

Chapter 1

Separation Processes

§1.0 INSTRUCTIONAL OBJECTIVES

After completing this chapter, you should be able to:

- Explain the role of separation operations in the chemical industries.
- Enumerate the four basic separation techniques.
- Explain the uses of energy-separating agents (ESA) and mass-separating agents (MSA).
- Calculate component material balances around a separation operation based on specifications of component recovery and/or product purity.
- Understand the concept of sequencing of separation operations, particularly distillation.

Separation processes for chemical mixtures have been known for millennia. Early civilizations developed processes for: (1) extraction of metals from ores, perfumes from flowers, dyes from plants, and potash from burnt plants; (2) evaporation of seawater to obtain salt; (3) refining of rock asphalt; and (4) distilling of liquors. The human body could not function if it had no kidney to separate water and waste products from blood.

Chemical engineers design large-scale facilities that employ separation methods that most often differ considerably from those used by chemists in laboratories. For example, chemists separate light-hydrocarbon mixtures by chromatography, while a manufacturing plant uses distillation to separate the same mixture. This book discusses methods for the design of large-scale separation operations for processes that manufacture chemical products economically. Included are all of the most common operations, including distillation (both continuous and batch), gas absorption, stripping, liquid–liquid extraction, membrane separations, and gas and liquid adsorption. These design methods are incorporated into commercial computer-aided **process simulators** such as Aspen Plus, Aspen HYSYS, CHEMCAD, ChemSep, ProSimPlus, and UniSim.

Chemical engineers also design small-scale industrial separation systems involving manufacture of specialty chemicals by batch processing; recovery of biological solutes; crystal growth of semiconductors; recovery of chemicals from wastes; and products such as lung oxygenators, espresso machines, and hemodialysis devices. A companion book, *Bioseparation Process Principles*, covers these smaller-scale processes and the principles of bioseparations. Included in that book are membranes and adsorption for bio applications, chromatography, electrophoresis, leaching, crystallization, drying, and mechanical separation operations. Both large- and small-scale operations are illustrated in examples and homework exercises in this book and in *Bioseparation Process Principles*.

§1.1 INDUSTRIAL CHEMICAL PROCESSES

Chemical companies manufacture products that differ from those in the feedstocks. Included can be (1) naturally occurring living or nonliving materials; (2) chemical intermediates that are precursors for producing other chemicals; (3) “chemicals in commerce” that can be purchased from the global market; or (4) waste products that can be processed into valuable products. Especially common are oil refineries, which process crude oil, synthetic crude oil from tar sands, and tight oil from the use of horizontal drilling followed by fracking of shale deposits, to produce a variety of hydrocarbon-based products. For example, starting in 1967, Canada began increasing its oil production dramatically by processing tar sands from the huge Athabasca deposit in the province of Alberta. Figure 1.1 shows the initial products produced by Great Canadian Oil Sands Ltd. (GCOS), now Suncor Energy, from its 1967 plant. The products are the result of numerous separation operations within the plant. By 2013, several Canadian companies produced more than 1.6 million barrels per day (bbl/day) of synthetic crude oil (syncrude). Because Canada produces more oil than it needs, it exports syncrude to the United States by the Keystone Pipeline. If phases 3 and 4 of the pipeline are completed, Canadian syncrude will flow more than 2,000 miles from the Keystone Hardisty Terminal in Alberta, Canada, to the Gulf near Houston, Texas.

Chemical plants operate in a **batchwise**, **continuous**, or **semicontinuous** manner. Plant operations may be **key operations** unique to chemical engineering because they involve changes in chemical composition, or **auxiliary operations** that are necessary to the success of the key operations but are often designed by mechanical engineers because they do not involve changes in chemical composition. The key operations involve (1) chemical reactions and (2) separation of chemical mixtures, such as the separation of a mixture of chemicals into pure, or nearly pure, species (components).

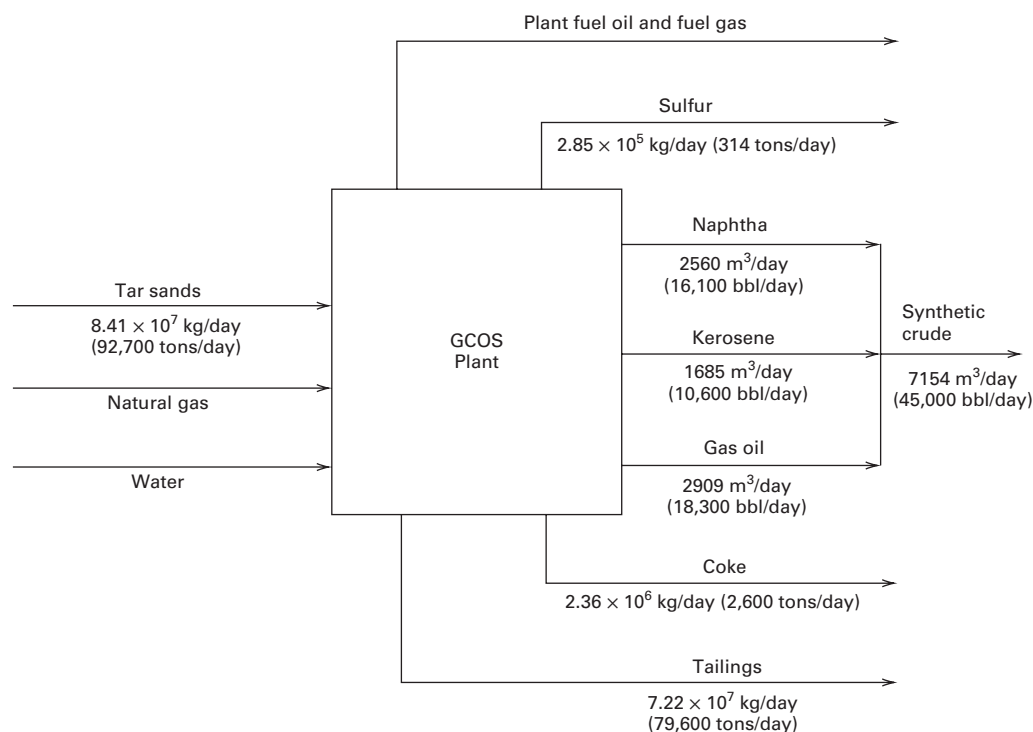


Figure 1.1 GCOS process for producing synthetic crude oil from Canadian Athabasca tar sands.

The auxiliary operations include phase separation (e.g., separation of gas from liquid or separation of two immiscible liquid phases), heat addition or removal (heat exchangers), shaft-work (pumps, compressors, turbines), mixing or dividing of streams, solids agglomeration, size reduction of solids, and separation of solids by size.

Block-flow diagrams can represent key operations in chemical processes. They indicate, by square or rectangular blocks, only the chemical reaction and separation steps and, by connecting lines, the process streams. More detail is shown in **process-flow diagrams**, which also include auxiliary operations and utilize icons that depict the particular type of equipment employed. Figure 1.2 shows a block-flow diagram for manufacturing hydrogen chloride gas from chlorine and hydrogen. Central to the process is a chemical reactor, where the gas-phase combustion reaction, $\text{H}_2 + \text{Cl}_2 \rightarrow 2\text{HCl}$, occurs. In this process, no separation operations are necessary because of the complete conversion of chlorine and the absence of any side reactions. A slight excess of hydrogen is used, and the

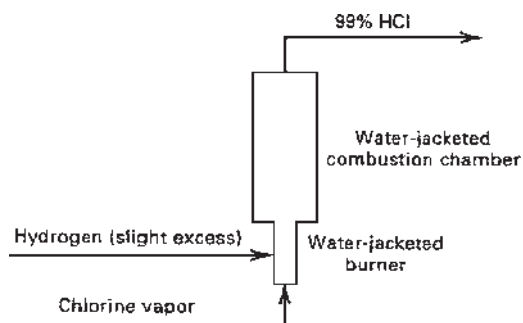


Figure 1.2 Synthetic process for anhydrous HCl production.

product, 99% HCl, with small amounts of H_2 , N_2 , H_2O , CO , and CO_2 , requires no purification. However, simple process flowsheets that do not require separation operations are rare. In most process flowsheets, separation operations dominate.

Most industrial chemical processes involve at least one chemical reactor accompanied by one or more separation operations. An example is the continuous hydration of ethylene with water to produce ethyl alcohol, shown in Figure 1.3. Central to the process is a reactor packed with catalyst particles, in which the reaction $\text{C}_2\text{H}_4 + \text{H}_2\text{O} \rightarrow \text{C}_2\text{H}_5\text{OH}$ occurs. Due to chemical equilibrium limitations, conversion of ethylene is only 5% per pass through the reactor. However, by recovering unreacted ethylene, in a partial condensation separation step, and recycling the ethylene to the reactor, nearly complete overall conversion of the ethylene feed is achieved. Recycling is a common element of chemical processes.

