Separation Process Engineering

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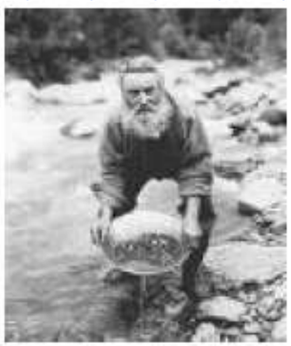
Processes and plants

- Process
  - All the **transformations** that allow to get the final product from raw materials.
- Plant
  - All the **equipment's** in which transformations take place: i.e. the physical realization of the process.
- Process development
  - A process development start from a well defined **project idea** (a patent, or technological knowledge of a company)
  - The project idea gives all the necessary information for the **operative conditions** for the transformation of interest
  - This is the starting point of the process development

What is Separation and Separation Processes

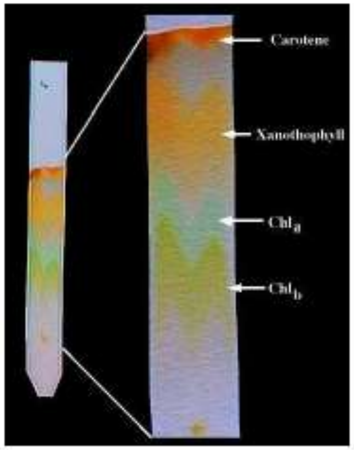
- Separate (definition from a dictionary)
  - to **isolate** from a mixture; [extract]
  - to **divide** into constituent parts

- Separation process
  - In chemistry and chemical engineering, a separation process is used to transform a **mixture** of substances into two or more **distinct products**.
  - The specific separation design may vary depending on what chemicals are being separated, but **the basic design principles** for a given separation method **are always the same**

Separations

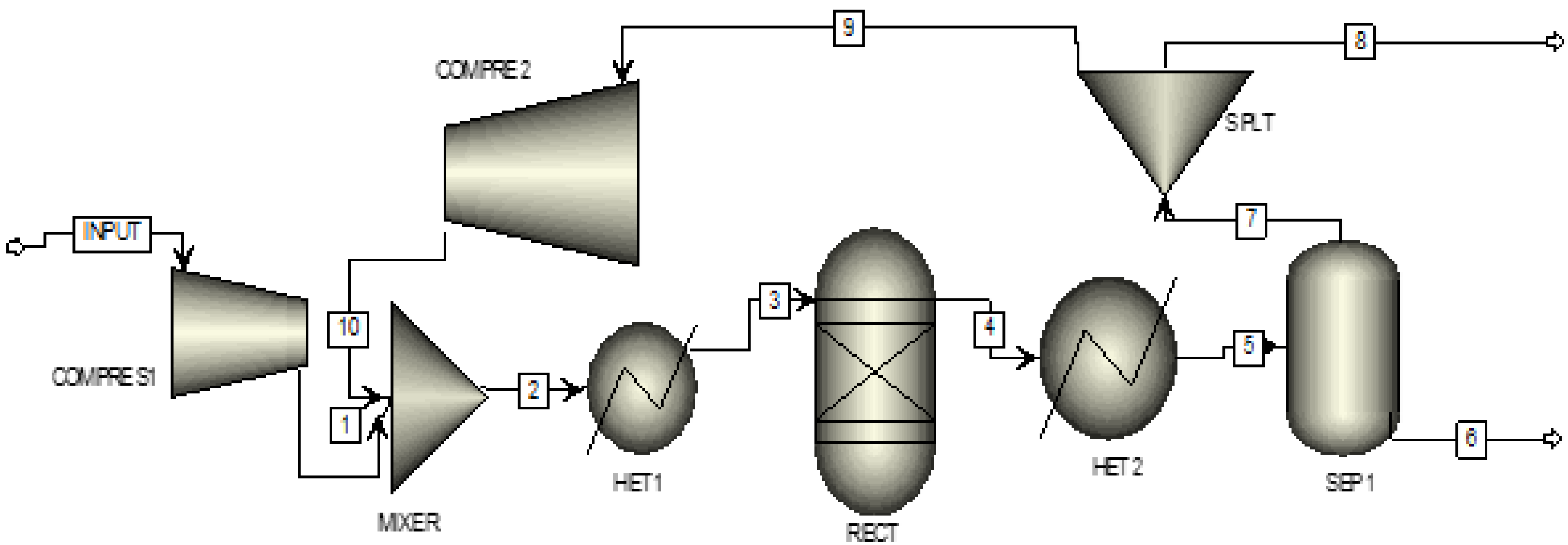
- Separations includes
  - **Enrichment** – Concentration
  - **Purification**
  - **Refining** – Isolation
- Separations are important to chemist & chemical engineers
  - **Chemist**: analytical separation methods, small-scale preparative separation techniques
  - **Chemical engineers**: economical, large scale separation methods





Why Separation Processes are Important ?

- Almost every element or compound is found naturally in an impure state such as a **mixture** of two or more substances. Many times the need to separate it into its **individual components** arises.
- A typical chemical plant is a chemical reactor surrounded by **separators**.

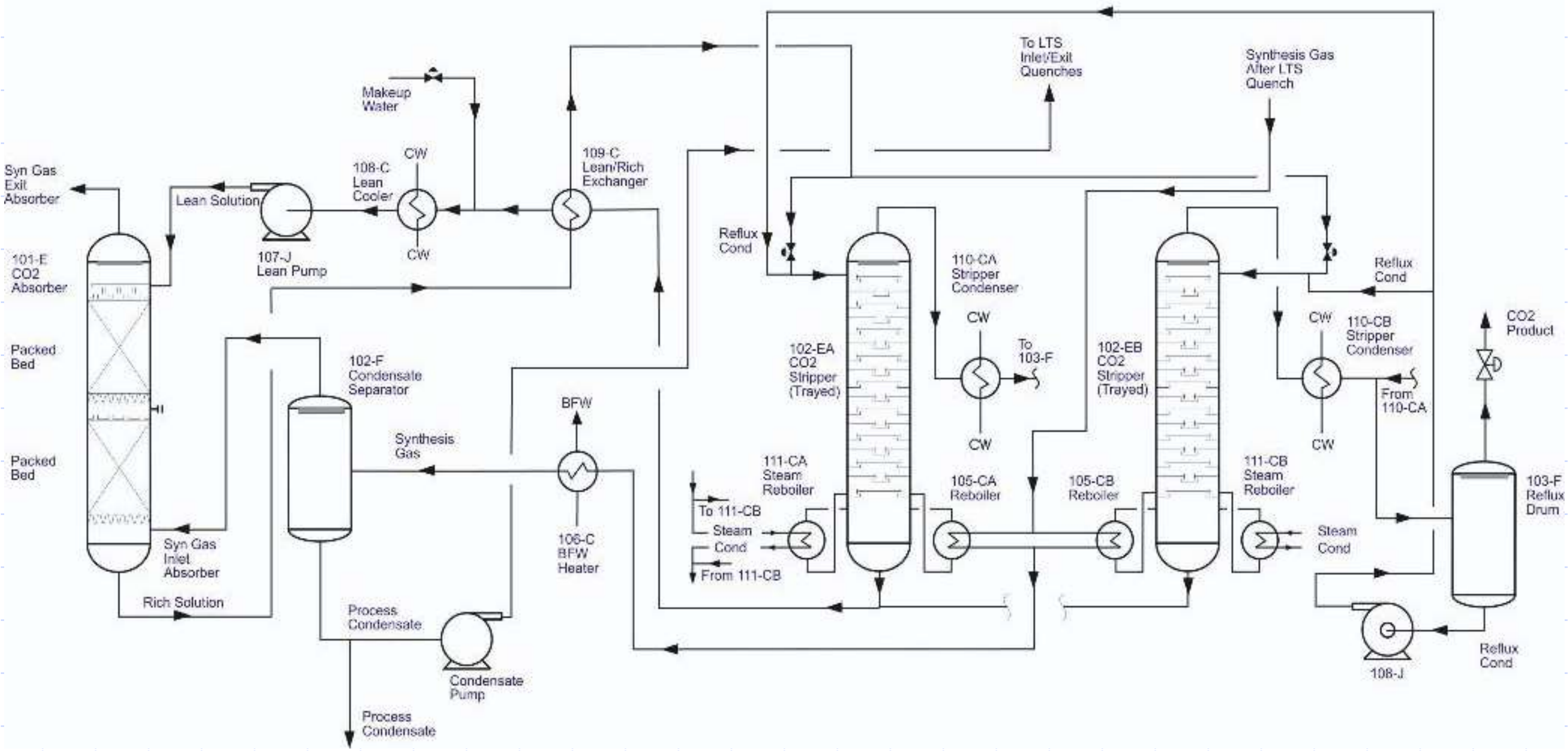


- Chemical plants commonly have 50-90% of their capital invested in separation equipment.

Separations – Why Needed?

- Separations are employed in chemical plants
  - where the separation of a mixture is required to obtain a **relatively pure chemical species.**
- Separations are usually **closely integrated** with other unit operations in the process flow,
  - obtaining **feeds from other unit operations**
  - and **separating the desired product**
  - or providing the required **product streams for feed to other units**.
- The number of other unit operations is often small compared to the number of unit operations involving separations
  - it is often relatively **easy to make something,**
  - but the subsequent **separation of the desired component** is often the most involved process!

Typical CO_2 removal system



Why Separation is Difficult to Occur?

