

Klaus Sattler, Hans Jacob Feindt

Thermal Separation Processes

Principles and Design



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Foreword

The separation of gaseous and liquid solutions into their components and the drying of wet products have always been an integral part of the manufacture of products in the chemical, petroleum, food, and pharmaceutical industries. As environmental protection has become an increasingly important consideration to industry, separation processes have become more important in direct proportion.

This book provides a clear fundamental development of the technology of important separation processes. As indicated by the title the book deals with separation processes in which heat is an input to the complete process of separating the constituents of a mixture. The flow of heat in the process is clear in distillation, crystallization and drying but is not so obvious in absorption, extraction and adsorption, where the heat flow is required to regenerate the solvent or adsorbent.

Each of these six subjects is given thorough coverage in its own chapter. These chapters follow a comprehensive development of the physical chemistry and engineering which provide the principles upon which the separation processes are based. The individual process treatments cover computational algorithms, equipment design criteria and energy conservation. The overall treatment permits the evaluation of competing separations techniques and the choice of the optimal process.

This book is intended as a college or university level text for students in chemical engineering and related fields. It is also complete enough and detailed enough in its development of each topic to be useful as a reference for practicing engineers both new to and experienced in the area of separations.

CCNY, New York
May 1994

Prof. H. Weinstein

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Preface

This book, transformed from the original German by Dr. H. J. Feindt, is based on two German editions "Thermische Trennverfahren", published by Prof. K. Sattler. They have been successfully used as textbooks for university and college students and as reference texts in seminars and training programs for practising engineers in Germany, Austria and Switzerland.

The book presents a clear and very practice-oriented overview of thermal separation technologies. An extensive introduction elucidates the physical, physico-chemical, and chemical engineering fundamentals and principles of the different unit operations used to separate homogenous gaseous and liquid mixtures. The introduction is followed by a concise text with many explanatory figures and tables referring to process and basic design, flow-sheets, basic engineering and examples for the application of the unit operations distillation, absorption, adsorption, drying, liquid-liquid and solid-liquid extraction, evaporation and crystallization of solutions, melt crystallization and desublimation. A comprehensive reference list allows follow up of special separation problems.

The book enables the reader to choose and evaluate thermal separation processes and to model and design the necessary separation plant equipment.

Chemical and mechanical engineers, chemists, physicists, bio-technologists in research and development, plant design, production, environmental protection and administration and students in engineering and natural sciences will find this treatment of exceptional value and practical use.

Due to the quantity of the topics covered exercises could not be included in this book. An additional collection of illustrations with reference to basic engineering and design of the necessary equipment of thermal separation units is available in German (Sattler, K.: Thermische Trennverfahren. Aufgaben und Lösungen, Auslegungsbeispiele) and will be translated into the English language.

We are very much obliged to Prof. H. Weinstein, City University of New York for his advice and his Foreword to this book. Many thanks are also given to Philomena Ryan-Bugler, Louise Elsam, Karin Sora and the production team of VCH Verlagsgesellschaft for the accurate lectorship and book production. Special thanks are also given to Paul Fursey, University of Bradford, United Kingdom, for his assistance in copy-editing.

Brühl, Ludwigshafen
December 1994

K. Sattler
H. J. Feindt

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Frequently Used Nomenclature

A	Area	m^2
A_Q	Cross sectional area, cross section	m^2
D	Diffusion coefficient	m^2/h
D, \dot{D}	Vapor; vapor flow rate	kg, kmol; kg/h, kmol/h
E	Enrichment ratio, stage efficiency factor	–
F	Force	N
F	Loading factor for column trays	$m/s \cdot \sqrt{kg/m^3} = \sqrt{Pa}$
F, \dot{F}	Feed; feed flow rate	kg, kmol; kg/h, kmol/h
F	Free internal energy	kJ
G, \dot{G}	Gas; gas flow rate	kg, kmol; kg/h, kmol/h
G	Free enthalpy, Gibbs free energy	kJ
H	Enthalpy	kJ
$HETS$	Height equivalent to one theoretical stage	m
HTU	Height of a transfer unit	m
K^*	Phase equilibrium constant, distribution coefficient	–
L, \dot{L}	Liquid; liquid flow rate	kg, kmol; kg/h, kmol/h
L_c	Characteristic length	m
M	Molar mass	kg/kmol
N	Number of stages	–
NTU	Number of transfer units	–
Q, \dot{Q}	Heat; heat flow rate	kJ; kJ/h, W
R, \dot{R}	Reflux; reflux flow rate	kg, kmol; kg/h, kmol/h
\bar{R}	Gas constant	kJ/(kmol · K)
S	Entropy	kJ/K