If pure ethylene were available as a feedstock and no side reactions occurred, the simple process in Figure 1.3 could be realized. It utilizes a reactor, a partial condenser for unreacted gaseous ethylene recovery, and distillation to produce an overhead distillate of aqueous ethyl alcohol of near-azeotropic composition (93 wt%), with a wastewater bottoms product. Unfortunately, impurities in the ethylene feed, together with side reactions involving ethylene and the impurities, increase the number of separators required for the process, as shown in Figure 1.4. Also, as shown, an additional reactor may be necessary to convert impurities or by-products to other chemicals that can be more easily separated from the main product. Such an escalation of the need for separation operations is common. Thus, most chemical processes include many more separation operations than chemical reactors.

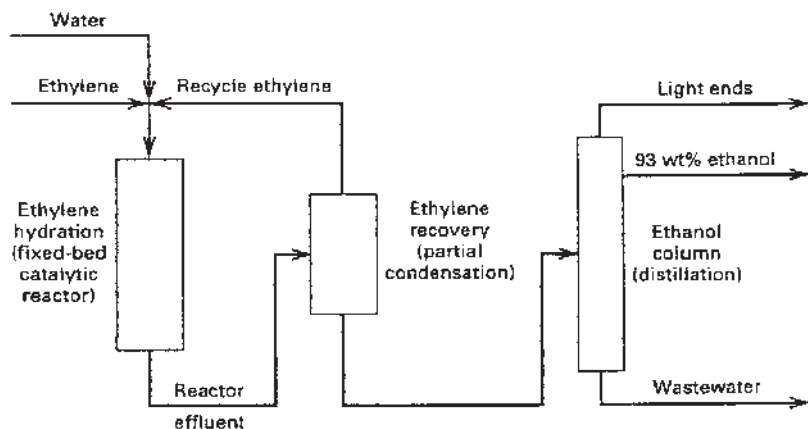


Figure 1.3 Process for hydration of ethylene to ethanol.

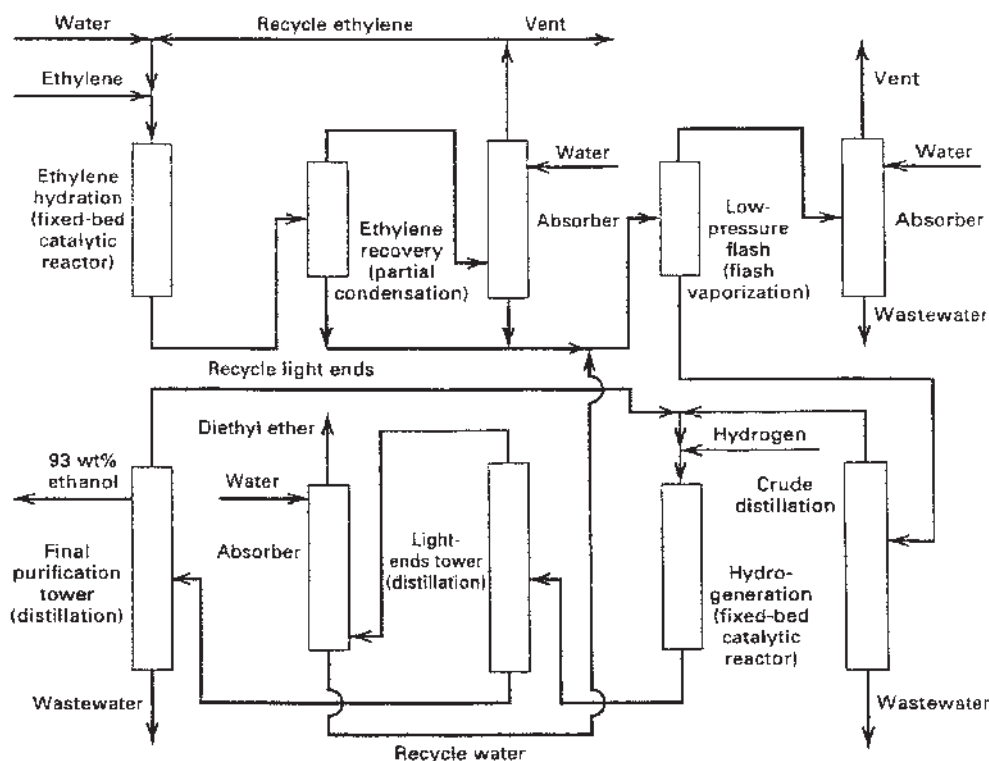


Figure 1.4 Industrial process for hydration of ethylene to ethanol.

Chemical engineers also design products that can involve separation operations. One such product is the espresso coffee machine. Very hot water rapidly leaches desirable chemicals from the coffee bean, leaving behind ingredients responsible for undesirable acidity and bitterness. The resulting cup of espresso has (1) a topping of creamy foam that traps the extracted chemicals, (2) a fullness of body due to emulsification, and (3) a richness of aroma. Typically, 25% of the coffee bean is extracted and the espresso contains less caffeine than filtered coffee. Cussler and Moggridge [1] and Seider, Seader, Lewin, and Widagdo [2] discuss other examples of products designed by chemical engineers that involve the separation of chemical mixtures.

§1.2 BASIC SEPARATION TECHNIQUES

The separation of a chemical mixture into its components is not a spontaneous process, like the mixing by diffusion of soluble components. Separations require energy in some form. A mixture to be separated into its separate chemical species is usually a single, homogeneous phase. If it is multiphase, it is often best to first separate the phases by gravity or centrifugation, followed by the separation of each phase mixture.

A schematic of a general separation process is shown in Figure 1.5. The phase state of the feed can be a **vapor**, **liquid**, or **solid** mixture. The products of the separation differ in composition from the feed and may differ in the state of the

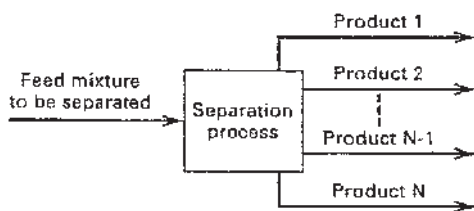


Figure 1.5 General separation process.

phase. The separation is accomplished by inducing the different chemical species in the feed to partition among the different product phases. Four basic methods for doing this are shown in Figure 1.6. Most common is the **phase creation** technique in Figure 1.6a. For example, the feed may be a liquid mixture. By heating the mixture to partially vaporize the liquid, the more-volatile components tend to move to the vapor, while less-volatile components tend to remain in the liquid phase. Alternatively, the feed may be a vapor that is partially condensed.

A second technique, labeled **phase addition**, is shown in Figure 1.6b. For example, a liquid solvent may contact a liquid feed to selectively dissolve certain species from the feed. Less common is the third separation technique that uses a **barrier**, such as a membrane, as shown in Figure 1.6c. The barrier allows passage of certain species in the feed while excluding or slowing other species. Finally, in a fourth technique, an **external force field** or gradient is used to preferentially attract certain species in the feed as indicated in Figure 1.6d. The force field might be electrical or centrifugal.

For the techniques of Figure 1.6, the **rate of separation** is governed by the rates of **mass transfer** of the different components, while the **extent of separation** is limited by **thermodynamic equilibrium**. These two topics are the subject of previous chemical engineering courses, but are reviewed here in Chapters 2 and 3. Also, fluid mechanics and heat transfer play important roles in the separation of mixtures, and their principles are applied, where appropriate, throughout this book.

The extent of separation achieved depends on the exploitation of differences in molecular, thermodynamic, and transport properties of the species in the feed mixture. Of importance are molecular weight, molecular shape, dipole moment, electric charge, vapor pressure, solubility, adsorptivity, and diffusivity. Values of these properties appear in handbooks, reference books, textbooks, and journals. Many can be obtained from physical property models in process-simulation programs. When properties are not available, they are estimated or determined experimentally.

§1.3 SEPARATIONS BY PHASE CREATION

The simplest and most widely used industrial separation technique is phase creation. The feed to the separator is a single-phase vapor, liquid, or solid. The second phase is created by the transfer of energy to or from the feed by an **energy-separating agent (ESA)**. The ESA can be heat transfer or shaft work by means of compression or by the reduction of pressure through a turbine or across a valve. After sufficient time and agitation to approach phase equilibrium, the product phases are separated. The size of the process vessel depends on the flow rate in and out of the vessel and the time for the phases to approach equilibrium.

The most common separation operations using phase creation are listed in Table 1.1. For these operations, design procedures are well established and are included as unit operation models in commercial process simulators.