- Second law of thermodynamics
  - Substances tend to **mix together naturally and spontaneously**
  - All natural processes take place to **increase the entropy**, or randomness, of the universe
  - To separate a mixture of species into products of different composition, we must **supply the equivalent of energy** (heat or work)

How Separations are Achieved?

- Enhancing the mass transfer rate of certain species
- Rate of Separation: *how fast ?*
  - Governed by **mass transfe**r (Rate-controlled separation)
- Extent of Separation: *how far ?*
  - Limited by **thermodynamics** (Equilibrium-staged separation)
- Properties of Importance

Molecular Properties	Thermodynamic and Transport Properties
Molecular weight van der Waals volume & area Molecular shape (Acentric factor) Dipole moment Polarizability Dielectric constant Electric charge Radius of gyration	Vapor pressure Solubility Adsorptivity Diffusivity

Data sources: Handbooks, journals, electronic databases, commercial process simulators

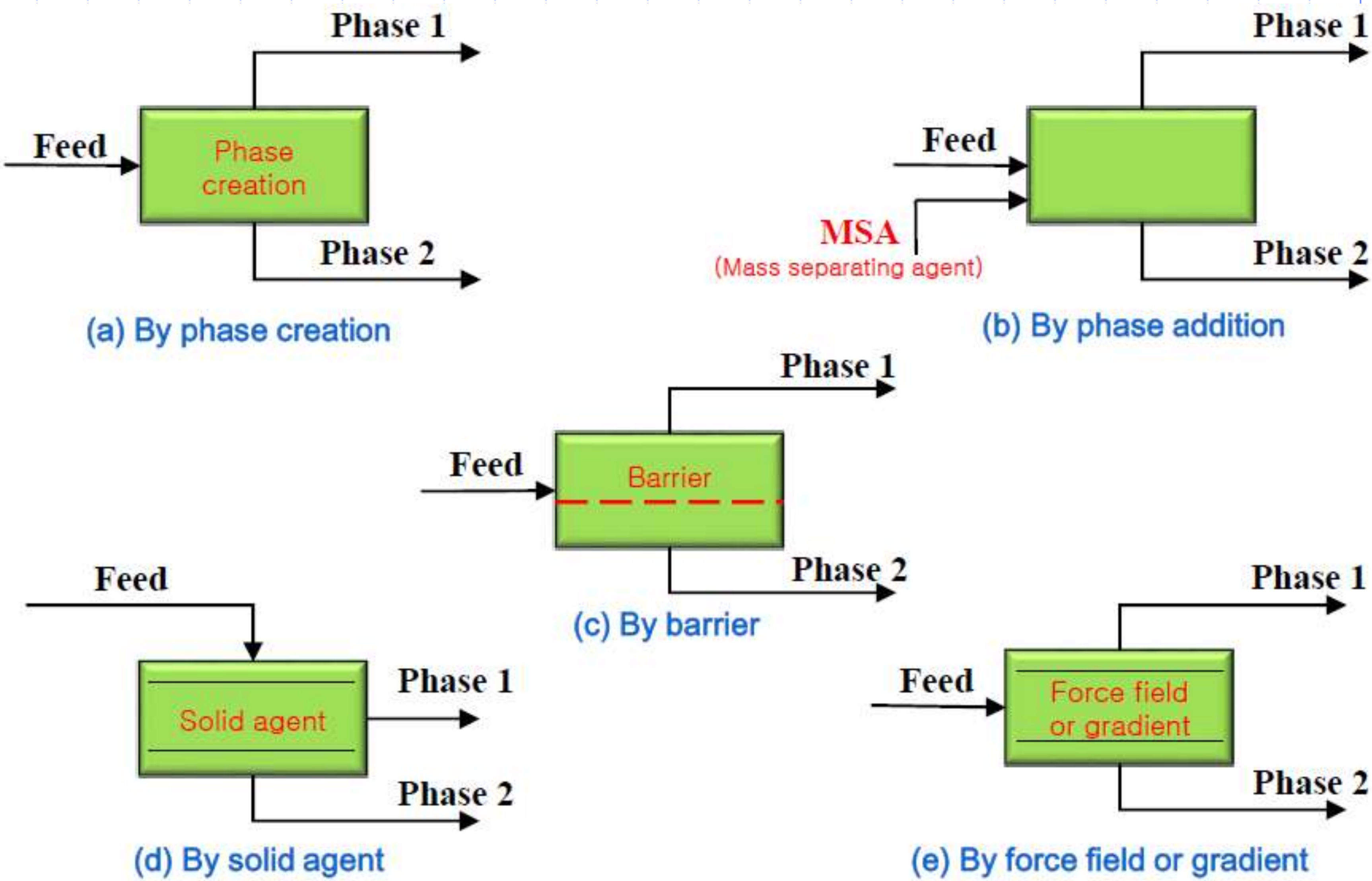
Which Separation Process Will You Choose?

- There are numerous approaches for separation.

  - **Supply heat** and boil water off, condensing the water at a lower temperature.
  - **Supply refrigeration** and freeze out pure ice, melting the ice at a higher temperature.
  - Pump the water to a **higher pressure** and force it through a thin solid membrane.

⇒ Consider product requirement, cost, environmental effects, etc.

General Separation Techniques



Separation processes

- Component separation from an homogenous mixture always requires the existence of a **second phase**
- The second phase
  - may be **added** (absorption, stripping, LL extraction)
  - may be **created** by heating / cooling (distillation)
- Component distribution among phases is crucial

- Criteria for the Selection of a Separation Method

- **Energy Separation Agent** (ESA)
  - Phase condition of feed
  - Separation Factor
  - Cost

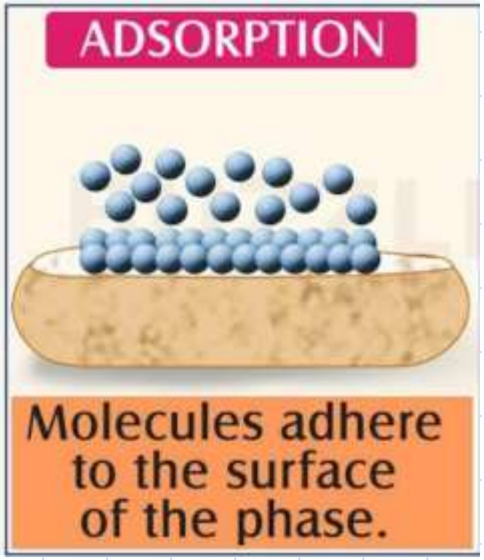
- **Mass Separation Agent** (MSA)
  - Phase condition of feed
  - Choice of MSA Additive
  - Separation Factor
  - Regeneration of MSA
  - Cost

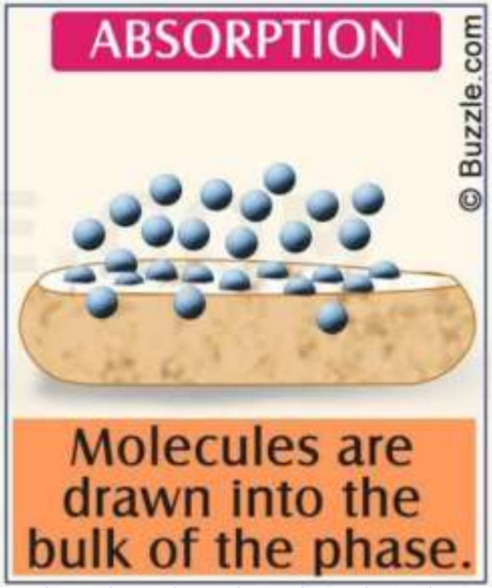
Separation Processes

- **Absorption** – Solutes removed from a gas into a liquid
  - Solutes removed from liquid into gas is called ***stripping*** *or desorption*
- **Distillation** – Thermal vapor-liquid separation processes (Ch 11); vapor phase generated from liquid
- **Liquid-liquid extraction** – Solute extracted from liquid A into an immiscible liquid B (a solvent)
- **Leaching** (extraction) – Solute extracted from a solid into a solvent phase (liquid, dense gas, or supercritical fluid)
- **Membrane processing** – Molecules separated using a dense (non porous film) or porous physical barrier
- **Filtration** – Suspended solids separated from a liquid or gas phase using a porous membrane

