.

- Following the change in temperature and /or pressure, phase equilibrium is rapidly attained .

- Mass-exchange units are among **the most common separation operations** used in the process industries.

- A mass exchanger is any **direct-contact mass-transfer unit that employs a mass separating agent** “MSA” (or a lean stream) to selectively remove certain components (e.g., impurities, pollutants, byproducts, products) from **a rich stream**.

- The designation of **a rich or a lean stream** is not tied to the composition level of the components to be exchanged. Instead, the definition is task related.

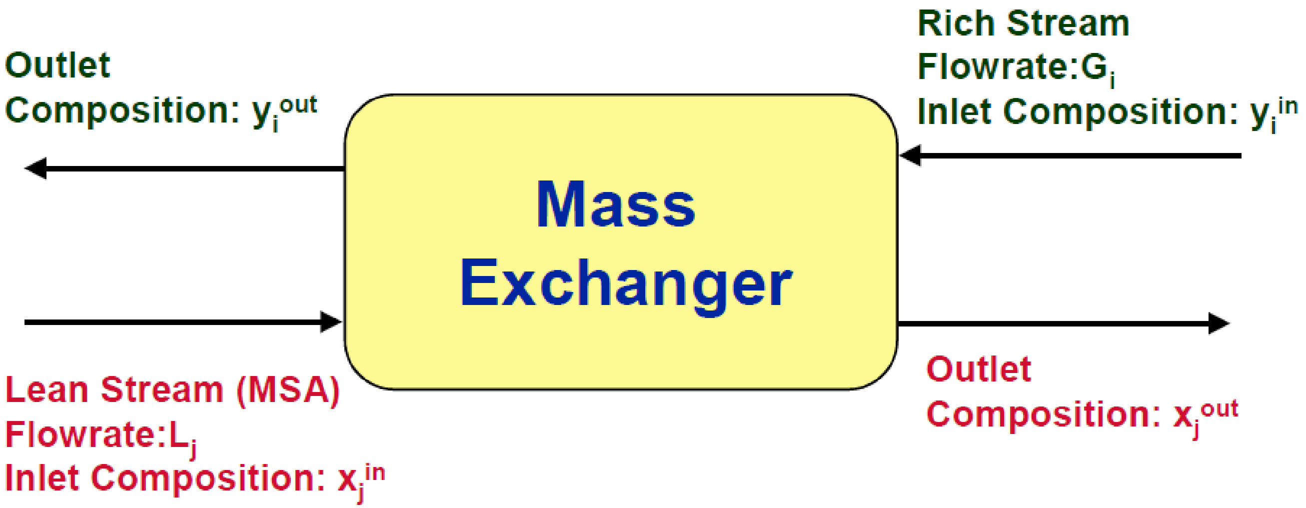
- The stream from which the targeted components **are removed** is designated as **the rich stream** while the stream to which the targeted components **are transferred** is referred to as **the lean stream** (or MSA).

- The MSA should be partially or **completely immiscible in the rich phase**. Examples of mass exchange operations include absorption, adsorption, stripping, ion exchange, solvent extraction, and leaching.

- Multiple mass exchange units are typically used in a processing facility. Therefore, their collective **selection, design, and operation must be coordinated and integrated.**

DESIGN OF INDIVIDUAL MASS EXCHANGERS

- Consider the mass exchanger shown in Fig. below. A certain component is transferred from the rich stream, i, to the lean stream, j.



- Two important aspects govern the performance of a mass exchanger: ***equilibrium function and material balance***.

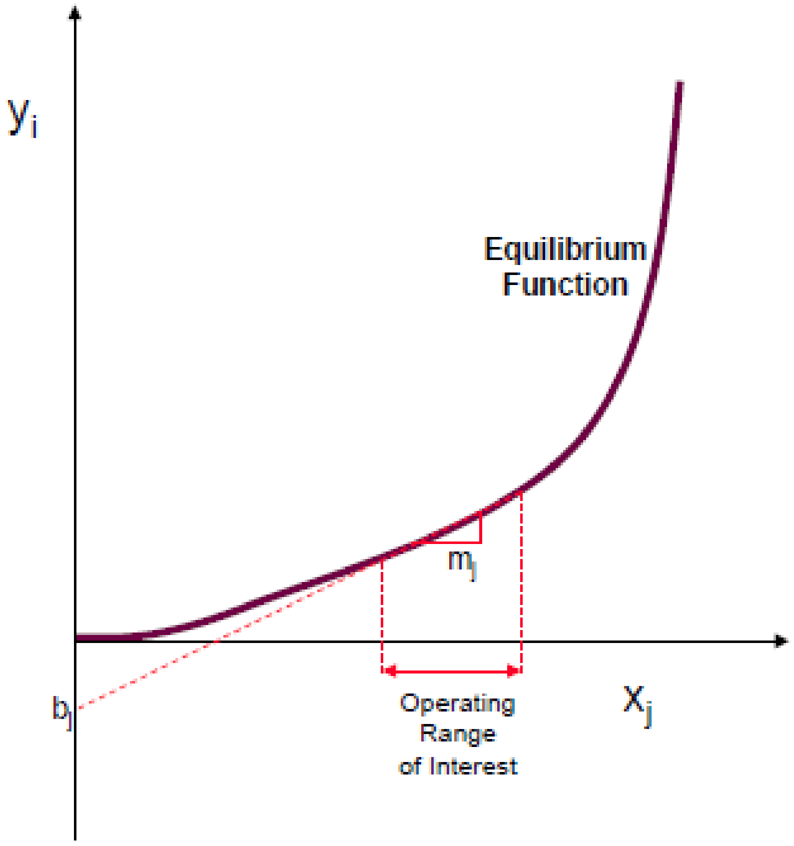
- Equilibrium refers to the state at which **there is no net interphase transfer** of the targeted species (solute).

- This situation corresponds to the state at which both phases have **the same value of chemical potential for the solute**. Mathematically, the composition of the solute in the rich phase, yi, can be related to its composition in the lean phase, xj, via an equilibrium distribution function, fj*.

- Hence, for a given rich-stream composition, yi, **the maximum achievable composition of the solute in the lean phase**, xj*, is given by:

$$y_i = f_j^*(x_j^*)$$

- Figure below is a schematic representation of an equilibrium function. In many cases, the equilibrium function can be linearized over a specific range of operation.



Linearized Segment of Equilibrium Function

- As shown by Fig., the linearized form has a slope of mj and an intercept of bj, i.e.

$$y_i = m_j \ x_j{}^* + b_j$$

- There are several important special cases of Eq. when the intercept, bj, is zero.

- These include Henry’s law, Raoult’s law, and extraction equilibrium with distribution coefficients.

- The material balance on the transferable solute accounts for the fact the mass of solute lost from the rich stream is equal to mass of solute gained by the lean stream, i.e.

$$G_i \left(y_i^{in} - y_i^{out}\right) = L_j \left(x_j^{out} - x_j^{in}\right)$$

- The material balance equation provides the mathematical description of the operating line.

- The operating line can be graphically represented on a y-x (McCabe-Thiele) diagram.

- Mass exchangers may be broadly classified into two categories:
    - **Stagewise units and**
    - **Differential (continuous) contactors.**

- **Stagewise units** are characterized by discrete solute transfer where mass exchange takes place in a stage followed by disengagement between the rich and lean phases then mass exchange and so on.