When the feed mixture is a vapor or a liquid with components that differ widely in volatility (e.g., hydrogen and benzene), **partial condensation** or **partial vaporization**, Operation (1) in Table 1.1, may be adequate to achieve the desired separation. Heat is transferred to or from the feed in a heat exchanger followed by phase separation by gravity in a vessel. Partial vaporization of a liquid occurs in **flash vaporization**, Operation (2), by reducing the feed pressure with a valve or turbine, followed by phase separation. Both of these separation operations are referred to as **single equilibrium stages** because experimental evidence shows that interphase

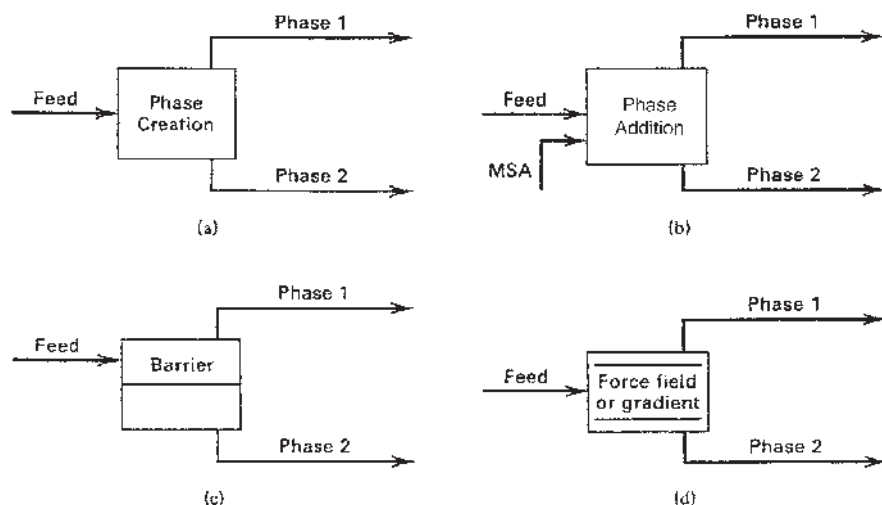


Figure 1.6 Basic separation process techniques: (a) separation by phase creation; (b) separation by phase addition; (c) separation by barrier; (d) separation by external force field or gradient.

Table 1.1 Common Separation Operations Based on Phase Creation

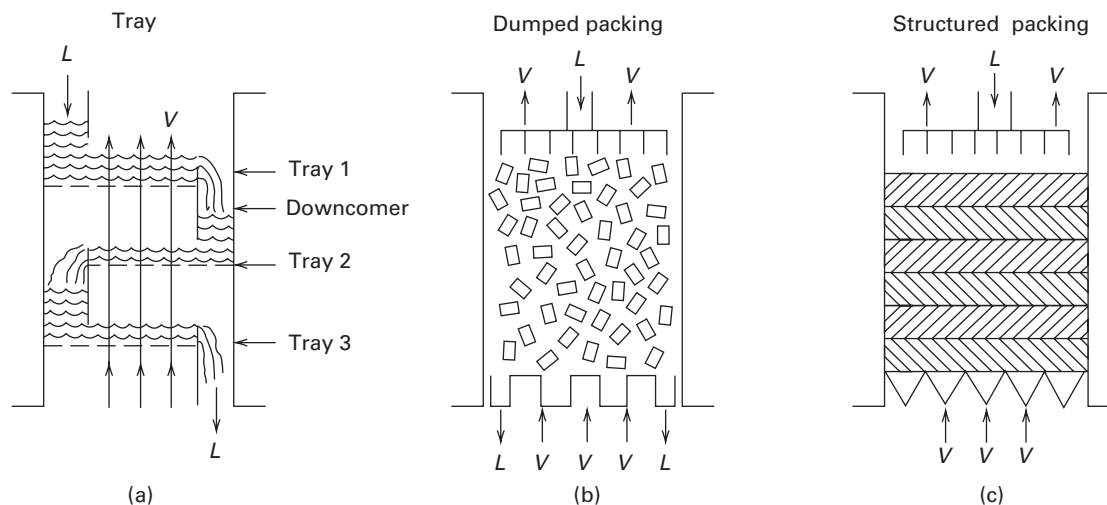
Separation Operation	Symbol	Feed Phase	Created Phase	Separating Agent(s)
(1) Partial condensation or vaporization		Vapor and/or liquid	Liquid or vapor	Heat transfer (ESA)
(2) Flash vaporization		Liquid	Vapor	Pressure reduction
(3) Distillation		Vapor and/or liquid	Vapor and liquid	Heat transfer (ESA) and sometimes shaft work (ESA)

mass transfer of species is so rapid that phase equilibrium is closely approached. The liquid product is enriched with respect to the less volatile species, while the vapor product is enriched with respect to the more volatile species. These operations are discussed in Chapter 4.

When the degree of separation by a single equilibrium stage is inadequate because the volatility differences among key species are insufficiently large (e.g., benzene and toluene), **distillation**, Operation (3) in Table 1.1, with multiple stages, often becomes the separation method of choice. It is and has long been the most widely utilized industrial separation method. Distillation involves vigorous mixing during contacts between countercurrently flowing liquid and vapor phases. Figure 1.7 shows schematics of three widely used phase-contacting methods. Figure 1.7a depicts a series of trays, where the phases are mixed and then disengaged. Each tray is some fraction of an equilibrium stage. In Figures 1.7b

and 1.7c, the phases are in continuous contact with other. In the first case, randomly dumped ceramic, metal, or plastic rings or saddles, called packing, promote turbulence as the vapor follows a tortuous path through the liquid, which flows over and through the packing. In the latter case, stacked, structured packing is used in the form of metal or plastic meshes, grids, or coils. Depending on the efficiency of the packing, inches or feet of packed height are equivalent to an equilibrium stage. In the icon for distillation in Table 1.1, horizontal lines within the column indicate the stages. As vapor flows up the column, it is increasingly enriched with respect to the more volatile species. The liquid flowing down the column is increasingly enriched with respect to the less-volatile species.

Feed to a distillation column enters at a stage somewhere between the top and bottom stages. Any vapor in the feed starts up the column; feed liquid starts down. Liquid is also required for making contacts with vapor above the feed stage, and vapor

**Figure 1.7** Phase-contacting methods in distillation columns.

is also required for making contacts with liquid below the feed stage. Therefore, at the top of the column, vapor is condensed to provide distillate product and down-flowing liquid (**reflux**). Similarly, part of the liquid at the bottom is removed as bottoms product, while the other part passes through a reboiler, where it is heated to provide up-flowing vapor (**boilup**). Continuous distillation is introduced in Chapter 5. The separation of a binary mixture by continuous distillation is discussed in Chapter 7, while the continuous distillation of multicomponent mixtures is discussed in Chapters 9–12. Batch distillation is discussed in Chapter 13.

§1.4 SEPARATIONS BY PHASE ADDITION

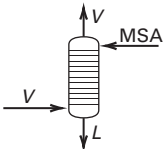
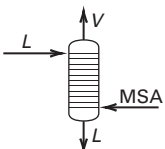
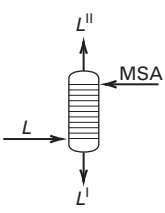
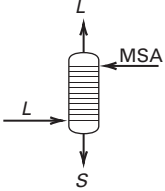
The next most widely used industrial separation technique is phase addition. The feed to the separator is a single-phase vapor, liquid, or solid. The second added phase is called a mass-separating agent (MSA). Certain components in the feed move from the feed phase to the MSA phase. After sufficient time and agitation to approach phase equilibrium, the product phases are separated. Disadvantages of using an MSA are (1) the need for an additional separator to recover the MSA for recycle, (2) the need for MSA makeup, (3) possible product contamination by the MSA, and (4) more complex design procedures. Table 1.2 lists the most common separation operations using phase addition. For these operations, design procedures are well established and are included in process simulators.

When the feed mixture is a vapor, and it is desired to remove the higher molecular weight (heavier) components from the lower molecular weight (lighter) components, **absorption**, Operation (1) in Table 1.2, is used. The feed gas enters at the bottom of a multistage column and flows up the column countercurrent to the MSA, called an absorbent, which enters at the top of the column. Typically, absorbers operate at near-ambient temperature at an elevated pressure. A subsequent separation, often distillation, separates the absorbate from the absorbent, which is then recycled to the absorber.

The inverse of absorption is **stripping**, Operation (2) in Table 1.2. Here, a liquid feed mixture is separated, at elevated temperature and near-ambient pressure, by contacting the feed, which enters at the top of the column, with a gas stripping MSA that enters at the bottom. A second separation operation may be needed if it is necessary to separate the stripping agent from the components stripped from the liquid feed and/or to recycle the stripping agent to the stripper. Absorption and stripping are discussed in Chapter 6. Column internals for absorption and stripping are those of Figure 1.7.

Liquid–liquid extraction, Operation (3) in Table 1.2, using a solvent as the MSA, can be used when distillation is impractical, e.g., because the feed is temperature-sensitive. A solvent (MSA) selectively dissolves only certain components in the feed. The products are an **extract**, L^I , containing the extracted components, and a **raffinate**, L^{II} , containing the unextracted species. Several countercurrently arranged stages may be necessary, either in a staged column with mechanical

Table 1.2 Common Separation Operations Based on Phase Addition

Separation Operation	Symbol	Feed Phase	Added Phase	Separating Agent(s)
(1) Absorption		Vapor	Liquid	Liquid absorbent (MSA)
(2) Stripping		Liquid	Vapor	Stripping vapor (MSA)
(3) Liquid–liquid extraction		Liquid	Liquid	Liquid solvent (MSA)
(4) Adsorption		Vapor or liquid	Solid	Solid adsorbent (MSA)

agitation or in a series of mixing and settling vessels. Additional separation operations, often distillation, are needed to recover the solvent for recycle. Liquid–liquid extraction is described in Chapter 8.