XVI Frequently Used Nomenclature

T	Absolute temperature	K
U	Internal energy	kJ
V, \dot{V}	Volume; volumetric flow rate	$\text{m}^3; \text{m}^3/\text{h}$
\bar{V}	Molar volume	m^3/kmol
W	Work	kJ
X	Ratio or loading of key component in liquid or heavy phase (moles i /moles inert, kg i /kg carrier (inert))	—
Y	Ratio or loading of key component in vapor or light phase (moles i /moles inert, kg i /kg carrier (inert))	—
Z	Length or height for heat and mass transfer	m
a	Activity	—
a	Specific volumetric area	m^2/m^3
c_p, \bar{c}_p	Specific heat	kJ/(kg · K), kJ/(kmol · K)
c	Molar concentration	kmol/ m^3
c_w	Resistance coefficient	—
d	Diameter	m
d_p	Particle diameter	m
d_s	Sauter diameter	m
f	Fugacity	bar
f, \bar{f}	Specific free internal energy	kJ/kg, kJ/kmol
g	gravitational acceleration	m/s^2
g, \bar{g}	Specific free enthalpy	kJ/kg, kJ/kmol
h, \bar{h}	Specific enthalpy	kJ/kg, kJ/kmol
$\Delta h, \Delta \bar{h}$	Latent heat	kJ/kg, kJ/kmol
k	Overall heat transfer coefficient	$\text{W}/(\text{m}^2 \cdot \text{K})$
k	Overall mass transfer coefficient	m/h
m, \dot{m}	Mass; mass flow rate	kg; kg/h
n, \dot{n}	Number of moles; molar flux	kmol; kmol/h
p	Total pressure	bar, Pa
p_i	Partial pressure of component i	bar, Pa

$p_{o,i}$	Saturated vapor pressure of component i	bar, Pa
Δp	Pressure drop	mbar, Pa
q	Specific heat requirement	kJ/kg
\dot{q}	Specific heat flux	kJ/(m ² · h), W/m ²
r	Radius	m
r	Reaction rate	kmol/(m ³ · h)
s, \bar{s}	Specific entropy	kJ/(kg · K), kJ/(kmol · K)
s	Characteristic distance (transfer distance)	m
t	Time	h
t_m	Mean residence time	h
u, \bar{u}	Specific internal energy	kJ/kg, kJ/kmol
w	Velocity	m/s
w	Mass fraction, weight fraction of component i	—
x	Molar fraction, heavy phase	—
y	Molar fraction, light phase	—
z	Variable distance length or height	m
Δz	Tray spacing	

Greek

α	Separation factor	—
α	Heat transfer coefficient	W/(m ² · K)
β	Mass transfer coefficient	m/h
γ	Activity coefficient	—
δ	Film thickness, layer thickness	m
ε	Porosity, void fraction of a bed of solids, fraction of free volume	—
η	Yield	—
η	Dynamic viscosity	Pa · s
ϑ	Temperature	°C
\varkappa	Slope, gradient angle, inclination	°
λ	Thermal conductivity	W/(m · K)
$\mu, \bar{\mu}$	Chemical potential	kJ/kg, kJ/kmol

XVIII Frequently Used Nomenclature

v	Reflux ratio, solvent ratio, adsorbent ratio	—
ν	Kinematic viscosity	m^2/s
ρ	Density	kg/m^3
σ	Surface tension	N/m
ϕ	Relative humidity	—

Subscripts

H	Steam
T	Carrier
g	Gas phase
i, j	Component
l	Liquid phase
o	Above, surface
p	Effective, practical
s	Solid phase
t	Theoretical
u	Below
v	Loss
α	Start, entry
ω	End, exit

1 Basic Concepts

1.1 Principles of Thermal Separation Processes

In a chemical *production plant*, products are produced by the chemical and physical conversion of raw materials or intermediate products. The production unit is a completely integrated technical operating unit on the site. It is connected with other units on the site by transportation and personnel routes, and pipelines for raw materials, auxiliary substances, products, utilities, and energy. It usually consists of the actual production unit and several off-site facilities, as shown in Fig. 1-1.

The main unit contains the *unit processes and operations*, such as separation, combination, division, formulation, heat transfer, conveying, storage, packing. Figure 1-2 shows a general set-up which is independent from the type of process. The combination of unit processes and operations with respect to product properties depends on the product produced.

During the chemical conversion of raw materials, *homogeneous* and *heterogeneous* mixtures (Figs. 1-2 and 1-3) are generated. Both reactants and products may be found in these mixtures, according to the yield and conversion of the chemical reaction. By means of thermal separation processes these mixtures must be treated to obtain the desired products to a demanded purity and to enable the raw materials to be recycled.

Processes to separate physically homogeneous (one phase) and heterogeneous (two or multiphase) mixtures are listed in Table 1-1. The driving force of the separa-

tion process usually forms the criteria for the separation. Homogeneous mixtures with a molecularly dispersed distribution of individual components may only be separated by means of a thermal separation process.