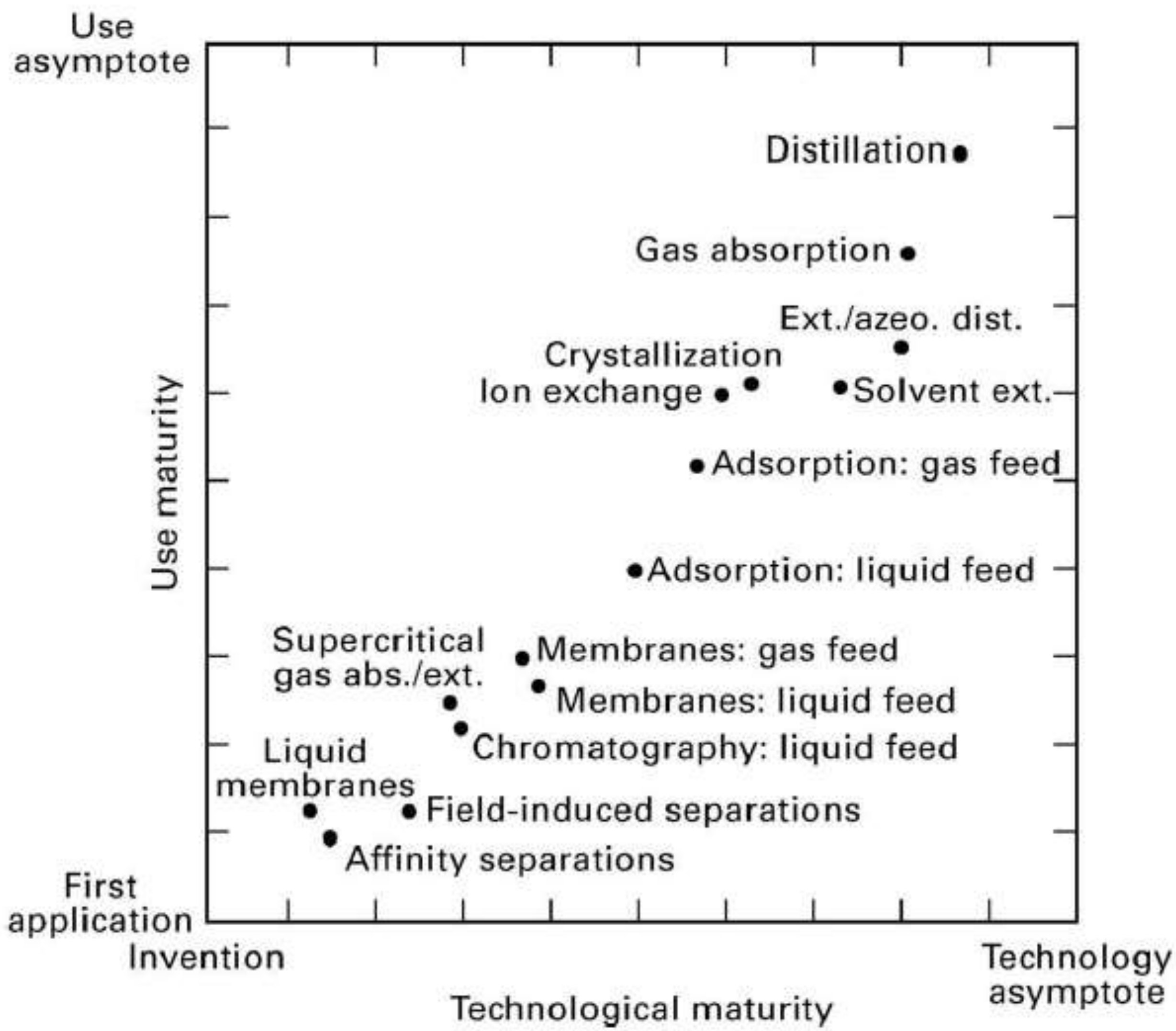
Adsorption vs. Absorption – D vs. B

- **ADsorption Process**
  - Components from a gas phase or a liquid phase are attached to the **surface of a solid phase** when the gas phase or the liquid phase is brought in contact to the solid phase. **Adsorption is a surface phenomenon.**
- **ABsorption Process**
  - Components **from a gas phase transfer into a liquid phase** when the gas phase and liquid phase are brought into contact.
- **Chemical vs Physical processes**
  - Adsorption and absorption may be **physical or chemical processes**, depending upon the fact that a chemical reaction may occur during the phase transfer





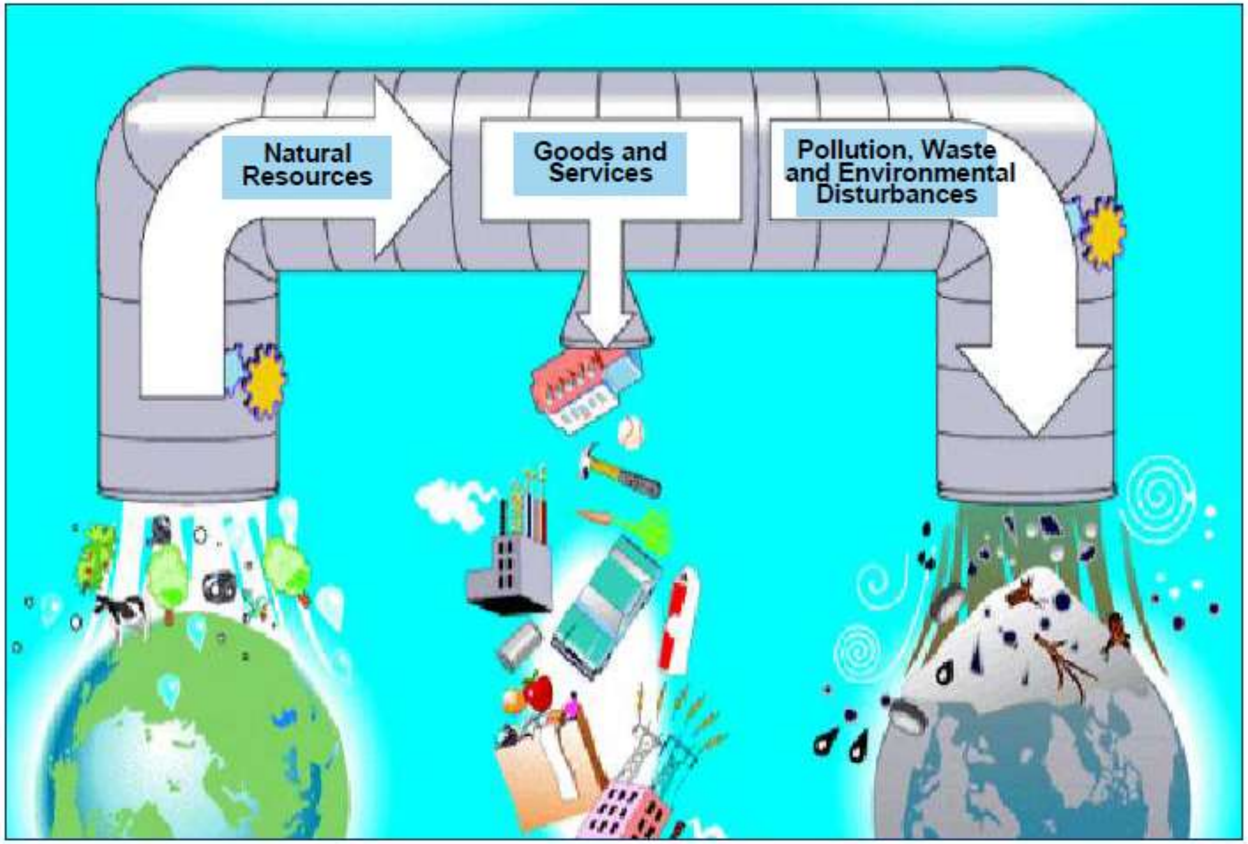
Technological and Use Maturities of Separation Processes



Seader & Henley, Separation Process Principles

Motivation: are our products and processes sustainable?

Only 25 wt% of what goes into the pipe comes out as goods and services – scope for significant improvements



Adapted from Drioli, 2005

Without significant improvements, our products-processes are not sustainable

A vision… how a future plant may look vs. a conventional plant.

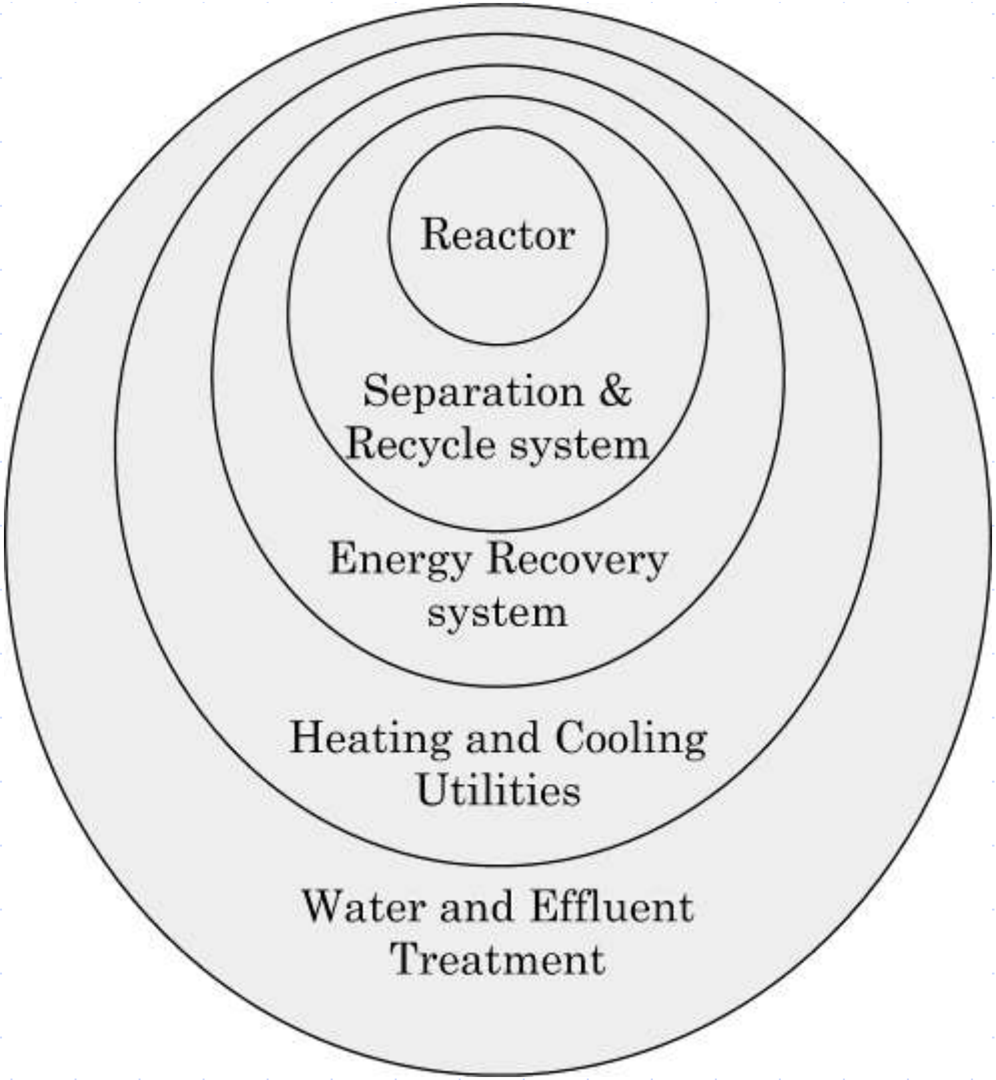
(Rendering courtesy of DSM)