The MSA may be a porous solid, in the form of granules, that selectively removes one or more components from a vapor or liquid feed mixture by **adsorption**, Operation (4) in Table 1.2. Adsorption is confined to the exterior and interior surfaces of the solid adsorbent, unlike absorption, which occurs throughout the liquid absorbent. Adsorption separations are often conducted batchwise or semicontinuously in vessels or columns. However, equipment is available to simulate continuous operation, as in Table 1.2. When the adsorbent, S, becomes saturated with the adsorbed solutes (adsorbate), it must be regenerated to recover the adsorbate and reuse the adsorbent. Alternatively, if the adsorbate is a waste product, the spent adsorbent may be discarded and replaced with fresh adsorbent. Adsorption is described in Chapter 15.

§1.5 SEPARATIONS BY BARRIER

The use of microporous and nonporous membranes as semi-permeable barriers for separating vapor or liquid mixtures is rapidly gaining adherents. The products are the **retentate** (components that do not pass through the membrane) and the **permeate** (components that do pass through the membrane). For microporous membranes, separation is effected by differing rates of species diffusion through the membrane pores. For nonporous membranes, separation is controlled by differences in solubility in the membrane and rates of species diffusion through the membrane material. The most complex and selective membranes are found in the trillions of cells in the human body.

Table 1.3 lists four of the most common industrial membrane separation operations. **Dialysis**, Operation (1) in Table 1.3, is the transport, by a concentration gradient, of small solute molecules through a porous membrane. The molecules unable to pass through the membrane are small, insoluble,

non-diffusible particles. **Reverse osmosis**, Operation (2), is the selective transport of a solvent, for example, water, through a microporous membrane after the pressure of the feed is increased to a value higher than the osmotic pressure of the solution. Solutes in the solvent do not permeate the membrane. It is widely used to produce drinkable water.

Separation of gases by selective **gas permeation** through nonporous membranes, Operation (3), using a pressure driving force, is a process first used by the U.S. government in the 1940s with porous fluorocarbon barriers to separate $^{235}\text{UF}_6$ and $^{238}\text{UF}_6$. Today, nonporous polymer membranes are employed to enrich mixtures containing hydrogen, recover hydrocarbons from gas streams, and produce oxygen-enriched air.

To achieve high purities, **pervaporation**, Operation (4), can be used. Certain species in the liquid feed diffuse through the nonporous membrane, where they are evaporated before exiting as permeate. This method uses low pressures to enhance vaporization and the heat of vaporization must be supplied. It is used to separate azeotropic mixtures.

All four of the membrane separation operations in Table 1.3 are described in Chapter 14.

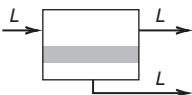
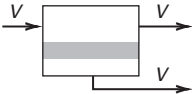
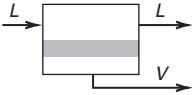
§1.6 SEPARATIONS BY AN EXTERNAL FIELD OR GRADIENT

External fields take advantage of differing degrees of response of certain molecules. **Centrifugation** establishes a pressure field that separates mixtures according to their size, shape, and density. It is used to separate $^{235}\text{UF}_6$ from $^{238}\text{UF}_6$, and can also separate large polymer molecules according to molecular weight.

If a temperature gradient is applied to a homogeneous solution, concentration gradients induce **thermal diffusion**. It has been used to enhance separation of isotopes in permeation processes.

When water containing 0.000149 atom fraction deuterium is decomposed by **electrolysis** into hydrogen and oxygen, the deuterium concentration in the hydrogen is lower than in water.

Table 1.3 Common Separation Operations Based on Barriers

Separation Operation	Symbol	Feed Phase	Barrier	Separating Agent(s)
(1) Dialysis		Liquid	Microporous membrane	Pressure (ESA)
(2) Reverse osmosis		Liquid	Microporous membrane	Pressure (ESA)
(3) Gas permeation		Vapor	Nonporous membrane	Pressure (ESA)
(4) Pervaporation		Liquid	Nonporous membrane	Pressure and heat transfer (ESA)

Until 1953, this process was the source of heavy water (D_2O). In **electrodialysis**, cation- and anion-permeable membranes carry a fixed charge that prevents migration of species with like charge. This phenomenon is applied to desalinate seawater. A related process is **electrophoresis**, which exploits the different migration velocities of charged colloidal or suspended species in an electric field.

These external field separation operations are not discussed further in this textbook, with the exception of electrodialysis, which is described in Chapter 14.

§1.7 BRIEF COMPARISON OF COMMON SEPARATION OPERATIONS

When selecting among feasible separation techniques for a given application, some major factors to consider are (1) technological maturity, which allows designers to apply prior knowledge; (2) cost; (3) ease of scale-up from laboratory experiments; (4) ease of providing multiple stages; and (5) need for parallel units for large capacities. A survey by Keller [3], Figure 1.8, shows that the degree to which a separation operation is technologically mature correlates with its extent of commercial use. Operations based on membranes are more expensive than those based on phase creation (e.g., distillation) or phase addition (e.g., absorption, extraction, and adsorption). All separation equipment is limited to a maximum size. For capacities requiring a larger size, parallel units must be provided. Except for size constraints or fabrication problems, capacity of a single unit can be doubled for an additional investment cost of about 60%. If two parallel units are installed, the additional investment for the second unit is 100% of the first unit, unless a volume-discount

Table 1.4 Ease of Scale-Up of the Most Common Separations

Operation in Decreasing Ease of Scale-Up	Ease of Staging	Need for Parallel Units
Distillation	Easy	No need
Absorption	Easy	No need
Liquid-liquid extraction	Easy	Sometimes
Membranes	Re-pressurization required between stages	Almost always
Adsorption	Easy	Only for regeneration cycle

applies. Table 1.4 lists operations ranked according to ease of scale-up. Those ranked near the top are frequently designed without pilot-plant or laboratory data. Operations near the middle usually require laboratory data, while those near the bottom require pilot-plant tests. Included in the table is an indication of the ease of providing multiple stages and whether parallel units may be required. Ultimately, the most cost-effective process, based on operating, maintenance, and capital costs, is selected, provided it is controllable, safe, and nonpolluting.

Also of interest are studies by Sherwood, Pigford, and Wilke [4], Dwyer [5], and Keller [3] that show that the cost of recovering and purifying a chemical depends strongly on its concentration in the feed. Keller's correlation, Figure 1.9, shows that the more dilute the feed in the product, the higher the product price. The five highest priced and most dilute chemicals shown are all proteins.

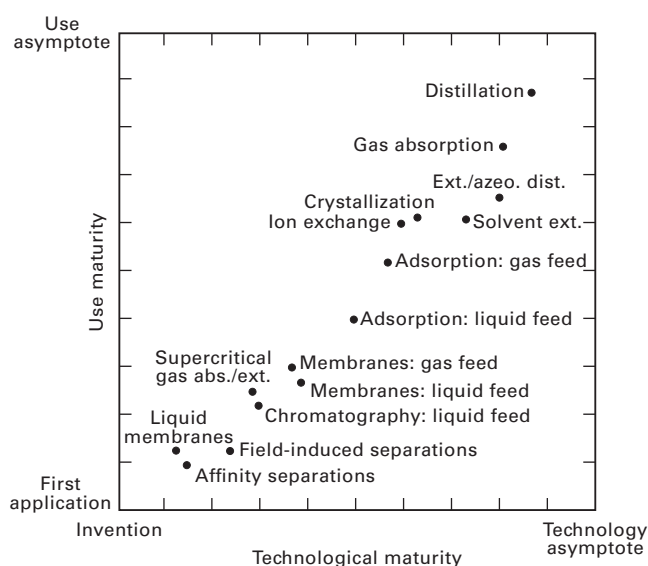


Figure 1.8 Technological and use maturities of separation processes.

[Reproduced from [3] with permission of the American Institute of Chemical Engineers.]

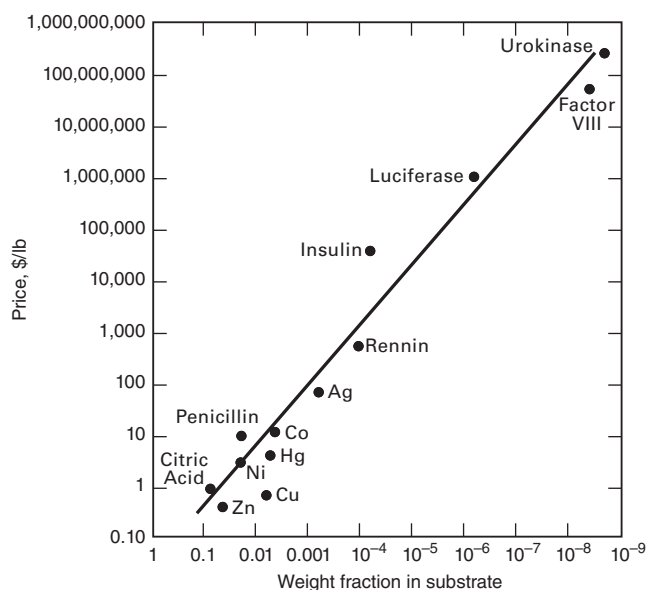


Figure 1.9 Effect of concentration of product in feed material on price.