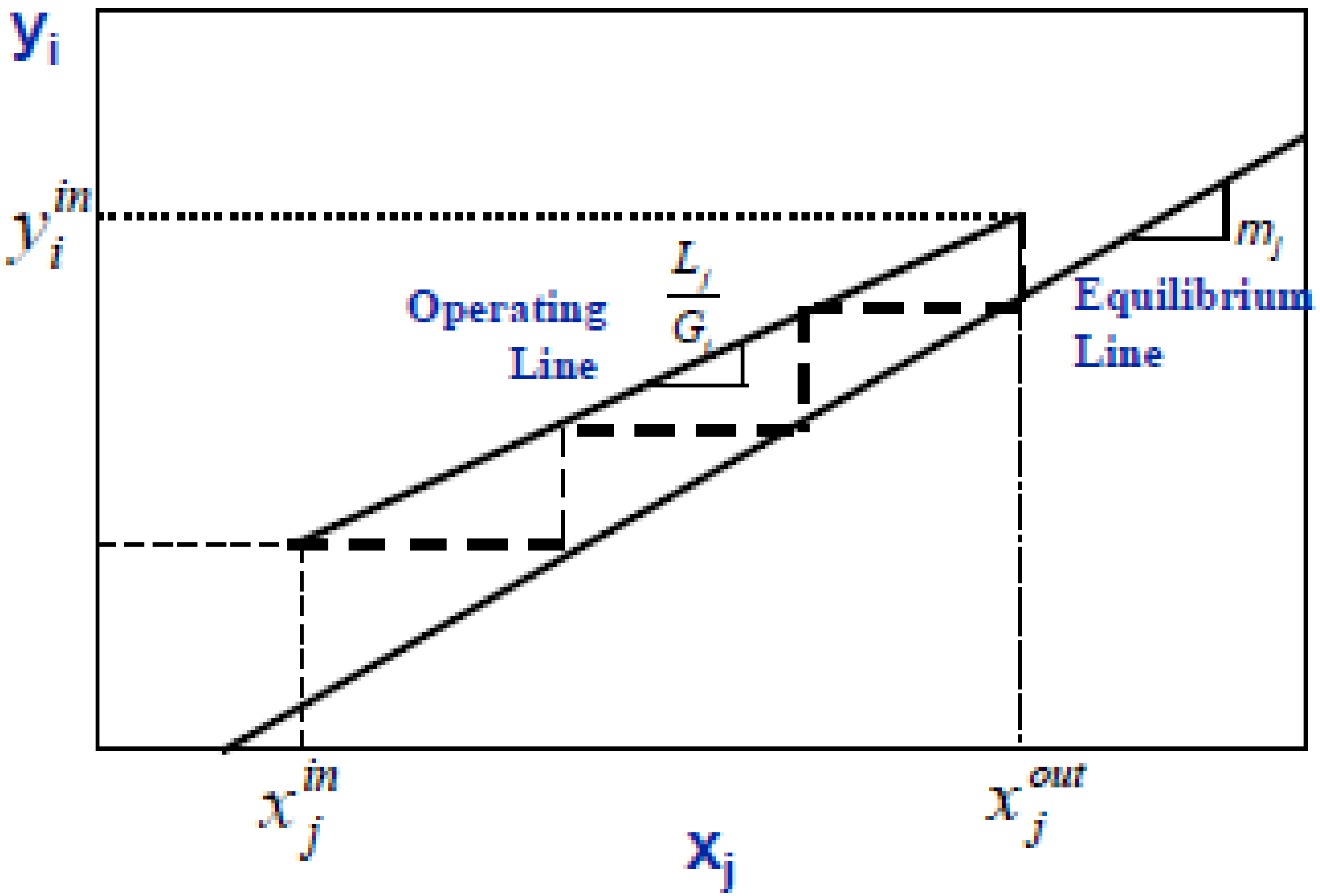
- Examples of stagewise units include tray columns and multistage mixer-settler arrangements.

- An important concept in stagewise operations is the notion of an **equilibrium stage or a theoretical plate**.

- With sufficient mixing time, the two phases leaving the theoretical stage are **essentially in equilibrium**; hence the name equilibrium stage.

- Each theoretical stage can be represented by **a step between the operating line and the equilibrium line**. Hence, the number of theoretical plates NTP can be determined by “stepping off” stages between the two ends of the exchanger.

- Equilibrium requires long-enough (infinite) contact time between the two phases. Therefore, to relate actual performance to equilibrium behavior it is necessary to calculate the number of actual plates “NAP” by incorporating **contacting efficiency**.



Equilibrium stage

- Once the number of plates is determined, the height of the mass exchanger can be determined by allowing **a plate spacing distance** between each two consecutive plates.

- The column diameter is normally determined by **selecting a superficial velocity** for one (or both) of the phases.

- The velocity is intended **to ensure proper mixing while avoiding hydrodynamic problems such as flooding**, weeping, or entrainment.

- Once a superficial velocity is determined, the cross-sectional area of the column is obtained **by dividing the volumetric flowrate** by the velocity.

- Continuous (differential) mass exchanger include packed units, spray exchangers, and bubble columns.

- The height of a differential contactor, H, may be estimated using

$$H = HTU_y \; NTU_y$$

$$= HTU_x \; NTU_x$$

- Where HTUy and HTUx are **the overall height of transfer units** based on the rich and the lean phases, respectively, while NTUy and NTUx are the **overall number of transfer units** based on the rich and the lean phases, respectively.

- The overall height of a transfer unit may be provided by the packing (or unit) manufacturer or estimated using empirical correlations (typically **by dividing superficial velocity of one phase by its overall mass transfer coefficient).**

- On the other hand, the number of transfer units can be theoretically estimated for the case of **isothermal, dilute mass exchangers with linear equilibrium** as follows:

$$NTU_y = \frac{y_i^{in} - y_i^{out}}{(y_i - y_i^*)_{\log mean}},$$

where

$$\left(y_i - y_i^*\right)_{\log mean} = \frac{\left(y_i^{in} - m_j x_j^{out} - b_j\right) - \left(y_i^{out} - m_j x_j^{in} - b_j\right)}{\ln\left(\dfrac{y_i^{in} - m_j x_j^{out} - b_j}{y_i^{out} - m_j x_j^{in} - b_j}\right)}$$

$$NTU_x = \frac{x_j^{in} - x_j^{out}}{(x_j - x_j^*)_{\log mean}},$$

where

$$\left(x_j - x_j^*\right)_{\log mean} = \frac{\left[x_j^{out} - \left(\frac{y_i^{in} - b_j}{m_j}\right)\right] - \left[x_j^{in} - \left(\frac{y_i^{out} - b_j}{m_j}\right)\right]}{\ln\left\{\frac{\left[x_j^{out} - \left(\frac{y_i^{in} - b_j}{m_j}\right)\right]}{\left[x_j^{in} - \left(\frac{y_i^{out} - b_j}{m_j}\right)\right]}\right\}}.$$

- If the terminal compositions or *Lj/Gi are unknown, it is convenient to use the following form:*

$$NTU_y = \frac{\ln\left[\left(1-\frac{m_j G_i}{L_j}\right)\left(\frac{y_i^{in}-m_j x_j^{in}-b_j}{y_i^{out}-m_j x_j^{in}-b_j}\right)+\frac{m_j G_i}{L_j}\right]}{1-\left(\frac{m_j G_i}{L_j}\right)}.$$

COST OPTIMIZATION OF MASS EXCHANGERS

- In assessing the economics of a mass exchanger, two types of cost must be considered: **fixed and operating**.

- The fixed cost (investment) refers to the cost of the mass exchanger (e.g., shell, trays, etc.), auxiliary devices (e.g., pump, compressor), installation, insulation, instrumentation, electric work, piping, engineering work and construction.

- Fixed capital investments are characterized by the fact that **equipment have to be replaced after a number of years commonly referred to as service life or useful life period** because of wear and tear or by virtue of becoming obsolete or inefficient.

- Therefore, it is useful to evaluate an annual cost associated with the capital investment of the mass exchanger, referred to as the **annualized fixed cost "AFC".**

- A simplified method for evaluating AFC is to consider **the initial fixed cost of the equipment (FCo) and its salvage value** (FCs) after n years of useful life period. Using an annual depreciation scheme, we get

$$AFC = \frac{FC_o - FC_s}{n}$$

- In addition to the fixed capital investment needed to purchase and install the mass exchange system and auxiliaries, there is a continuous expenditure referred to as **operating cost which is needed to operate the mass exchanger**.
- The operating cost includes **mass-separating agents** (makeup, regeneration, etc.) and **utilities** (heating, cooling, etc.).

- By combining the fixed and operating costs, we get **the total annualized cost of a mass exchange system**:

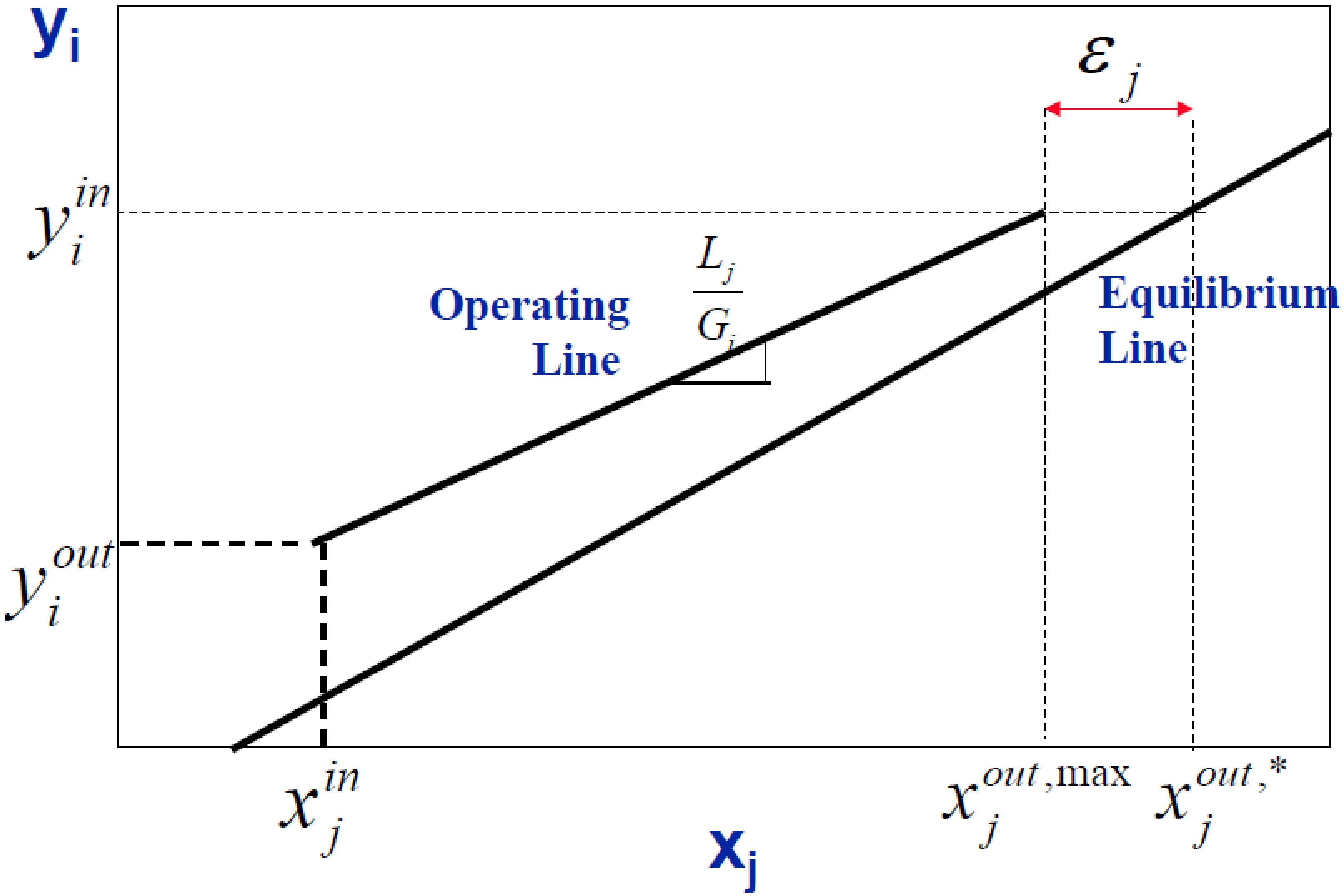
Total annualized cost = Annualized fixed cost + Annual operating cost

- In order to minimize TAC, it is necessary **to trade off the fixed cost versus the operating cost**.

- Such tradeoffs can be established **by identifying the role of the mass-exchange driving force** between the actual operation and the equilibrium limits.

- In order to reach equilibrium compositions, an infinitely-large mass exchanger is required. Therefore, the operating line must have **a positive driving force with respect to the equilibrium line**.

- The minimum driving force between the operating line and the equilibrium line is referred to as **the minimum allowable composition difference** and is designated by ε_j as shown by the Fig. below.



Establishing a Minimum Allowable Composition Difference

- The minimum allowable composition difference can be **used to tradeoff capital versus operating costs**. In order to demonstrate this concept, let us consider the mass exchanger represented on the y-x diagram of the above Fig. .

- For the rich stream, the inlet and outlet compositions as well as the flowrate are all given. For the lean stream, **the inlet composition is given while the flowrate and the outlet compositions are unknown** (x_j^{out} and Lj).

- The maximum theoretically attainable outlet composition in the lean phase **(x_j^{out*}) is the equilibrium value corresponding to the inlet composition of the rich stream** .

- As mentioned earlier, **achieving this equilibrium value requires an infinitely-large mass exchanger**.

- Once the minimum allowable composition difference is selected, the maximum practically feasible outlet composition in the lean stream (x j $^{\text{out max}}$) can be determined as:

$$x_j^{out,max} = x_j^{out,*} - \varepsilon_j$$

And

$$y_i^{in} = m_j \, x_j^{out,*} + b_j$$

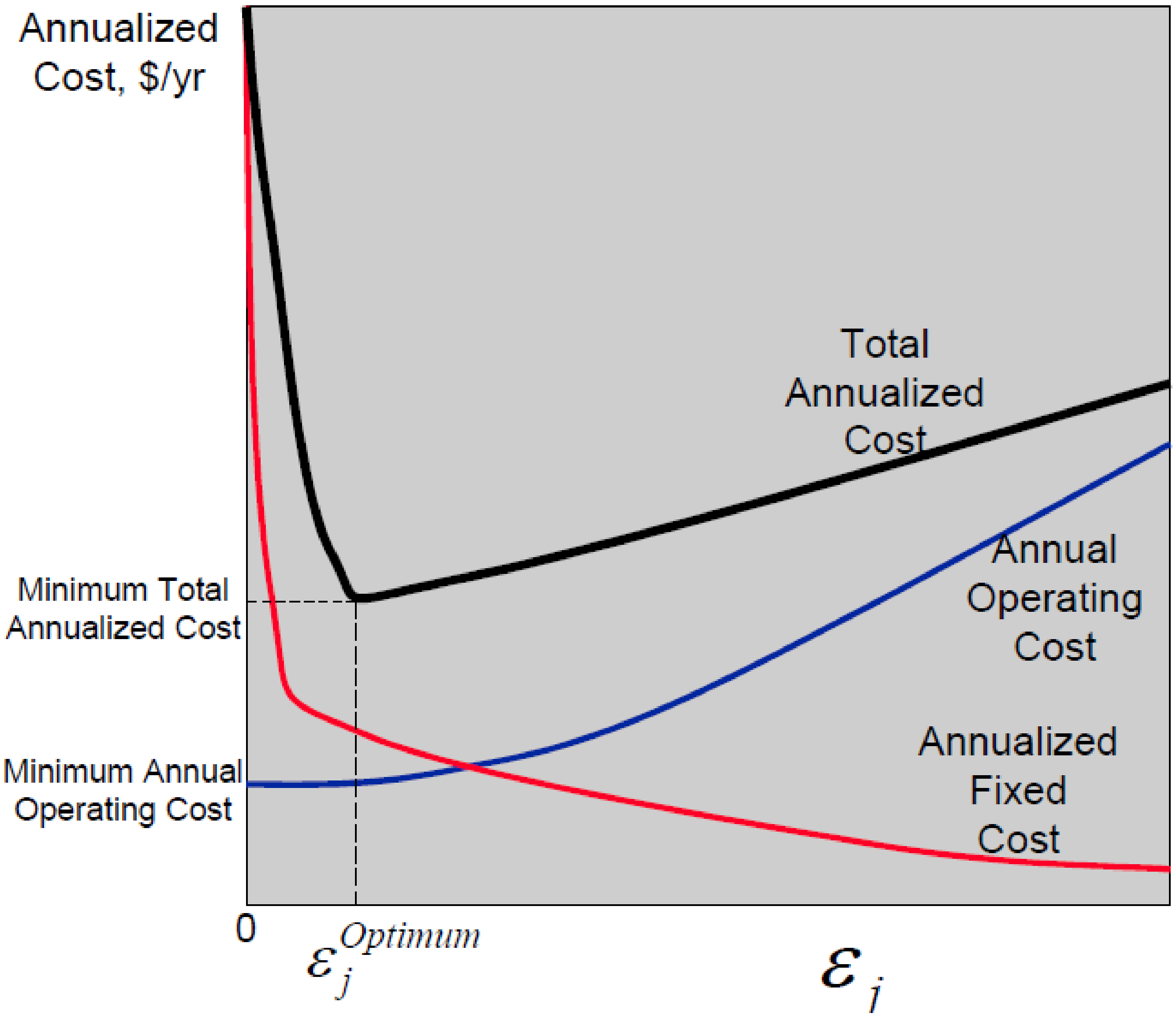
Thus, the general equation will be :

$$x_j^{out,\mathrm{max}} = \frac{y_i^{in} - \mathrm{b_j}}{\mathrm{m_j}} - \varepsilon_\mathrm{j}$$

- **As ε_j increases, the slope of the operating line increases and the flowrate of the MSA increases leading to an increase in the operating cost** of the mass exchanger.

- Meanwhile, as ε_j increases **the number of theoretical plates decreases** thereby leading to **a reduction in the fixed cost**.

- By varying ε_j and evaluating the corresponding annualized fixed cost, annual operating cost, and total annualized cost, we can determine **the optimum value of minimum allowable composition difference**, Optimum ε_j , which corresponds to **the minimum total annualized cost**.



Typical diagram of TAC vs εj

SYNTHESIS OF MASS-EXCHANGE NETWORKS

- In many processing facilities, mass exchangers are used to separate targeted species **from a number of rich streams**.