Thermal separation processes are mass transfer operations, driven by molecular forces. Mass, and often heat, is exchanged between at least two phases of different composition. The phases are the mixture phase(s) and a selective auxiliary phase. The auxiliary phase is generated by either adding heat and/or by means of an auxiliary substance. The required driving forces, concentration, and temperature gradients, are formed due to the auxiliary phase.

In Fig. 1-4 thermal separation processes are listed and are denoted by the phases contributing to mass transfer in Table 1-2.

Thermal separations of mixtures are carried out in the following individual steps:

- *Step 1*: An additional phase is generated by supplying energy to the system, or by adding an auxiliary component.
- *Step 2*: Mass, and often simultaneously heat, is exchanged between phases. This is achieved by the addition or removal of energy.
- *Step 3*: After completion of the interchange process, the phases are separated. Together with the separation of the phases a (partial) separation of the mixture occurs.

All thermal separation processes follow this order of events. The *basic principles* of thermal separation processes are now formulated and will be discussed in detail.

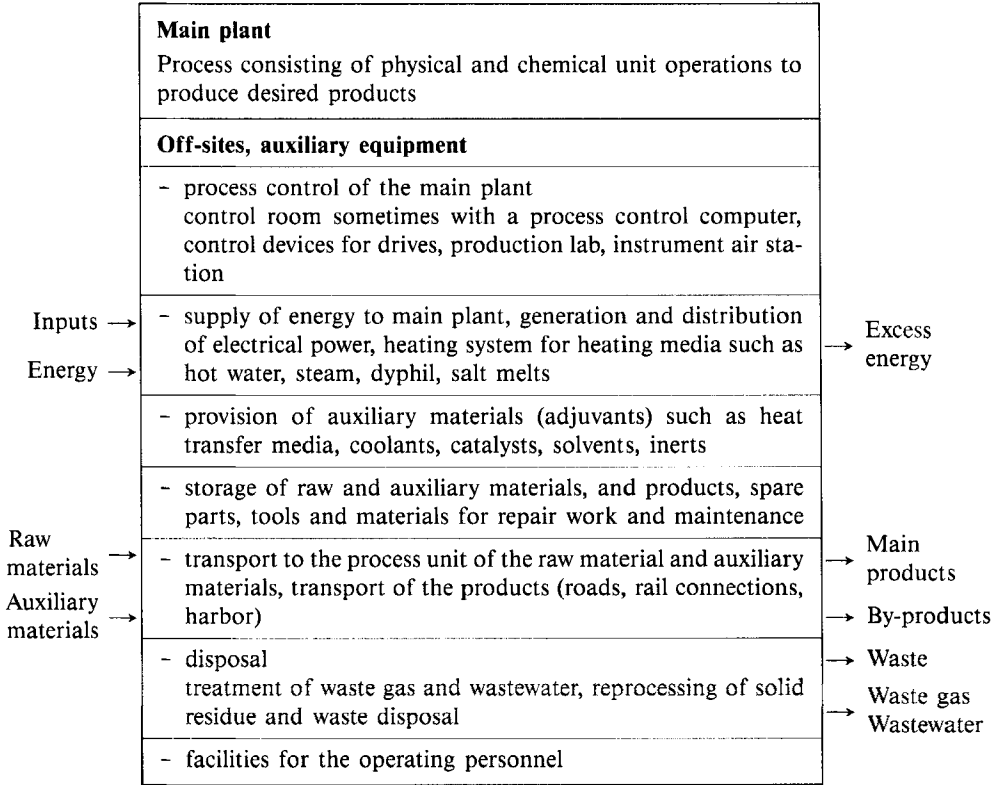


Fig. 1-1. General production process set-up.