[Reproduced from [3] with permission of the American Institute of Chemical Engineers.]

§1.8 SEPARATION PROCESSES, PRODUCT PURITY, COMPONENT RECOVERY, AND SEPARATION SEQUENCES

Separation processes usually consist of more than one operation and may produce more than one product. The process is designed to meet product specifications, given as **product purities** and compositions. The process strives to do this with high **component recoveries**.

Consider the continuous hydrocarbon recovery process shown in Figure 1.10, which consists of a sequence of three multistage distillation operations (C1, C2, and C3). The feed consists of propane (C_3H_8), iso-butane (iC_4H_{10}), normal-butane (nC_4H_{10}), iso- and normal-pentane (iC_5H_{12} and nC_5H_{12}), higher molecular weight (heavier) hydrocarbons (C_6^+), and a small amount of ethane (C_2H_6). The components in the feed are rank-ordered by decreasing volatility (increasing normal boiling point), with ethane being the most volatile. The distillation columns were designed with the Aspen Plus simulator, using mathematical models described in Chapter 10, to produce four products: a C_5^+ -rich bottoms from C1, a C_3 (propane)-rich distillate from C2, and an iC_4 (isobutane)-rich distillate and an nC_4 (normal butane)-rich bottoms from C3. A material balance for the process is given in Table 1.5. Streams 4, 6, and 7 are final products, while Stream 2 is an intermediate that requires further processing. Specifications for the products are included in Table 1.6. The

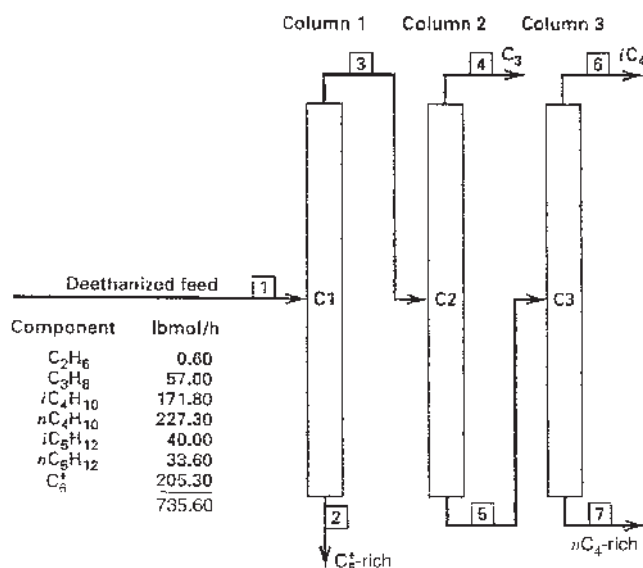


Figure 1.10 Hydrocarbon recovery process using distillation.

C_5^+ -rich component for Stream 2 indicates that all components less volatile than the pentanes are included.

Each column separates two **key components** in the column feed. In Column C1, the separation is between nC_4H_{10} (**light key**) and iC_5H_{12} (**heavy key**). This is indicated by the horizontal line separating the flow rates of these two components

Table 1.5 Material Balance for Hydrocarbon Recovery Process of Figure 1.10

Stream Component	Flow rates in lbmol/h						
	1 Feed to C1	2 C_5^+ -rich Product	3 Feed to C2	4 C_3 -rich Product	5 Feed to C3	6 iC_4 -rich Product	7 nC_4 -rich Product
C_2H_6	0.60	0.00	0.60	0.60	0.00	0.00	0.00
C_3H_8	57.00	0.00	57.00	54.80	2.20	2.20	0.00
iC_4H_{10}	171.80	0.10	171.70	0.60	171.10	162.50	8.60
nC_4H_{10}	227.30	0.70	226.60	0.00	226.60	10.80	215.80
iC_5H_{12}	40.00	11.90	28.10	0.00	28.10	0.00	28.10
nC_5H_{12}	33.60	16.10	17.50	0.00	17.50	0.00	17.50
C_6^+	205.30	205.30	0.00	0.00	0.00	0.00	0.00
Totals	735.60	234.10	501.50	56.00	445.50	175.50	270.00

Table 1.6 Comparison of Process Product Purities with Product Specifications

Component	mol% in Product					
	Propane-Rich		Iso-Butane-Rich		Normal-Butane-Rich	
	Data	Spec	Data	Spec	Data	Spec
C_2H_6	1.07	5 max	0		0	
C_3H_8	97.86	93 min	1.25	3 max	0	1 max
iC_4H_{10}	1.07	2 max	92.59	92 min	83.11	80 min
nC_4H_{10}	0		6.16	7 max		
C_5^+	0		0		16.89	20 max
Total	100.00		100.00		100.00	

in the “Feed to C1” column of Table 1.5. Column C1 produces a C_3^+ -rich product (Stream 2) as bottoms. The distillate from C1 is the feed to C2. Column C2 separates between C_3H_8 (light key) and iC_4H_{10} (heavy key) to produce a C_3 -rich product (Stream 4) as distillate. The bottoms from C2 is the feed to C3, which produces two products by separating between iC_4H_{10} (light key) and nC_4H_{10} (heavy key).

In Table 1.5, it should be noted that an overall material balance equation is satisfied for each of the six components and the C_6^+ multicomponent. For Figure 1.10, that equation in terms of $n_{i,j}$, the lbmol/h flow rate of component i in stream j is

$$n_{i,1} = n_{i,2} + n_{i,4} + n_{i,6} + n_{i,7}$$

For example, for propane (C_3H_8), $57.00 = 0.00 + 54.80 + 2.20 + 0.00$.

From the data in Table 1.5, product purities and component recoveries for the process can be computed. For example, the mol% purity of propane in the C_3 -rich product, Stream 4, is $54.80/56.00 = 0.9786 = 97.86$ mol%. The propane recovery in this product is $54.80/57.00 = 0.9614 = 96.14$ %. Other components in the propane product are ethane and isobutene. Table 1.6 compares the product purities of the final products of Streams 4, 6, and 7 with commercial product specifications. Note that all product specifications are achieved.

The following example illustrates the use of material balances for a separator when product purities and/or component recoveries are specified.

EXAMPLE 1.1 Material balances around a separator

A feed, F , of 100 kmol/h of air containing 21 mol% O_2 (1) and 79 mol% N_2 (2) is to be partially separated by a gas permeation membrane unit, Operation (3) in Table 1.3, according to each of three sets of specifications. Compute the flow rates (n_p and n_R) in kmol/h and compositions in mol% of the two products (retentate, R , and permeate, P). In Figure 1.6(c), Phase 1 is the retentate while Phase 2 is the permeate. The membrane is more permeable to O_2 than to N_2 .

Case 1: 50% recovery of O_2 to the permeate and 87.5% recovery of N_2 to the retentate.

Case 2: 50% recovery of O_2 to the permeate and 50 mol% purity of O_2 in the permeate.

Case 3: 85 mol% purity of N_2 in the retentate and 50 mol% purity of O_2 in the permeate.

Solution

The feed (F) rates of oxygen (1) and nitrogen (2) are

$$n_{1,F} = 0.21(100) = 21 \text{ kmol/h}$$

$$n_{2,F} = 0.79(100) = 79 \text{ kmol/h}$$

Case 1: Because two recoveries are given:

$$n_{1,P} = 0.50(21) = 10.5 \text{ kmol/h}$$

$$n_{2,R} = 0.875(79) = 69.1 \text{ kmol/h}$$

$$n_{1,R} = 21 - 10.5 = 10.5 \text{ kmol/h}$$

$$n_{2,P} = 79 - 69.1 = 9.9 \text{ kmol/h}$$

For the permeate: $n_p = 10.5 + 9.9 = 20.4 \text{ kmol/h}$

$$\text{mol\% } O_2 = 10.5/20.4 = 0.515 = 51.5\%$$

$$\text{mol\% } N_2 = 100 - 51.5 = 48.5\%$$

For the retentate: $n_R = 69.1 + 10.5 = 79.6 \text{ kmol/h}$

$$\text{mol\% } O_2 = 10.5/79.6 = 0.132 = 13.2\%$$

$$\text{mol\% } N_2 = 100 - 13.2 = 86.8\%$$

Case 2: O_2 recovery is given; its distribution to the products is

$$n_{1,P} = 0.50(21) = 10.5 \text{ kmol/h}$$

$$n_{1,R} = 21 - 10.5 = 10.5 \text{ kmol/h}$$

Using the purity of O_2 in the permeate, the total permeate flow rate is

$$n_p = 10.5/0.5 = 21 \text{ kmol/h}$$

By a total permeate material balance:

$$n_{2,P} = 21 - 10.5 = 10.5 \text{ kmol/h}$$

By an overall N_2 material balance:

$$n_{2,R} = 79 - 10.5 = 68.5 \text{ kmol/h}$$

For the permeate: $n_p = 21 \text{ kmol/h}$

$$\text{mol\% } O_2 = 10.5/21 = 0.50 = 50\%$$

$$\text{mol\% } N_2 = 100 - 50 = 50\%$$

For the retentate: $n_R = 100 - 21 = 79 \text{ kmol/h}$

$$\text{mol\% } O_2 = 10.5/79 = 0.133 = 13.3\%$$

$$\text{mol\% } N_2 = 100 - 13.3 = 86.7\%$$

Case 3: Two material-balance equations, one for each component, can be written:

For nitrogen, with a purity of 0.85 in the retentate and $1.00 - 0.50 = 0.50$ in the permeate,

$$n_{2,F} = 0.85n_R + 0.50n_p = 79 \text{ kmol/h} \quad (1)$$

For oxygen, with a purity of 0.50 in the permeate and $1.00 - 0.85 = 0.15$ in the retentate,

$$n_{1,F} = 0.50n_p + 0.15n_R = 21 \text{ kmol/h} \quad (2)$$

Solving (1) and (2) simultaneously for the total flow rates of the products gives

$$n_p = 17.1 \text{ kmol/h and } n_R = 82.9 \text{ kmol/h}$$

Therefore, the component flow rates are

$$n_{1,P} = 0.50(17.1) = 8.6 \text{ kmol/h}$$

$$n_{2,R} = 0.85(82.9) = 70.5 \text{ kmol/h}$$

$$n_{1,R} = 82.9 - 70.5 = 12.4 \text{ kmol/h}$$

$$n_{2,P} = 17.1 - 8.6 = 8.5 \text{ kmol/h}$$

For the permeate: $n_p = 17.1 \text{ kmol/h}$

$$\text{mol\% } O_2 = 8.6/17.1 = 0.503 = 50.3\%$$

$$\text{mol\% } N_2 = 100 - 50.3 = 49.7\%$$

For the retentate: $n_R = 100 - 17.1 = 82.9 \text{ kmol/h}$

$$\text{mol\% } O_2 = 12.4/82.9 = 0.150 = 15.0\%$$

$$\text{mol\% } N_2 = 100 - 15.0 = 85.0\%$$

§1.8.1 Purity and Composition Designations

The product purities in Table 1.6 are given in **mol%**, a designation usually restricted to gas mixtures, for which **vol%** is equivalent to **mol%**. For liquids, purities are more often specified in mass fractions or **wt%**. Although **vol%** is also common for liquid mixtures, it is not easily calculated when the liquid mixture is a nonideal solution. To meet environmental regulations, the allowable concentration of an impurity in gas, liquid, or solids streams is typically specified in parts of impurity per million parts (**ppm**) or parts of impurity per billion parts (**ppb**), where if a gas, the parts are moles or volumes; if a liquid or solid, then mass or weight. For aqueous solutions, especially those containing acids and bases, common designations for composition are **molarity (M)**, which is the molar concentration in moles of solute per liter of solution; **molality (m)** in moles of solute per kilogram of solvent; and **normality (N)** in number of equivalent weights of solute per liter of solution. Concentrations (*c*) in mixtures can be in units of moles or mass per volume (e.g., **mol/L**, **g/L**, **kg/m³**, **lbmol/ft³**, **lb/ft³**). For some chemical products, an attribute, such as color, may be used in place of purity in terms of composition.

§1.8.2 Alternative Separation Sequences

The three-distillation-column recovery process shown in Figure 1.10 is only one of five alternative sequences of distillation operations that can separate the process feed into four

products when each column has a single feed that is separated into one distillate and one bottoms. Consider a hydrocarbon feed that consists, in the order of decreasing volatility, propane (C_3), isobutane (iC_4), n -butane (nC_4), isopentane (iC_5), and n -pentane (nC_5). A sequence of distillation columns is used to separate the feed into three nearly pure products of C_3 , iC_4 , and nC_4 ; and one multicomponent product of combined iC_5 and nC_5 . Figure 1.11 shows the five alternative sequences. When two final products are desired, only a single distillation column is needed. For three final products, there are two alternative sequences. As the number of final products increases, the number of alternative sequences grows rapidly as shown in Table 1.7.

For the initial selection of a feasible sequence, the following heuristics (plausible but not infallible rules) are useful and easy to apply, and do not require an economic evaluation:

1. Remove unstable, corrosive, or chemically reactive components early in the sequence. Then the materials of construction used in later columns will be less expensive. Also remove very volatile components early in the sequence so that column pressures can be reduced in later columns.
2. Remove final products one by one, in order of decreasing volatility or increasing boiling point, as overhead distillates.
3. Remove, early in the sequence, those components of greatest molar percentage in the feed. The remaining columns will be smaller in diameter.

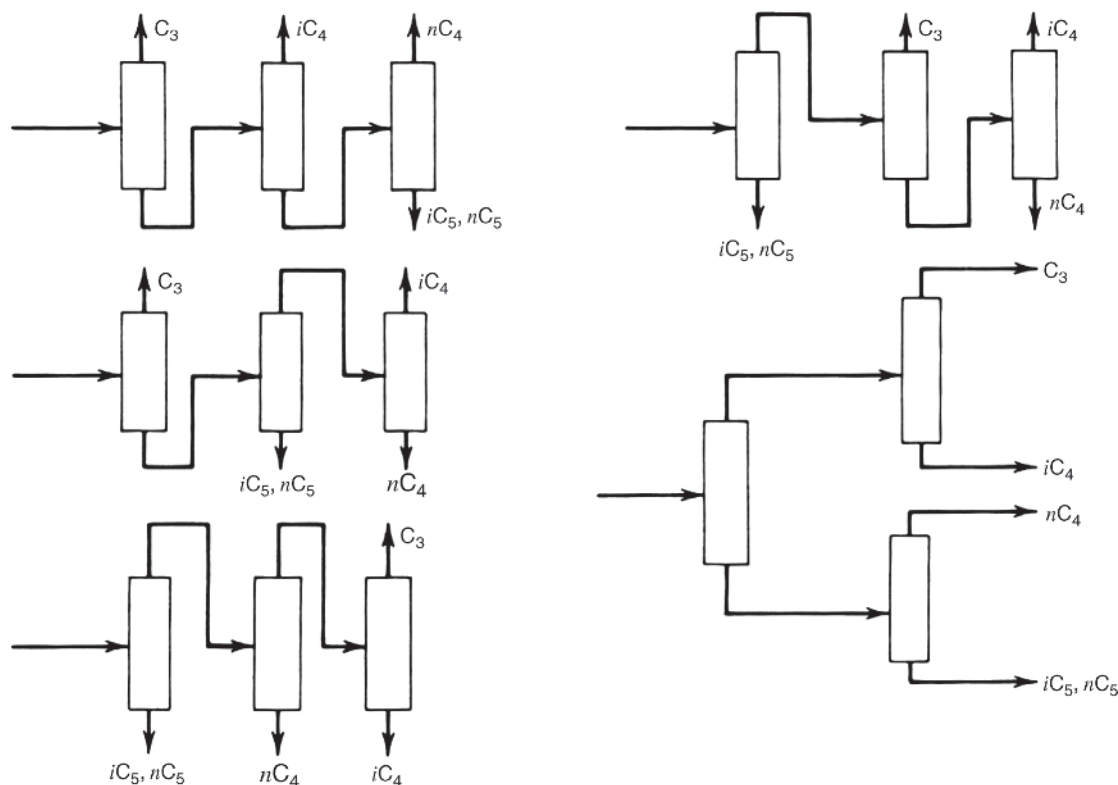


Figure 1.11 Alternative distillation sequences to produce four products.

Table 1.7 Number of Alternative Sequences

Number of Final Products	Number of Columns	Number of Alternative Sequences
2	1	1
3	2	2
4	3	5
5	4	14
6	5	42

4. Make the most difficult separations in the absence of the other components. This will usually lower the diameter of the tallest column.
5. Leave later in the sequence those separations that produce final products of the highest purities. This will also lower the diameter of the tallest column.
6. Select the sequence that favors near-equimolar amounts of distillate and bottoms in each column. Then the two sections of the column will tend to have the same diameter.

Unfortunately, these heuristics sometimes conflict with one another so that one clear choice may not be possible. If applicable, Heuristic 1 should always be employed. The most common industrial sequence is that of Heuristic 2. When energy costs are high, Heuristic 6 is favored because of lower utility costs. When one of the separations is particularly difficult, such as the separation of isomers, Heuristic 4 is usually

SUMMARY

1. Industrial chemical processes include equipment for separating chemical mixtures in process feed(s) and/or produced in reactors within the process.
2. The more widely used separation operations involve transfer of species between two phases, one of which is created by an energy separation agent (ESA) or the introduction of a mass-separating agent (MSA).
3. Less commonly used operations employ a barrier to preferentially pass certain species or a force field to cause species to diffuse to another location at different rates.

REFERENCES

1. CUSSLER, E.L., and G.D. MOGGRIDGE, *Chemical Product Design*, Cambridge University Press, Cambridge, UK (2001).
2. SEIDER, W.D., J.D. SEADER, D.R. LEWIN and S. WIDAGDO, *Product & Process Design Principles* 3rd ed., John Wiley & Sons, Hoboken, NJ (2009).