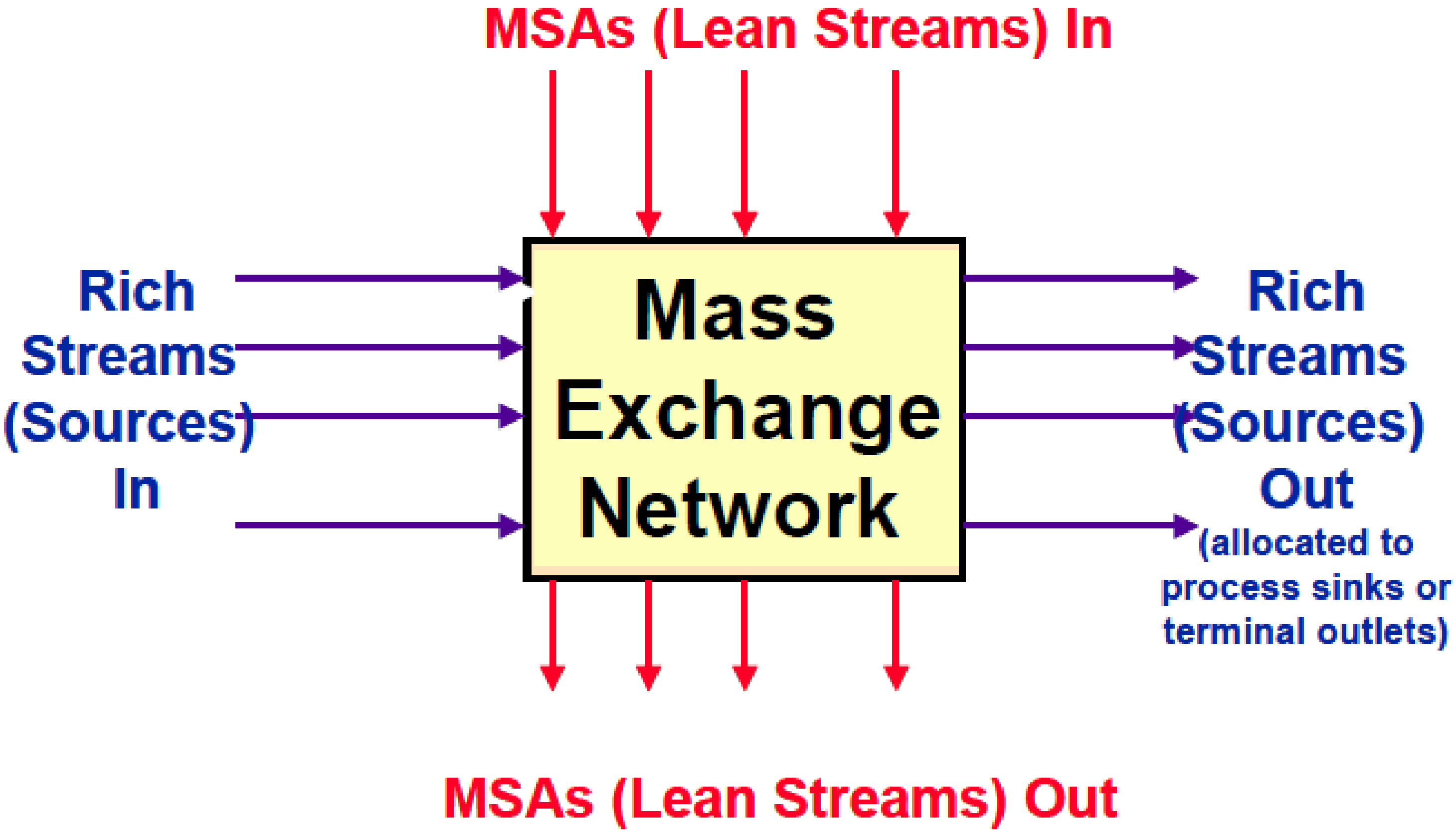
- More than one mass-exchange technology and **more than one MSA** may be considered. In such situations, it is **necessary to integrate the decisions and design** of the multiple mass exchangers.

- This requires a holistic approach to **consider all separation tasks from all rich stream**, simultaneously **screen all candidate mass exchange operations and MSAs**, and **identify the optimum network of mass exchangers.**

- El-Halwagi and Manousiouthakis (1989) introduced the problem of synthesizing mass-exchange network **“MENs” and developed systematic** techniques for their optimal design.

The problem of synthesizing MENs can be stated as follows:

- Given a number N_R of rich streams (sources) and a number N_S of MSAs (lean streams), it is desired to synthesize a cost-effective network of mass exchangers that can preferentially transfer certain species from the rich streams to the MSAs.
- Given also are the flowrate of each rich stream, Gi, its supply (inlet) composition yi^s, and its target (outlet) composition yi^t, where $i = 1,2,...,N_R$. In addition, the supply and target compositions, xj^s and xj^t, are given for each MSA, where $j = 1,2,...,N_S$. **The flowrate of each MSA is unknown and is to be determined so as to minimize the network cost**.



MSAs (Lean Streams) In

Rich Streams (Sources) In

Mass Exchange Network

Rich Streams (Sources) Out (allocated to process sinks or terminal outlets)

MSAs (Lean Streams) Out

- The candidate lean streams can be classified into N_{SP} **process MSAs** and N_{SE} **external** MSAs (where $N_{SP} + N_{SE} = N_S$).

- The process MSAs already exist on plant site and can be used for the removal of the undesirable species at a very low cost (virtually free).

- The flowrate of each process MSA that can be used for mass exchange is bounded by its availability in the plant, i.e.,

$$L_j \leq L_j^c \qquad j = 1,2,\ldots,N_{SP}$$

where Lj^c is the flowrate of the jth MSA that is available in the plant.

- On the other hand, the external MSAs can be purchased from the market. Their flowrates are to be determined according to the overall economic considerations of the MEN

- Typically, rich streams leaving the MEN are either allocated to **process sinks (equipment) or assigned to be terminal streams** (e.g., products, wastes).

- When the outlet rich streams are allocated to process sinks, **the target composition of the rich stream are selected so as to satisfy the constraints on the feed** to these sinks.

- In case of final discharge, the target composition of the undesirable species in each rich stream **corresponds to the environmental regulations**.

- Finally, if the outlet rich stream corresponds to a terminal product, the target composition is set **to satisfy quality requirements for the product**.

- The target composition of each MSA is an upper bound on the actual outlet composition of the MSA. The value of the target composition is selected based on a number of factors whose nature may be:
  - **Physical** (e.g., saturation compositions, solubility limits, precipitation conditions)
  - **Operational**: If the outlet MSA is used in a subsequent unit, its content of certain species must conform to the constraints on the feed to the subsequent unit
  - **Safety** (e.g., to stay away from flammability/explosion limits)
  - **Health** (e.g., to avoid reaching toxic compositions)
  - **Environmental** (e.g., to satisfy emission regulations)
  - **Economic** (e.g., to minimize the cost of the mass-exchange and regeneration systems)
  - **Technical feasibility** (e.g., to satisfy thermodynamic constraints and minimum driving force)

- The MEN synthesis task entails answering several design questions and challenges:
  - **Which mass-exchange technologies** should be utilized (e.g., adsorption, solvent extraction ion exchange, etc.)?

  - **Which MSAs** should be selected (e.g., which solvents, adsorbents)?

  - What is the **optimal flowrate** of each MSA?

  - How should these MSAs **be matched with the rich streams**?

  - What is the **optimal system configuration** (e.g., how should these mass exchangers be arranged? Is there any stream splitting and mixing?) ?

MASS-EXCHANGE PINCH DIAGRAM

- The mass exchange pinch analysis (El-Halwagi and Manousiouthakis, 1989) provides **a systematic approach to synthesizing MENs**.
- It also enables the identification of rigorous targets such as **minimum cost of MSAs**.
- The first step in the analysis is **to develop an integrated view of all the separation task**s for the rich streams.
- This can be achieved by developing a composite representation of mass exchanged from all the rich streams.
- Mass of targeted species removed from the ith rich stream is given by:

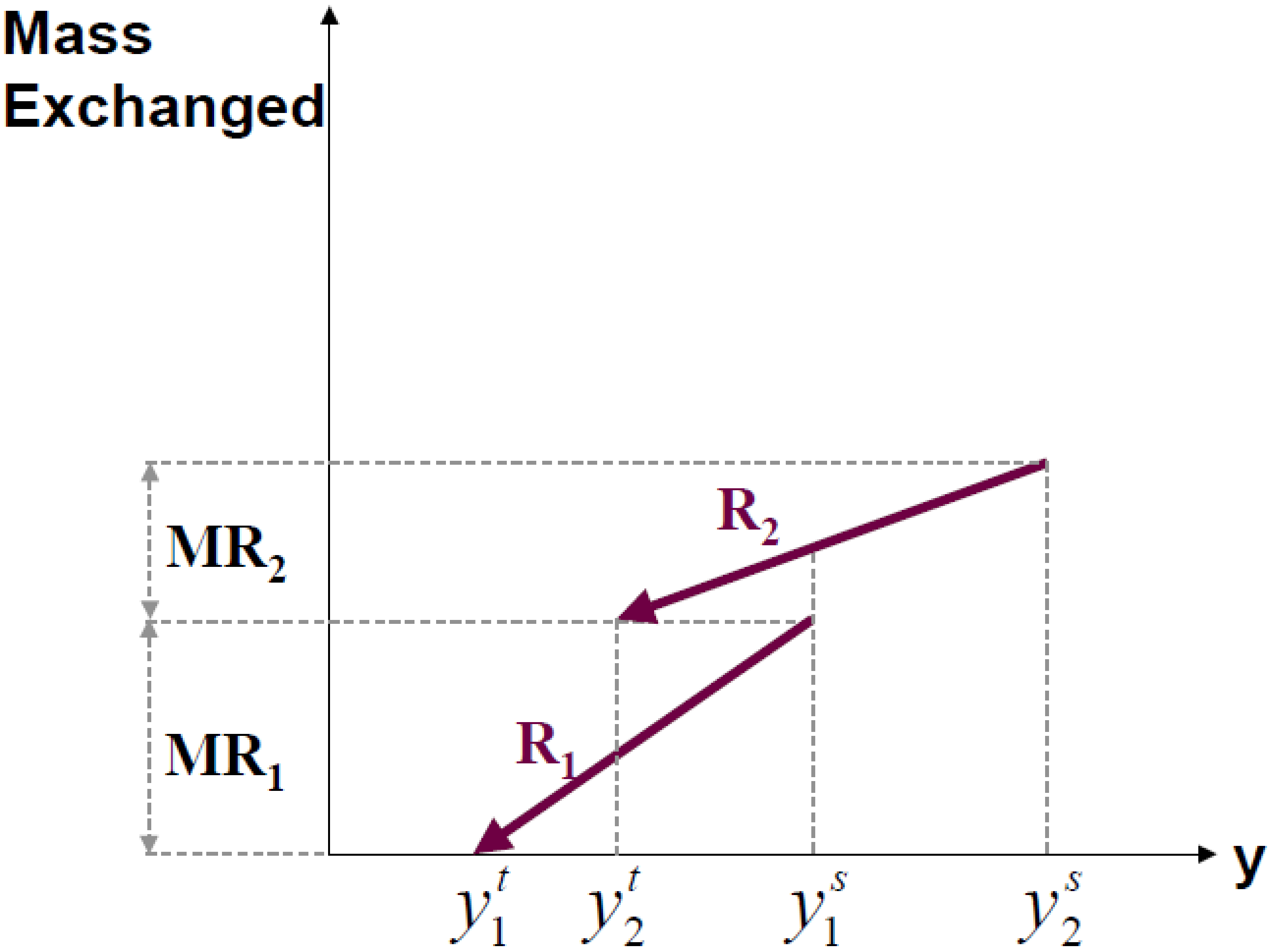
$$MR_i = G_i\,(y_i^s - y_i^t),$$

- By plotting mass exchanged versus composition, each rich stream is represented **as an arrow** whose **tail corresponds to its supply composition** and **its head to its target composition**.

- The slope of each arrow is equal to the **stream flowrate**.

- The vertical distance between the tail and the head of each arrow represents **the mass of targeted species that is lost by that rich stream**.

- In this representation, the **vertical scale is only relative**. Any stream can be moved up or down while preserving the same vertical distance between the arrow head and tail and maintaining the same **supply and target compositions**.



Representation of mass exchanged by two rich streams

- A stream **cannot be moved left or right**, otherwise stream composition will be altered.

- A convenient way of vertically placing each arrow is to rank the rich streams **in ascending order of their targeted composition** then we stack the rich streams on top of one another, starting with **the rich stream having the lowest target composition**.

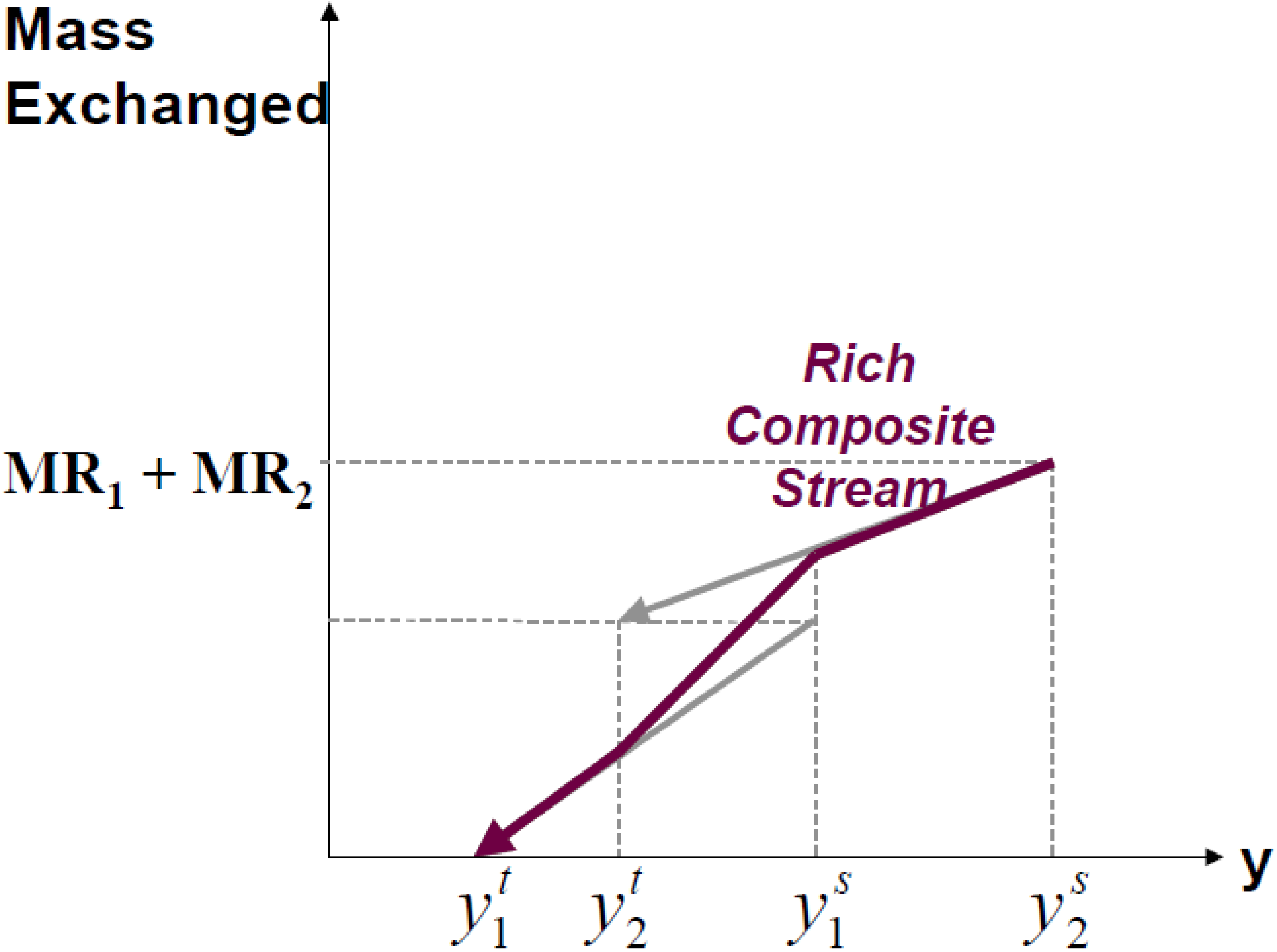
- Once the first rich stream is represented, we draw a horizontal line passing through the arrow tail of the stream.

- Next, the second rich stream is represented as an arrow extending between its supply and target compositions and having a vertical distance equal to the mass of the targeted species to be removed from this stream.

- The arrowhead of the second rich stream is placed on the horizontal line passing through the arrow tail of the first rich stream.

- After all the rich streams have been represented, it is necessary to develop a combined representation of the rich streams that allows us **to observe the separation tasks of all rich streams as a function of composition**.

- A rich composite stream can be constructed using "diagonal rule" for superposition to add up mass in the overlapped regions of streams


Mass
Exchanged
$MR_1 + MR_2$
Rich
Composite
Stream
y
y_1^t
y_2^t
y_1^s
y_2^s

- In the region between y_t^1 and y_t^2 , there is only R1.

- Therefore, the composite representation is exactly the same as R1.

- Similarly, in the region between y_s^1 and y_s^2 there is only R2 and, hence, the composite representation is exactly the same as R2. In the overlapping region of the two rich streams (between y_t^2 and y_s1), the composite representation of the two streams is the diagonal (hence the name diagonal rule).

- By connecting these three linear segments, we now have a rich composite stream which represents the **cumulative mass of the targeted species removed from all the rich streams**.

- It captures **the relevant characteristics of the rich streams** and enables **the simultaneous consideration of all rich streams** and developing an integrated mass-exchange strategy for all of them.