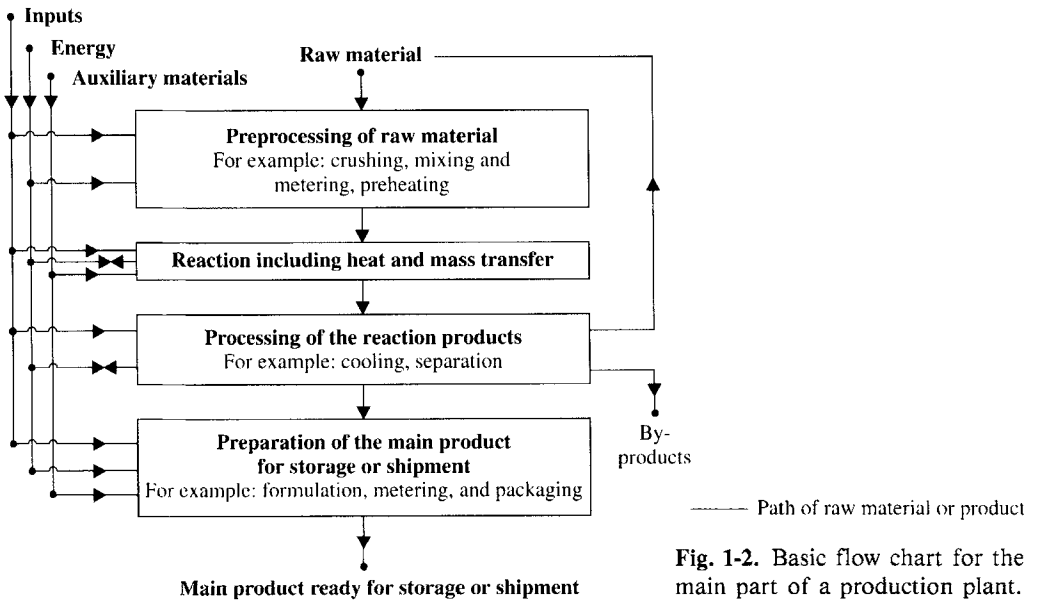


Fig. 1-2. Basic flow chart for the main part of a production plant.

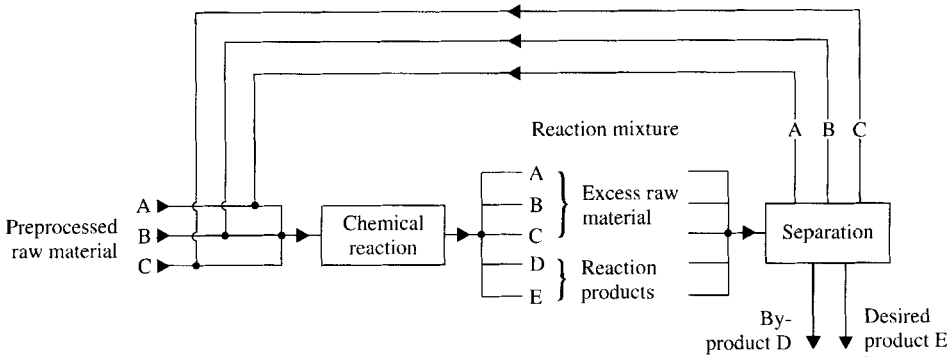


Fig. 1-3. Reaction and separation steps for an incomplete reaction of type $A + B + C \rightleftharpoons D + E$.

Phases in Cocurrent Flow
(Principle of Parallel Flow)

Phases taking place in mass and heat transfer are guided in cocurrent flow through the separation apparatus. The maximum efficiency of this separation apparatus is the same as that for a single theoretical separation stage.

Phases in Countercurrent Flow
(Principle of Counterflow)

Phases taking place in mass and heat transfer are guided in countercurrent flow through the separation apparatus. In this case it is important to disperse the phases with the aid of internals, thereby achieving intensive mixing of the phases. Thus the

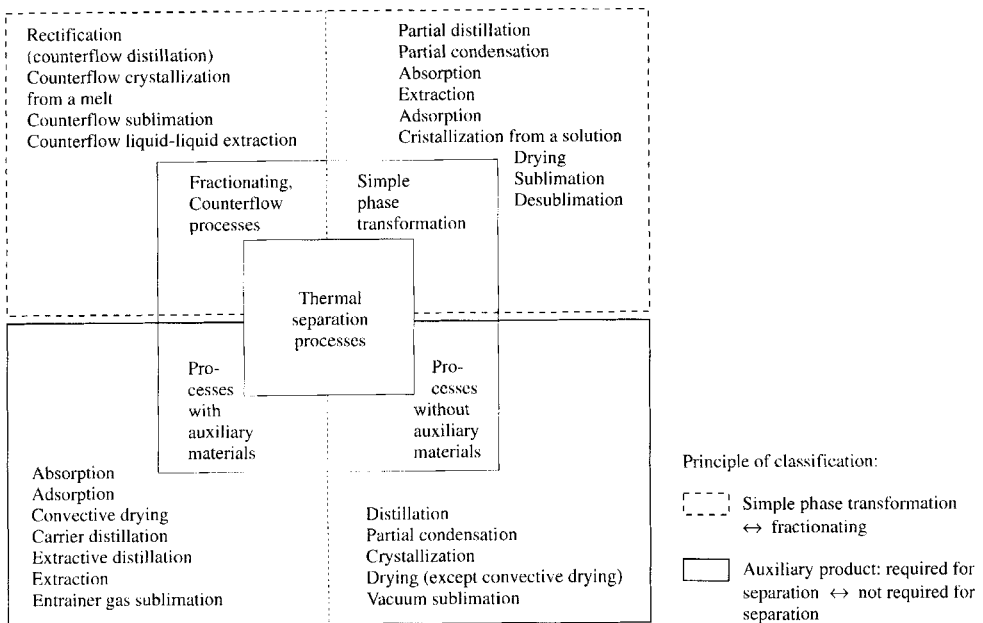


Fig. 1-4. Summary of thermal separation processes.