OPERATING with NON POLLUTING PROCESSES involving

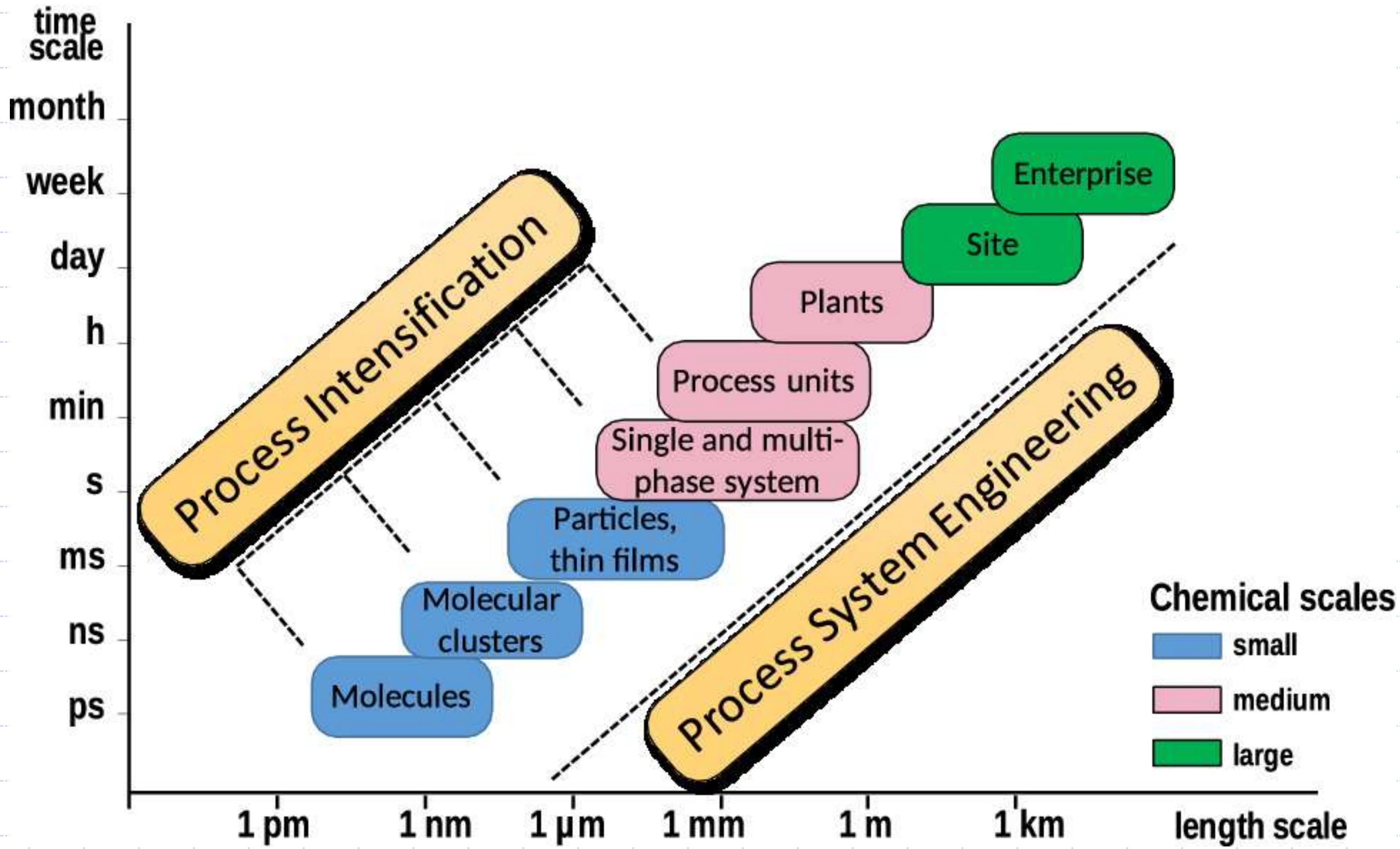
PROCESS INTENSIFICATION

SAVINGS ABOUT 30 % (RAW MATERIALS + ENERGY + OPERATING COSTS)

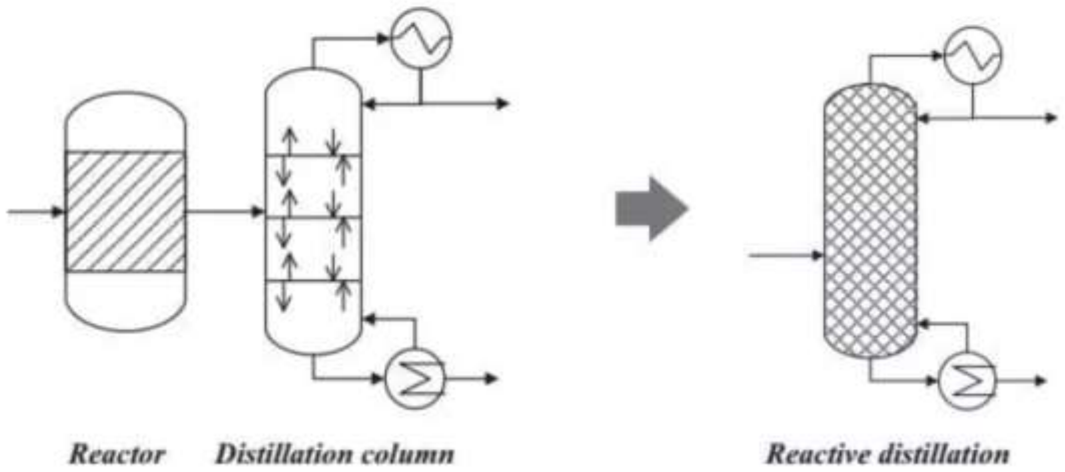
A process is more than just reaction and separation …