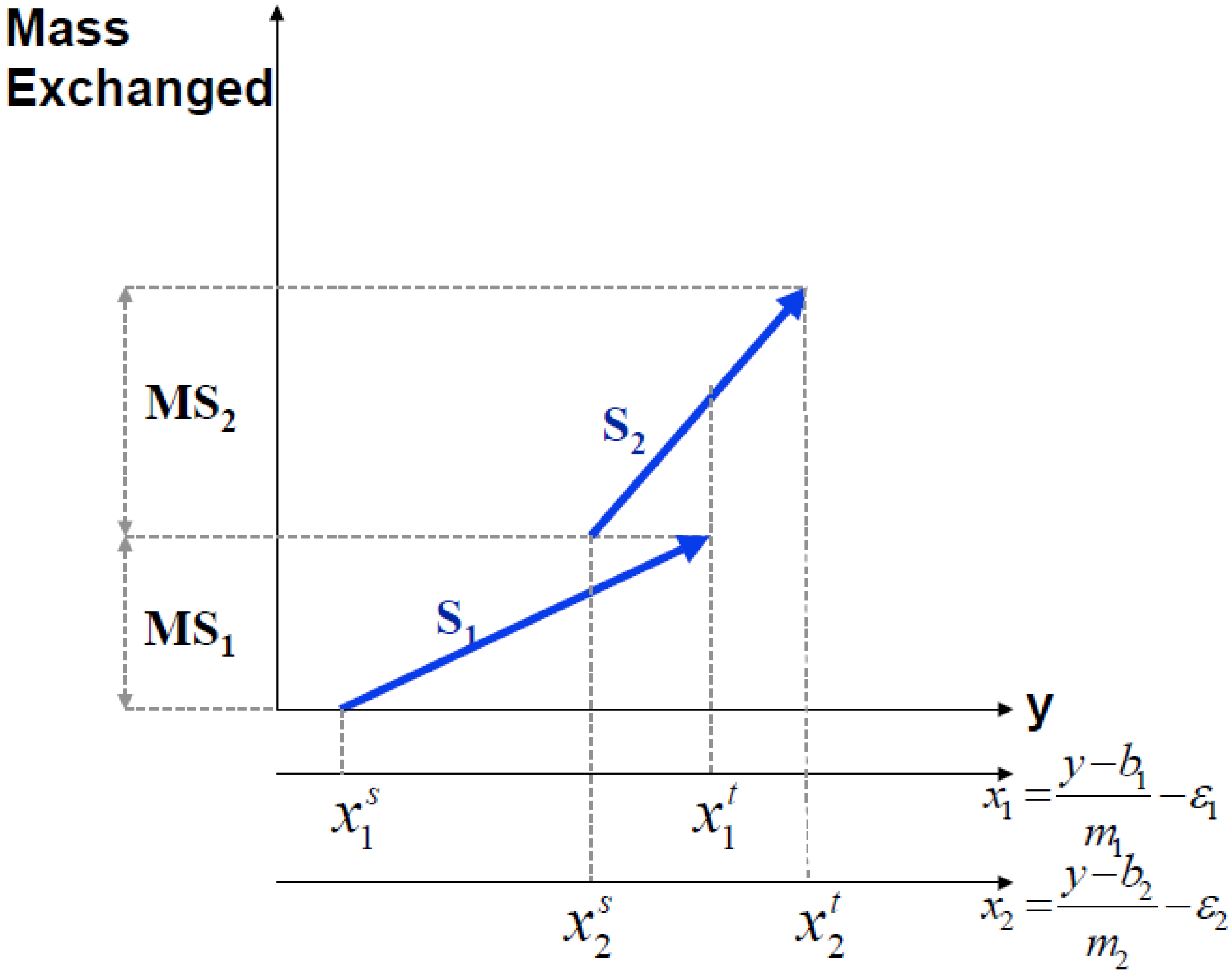
- Next, attention is turned to the lean streams. Since the **process MSAs are available on-site and may be used virtually for no or little operating cost, we will first consider maximizing their use**.

- The remaining load will then be removed using external MSAs. Therefore, we first establish ***NSP* lean composition scales (one for each process MSA)** that are in one-to-one correspondence with the rich scale.

- Next, the mass of targeted species that can be gained by each process MSA is plotted versus the composition scale of that MSA. Hence, **each process MSA is represented as an arrow extending between supply and target compositions**

- The vertical distance between the arrow head and tail is given by Mass of solute that can be gained by the jth process MSA

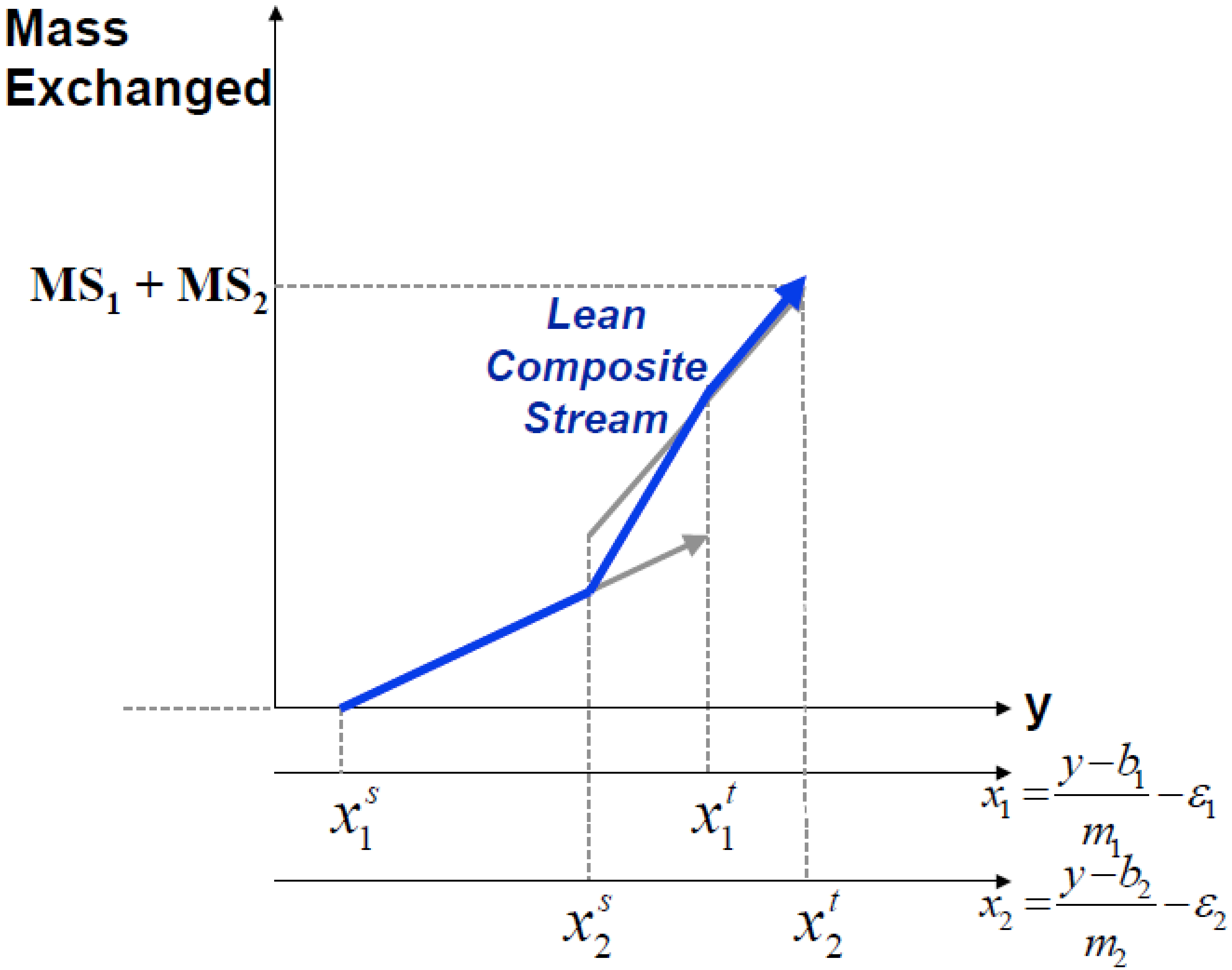
$$MS_j = L_j^c \,(x_j^t - x_j^s) \qquad j = 1,2,\ldots, N_{SP}.$$


Mass Exchanged
MS_2
MS_1
S_2
S_1
y
x_1^s
x_1^t
x_2^s
x_2^t
$x_1 = \frac{y - b_1}{m_1} - \varepsilon_1$
$x_2 = \frac{y - b_2}{m_2} - \varepsilon_2$

- Once again, the vertical scale is only relative and any stream can be moved up or down on the diagram.

- A convenient way of vertically placing each arrow is to stack the process MSAs on top of one another **starting with the MSA having the lowest supply composition** .