Table 1-1. Summary of separation processes.

Classes of separation processes	Driving force of separation process	Nature of mixture	Separation processes
Mechanical separations	Gravity Centrifugal force Pressure	Heterogeneous	Sorting (s - s) Dense-media separation (s - l) Flotation (s - l - g) Sedimentation (s - l) Filtration (s - l) Pressing (s - l) Centrifugation (s - l) Hydrocyclone separation (s - l) Classification Sieving (s - s) Air classification (s - g) Hydraulic classification (s - l)
Membrane separation	Pressure Electrical field Concentration gradient	Heterogeneous Homogeneous	Ultrafiltration (s - l) Reverse osmosis (hyperfiltration) (s - l) Dialysis (s - l) Electrodialysis (s - l) Electrophoresis (s - l) Permeation (l - l, g - g) Gas diffusion (g - g)
Electrical separation	Electrical field	Heterogeneous	Electro osmosis (s - l) Electrical dust removal (s - g)
Magnetic separation	Magnetic field	Homogeneous	Magnetic separation (s - s)
Thermal separation	Concentration gradient Temperature gradient	Homogeneous	Distillation (l - l) partial condensation (g - g) Absorption (g - g), (A) Adsorption (g - g, s - l), (A) Chromatography (g - g, l - l) Extraction (s - s, l - l), (A) Sublimation (g - g) Crystallization (s - l, l - l) Drying (s - l) Thermal diffusion (g - g, l - l)

Abbreviations: s solid, l liquid, g gas to characterize the state of the components of the mixture to be separated, (A) thermal separation process with auxilliary component.

maximum possible interfacial area (phase boundary) for mass transfer is obtained and, hence, the highest possible mass transfer coefficient values. Figure 1-5 shows a "separation column" with stages connected in series in which the key component i of a

mixture is exchanged from the heavy phase to the light phase. Both phases may contain all components of the mixture.

A closer inspection of stage n shows that the heavy phase, with a mole fraction of x_n is in contact with the light phase with a

Table 1-2. Characteristics of thermal separation processes by the phases in which mass and heat transfer occurs.

	Phase 1	Phase 2	All components are contained in both phases	Not all components are in both phases		
				Phase 1 pure	Phase 2 pure	One (several) component (s) is (are) in both phases
Immiscible phases in contact	g	l	Distillation Partial condensation	Concentration of solutions	Gas drying	Absorption Desorption by stripping
	g	s	Counter current sublimation	—	—	Adsorption Drying
	g	g	—	—	—	—
	l	l	Liquid-liquid extraction	—	—	—
	l	s	Crystallization from a melt	—	Crystallization from a solution	Solid-liquid extraction (leaching) Adsorption
	s	s	—	—	—	—
Miscible phases in contact	g	g	Thermal diffusion			
	l	l	Thermal diffusion			
	s	s	—			

Abbreviations: g gas phase, l liquid phase, s solid phase.

mole fraction of y_{n-1} . If x_n and y_{n-1} are not phase equilibrium concentrations, the fed phases of stage n are not in phase equilibrium, and mass and heat transfer take place. The key component i becomes enriched in the light phase up to a final concentration y_n , while the heavy phase is reduced in component i from x_n to x_{n-1} .

With stage n as a theoretical separation stage, the leaving phases are in equilibrium and no further mass or heat transfer is possible. Therefore, y_n and x_{n-1} are phase equilibrium concentrations.

The heavy phase, with concentration x_{n-1} , arrives at stage $n - 1$ and comes into contact with the light phase, with concen-

tration y_{n-2} . An exchange, similar to that in stage n , takes place.

The discussed example shows that for countercurrent phase flow, single stages are connected in series in one separation apparatus. The light phase leaving a stage is guided to the following stage whereas the heavy phase is guided to the previous stage.

A *theoretical stage* is that part of a separation apparatus where mass or heat transfer take place in which entering phases are not in phase equilibrium, while the leaving phases have reached *phase equilibrium* (see Chapter 1.4).

In a practical separation stage, equilibrium is often not achieved. The efficiency

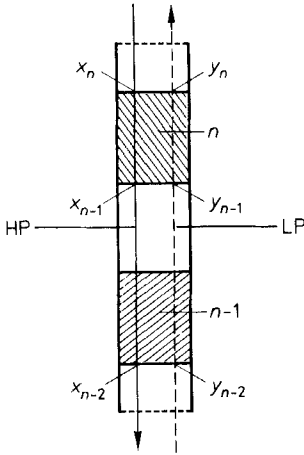


Fig. 1-5. Countercurrent flow of two phases in a separation apparatus.