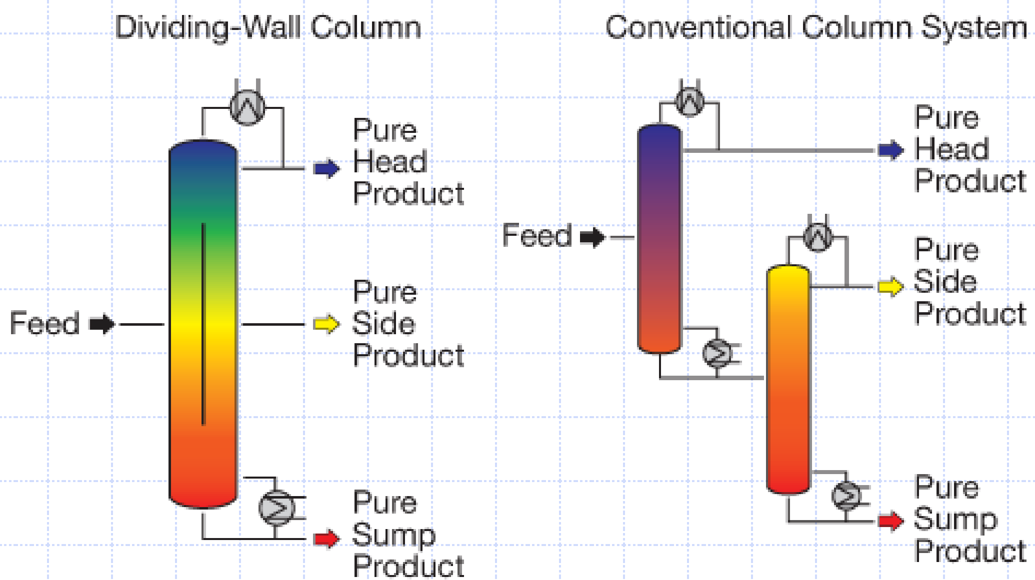


Process intensification & Process system engineering



Process intensification: simple examples

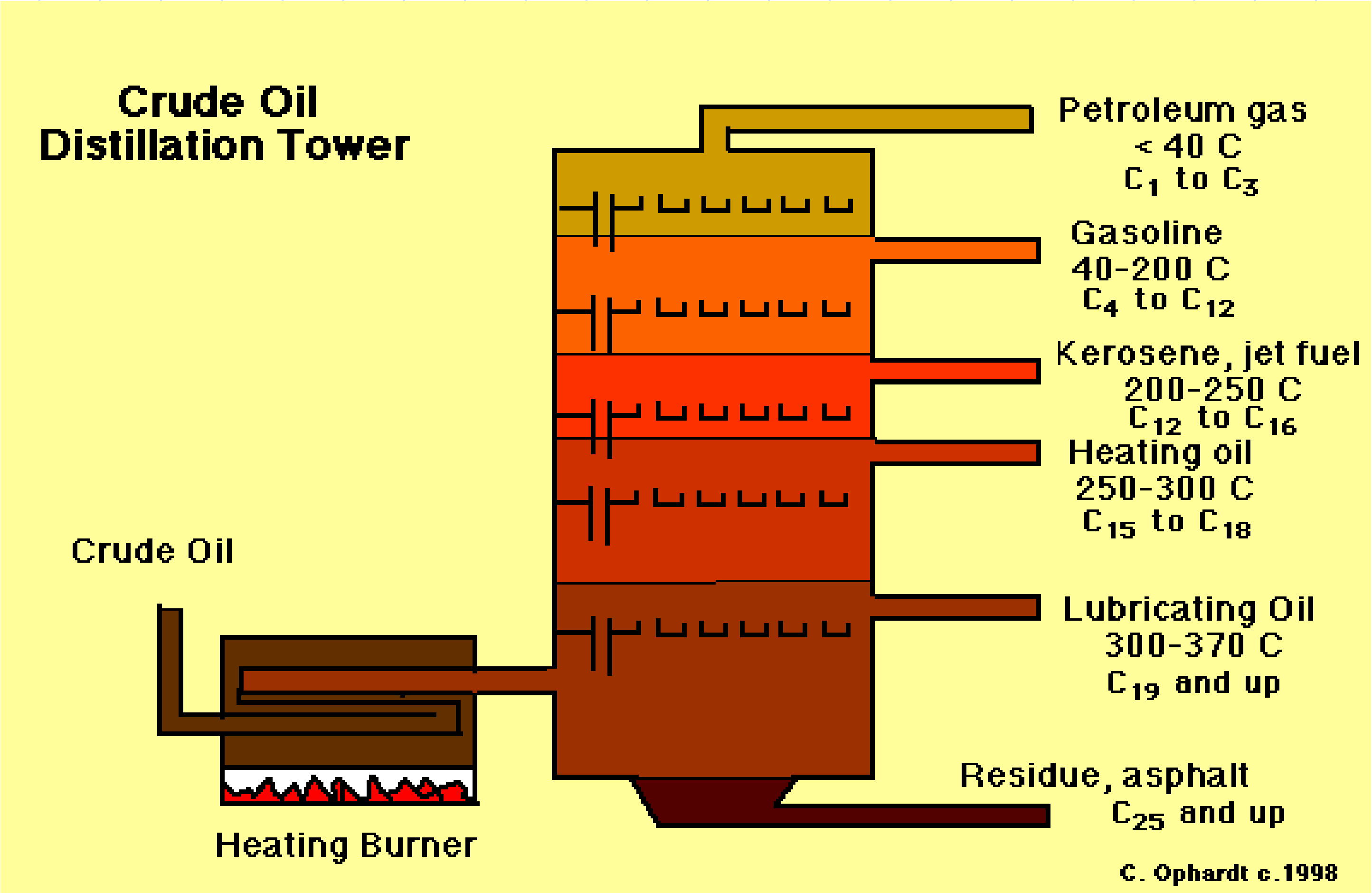




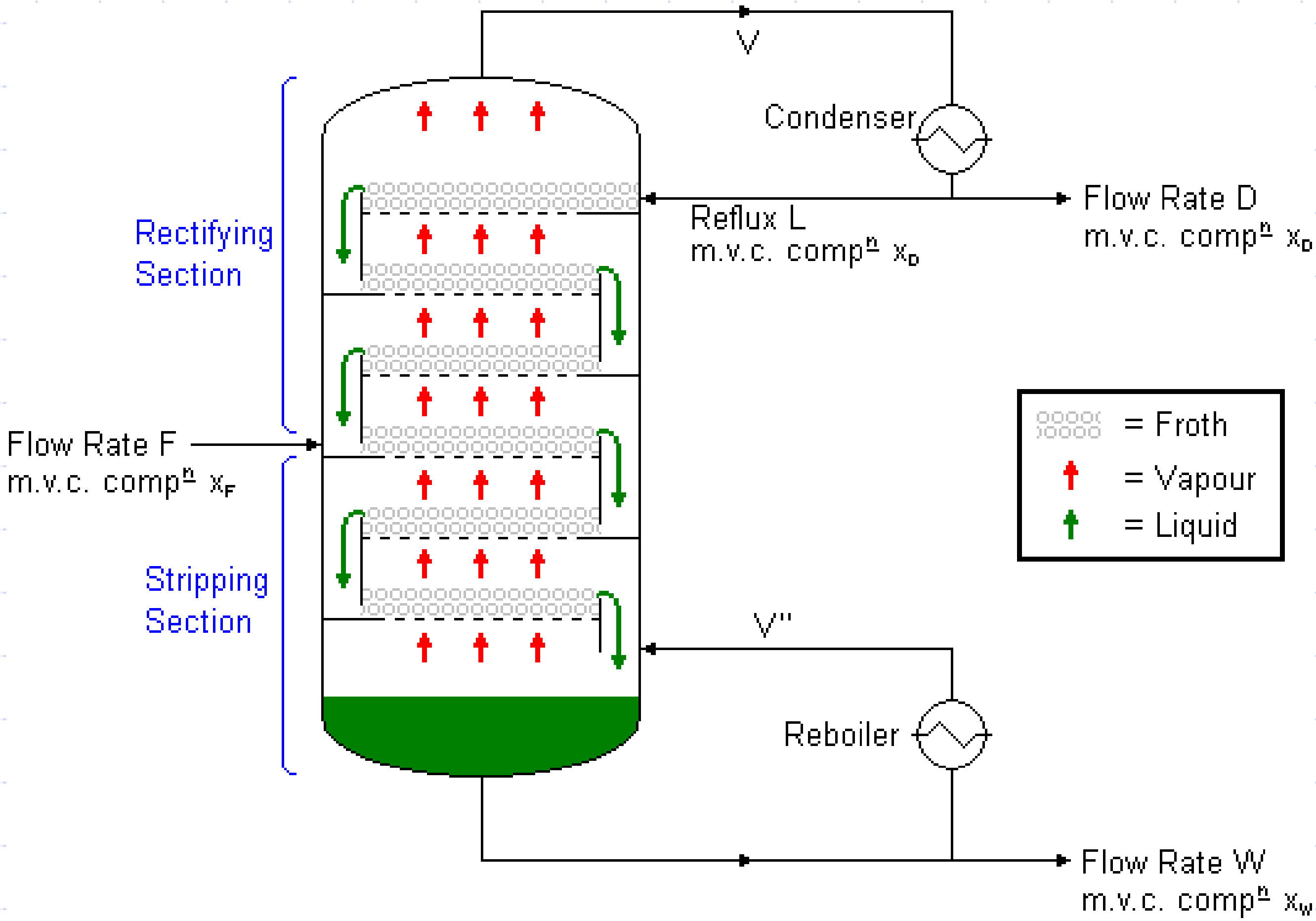
Equilibrium Staged Separations

- What do we mean by **separations?**
- What do we mean by **equilibrium?**
- What do we mean by **staged?**