- Hence, a lean composite stream representing the cumulative mass of the targeted species gained by all the MSAs is obtained by using **the diagonal rule for superposition**

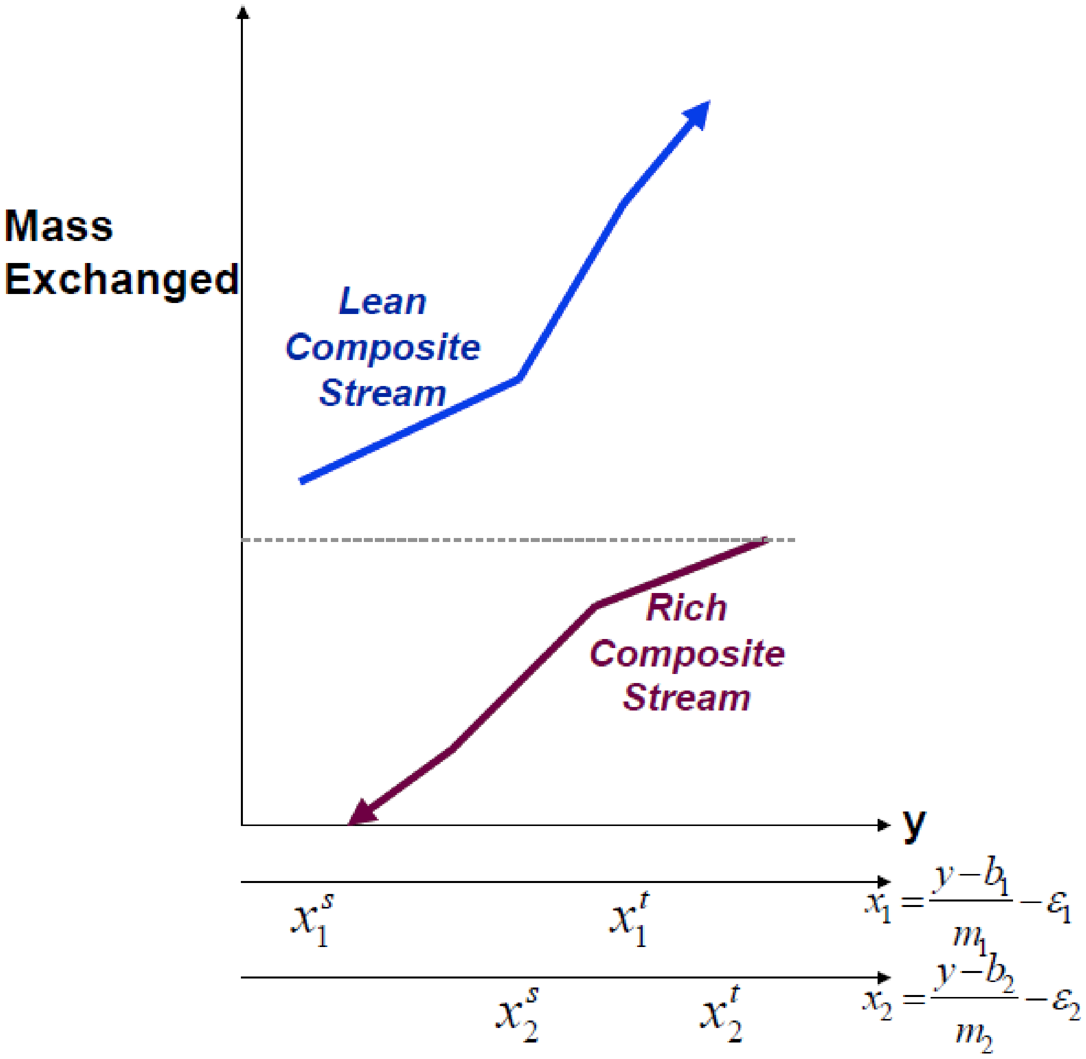

Mass Exchanged
$MS_1 + MS_2$
Lean Composite Stream
y
x_1^s
x_1^t
$x_1 = \frac{y - b_1}{m_1} - \varepsilon_1$
x_2^s
x_2^t
$x_2 = \frac{y - b_2}{m_2} - \varepsilon_2$

- Next, both composite streams are plotted **on the same diagram** (shown in the next Fig.).

- On this diagram, thermodynamic feasibility of mass exchange is guaranteed when **at any mass-exchange level (which corresponds to a horizontal line), the composition of the lean composite stream is located to the left of the rich composite stream**.

- For a given set of corresponding composition scales it is thermodynamically and practically feasible to **transfer the targeted species from any rich stream to any MSA**.

- In addition, it is also feasible to transfer the targeted species from any rich stream of a composition y_i to any MSA which has a composition less than the x_j.



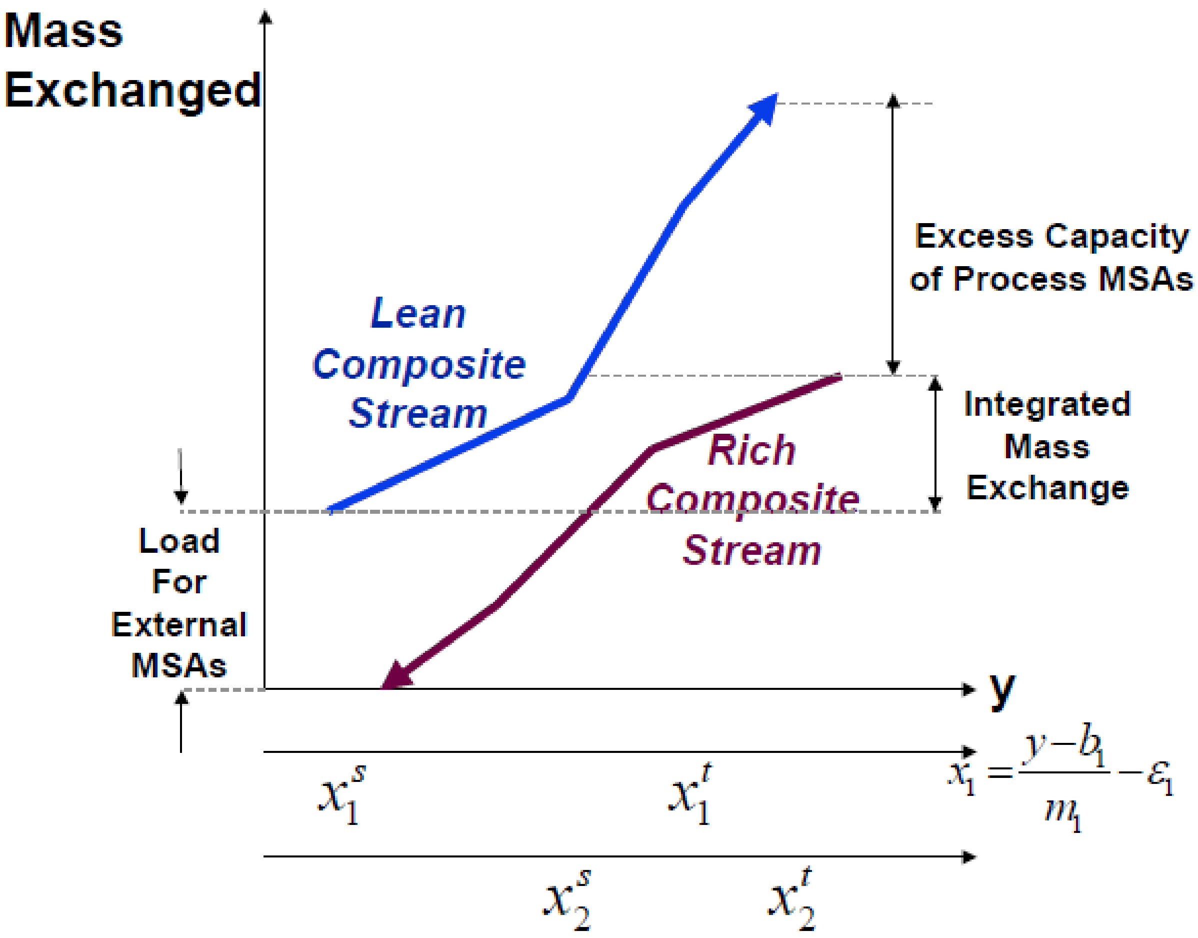
No Integration between Rich and Process MSAs

- The lean composite stream can be moved up and down **which implies different mass exchange decisions**.

- For instance, if we move the lean composite stream upwards in a way that leaves no horizontal overlap with the rich composite stream, then there is **no integrated mass exchange between the rich composite stream and the process MSAs** as seen in the above Fig.