$n - 1, n$ Stages connected in series

LP Upflowing light phase

HP Downflowing heavy phase

x Molar fraction of the key component in the heavy phase

y Molar fraction of the key component in the light phase

transfer ratio, depending on whether y is only locally valid or constant across the cross section of the column.

Phases in Cross Flow
(Cross Flow Principle)

Phases taking part in mass and heat transfer flow across through the separation apparatus at an angle of 90° to each other.

The separation efficiency depends on the equilibrium location and the ratio of the phase fluxes, but is often low in an individual separation stage. To separate a mixture and obtain pure products, several separation stages are connected in series. This is done most effectively with countercurrent phase flow. Phase cross flow and parallel feed of one phase to individual separation stages are sometimes used. However, cocurrent flow is of non importance.

compared with a theoretical stage is expressed as the *stage efficiency factor*, E (exchange ratio, enrichment ratio, MURPHREE efficiency) (Fig. 1-5):

$$E = \frac{\text{separation effect of a practical stage}}{\text{separation effect of a theoretical stage}}$$

$$E = \frac{y_n - y_{n-1}}{y_n^* - y_{n-1}} \tag{1-1}$$

where

- $y_n^* - y_{n-1}$ possible theoretical enrichment of the key component in the light phase (y_n^* phase equilibrium concentration at x_{n-1})
- $y_n - y_{n-1}$ actual enrichment of the key component in the light phase

This often has to be distinguished as a local transfer ratio, as opposed to an overall

Time Requirement

The time needed to separate a mixture in a discontinuous operation is the effective residence time. For continuous operation, it is the *mean residence time* t_m , of the mixture in the separation apparatus:

$$t_m = \frac{V}{\dot{V}} \tag{1-2}$$

where

- V filled volume of the mixture in the separation apparatus (determined by the volume of the apparatus and the degree of filling)
- \dot{V} effective volumetric flow of the mixture

Short-, medium- and long-term separation processes can be distinguished depending on the time requirement:

- *Short-term processes* ($t_m < 30$ sec).
Examples: Spray drying, gas adsorption, precipitation crystallization.
- *Medium-term processes* ($30 \text{ sec} < t_m < 2\text{h}$).
Examples: Absorption, rectification, drum drying, pneumatic-conveyor drying, sublimation, extraction, crystallization, liquid adsorption.
- *Long-term processes* ($1 \text{ h} < t_m < 1 \text{ d}$).
Examples: Rotary drum drying, vacuum tumbling drying, vacuum freeze drying, fractionation crystallization.
- *Flow energy*, for pressure drops in the apparatus and the connecting pipework.
- *Mechanical energy*, for example for dispersing, pulsing, stirring and pump circulation devices.
- *Work*, to operate peripheral machines such as compressors and vacuum pumps.

1.2 Thermal Separation Process Modes

Energy Supply

For the thermal separation of a mixture in an apparatus, energy has to be supplied in the form of:

- *Heat*, to increase the sensible heat of the flowing masses and to supply latent heat.

Apparatus for the thermal separation of mixtures may be operated both *discontinuously* (intermittently, batch production, stagewise operation) and *continuously* (steady-state). In the following section, the operating modes are briefly illustrated. The advantages and disadvantages are listed in Table 1-3.

Table 1-3. Comparison of continuous and discontinuous operation to achieve the same separation problem.

Comparison criteria	Operating mode	
	Continuous	Discontinuous
Mathematical description of the separation process, modeling	Simpler	
Investment cost of separation unit	Less	
Operating cost of separation unit	Less	
Operation of separation unit	Easier	
Automatic control of separation process	Possible with less expense	
Working stress on unit components	Less	
Environmental pollution, possibility of accident	Less	
Operation reliability, flexibility in the case of breakdown of separation unit parts, safety buffer		Higher
Flexibility to adjust to other mixtures to be separated		Better

Continuous Operation: In continuous operation the mixture being separated is continuously fed to the separation device. It is continuously separated into two or more fractions, which are continuously withdrawn from the separation device.

An ideal binary mixture can be separated into almost pure components in a separation column operated continuously with countercurrent flow. To separate a mixture of k components, $k - 1$ columns connected in series are needed.

Discontinuous Operation: With discontinuous operation the mixture being separated is charged to the separation device. During a time period, the “batch period”, the mixture is separated mainly into two fractions of defined different compositions. One fraction is continuously withdrawn from the separation device, while the other remains in the device and is withdrawn at the end of the batch time.

Discontinuously operated processes – mainly in one stage – allow incomplete separation of a mixture; the obtained fractions are treated in subsequent stages (this is the case for multistage discontinuous separation).