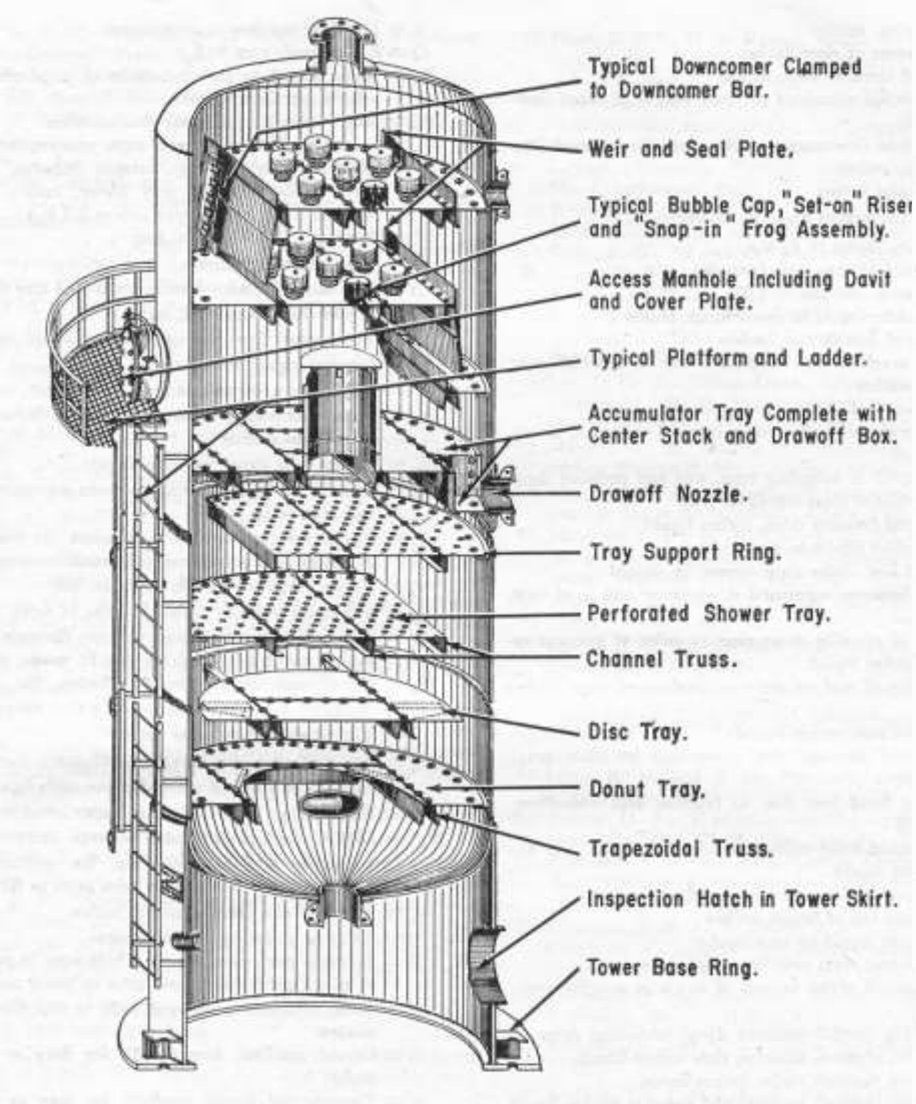
Separations – Distillation

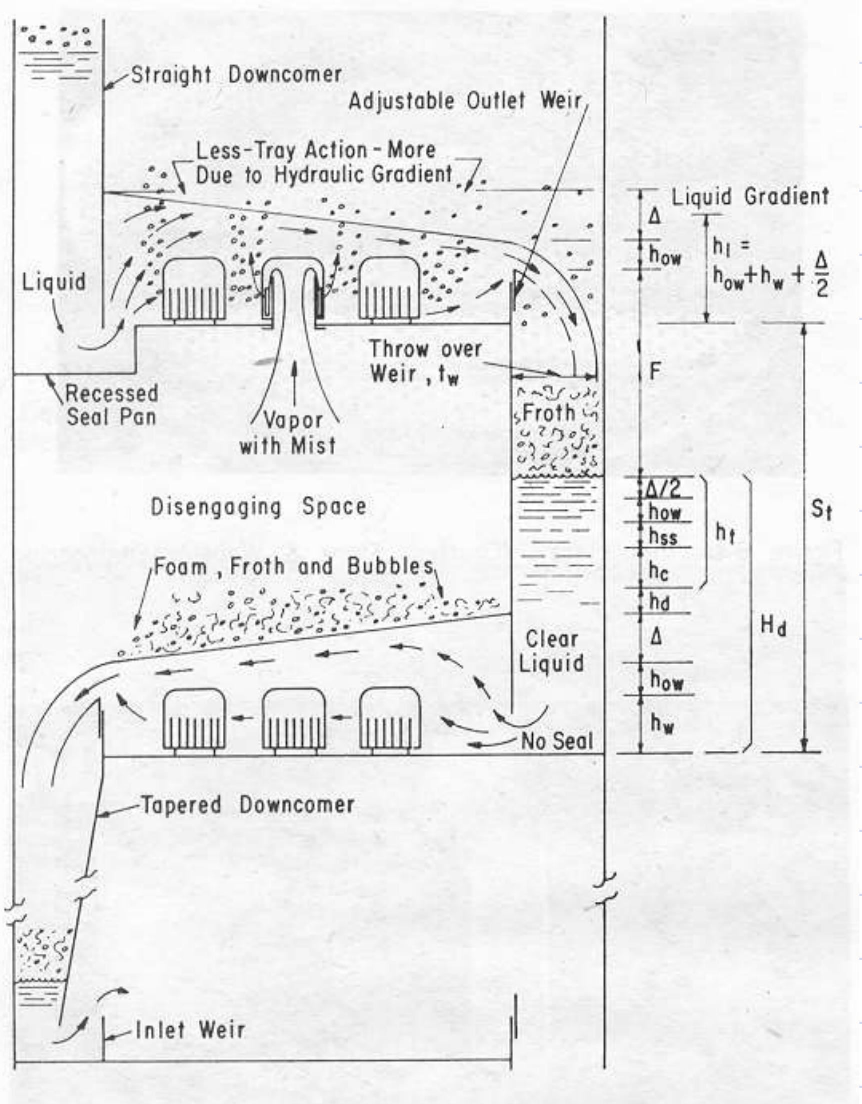


Staged Separations – Distillation

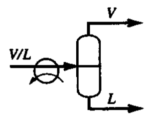
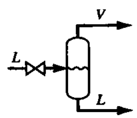
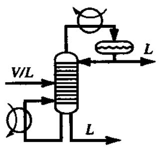
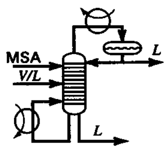
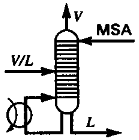
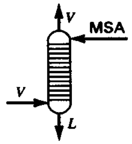
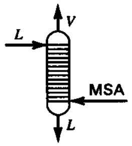


Separations – Distillation Design

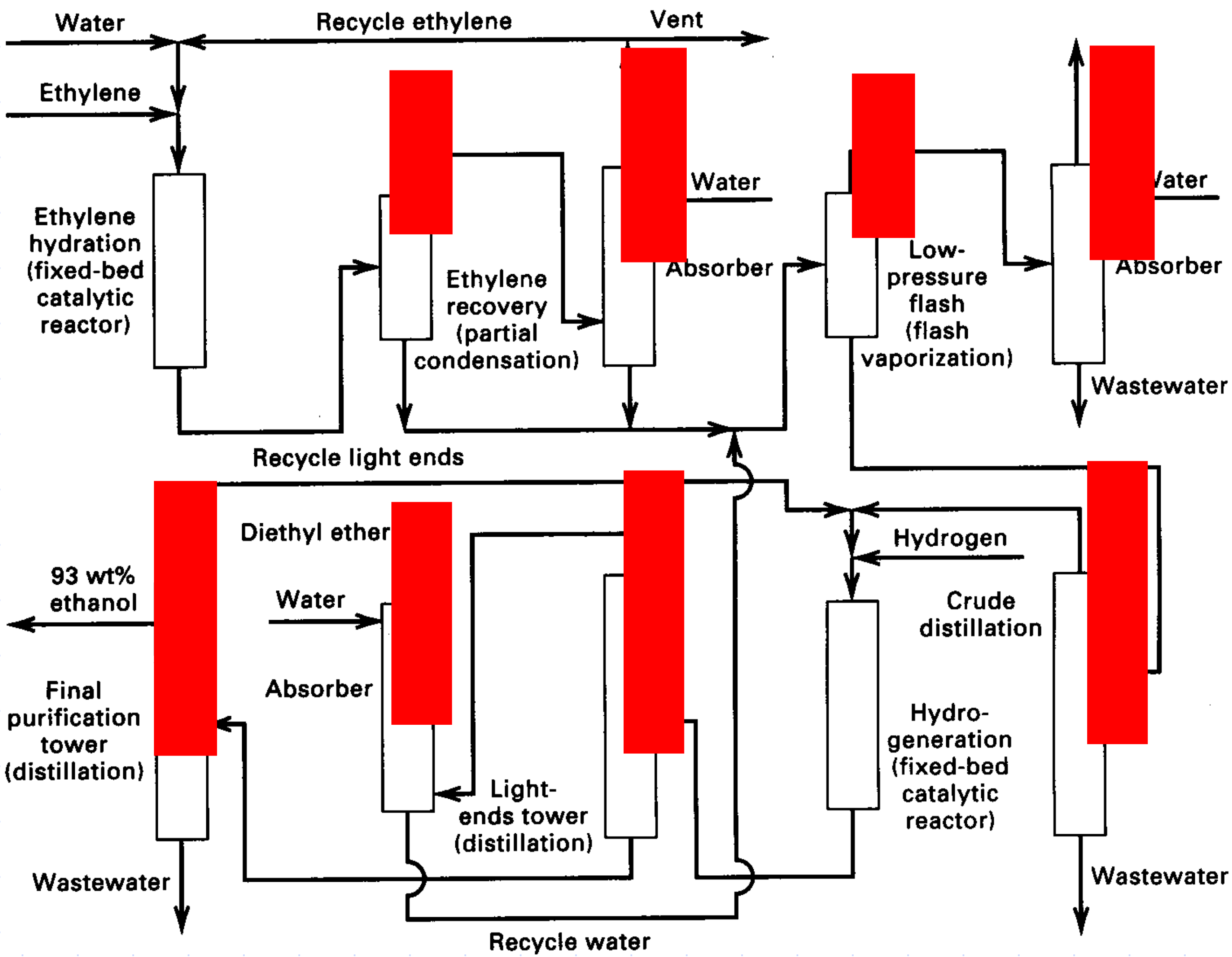




Separation Operations based on phase creation or addition

Separation Operation	Symbol[a]	Initial or Feed Phase	Created or Added Phase	Separating Agent(s)	Industrial Example[b]
Partial condensation or vaporization* (1)		Vapor and/or liquid	Liquid or vapor	Heat transfer (ESA)	Recovery of H_2 and N_2 from ammonia by partial condensation and high-pressure phase separation (Vol. 2, pp. 494–496)
Flash vaporization* (2)		Liquid	Vapor	Pressure reduction	Recovery of water from sea-water (Vol. 24, pp. 343–348)
Distillation* (3)		Vapor and/or liquid	Vapor and liquid	Heat transfer (ESA) and sometimes work transfer	Purification of styrene (Vol. 21, pp. 785–786)
Extractive distillation* (4)		Vapor and/or liquid	Vapor and liquid	Liquid solvent (MSA) and heat transfer (ESA)	Separation of acetone and methanol (Suppl. Vol., pp. 153–155)
Reboiled absorption* (5)		Vapor and/or liquid	Vapor and liquid	Liquid absorbent (MSA) and heat transfer (ESA)	Removal of ethane and lower molecular weight hydrocarbons for LPG production (Vol. 14, pp. 384–385)
Absorption* (6)		Vapor	Liquid	Liquid absorbent (MSA)	Separation of carbon dioxide from combustion products by absorption with aqueous solutions of an ethanolamine (Vol. 4, pp. 730–735)
Stripping* (7)		Liquid	Vapor	Stripping vapor (MSA)	Stream stripping of naphtha, kerosene, and gas oil side cuts from crude distillation unit to remove light ends (Vol. 17, pp. 199–201)

Industrial process for hydration of ethylene to ethanol



Separations – Why Design?