STUDY QUESTIONS

- 1.1. What are the two key process operations in chemical engineering?
- 1.2. What are the main auxiliary process operations in chemical engineering?

applied. For determining an optimal sequence, Seider et al. [2] present rigorous methods that do require column designs and economic evaluations. They also consider complex sequences that include separators of different types and complexity.

EXAMPLE 1.2 Selection of a Separation Sequence using Heuristics.

A distillation sequence is to produce four final products from five hydrocarbons. Figure 1.11 shows the five possible sequences. The molar percentages in the process feed to the sequence are C_3 (5.0%), iC_4 (15%), nC_4 (25%), iC_5 (20%), and nC_5 (35%). The most difficult separation by far is that between the isomers, iC_4 and nC_4 . Use the heuristics to determine the best sequence(s). All products are to be of high purity.

Solution

Heuristic 1 does not apply. Heuristic 2 favors taking C_3 , iC_4 , and nC_4 as distillates in Columns 1, 2, and 3, respectively, with the multicomponent product of iC_5 and nC_5 taken as the bottoms in Column 3. Heuristic 3 favors the removal of the multicomponent product (55% of the feed) in Column 1. Heuristic 4 favors the separation of iC_4 from nC_4 in Column 3. Heuristics 3 and 4 can be combined with C_3 taken as distillate in Column 2. Heuristic 5 does not apply. Heuristic 6 favors taking the multicomponent product as bottoms in Column 1 (45/55 mole split), nC_4 as bottoms in Column 2 (20/25 mole split), and C_3 as distillate with iC_4 as bottoms in Column 3. Thus, the heuristics lead to three possible sequences as most favorable.

4. Separation operations are designed to achieve product purity and to strive for high recovery.
5. A sequence of separators is usually required when more than two products are to be produced or when the required product purity cannot be achieved in a single separator.
6. The cost of purifying a chemical depends on its concentration in the feed. The extent of industrial use of a particular separation operation depends on its cost and technological maturity.

3. KELLER, G.E., II, *AIChE Monogr. Ser.*, **83**(17) (1987).
4. SHERWOOD, T.K., R.L. PIGFORD, and C.R. WILKE, *Mass Transfer*, McGraw-Hill, New York (1975).
5. DWYER, J.L., *Biotechnology*, **1**, 957 (Nov. 1984).

- 1.3. What are the four general separation techniques and what do they all have in common?
- 1.4. Why is the rate of mass transfer a major factor in separation processes?

- 1.5. What limits the extent to which the separation of a mixture can be achieved?
- 1.6. What are the most common methods used to separate two fluid phases?
- 1.7. What is the difference between an ESA and an MSA? Give three disadvantages of using an MSA.
- 1.8. What is the most widely used industrial separation operation?

EXERCISES

Section 1.1

1.1. Description of the ethanol process.

Considering possible side reactions, describe as best you can what takes place in each block of the process shown in Figure 1.4.

Section 1.2

1.2. Mixing vs. separation.

Explain, using thermodynamic principles, why mixing pure chemicals to form a homogeneous mixture is a spontaneous process, while separation of that mixture into its pure species is not.

1.3. Separation of a mixture requires energy.

Explain, using the laws of thermodynamics, why the separation of a mixture into pure species or other mixtures of differing compositions requires energy transfer to the mixture.

Sections 1.3 and 1.4

1.4. ESA vs. MSA.

Compare the advantages and disadvantages of making separations using an ESA versus using an MSA.

1.5. Differences among distillation and liquid–liquid extraction.

Under what conditions should liquid–liquid extraction be considered over distillation?

Section 1.5

1.6. Osmotic pressure.

The osmotic pressure, π , of seawater is given by $\pi = RTc/M$, where c is the concentration of the dissolved salts (solutes) in g/cm^3 , M is the average molecular weight of the solutes as ions, T is temperature in Kelvin, and R is the ideal gas constant. Consider recovering pure water from seawater containing $0.035 \text{ g of salts}/\text{cm}^3$ of seawater and $M = 31.5$, at 298 K . What is the minimum required pressure difference across the membrane in kPa to just overcome the osmotic pressure?

1.7. Basic separation techniques.

For each of the following separation operations, state the basic separation process technique shown in Figure 1.6: absorption, adsorption, dialysis, distillation, flash vaporization, gas permeation, liquid–liquid extraction, pervaporation, reverse osmosis, and stripping.

Section 1.7

1.8. Removing organic pollutants from wastewater.

The need to remove organic pollutants from wastewater is common to many industrial processes. Separation methods to be considered are: (1) adsorption, (2) distillation, (3) liquid–liquid extraction, (4) membrane separation, (5) stripping with air, and (6) stripping with steam. Discuss the advantages and disadvantages of each method. Consider the fate of the organic material.

- 1.9. What is the difference between adsorption and absorption?
- 1.10. The degree of separation in a separation operation is often specified in terms of product purities and component recoveries. How do these two differ?
- 1.11. What is a key component? What is a multicomponent product?
- 1.12. Why are sequences of separators sometimes necessary to separate a feed mixture?

1.9. Removal of VOCs from a wastewater stream.

Many waste gas streams contain volatile organic compounds (VOCs), which must be removed. Recovery of the VOCs may be accomplished by (1) absorption, (2) adsorption, (3) condensation, (4) freezing, (5) membrane separation, or (6) catalytic oxidation. Discuss the pros and cons of each method, paying particular attention to the fate of the VOC. For the case of a stream containing $3 \text{ mol\%}$ acetone in air, draw a flow diagram for a process based on absorption. Choose a reasonable absorbent and include in your process a means to recover the acetone and recycle the absorbent.

1.10. Separation of air.

Describe three methods suitable for the separation of air into nitrogen and oxygen.

Section 1.8

1.11. Material balance for a distillation sequence.

The feed to Column C3 in Figure 1.10 is given in Table 1.5. The separation is to be altered to produce a distillate of $95 \text{ mol\%}$ pure isobutane with a recovery in the distillate of 96% . Because of the sharp separation in Column C3 between $i\text{C}_4$ and $n\text{C}_4$, assume all propane goes to the distillate and C_3s to the bottoms.

- (a) Compute the flow rates in lbmol/h of each component in each of the two products leaving Column C3.
- (b) What is the percent purity of the n -butane bottoms product?
- (c) If the isobutane purity in the distillate is fixed at 95% , what percent recovery of isobutane in the distillate will maximize the percent purity of normal butane in the bottoms product?

1.12. Material balance for a distillation sequence.

Five hundred kmol/h of liquid alcohols containing, by moles, 40% methanol (M), 35% ethanol (E), 15% isopropanol (IP), and 10% normal propanol (NP) is distilled in two distillation columns in series. The distillate from the first column is 98% pure M with a 96% recovery of M. The distillate from the second is 92% pure E with a 95% recovery of E from the process feed. Assume no propanols in the distillate from Column C1, no M in the bottoms from Column C2, and no NP in the distillate from Column C2.

- (a) Compute flow rates in kmol/h of each component in each feed, distillate, and bottoms. Draw a labeled block-flow diagram. Include the material balances in a table, similar to Table 1.5.
- (b) Compute the mole-percent purity of the propanol mixture leaving as bottoms from the second column.
- (c) If the recovery of ethanol is fixed at 95% , what is the maximum purity of the ethanol in the distillate from the second column?
- (d) If instead, the purity of the ethanol is fixed at 92% , what is the maximum recovery of ethanol (based on the process feed)?

1.13. Material balance for separation by pervaporation.

Ethanol and benzene are separated in a network of distillation and membrane separation steps. In one step, a near-azeotropic liquid

mixture of 8,000 kg/h of 23 wt% ethanol in benzene is fed to a pervaporation membrane consisting of an ionomeric film of perfluoro-sulfonic polymer cast on a Teflon support. The membrane is selective for ethanol, producing a vapor permeate containing 60 wt% ethanol, while the non-permeate (retentate) liquid contains 90 wt% benzene.

- Draw a flow diagram of the pervaporation using symbols from Table 1.3 and include all process information.
- Compute the component flow rates in kg/h in the feed stream and in the product streams, and enter these results in the diagram.
- What separation operation could be used to purify the vapor permeate?

1.14. Material balance for an absorption-distillation sequence.

1,000 kmol/h of a gas mixture containing 90 mol% acetone and 10% nitrogen is to be processed to recover the acetone. First the gas is fed to a multistage absorber where 95% of the acetone is absorbed with 500 kmol/h of a liquid absorbent of methyl-isobutyl-ketone (MIBK). Two percent of the entering MIBK is lost to the gas leaving the top of the absorber. Nitrogen does not dissolve in the MIBK.

The liquid leaving the bottom of the absorber is sent to a distillation column to separate the acetone from the MIBK. In this column, a distillate of 98 mol% acetone and a bottoms of 99 mol% MIBK is obtained.

- Draw a flow diagram of the separation process and place the above data on the diagram.
- For each stream in the process, calculate by material balances the flow rate of each component and enter your results in a table similar to Table 1.5.