Alternating Operation: If in a separation apparatus after a loading process (separation of a mixture) an unloading process (the regeneration of a substance aiding separation) is required, at least two sets of equipment are operated alternately. Therefore, steady separation of the mixture is guaranteed.

In the case of the adsorption of a substance from a gas phase in a container adsorber (see Chapter 4), for example, a solid

adsorbent adsorbes adsorbate (key component in the gas phase). Adsorption continues to an upper loading limit. After the maximum load has been reached in the first adsorber, operation is switched to the second adsorber. The loaded adsorber is then regenerated by dampening, drying and cooling. After the regeneration cycle is finished, the adsorber is ready for loading again.

1.3 Mass Balance, Energy Balance, Exergy Balance

In general, the first step in the design of a separation plant is the *balancing* of individual apparatuses and parts of the plant. Balances are done with respect to energy and mass fluxes, in connection with a schematic representation of the process (flow diagram).

1.3.1 Mass, Energy and Heat Balances

The balancing of chemical engineering systems follows the sequence listed in Fig. 1-6.

Of the variables listed for process design, mass and energy (usually in the form of heat, enthalpy, and exergy) are of most interest. These variables may also be used for planning, evaluation of systems, analysis, and synthesis.

Based on the laws of conservation of mass, energy and momentum, balance equations are set up [1.1] – [1.5]. For a general open system

$$\left\{ \begin{array}{l} \text{sum of amount} \\ \text{entering} \\ \text{the system} \\ \text{(transport)} \end{array} \right\} + \left\{ \begin{array}{l} \text{sum of amount} \\ \text{generated in} \\ \text{the system} \\ \text{(transformation)} \end{array} \right\} = \left\{ \begin{array}{l} \text{sum of amount} \\ \text{leaving} \\ \text{the system} \\ \text{(transport)} \end{array} \right\} + \left\{ \begin{array}{l} \text{increase of} \\ \text{mass stored} \\ \text{in the system} \\ \text{(accumulation)} \end{array} \right\}$$