- Why Design?
  - The first goal in separation design is to **obtain the required products** from the given feeds.
  - The second goal is to **minimize the cost of the equipment** – that is design it such that it works, but also such that it is not oversized or undersized – thus, minimizing construction costs and problems.
  - The third goal is to **minimize operating costs** since separations, such as distillation – the most common separation method – consume enormous amounts of energy, often up to 50% of a plant's operating costs.
- Mechanism of separation
  - One takes advantage of **differences in the chemical and physical properties** of chemical species to enable their separation – these differences can involve their molecular, thermodynamic, and transport properties.
  - If these differences are not significantly large at given conditions, one may increase the magnitude of these differences by **altering the system conditions or by the addition of other species**.

Separation via Thermodynamic Properties

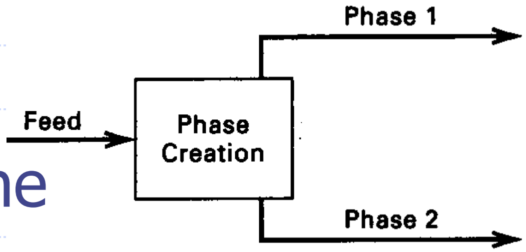
- One of the most powerful differences that we can take advantage of for separation is the differences in the **thermodynamic properties** of different chemical species.
- The thermodynamic properties of the chemical species can be altered by **changes in the system temperature and pressure**, which often can be readily and relatively easily varied in a process.
- What thermodynamic properties?
  - The **relative volatility** of chemical species at a particular temperature and pressure is one of the most useful thermodynamic properties for separation.
  - Volatility is the **tendency of a chemical species to vaporize to a gas**; thus, it is related to its boiling point.
  - Most chemical species have **a different volatility** or boiling point than others.

Separations – Distillation

Crude Oil Refining		
Distillate Fraction	**Boiling Point (°C)**	**Carbon Atoms per Molecule**
Gases	below 30	1-4
Gasoline	30-210	5-12
Naphtha	100-200	8-12
Kerosene & Jet Fuel	150-250	11-13
Diesel & Fuel Oil	160-400	13-17
Atmospheric Gas Oil	220-345	
Heavy Fuel Oil	315-540	20-45
Atmospheric Residue	over 450	over 30
Vacuum Residue	over 615	over 60

Vapor-Liquid Phase Separations

- If a mixture of components is allowed to separate into vapor and liquid phases, **the more volatile component** – the one with the lower boiling point – will tend to be **more highly concentrated in the vapor phase.**



- If we then separate the vapor from the liquid, we have **increased the concentration** of the component in the vapor phase with respect to the liquid phase.
- The separation of the vapor from the liquid is readily accomplished by their **differences in density** – a vapor phase comes of the top and the liquid phase off the bottom of the separator.
- It is that simple! Except that…

Equilibrium: the Determining Separation Factor

- The ultimate concentrations of the chemical species with respect to the phases are determined by **thermodynamic equilibrium** for a given set of conditions.
  - Given the temperature, pressure, and concentrations of a mixture, we can use equilibrium relationships to determine the liquid and vapor phase concentrations of the chemical species as they separate.
  - By assuming equilibrium in our separation designs, we can thus solve design problems.

Staged Separations

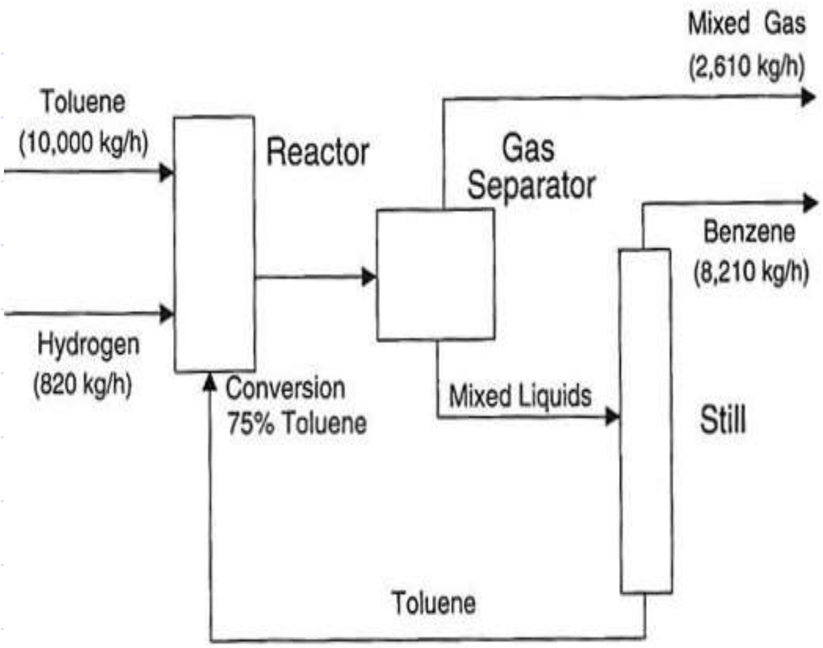
- **Equilibrium is the determining factor** as to what concentrations can be obtained in the liquid and vapor phases at a given set of conditions.
- **Equilibrium behavior**, and thus the concentrations in the liquid and vapor phases, can be changed by altering the conditions of the system – for example, **the temperature and/or pressure**.
- Multiple separations may be **employed in series**, each at different conditions, to take advantage of even slight differences in the equilibrium concentrations to ultimately obtain high levels of separation.
- Each separator can be thought of as an **equilibrium "stage"** in the overall separation.
- We can even combine these stages **into an overall single separator**, as we will see, for example, in distillation.

Process schemes

- Block Flow Diagram (BFD): **illustrative, very general**
- Process Flow Diagram (PFD): technical, they allow design
  - **Simplified**: reports unit operations and necessary equipment
  - **Quantified**: add to the simplified flow diagram material and energy balances
  - **Instrumental**: add the quantified flow diagram the control systme
- Piping and Instrumentational Diagram (P&I)
  - **Main document** of the design, the PFD is enhanced and quantified:
  - **Instrumentation** and **control** system is added
  - Is a '**technical photo**' of the process
  - Allows to identify and **recognize all units and piping** of the plant

Block Flow Diagram BFD

- Series of boxes labeled with the **name of the operation**
- **Streams** indicated as continuum lines with arrows
  - At least two **sections**: reaction and separation
  - At least 2 **streams**: feed and product
- Usually at least **one recycle** and **one purge**

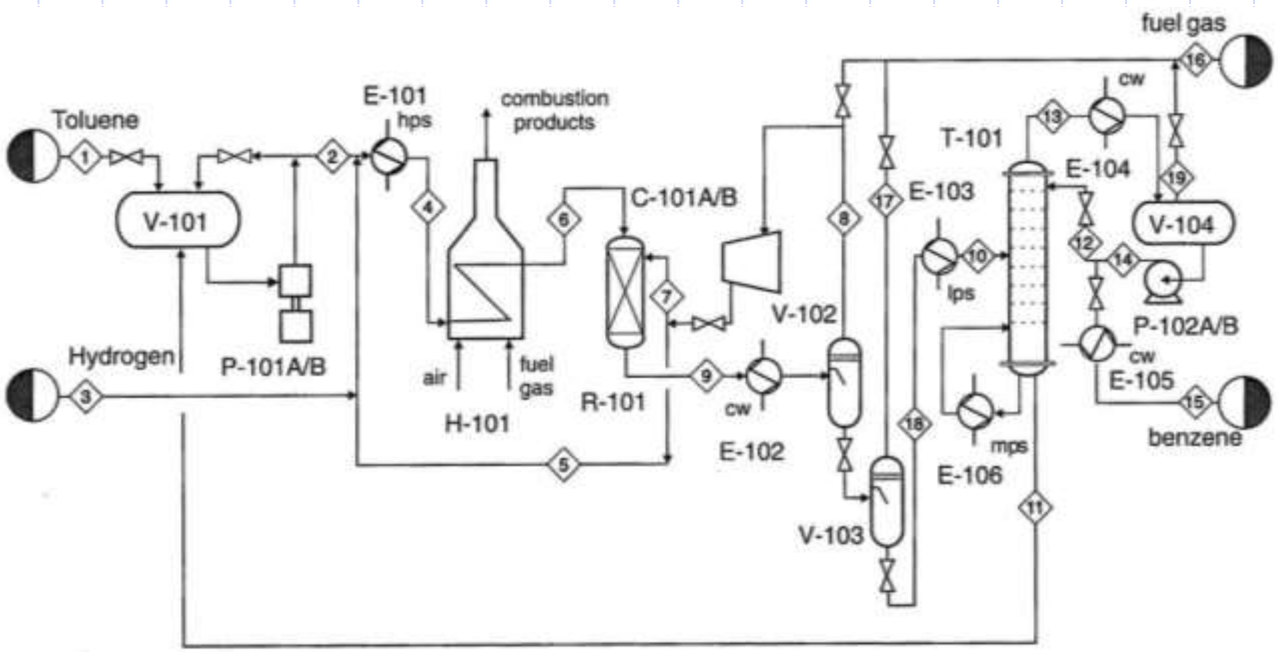


- More BFD could be generated from one patent or know-how
  - The optimal one will be selected based on **economical considerations**