1.15. Separation by Gas Permeation.

The Prism gas permeation process developed by the Monsanto Company is selective for hydrogen when using hollow-fiber membranes made of silicone-coated polysulphone. A feed gas at 16.7 MPa and 40°C containing 42.4 H₂, 7.0 CH₄, and 0.5 N₂ kmol/h is separated by the membrane into a retentate gas at 16.2 MPa and a permeate gas at 4.56 MPa.

- Assume the membrane is non-permeable to nitrogen, the recovery of H₂ is 60.38%, and the mole ratio of CH₄ to H₂ in the permeate is 0.0117. Calculate the total flow rate of the retentate and permeate gases and the flow rates of each component in them.
- Compute the percent purity of the hydrogen in the permeate gas.
- Draw a process-flow diagram that displays pressure and component flow rates.

1.16. Separation by membrane separation and adsorption.

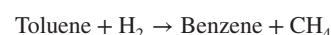
Nitrogen is injected into oil wells to increase the recovery of crude oil (enhanced oil recovery). It mixes with the natural gas that is produced along with the oil. The nitrogen must be separated from the natural gas. A total of 170,000 SCFH (based on 60°F and 14.7 psia) of natural gas containing 18% N₂, 75% CH₄, and 7% C₂H₆ at 100°F and 800 psia is to be processed in two steps to reduce nitrogen content to 3 mol%: (1) membrane separation with a nonporous glassy polyimide membrane, followed by (2) adsorption using molecular sieves to which the permeate gas is fed. The membrane separator is highly selective for N₂ (90.83% recovery), and completely impermeable to ethane. The mole ratio of CH₄ to N₂ in the permeate is 1.756. The adsorption step selectively adsorbs methane, giving 97% pure methane in the adsorbate, with an 85% recovery of CH₄ fed to the adsorber. The non-permeate (retentate) gas from the membrane step and adsorbate from the adsorption step are combined to give a methane stream that contains 3.0 mol% N₂. The pressure drop across the membrane is 760 psia. The permeate at 20°F is compressed to 275 psia and cooled to 100°F before entering

the adsorption step. The adsorbate, which exits the adsorber during regeneration at 100°F and 15 psia, is compressed to 800 psia and cooled to 100°F before being combined with non-permeate gas to give the final pipeline natural gas.

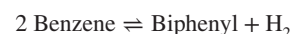
- Draw a process flow diagram of the process using appropriate symbols. Include compressors and heat exchangers. Label the diagram with the data given and number all streams.
- Compute component flow rates of N₂, CH₄, and C₂H₆ in lbmol/h and create a material-balance table similar to Table 1.5.

1.17. Partial condensation of a reactor effluent.

Toluene (methylbenzene) is used as a solvent and a fuel and is a precursor for making benzene by hydrodealkylation; benzene and xylenes by disproportionation; and 2,4,6-trinitrotoluene (TNT)—an explosive used by the military and construction companies—by nitration. Consider a hydrodealkylation process, where the main reaction is:



with an undesirable side reaction:



The reactor effluent is cooled and partially condensed. The liquid phase is then distilled in a sequence of three distillation columns to give the following four products, one of which is recycled back to the reactor.

Component	Off-gas, kmol/h	Benzene Product, kmol/h	Toluene Recycle, kmol/h	Biphenyl By-product, kmol/h
Hydrogen	1.48	0.00	0.00	0.00
Methane	11.07	0.06	0.00	0.00
Benzene	0.80	264.72	1.33	0.00
Toluene	0.00	0.09	88.79	0.45
Biphenyl	0.00	0.00	0.02	4.59
Total	13.35	264.87	90.14	5.04

- Calculate for the sequence the percent recoveries for benzene and biphenyl.
- Calculate the mol% purities for benzene and biphenyl.
- Why is there a toluene recycle stream?
- What would happen if the biphenyl was not separated from the toluene, but was recycled back to the reactor with the toluene?

1.18. Separation by Gas Permeation.

In a hydrodealkylation process of the type stated in Exercises 1.17 and 1.22, a gas rich in hydrogen and methane is separated from the reactor effluent by partial condensation. The gas is then sent to a gas permeation membrane separator (Operation 3 in Table 1.3) to separate the hydrogen from the methane. The permeate is recycled to the reactor and the retentate is used for fuel. The component flow rates for the membrane feed, retentate, and permeate are as follows:

Component	Feed, kmol/h	Retentate, kmol/h	Permeate, kmol/h
Hydrogen	1575.7	157.6	1418.1
Methane	2388.1	2313.5	74.6
Benzene	29.8	29.8	0.0
Toluene	3.5	3.5	0.0
Biphenyl	0.0	0.0	0.0
Total	3997.1	2504.4	1492.7

- (a) Calculate the percent recoveries and mol% purities of hydrogen in the permeate and methane in the retentate. (b) Why not separate hydrogen from methane by distillation or absorption?

1.19. Distillation sequences.

The feed stream in the table below is to be separated into four nearly pure products. None of the components is corrosive and, based on the boiling points, none of the three separations is difficult. As seen in Figure 1.11, five distillation sequences are possible. (a) Determine a suitable sequence of three columns using the heuristics of §1.8.2. (b) If a fifth component were added to give five products, Table 1.7 indicates that 14 alternative distillation sequences are possible. Draw, in a manner similar to Figure 1.10, all 14 of these sequences.

Component	Feed rate, kmol/h	Normal boiling point, K
Methane	19	112
Benzene	263	353
Toluene	85	384
Ethylbenzene	23	409

1.20. Distillation sequence for a solution from fermentation.

Starch from corn or sugarcane can be fermented to an aqueous solution of acetone (A), *n*-butanol (B), and ethanol (E). Typically, the mass ratio of bioproducts is 3(A):6(B):1(E). The solution contains 33 g of bioproducts per liter of water. After removal of solid particles from the broth by centrifugation, the remaining liquid is distilled in a sequence of distillation columns to recover (1) acetone with a maximum of 10 wt% water; (2) ethanol with a maximum of 10 wt% water; (3) *n*-butanol (99.5 wt% purity with a maximum of 0.5% water); and (4) water (W), which can be recycled to the fermenter. If the four products distill according to their normal boiling points in °C of 56.5 (A), 117 (B), 78.4 (E), and 100 (W), devise a suitable distillation sequence using the heuristics of §1.8.2.

1.21. Separation by a distillation sequence.

A light-hydrocarbon feed stream contains 45.4 kmol/h of propane, 136.1 kmol/h of isobutane, 226.8 kmol/h of *n*-butane, 181.4 kmol/h of isopentane, and 317.4 kmol/h of *n*-pentane. This stream is to be separated by a sequence of three distillation columns into four products: (1) propane-rich, (2) isobutane-rich, (3) *n*-butane-rich, and (4) combined pentanes-rich. The first-column distillate is the propane-rich product; the distillate from Column 2 is the isobutane-rich product; the distillate from Column 3 is the *n*-butane-rich product, and the combined pentanes are the Column 3 bottoms. The recovery of the main component in each product is 98%. For example, 98% of the propane in the feed stream appears in the propane-rich product.

- (a) Draw a process-flow diagram for this distillation sequence, similar to Figure 1.10.

- (b) Complete a material balance for each column and summarize the results in a table similar to Table 1.5. To complete the balance, you must make assumptions about the flow rates of: (1) isobutane in the distillates for Columns 1 and 3 and (2) *n*-butane in the distillates for Columns 1 and 2, consistent with the specified recoveries. Assume no propane in the distillate from Column 3 and no pentanes in the distillate from Column 2.

- (c) Calculate the mol% purities of the products and summarize your results as in Table 1.6, but without the specifications.

1.22. Distillation sequence for a wide-boiling mixture.

The effluent from the reactor of a toluene hydrodealkylation process of the type stated in Exercise 1.17 is as follows:

Component	Boiling Point, °C	Flow Rate, kmol/h
Hydrogen	−252.8	1577.1
Methane	−169.0	2399.2
Benzene	80.1	296.6
Toluene	110.6	92.8
Biphenyl	255.2	4.6
Total		4370.3

The reactor effluent at 1265°F and 500 psia is to be separated into four products: (a) a gaseous mixture rich in hydrogen and methane; (b) a benzene-rich product of high purity; (c) a toluene-rich mixture of low purity that is recycled to the reactor; and (d) a biphenyl by-product of high purity. Using the heuristics of §1.8.2, determine one or two favorable sequences. A sequence can contain one single-stage partial condenser in place of one multistage distillation column.

1.23. Distillation sequence for an alcohol mixture.

A mixture of five alcohols is to be separated into nearly pure products in a sequence of distillation columns, each of which produces a distillate and a bottoms. According to Table 1.7, four columns are needed that can be arranged into 14 different sequences. Using the heuristics of §1.8.2, determine one or two feasible sequences. The following table lists the alcohols in the order of volatility. The volatilities relative to the fifth (least volatile) alcohol, 1-hexanol, are shown. Also included are the flow rates of the five alcohols in the feed to the sequence.

Alcohol	Feed flow rate, kmol/h	Volatility relative to 1-hexanol
1-Butanol	360	3.6
2-Methyl-1-Butanol	180	2.5
3-Methyl-2-Butanol	360	2.3
1-Pentanol	2520	1.8
1-Hexanol	3600	1.0
Total	7020	