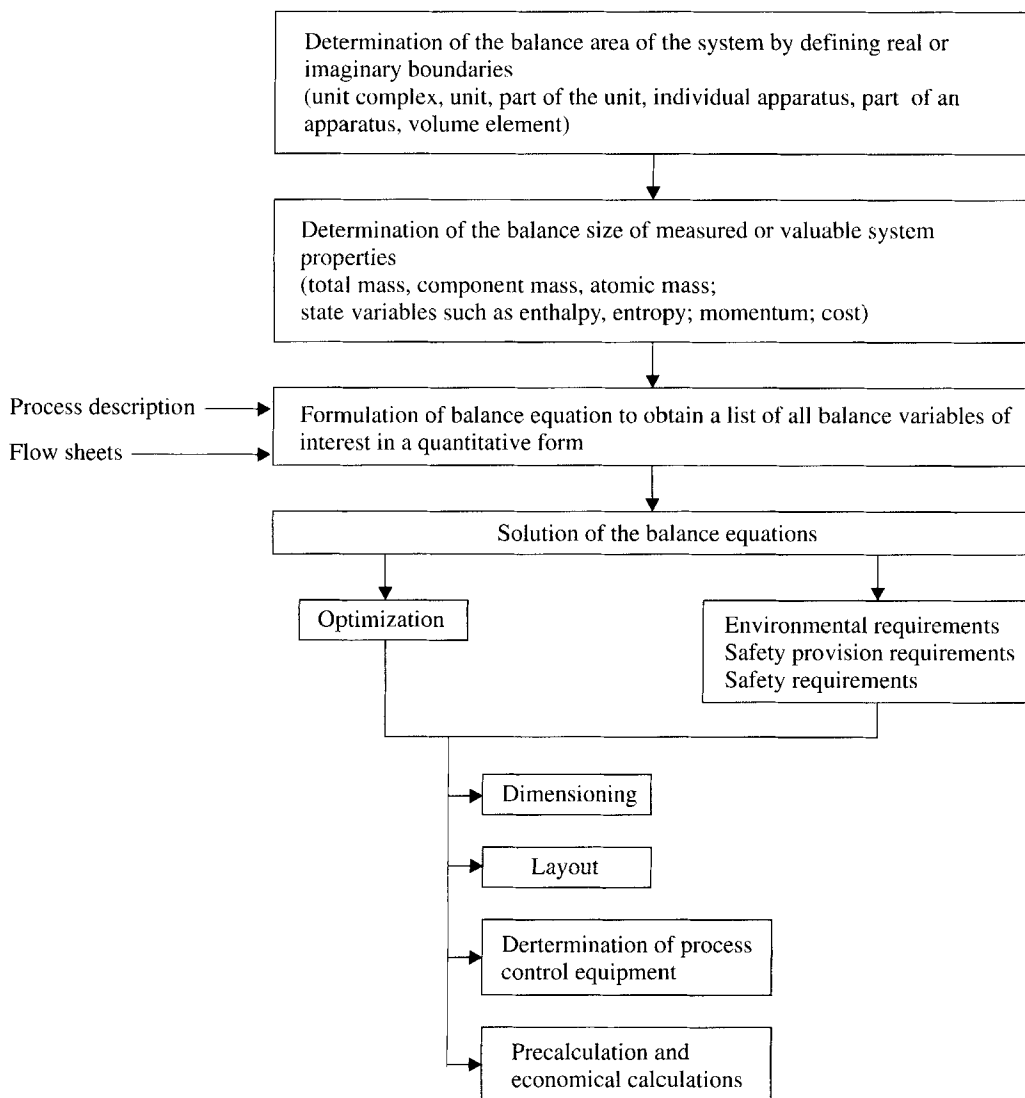


Fig. 1-6. Balancing of processes (schematic simplification of the concepts) [1.1].

Depending on the problem or task a *integral* or *differential balance equation* is generated:

- Differential, to investigate a process in a differential volume element or at an interfacial surface element

- Integral to determine the streams entering and leaving the system

Differential balance equations lead to velocity, concentration, and temperature profiles in the system, or at the boundary surfaces, after solving the corresponding differential equation system with suitable bound-

ary conditions. Integral balance equations give a basis for evaluation of the total system with respect to energy and mass.

Results of integral balance equations are often presented in a table or in a flowchart (product and energy scheme, mass and heat flowchart, etc.).

Mass Balance

Material balances (mass or quantity balances) can be *general* or *total material balances* over the complete system and must be distinguished from material balances for individual *mixture components* (Fig. 1-7). Using the terms in Fig. 1-7, for an open system the general integral balance equation is

$$\sum_i \dot{m}_{i,\alpha} + \dot{m}_Q = \sum_i \dot{m}_{i,\omega} + \dot{m}_S \quad (1-3)$$

In steady-state operation (continuous feed and withdrawal of material without start up and shut down procedures) the accumulation terms \dot{m}_Q and \dot{m}_S are not required.

The material balance for an individual component k is

$$\sum_i \dot{m}_{i,\alpha} \cdot w_{i,k,\alpha} + \dot{m}_{Q,k} = \sum_i \dot{m}_{i,\omega} \cdot w_{i,k,\omega} + \dot{m}_{S,k} \quad (1-4)$$

where

$\dot{m}_{i,\alpha}, \dot{m}_{i,\omega}$ mass flux i (α feed, ω product)
 $w_{i,k,\alpha}, w_{i,k,\omega}$ mass fraction of component k in stream i
 $\dot{m}_Q, \dot{m}_S, \dot{m}_{Q,k}, \dot{m}_{S,k}$ intensity of sources (Q) and sinks (S) inside the balance area generally with respect to component k

$\dot{m}_{Q,k}$ and $\dot{m}_{S,k}$ take, for example, chemical reactions involving k into consideration. If k is a reactant in j single reactions taking place simultaneously in the balance area, for steady-state operation,

$$\dot{m}_{Q,k} - \dot{m}_{S,k} = V \cdot M_k \cdot \sum_j r_j \cdot v_{j,k} \quad (1-5)$$

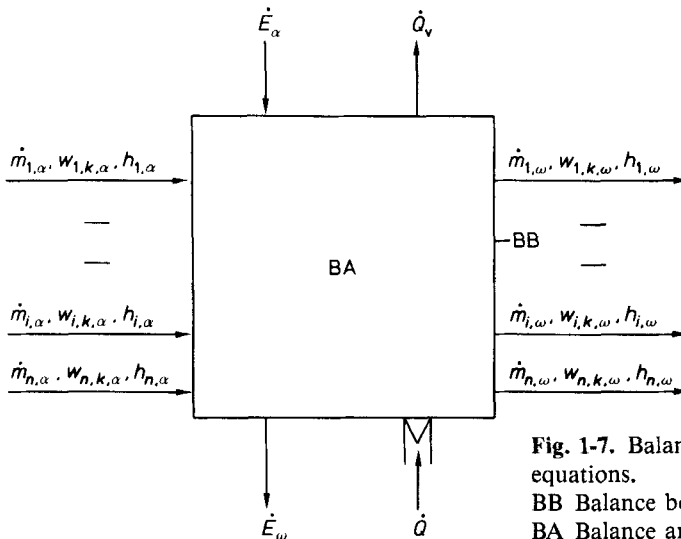


Fig. 1-7. Balance scheme to derive the balance equations.

BB Balance boundary

BA Balance area