Process Flow Diagram PFD

- Complete flow diagram in which **all units and streams** involved are represented.
- Units are shown schematically and **conventionally labeled**
  - (in Italy UNICHIM - Tab. 1)

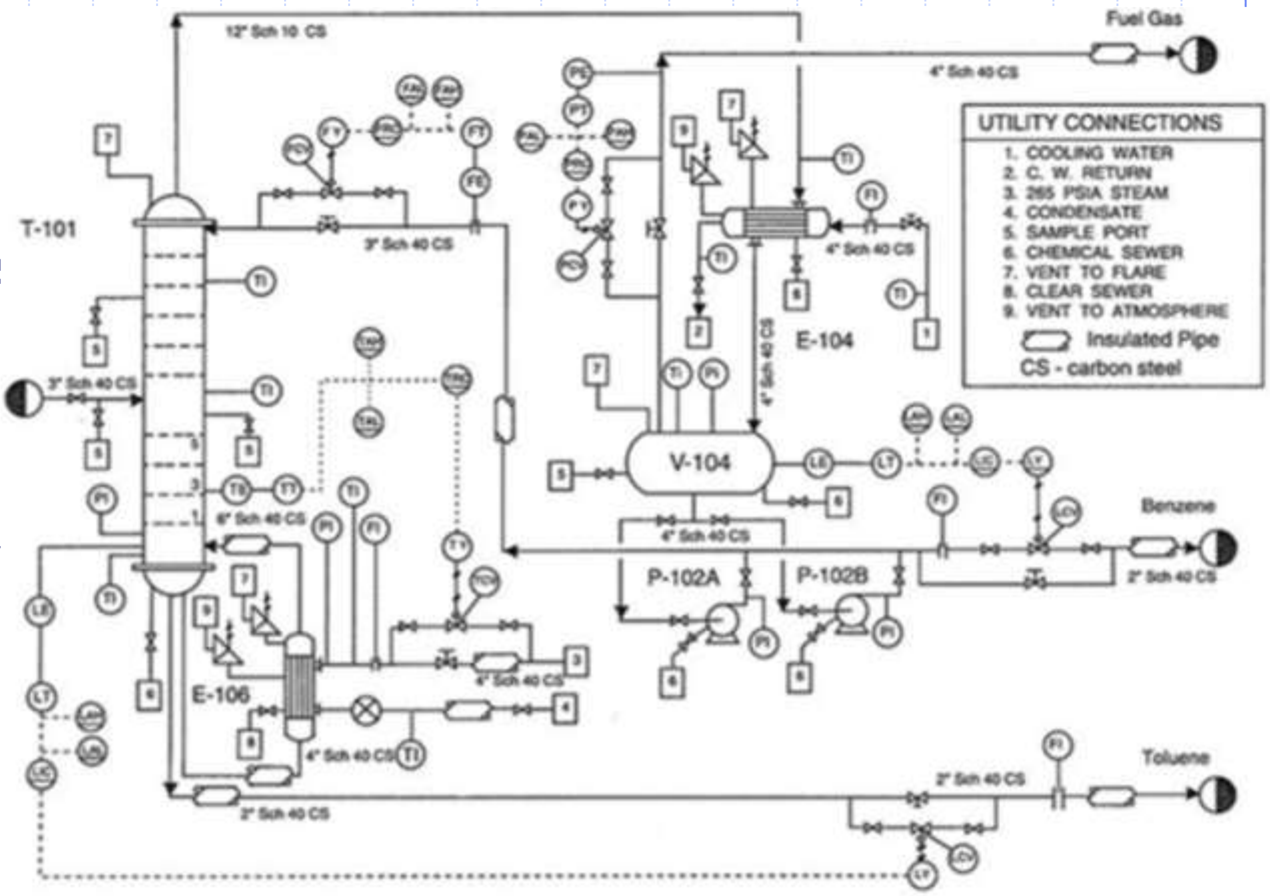
APPARECCHI	SIGLA
Agitatori	AG
Centrifughe	CE
Compressori alternativi	CA
" centrifughi	CC
" rotativi	CR
Recipienti e separatori	D
Essiccatori	DE
Scambiatori di calore	E
Eiettori	EJ
Evaporatori	EV
Forni	F
Filtri	FI
Pompe dosatrici	PA
" centrifughe	PC
" volumetriche	PV
Reattori	R
Serbatoi stoccaggio	S
Trasportatori di solidi	TS
Ventilatori	V



PFD of toluene de-alchilation process

Piping and Instrumentational Diagram P&I

- Service lines are indicated connecting the units that need the specific service
- Indication of: by-pass, pipes slopes, insulations, condensate drains, …
- Complex schema: clarity, sufficient spacing, no winding paths



P&I of a distillation column for toluene de-alchilation process

Course Objectives

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So what is this course about?

- We will …
  - Use **equilibrium relationships** to determine the phase behavior of mixtures of chemical species as they separate.
  - Use this behavior in conjunction with **mass and energy balances** to solve separation problems.
  - Incorporate **staged separations** to achieve the desired level of separation.
  - Use **mass transfer principles** for the design of columns internals
  - **Design separators** using all of the above.
- This course covers the basic "work horse" separation methods most commonly used in the chemical industry
  - **Distillation**
  - **Absorption**
  - **Extraction**
- It does **not cover** newer "advanced" separations methods:
  - Adsorption (e.g., PSA) Note the difference: AB vs. AD
  - Membrane separation
  - Electrophoresis
  - Washing, leaching, SFE
  - Chromatography, ion exchange

Main Objectives

- To provide to students **basic theoretical competence** for the design of unit operations used in the fundamental **separation processes of chemical and environmental engineering.**
- To provide to students understanding of the **underling physical and chemical phenomena** of the unit operations.

Specific Objectives

- D1. Knowledge and understanding:
  - to **solve phase equilibrium problems** for multicomponent mixtures;
  - to **select the correct thermodynamic** and **transport models** for the specific separation process;
  - to setup and **solve material and energy balances** for single stage and multi stage separation processes.
- D2. Applying knowledge and understanding:
  - to design unit operations and separation devices using **traditional methods**
  - to design unit operations and separation devices using **process simulators**.
- D3. Making judgments:
  - to select the right tools for **sizing and designing** separation unit operations
  - to describe inherent **thermodynamic** and **transport phenomena**.
- D4. Communication skills:
  - to be able to **describe and present** in an oral, written and graphical form the concepts learned in class.
- D5. Learning skills:
  - to be able to **identify all the necessary information** for the design of separation processes, including thermophysical data and thermodynamic models for the solution of the material and energy balances describing the behavior of the unit operations.

Course program

- Introduction to separation process engineering
- Phase equilibrium thermodynamic for multicomponent mixtures
- Single stage separation: flash distillation
- Introduction to column distillation
- Column distillation: internal stage –by-stage balances
- Multicomponent distillation: rigorous methods
- Multi component distillation: short-cut methods.
- Special separation processes: azeotropic, extractive, reactive distillation,
- Batch distillation
- Staged and packed column design
- Absorption and stripping
- Liquid-liquid extraction.

- Use of simulation package ASPEN+

Logistics

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Teaching methodology

- Classroom lectures, demo and hands-on sessions.
  - PPT slides available in MOODLE: https://moodle2.units.it//
  - Class exercises: separation process projects to be done in class
  - Home work: separation process projects to be done in autonomy
- Timetable
  - Monday 15.30 – 18.00 (with 15 minutes break) – Ed. B
  - Tuesday 9.30 – 12.00 (with 15 minutes break) – Ed. B
  - Thursday 8.30 – 10.00 – Room Green ed. C5
- Demo of user interface of commercial SW
  - Aspen Properties
  - Aspen Plus
- Hands on
  - Thermodynamics, physical properties and phase equilibria
  - Unit operations

Text books

- Reference text book :
  - **Wankat P.C.**:"Separation process Engineering", Prentice Hall, 4 ed. 2016.
- Other text books:
  - Seader J.D., Henley E.J.:"Separation Process Principles", John Wiley & Sons.
  - Geankoplis C.J.:"Transport Processes and Unit Operations", 3rd ed., Prentice Hall.
  - Perry R.H., Green D.W.:"Perry's chemical engineers' Handbook", 9th ed., McGraw-Hill.
  - Metcalf & Eddy: "Wastewater Engineering",McGraw-Hill
  - AspenPlus manuals

Exam methodology

- Home works
  - Assigned in class, in some cases continuation of exercises started in class
  - To be sent BEFORE a given deadline via email; in case of unjustified delay the exercise is not considered in the evaluation
- Oral Exam (at the end of the course)
  - Presentation of a separation process developed using Aspen+
- Grading (x/30) is given by the following contributions:
  - Home works 40%
  - Oral exam 60%