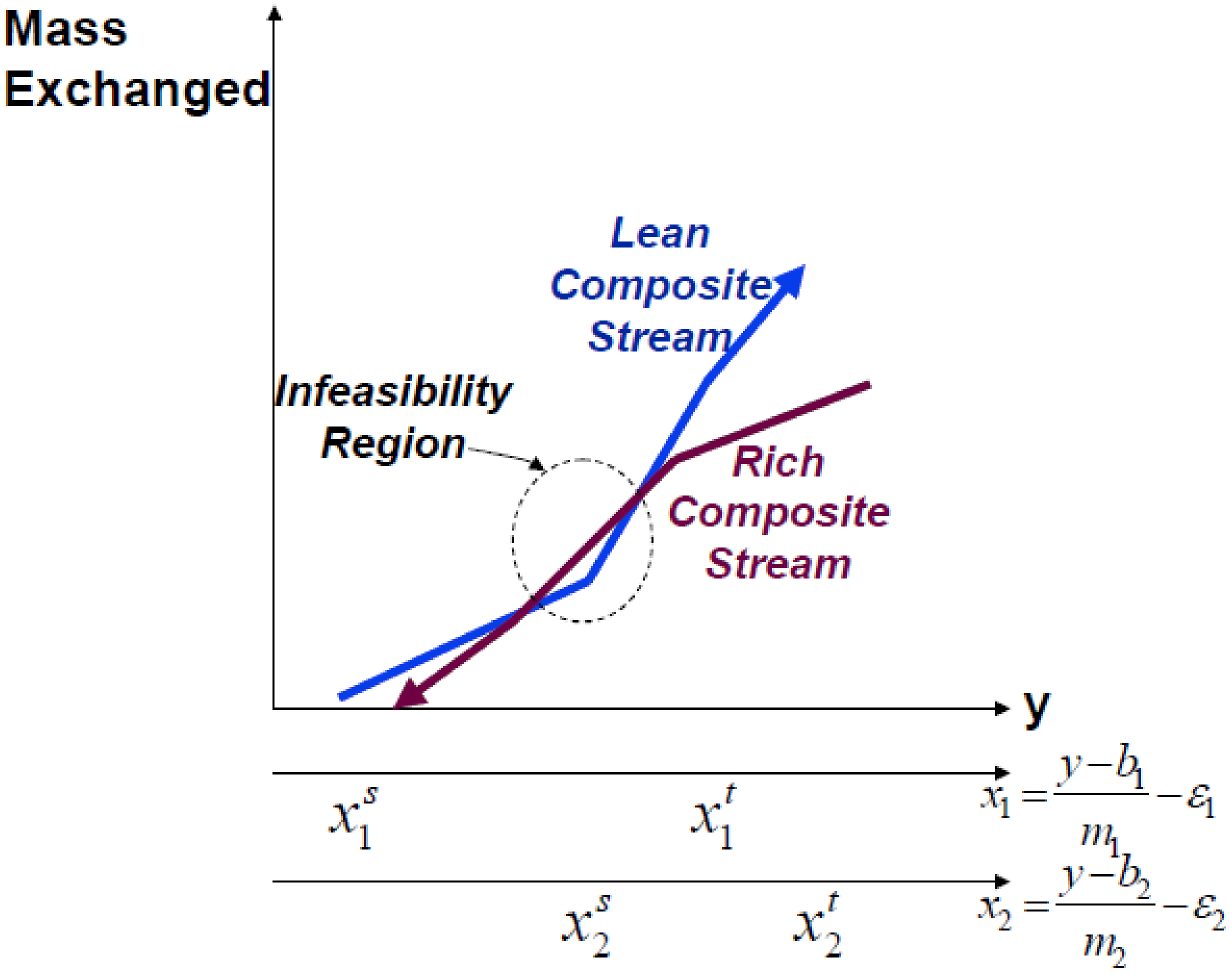
- When the lean composite stream is moved downwards so as to provide some horizontal overlap (see next Fig.), some **integrated mass exchange can be achieved**.

- The remaining load of the rich composite stream has to be removed by the external MSAs.



Partial Integration of Rich and Lean Streams (Passing Mass through the Pinch)

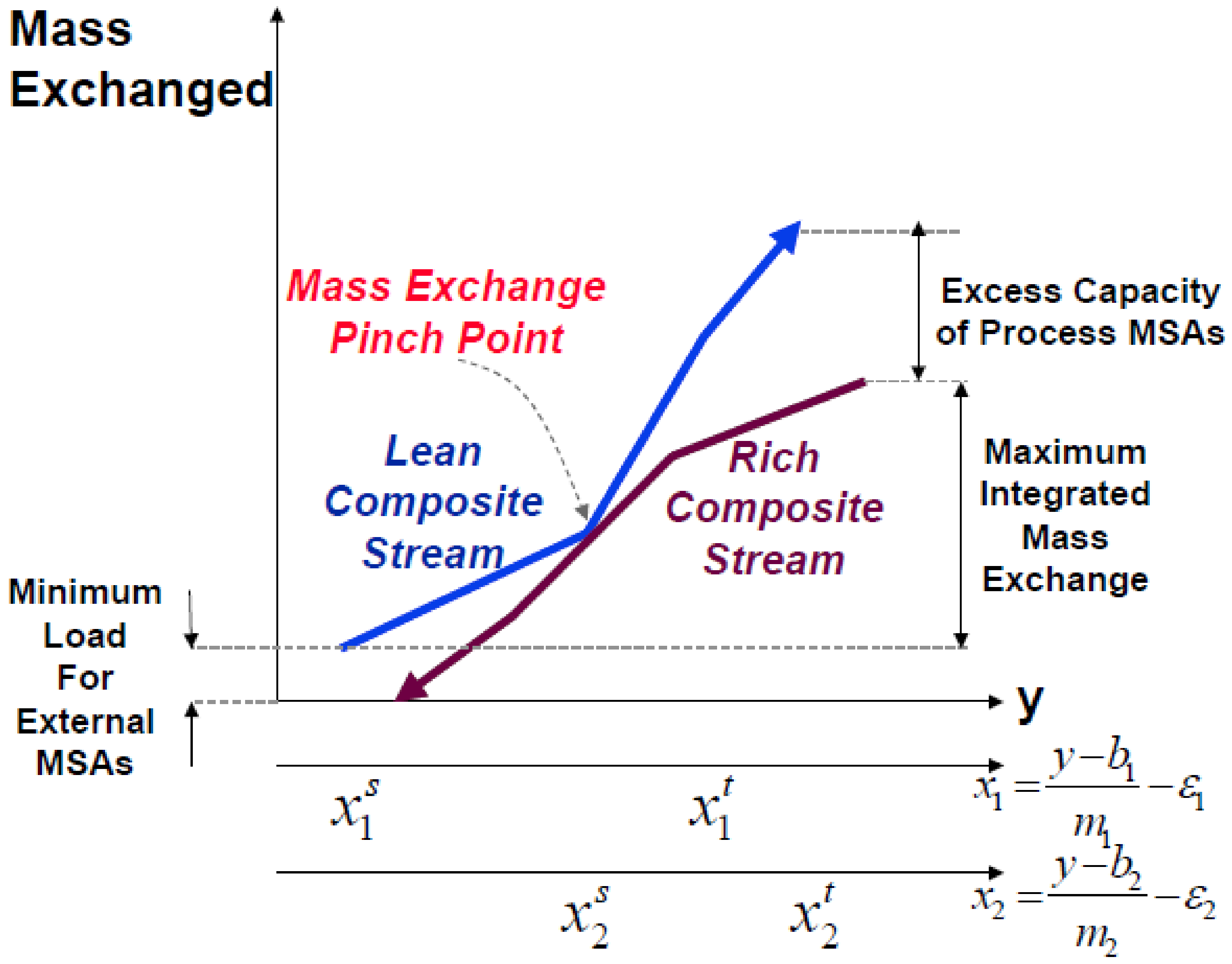
- However, if the lean composite stream is moved downwards such that a portion of the lean is placed to the right of the rich composite stream, **thereby creating infeasibility** (see next Fig.).



Causing Infeasibility by Placing Lean to the Rich of Rich

- Therefore, the **optimal situation is constructed when the lean composite stream is slid vertically until it touches the rich composite stream while lying completely to the left of the rich composite stream at any horizontal level**.

- The point where the two composite streams touch is called the "**mass-exchange pinch point**".; hence the name "pinch diagram“ (see next figure)



The Mass-Exchange Pinch Diagram

- On the pinch diagram, the vertical overlap between the two composite streams represents the **maximum amount of the targeted species that can be transferred from the rich streams to the process MSAs**. It is referred to as the "integrated mass exchange."

- The vertical distance of the lean composite stream which lies above the upper end of the rich composite stream is referred to as "excess process MSAs." It corresponds to **that capacity of the process MSAs to remove the targeted species that cannot be used because of thermodynamic infeasibility**.

- The above discussion indicates that in order to achieve the targets for maximum integration of mass exchange from rich stream to process MSAs and minimum load to be removed by the external MSAs, the following **three design rules are needed:**

  - **No mass should be passed through the pinch (i.e. the two composites must touch)**

  - **No excess capacity should be removed from MSA's below the pinch**

  - **No external MSAs should be used above the pinch**

- Above the pinch, exchange between the rich and the lean process streams takes place. External MSAs are not required.

- Using an external MSA above the pinch will incur a penalty eliminating an equivalent amount of process lean streams from service.

- On the other hand, below the pinch, both the process and the external lean streams should be used.

- Therefore, to minimize the cost of external MSAs, mass should not be transferred across the pinch.